SEMICONDUCTOR NANOWIRES:
SYNTHESIS AND QUANTUM TRANSPORT

by

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(date) 01/09/2012

*We also certify that written approval has been obtained for any proprietary material contained therein.
To my parents and my wife Chao Ru
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The transition from weak anti-localization to weak localization occurs when \( l_{so} \) changes from the value smaller than \( l_p \) to the value larger.

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# List of Abbreviations

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<tr>
<td>NW</td>
<td>Nanowire</td>
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<tr>
<td>1D, 2D, 3D</td>
<td>One dimension, two dimension, three dimension</td>
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<tr>
<td>InAs</td>
<td>Indium Arsenide</td>
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<tr>
<td>Bi$_2$Se$_3$, Bi$_2$Te$_3$</td>
<td>Bismuth Selenide, Bismuth Telluride</td>
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<tr>
<td>CVD</td>
<td>Chemical vapor deposition</td>
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<tr>
<td>VLS, VSS</td>
<td>Vapor liquid solid, vapor solid solid</td>
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<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
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<tr>
<td>(HR)TEM</td>
<td>(High resolution) transmission electron microscopy</td>
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<tr>
<td>EDX</td>
<td>Energy dispersive X-ray spectroscopy</td>
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<td>UV-Vis</td>
<td>Ultraviolet and visible spectroscopy</td>
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<td>FET</td>
<td>Field effect transistor</td>
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<td>HF</td>
<td>Hydrofluoric acid</td>
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<td>Carbon nanotube</td>
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<td>Isopropyl alcohol</td>
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Semiconductor Nanowires: Synthesis and Quantum Transport

Abstract

by

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Semiconductor nanowires are believed to be one of most promising building blocks in nanotechnology. In this dissertation, we report the controlled synthesis and quantum transport in InAs nanowires and topological insulator Bi$_2$Se$_3$ nanoribbons, two small band gap semiconductors with important applications in high speed transistor, spintronics, thermoelectric, etc.

First, InAs nanowires and Bi$_2$Se$_3$ nanoribbons were synthesized based on the Au nanoparticle catalyzed vapor-liquid-solid mechanism in a chemical vapor deposition system. We first found that small vacuum leakage in the system incorporated oxygen in the InAs nanowires. Such nanowires exhibit low electron mobility (~100 cm$^2$/Vs). Upon improving the system vacuum sealing, we showed that pure InAs nanowires with correct stoichiometry and superior mobility (~1000 cm$^2$/Vs) can be consistently grown. Particularly, we studied the effect of Au nanoparticles’ shape on InAs nanowire growth and found that shaped Au nanoparticles can double the average growth rate compared
with spherical ones. We attributed this enhanced growth rate to the better wetting ability of non-melted flat facets in shaped Au nanoparticles.

Secondly, due to the small diameter (<100nm) of nanowires, low temperature electronic transport of the nanowires can be low dimensional and quantum mechanical in nature. For low mobility InAs nanowires, one-dimensional weak localization was observed. The anisotropic suppression of weak localization in these nanowires was studied and attributed to the radial size confinement of time reversed electron diffusion paths. For pure InAs nanowires with high mobility, weak anti-localization was observed due to strong intrinsic spin orbit interaction. We further demonstrated the application of a surrounding electrolyte gate scheme to tune the Rashba spin orbit interaction by six fold within 1 V of gate voltage.

Thirdly, we performed magneto-transport study of nanoribbons of topological insulator materials (Bi$_2$Se$_3$, Bi$_2$Te$_3$, etc) which have novel two-dimensional helical Dirac surface states with linear dispersion. For the first time, a linear magneto-resistance persisting up to room temperature was discovered in a single Bi$_2$Se$_3$ nanoribbon, resembling the quantum linear magneto-resistance of other Dirac materials (graphene, etc). Angular dependent magneto-resistance and Shubnikov-de Haas oscillations indicate a two dimensional transport origin of this linear magneto-resistance.
Chapter 1 Introduction

1.1 Introduction to nanotechnology

“There is plenty of room at the bottom” was the title of a visionary lecture by Richard Feynman in 1959 in which the initial idea of nanotechnology was elaborated. Nanotechnology deals with materials or structures with the size on the order of tens of nanometer, about one hundred millionth of the thickness of hair. Figure 1.1 shows various natural or manmade objects with the length scale ranging from microscopic (nanoscale) level to macroscopic level. Nowadays, human beings are able to push the research frontiers from macroscopic (>mm) towards down to microscopic (nanoscale) regime, making revolutionary progress in many areas including electronics[1,2] and photonics[3-5], biology and medicine[6-8], energy harvesting[9,10] and conversion industry[11]. Nanotechnology enables integration of thousands of thousands of electronic and photonic components on a tiny chip; Nanotechnology enables manipulation of a single DNA molecule[12] and drug delivery to designed organs[13]; Nanotechnology enables generation of light confined in subwavelength scale[14]; Nanotechnology enhances the efficiency of thermoelectronics[15,16], photovoltaics[17], battery[18,19] and hydrogen generation[20]…
Figure 1.1 The length scale of things. Things on the upper level are natural; Things on the lower level are manmade. The thesis mainly focuses on the nanoscale things highlighted by red area. Figure is cited from[21] and modified.

In addition to application, nanotechnology offers a new platform to examine the quantum world. As the size of materials shrinks in one, two or even three directions, quantum confinement effect kicks in and quantum nature of matter[22-25] starts to take effect in materials’ electrical and optical properties which people are very excited.

The key challenge in nanotechnology is to make the materials into nanosize. Two approaches are available to achieve the goal.

The first one is so-called top down approach, which utilizes tools to “cut” the materials to nanoscale, such as lithography plus etching technique. The advantage of top down approach is its mass production compatible with modern industrial production line. The disadvantage is introduction of imperfections or defects in crystal structure of nanomaterial and physical limitation to approach sub ten-nanometer.
The other is the bottom up approach, in which nanostructures or materials are self-assembled by the building blocks of atom or molecule. This approach permits us to obtain nanostructures with less defects and better structural/surface morphology. The disadvantage lies in the randomness and less efficient controllability in synthesis and device fabrication, leading to difficulties in large scale integration or mass production.

The way out can rely on the combination of both top down and bottom up approaches where only the advantages of these two approaches are exploited. The interesting and intriguing parts of this endeavor to blend the top down and bottom up approaches together are the achievement of a broad range of novel integrated and flexible nano-devices as shown in Figure 1.2.

Figure 1.2 Bottom up plus top down approaches create flexible nano-devices for application in electronics[2], photonics[14], thermoelectric[16], biosensor, energy harvesting[17] and storage[11].
1.2 Semiconductor nanowires (NWs) as building blocks of nanotechnology

Semiconductors are materials with controllable charge carriers (negatively charged electrons or positively charged holes). This unique feature leads to the invention of diodes and transistors which serve as building blocks of semiconductor devices for modern electronics[26]. Mankind would not have entered the era of highly developed information technology if semiconductor was not discovered.

Semiconductor injects new blood to nanotechnology when it is downsized to nanoscale. Semiconductor nanowires (NWs) are believed to be one of the most promising building blocks in nanotechnology[27,28]. Thanks to bottom up approaches which provide ease of access to high quality nanomaterials, semiconductor NWs[29] have become one of the most powerful and versatile class of functional materials besides carbon nanotube[30] and graphene[31], etc. Bottom up growth techniques render highly controllable synthesis of semiconductor NWs with single crystalline quality[32]. The NW materials’ characteristics such as wire diameter, length, crystal orientation, morphology and doping can be tuned by controlling the growth parameters including temperature[33], pressure[34], growth duration time, catalyst material[35] and size [36,37], precursor concentration[38]. Such an elaborated control facilitates extensive device fabrication based on semiconductor NWs. For example, semiconductor NWs were utilized to fabricate nanoscale field effect transistors[39,40], NW p-n junctions for light-emitting diode[41] and lasers[42], quantum qubit device[43,44], logic circuits[45], chemical
sensors[46] and biosensors[6], etc. The flexibility and versatility offer great promise for the semiconductor NWs to be the building blocks of nanoscience and technology.

In this Ph.D. dissertation, we report studies on the controlled synthesis and electronic transport properties of InAs NWs and Bi$_2$Se$_3$ nanoribbons, two small band gap semiconductors with important applications in high speed transistor, spintronics, far-infrared detector and thermoelectronics etc. Through this Ph.D. work, we not only obtained improved understanding of the role of nano-catalysts in NW growth, but also unraveled various interesting electronic transport behavior related to the low dimensional quantum nature of electrons in NWs or the existence of conducting surface states. The thesis is structured as follows:

In Chapter 2, at first, the synthesis of semiconductors InAs NWs is discussed. Particularly, shape controlled Au nanoparticles are introduced to control the InAs NW growth. We demonstrate that using shaped Au nanoparticles doubles the average growth rate of InAs NWs compared with commercial spherical nanoparticles. Then the synthesis of topological insulator Bi$_2$Se$_3$ nanoribbons is introduced.

In Chapter 3, the basic principle and procedure are described for lithographic fabrication of NW field effect transistors by using as-grown InAs NWs. The characterization and performance of InAs NW field effect transistor is introduced as well. Then low noise lock-in measurement technique is explained. The last part focuses on the application of single InAs NW transistor as gas sensor.

In Chapter 4, basic knowledge of electron and spin transport in disordered low dimensional systems is introduced. Quantum corrections to the classical conductance
become prominent at low temperature in disordered systems when electrons have increased coherence time/length. Depending on the strength of spin orbit interaction, the quantum correction manifests as weak localization or weak anti-localization. The suppression of weak localization and weak anti-localization in magnetic field is also discussed.

In Chapter 5, magneto-transport of a single InAs NW is discussed. Weak localization was observed in low mobility InAs NWs with O incorporation. Then weak anti-localization was observed in high mobility pure InAs NWs due to strong spin orbit interaction. Cross over from weak localization to weak anti-localization was achieved by applying solid electrolyte surrounding gate. This experiment demonstrated the strong tuning of Rashba spin orbit interaction in pure InAs NWs.

In Chapter 6, the concept of topological insulator is introduced. Magneto-transport in topological insulator Bi₂Te₃ thin film and Bi₂Se₃ nanoribbons is discussed. For the first time, linear magneto-resistance was discovered in a single Bi₂Se₃ nanoribbon. Angular dependent magneto-resistance and Shubnikov-de Haas oscillation indicate a two dimensional origin for the linear magneto-resistance. The related mechanisms are discussed.
Chapter 2 Synthesis and characterization of InAs NWs and Bi₂Se₃ nanoribbons

2.1 Introduction to compound semiconductors InAs and Bi₂Se₃

Compound semiconductors are semiconductors formed by elements from two different groups. They possess diverse advantages, such as flexible band gap by controlling the composition ratio of compound materials, high carrier mobility and ease to form heterostructures, leading to widely usage in electrical and optical devices. Here we focus on semiconductor compound Indium Arsenide (InAs) and Bismuth Selenide (Bi₂Se₃).

Bulk semiconductor InAs is formed by indium (group III) and arsenic (group V) in the form of zinc blende structure where indium atoms and arsenic atoms are arranged alternatively in diamond crystal structure as shown in Figure 2.1. The lattice constant at room temperature is 6.0583 Å. The density is about 5.68 g·cm⁻³[47]. InAs possesses narrow band gap of 0.35 eV, large spin orbit splitting energy of 0.41 eV, small electron effective mass 0.023ₘₑ where ₘₑ is electron mass[48]. Therefore, InAs exhibits large electron Hall mobility of the order 20000 cm²/Vs and strong spin orbit interaction effects in transport. InAs is an excellent material for high speed electronics. Since the quantized energy of electrons in quantum well is proportional to 1/ₘₑ*, such a small effective mass indicates ease to reach quantum confinement in InAs nanostructures. Similarly, excitons in InAs quantum wire or well are expected to have a large Bohr radius due to small
effective mass, resulting in more considerable quantum effects than many other materials. Strong intrinsic spin orbit interaction makes InAs a promising candidate for spintronics. One more peculiarity is that InAs possesses surface accumulation layers due to Fermi level pinning[49]. Such surface states make InAs’s surface intrinsically n-type and ease to form Ohmic contact with metal electrodes.

![Figure 2.1](image)

Figure 2.1 (a) Zinc blende crystal structure of InAs. Yellow balls and gray balls represent indium and arsenic, respectively[47]. (b) Schematic band structure of InAs at 300 K[48].

Compound semiconductor Bi$_2$Se$_3$ is composed of Bismuth (group V) and Selenide (group VI) in the form of rhombohedral crystal structure with space group $D^{5d}_{3d}$. The lattice constant at room temperature is 9.84 Å[50]. The density is about 6.82 g·cm$^{-3}$. The direct band gap for Bi$_2$Se$_3$ is about 0.35 eV[51], similar to InAs. Bi$_2$Se$_3$ also exhibits strong spin orbit interaction due to strong interaction of $p$-orbital electrons with Bi and Se nuclei. Bi$_2$Se$_3$ features layered structures with a triangle lattice within one layer. Five atom layers constitute one unit known as quintuple layer with thickness 1 nm. Two atom layers
couple by covalent bond within quintuple layer and by Van der Waals force between two quintuple layers[52]. Figure 2.2(a) shows the crystal structure of Bi$_2$Se$_3$.

Recently, Bi$_2$Se$_3$ is discovered as one of a new class of materials named topological insulator in which the conducting topological surface states arise due to the strong spin orbit interaction induced band inversion in the bulk. Three dimensional topological insulators are quantum materials which possess conducting gapless states on the surface of insulating bulk. In other words, the interior of these materials is insulating while conduction of charge carriers on the surface is allowed. They feature helical Dirac cone near $\Gamma$ point in the band gap and spin-locked momentum due to strong spin orbit interaction[51]. In particular, Bi$_2$Se$_3$ is topological insulator with promising applications in spin-related device because of simple spin-helical Dirac cone within the energy band gap as shown in Figure 2.2(b).

![Crystal structure of Bi$_2$Se$_3$.](image)

**Figure 2.2** (a) Crystal structure of Bi$_2$Se$_3$. Five atom layers consist of quintuple layer indicated by red rectangle. (b) Band structure of Bi$_2$Se$_3$. Red line shows the topological surface Dirac cone is located between bulk conduction and valence bands indicated by red area. (Figures cited from ref.[52])
2.2 Au nanoparticles catalyzed NW growth via vapor-liquid-
solid mechanism

A number of growth techniques have been developed for NW growth. These techniques include chemical vapor deposition (CVD), molecular beam epitaxy (MBE) or their variations. Regular CVD employs gas phase pre-cursor or thermal evaporation of solid source as reactant supply; while metalorganic CVD (MOCVD), also known as metalorganic vapor phase epitaxy (MOVPE), employs metalorganic compound as direct vapor source supply. Laser ablation CVD utilizes laser power to evaporate the source. The CVD growth yields higher deposition or growth rate in vacuum with moderate pressure (several Torr or higher). On the contrary, MBE require ultra-high vacuum ($10^{-7}$ Torr or lower) to ensure the slow deposition rate (down to atomic layer per second).

For all the growth techniques, metallic nanoparticles are most commonly used as catalysts for NW growth via the vapor-liquid-solid (VLS) or vapor-solid-solid(VSS) mechanism, even though catalyst-free growth has been reported\[53-56\]. Au, Ni, Cu, etc nanoparticles are one of the most widely used catalysts.

VLS growth mechanism was first proposed in 1960s by Wagner and Ellis for the growth of silicon whisker catalyzed by Au particles\[57\]. In VLS mechanism, the growth is initiated and facilitated by the eutectic reaction between source reactant (Si, Ge, In, etc) and metallic nanoparticles (Au, Cu, Ni, etc). Eutectic reaction is the formation of alloy of two different elements which has a single crystallization and uniform chemical composition melting at the lower temperature than any other composition\[58\]. The lowest melting temperature is referred to as eutectic temperature. For example, the growth of Si
or InAs NWs by Au nanoparticles involves formation of Si-Au or In-Au alloy. NW growth is carried out at the temperature above the eutectic temperature based on VLS. The growth can be indentified in three steps[59]. (I) **Alloying**: metal nanoparticles stay solid at the beginning at temperature higher than eutectic temperature and then partially form alloy with vapor phase source reactant as the supply continues. The metal nanoparticle is partially liquefied as soon as the reactant concentration in the alloy increases and reaches eutectic composition. At this moment, the binary phase mixture is composed of metal solid and reactant/metal alloy liquid. The alloying proceeds till the whole metal nanoparticle is alloyed and ends with alloy phase droplet. (II) **Super-saturation and nucleation**: continuous accumulation of reactant in the alloy super-saturates the alloy droplet to such an extent that the alloy is not able to hold any more reactant. The reactant starts to precipitate out of the alloy and nucleates to solid crystal. The interface shows up between the alloy and precipitated reactant crystal. The growth of NWs is initiated. (III) **Axial growth**: continuous supply of source reactant sustains the super-saturation, precipitation and nucleation. The incoming reactant tends to diffuse and precipitate at the initial interface due to the lower energy cost than forming new interface. With the process going on, the NWs are elongated axially. The growth is illustrated by an example of Si NW growth in Figure 2.3.
Figure 2.3 The VLS Si NW growth procedure is shown in Au-Si phase diagram[60]. The blue region represents the liquid phase of Au-Si alloy. Red arrow guides the growth procedure.

In VSS mechanism, the growth is performed at the temperature lower than eutectic point so that the metal nanoparticles remain solid during the growth [61-63]. The neck between the solid nanoparticle and NW body may play a critical role in growth[64]. Debate is still going on with the mechanism.

The conventional picture on difference between VLS and VSS growth is illustrated in Figure 2.4. If shaped nanoparticles are used for growth, they will be melted during the VLS growth. The shape will be missing for VLS growth while the shape can be maintained by VSS growth. Recently our investigation of shaped Au nanoparticles on InAs nanowire growth in Section 2.4 shows the shape of nanoparticles is partially preserved even in VLS growth. Moreover, the metal nanoparticles after growth are alloy phase containing considerable amount of reactant for VLS mechanism while remain the initial particle phase containing little amount of reactant for VSS mechanism.
Figure 2.4 The conventional thinking of NW growth via (a)VLS and (b)VSS by using shaped catalysts. Our study in Section 2.4 casts doubt on this conventional thinking by showing the shape of nanoparticles is preserved even in VLS growth.

2.3 CVD growth of InAs NWs by Au nanoparticles

In this section, we mainly focus on the CVD NW growth of InAs NWs catalyzed by Au nanoparticles. The growth is on the basis of eutectic reaction of AuIn and AuIn$_2$ via VLS mechanism as shown by Figure 2.5(a).

The CVD NW growth system consists of horizontal single zone Lindberg Blue M tube furnace, 24 inches long, 1 inch outer diameter (O.D.) quartz tube, source transfer chamber and associated vacuum system. The vacuum system is integrated to quartz tube and source transfer chamber. InAs powder source is placed on the groove at one end of quarter inch O.D. quartz rod and transferred to the center of furnace by pushing the rod into the quartz tube through source transfer chamber. Edward RV5 rotary vane pump is used to evacuate the system. The schematic of the CVD system is shown in Figure 2.5.
Si (110) substrates were coated with 100 nm Al₂O₃ by e-beam evaporation and cut to be 3.5 cm×1 cm in size. Before synthesis, the substrates were boiled in boiling water for 5 minutes, then coated with commercial spherical (Ted Pella, Inc.), and placed at a distance of 13.5 ~14 cm from the center of the furnace. The commercial Au particles were diluted with distilled water (approx. 1 to 4 by volume). The quartz tube together with the source transfer chamber was initially pumped down to the base pressure. Then, the temperature of the furnace was increased to 610~680 °C. As the temperature was elevated, the quartz tube was flushed with Ar gas flow at 100 standard cubic centimeters per minute (sccm); the quartz tube then was purged with large flow (900 sccm), which was repeated 3 times to remove any oxygen and water in the furnace. Finally, the flow rate was set at 40 sccm and the pressure at 1.5 Torr. Then InAs powder source were transferred to the center of the furnace. Wires were grown by flowing Ar gas over a thermally vaporized solid InAs source (Alfa Aesar) at a pressure of 1.5 Torr and temperature of 610~680 °C. The growth was maintained about 1 hour and then was terminated by pulling the InAs source out of furnace. The reactor was cooled naturally to room temperature, then the pressure was raised, and the substrates were removed for ex situ materials characterization.
Figure 2.6 (a) SEM image of InAs NWs with O incorporation. Scale bar is 3 µm. (b) HRTEM of as-grown NWs. Growth direction is along <111>. Scale bar is 10 nm. (c) EDX of InAs NWs in (b). Carbon and Cu are from TEM grid.

For first several growths, the base pressure of vacuum system was 10~20 mTorr. The typical scanning electron microscopy (SEM) images of as-grown 20nm “InAs” NWs were taken by Hitachi S4500 SEM shown in Figure 2.6(a). The NWs are straight and uniform in diameter. The NW was grown along <111> direction and coated with relatively thick oxide layer as shown in high resolution transmission electron microscopy (HRTEM) images in Figure 2.6(b) were taken by TECNAI F30. Further energy dispersive X-ray spectroscopy (EDX) equipped in TEM shows oxygen (O) has incorporated in the NW shown in Figure 2.6(c). This was due to the small leakage of the vacuum system during growth. Even though we cannot rule out the possibility that most oxygen is from the native oxide layer or the formation of In₂O₃ nanowire, it is possible
that oxygen heavily doped or incorporated as solute in the InAs NW solid solution. Electrical transport of these wires shows low mobility of 100~200 cm²/Vs in Section 3.3. We further investigate magneto-transport properties of these NWs in Section 5.2 and 5.3. All these wires exhibit weak localization effect, other than weak anti-localization at low temperature. We believe that the absence of weak anti-localization is due to the incorporation of oxygen renders the NWs highly disordered with low mobility and reduces the strength of spin orbit interaction.

Later on, we improved the vacuum sealing of the system. The base pressure was able to reach 5-7 mTorr. The carrier gas was changed to mixture of H₂ and Ar (10% H₂ in Ar) to further reduce oxygen in the vacuum system. Under the same growth condition, we succeeded in growing InAs NW with correct stoichiometric ratio. SEM image of as-grown InAs NWs is shown in Figure 2.7(b). Analysis of fast Fourier transformed (FFT)
diffraction pattern indicates the NWs are zinc blende crystal structures along <111> growth direction as shown in Figure 2.7(c). Energy dispersive X-ray (EDX) spectroscopy equipped in TEM shows the composition ratio of In to As is 1:1 in Figure 2.7(a), confirming the InAs composition. Further electrical transport of these InAs NWs does show high mobility of 500~1000 cm²/Vs (Section 3.3) and weak anti-localization effect at low temperature due to strong spin orbit interaction of InAs[65,66]. Sometimes, the wires show stacking faults due to the alternative change between zinc blende and wurtzite crystal structure, shown in Figure 2.7(d).

Figure 2.8  EDX of Au nanoparticle after growth. Carbon and Cu are from TEM grid. Inset is the InAs NW with nano-catalyst on the tip. The red circle indicates the area where the EDX is performed. The scale bar is 20 nm.

It is clear that the hemi-spherical Au nanoparticle sits on the top of the NW in the inset of Figure 2.8. EDX in Figure 2.8 shows the Au nanoparticle after growth consists of In and Au with ratio of 1:1, indicating the formation of AuIn alloy. In addition, when setting the furnace or source temperature of 610~680 ºC, the temperature on growth substrate is
measured to be around 480~540°C, higher than Eutectic temperature. We believe the growth is based on VLS mechanism. Combined with Au-In phase diagram, the results indicate the growth was facilitated at the eutectic reaction between phase AuIn and AuIn$_2$. We will discuss this in next Section.

### 2.4 The effect of Au nanoparticles’ shape on InAs NW growth

Typically, semiconductor NWs are grown via the VLS mechanism which involves the eutectic reaction of a semiconductor vapor source with a metallic seed particle. In VLS growth, it is generally assumed that the semiconductor reactant and metal particle form a liquid droplet and supersaturation of semiconductor atoms leads to nucleation of a NW[59,67,68]. NW growth can also occur below the eutectic temperature, known as vapor-solid-solid (VSS) growth, where the seed particle remains solid[61-64]. Recently, the VSS growth mechanism has been exploited to form compositionally abrupt interfaces in NWs[69]. While the state of the seed particle during NW growth remains controversial[70], it is clear that the seed particle influences growth kinetics, orientation, and morphology, all of which are critical to applications.

Here, we show that the *shape* of the seed Au particle has a dramatic effect on NW growth. Our study is inspired by previous reports of carbon nanotube growth with highly faceted catalyst particles[71]. In addition, there has been some empirical evidence of the seed particle changing shape during NW growth[62,69]. However, to our knowledge, the seed particle shape has not been controlled *a priori*. Recent advances in particle synthesis allow the preparation of shape-controlled particles whose surface morphology is well-defined[72]. We compared the growth of InAs NWs under identical growth
conditions with similarly-sized shaped and spherical Au particles. Post-growth characterization of the wires shows that the shaped Au particles enhance the initial growth kinetics. Surprisingly, compositional analysis of the particles (shaped and spherical) indicates the formation of alloys, in accordance with VLS growth, but TEM images confirm that only the shaped particles remain highly faceted after growth. These results suggest that the initial morphology of the seed particle is retained, even in the case of VLS growth, and can be used to control NW growth.

Au particles, prepared by reducing a gold precursor (e.g. HAuCl₄) in the presence of a surfactant[73,74], or using commercially available material, have been used to grow InAs NWs[75,76]. As shown in Figure 2.9(a), these Au particles are “spherical” in that their surfaces do not exhibit significant faceting. In comparison, shaped Au particles are synthesized by incorporating a surfactant in the synthesis such as cetyl tetraammonium bromide (CTAB) which preferentially binds to the Au (100) plane. Figure 2.9(b) shows that shaped particles are uniform in diameter and exhibit highly faceted surfaces representative of well-defined crystalline planes. Although different shapes (i.e. cube, triangle, hexagonal, etc.) are evident (see Figure 2.9(b)) and can be controlled by varying the amount of CTAB relative to another surfactant such as ascorbic acid, we found that it is difficult to obtain pure samples of a single shape. For this reason, we focused our study on batches of shaped particles containing a mixture of different shapes and compared the InAs NW growth with commercial spherical particles of similar diameter (~20 nm).
InAs NWs were grown by the physical vapor transport method. The shaped Au particles or commercial spherical Au particles were deposited from solution onto Si (100) substrates by drop casting. To ensure the same density of particles on the substrates, the solutions were diluted appropriately to obtain similar particle concentrations as determined by ultraviolet and visible spectroscopy (UV-Vis) absorbance; we note that the optical absorption spectra of the shaped and spherical Au particles were identical. Multiple sample substrates were placed together in a tube furnace reactor to grow NWs at identical growth conditions. Wires were synthesized by flowing a mixture of H\textsubscript{2} and Ar (10\% H\textsubscript{2} in Ar) over a heated solid InAs source at a pressure of 1.5 Torr and temperature of 610 °C. The substrate was positioned approximately 13.5 cm from the InAs source with a measured temperature of 480°C. After growth, the supply of InAs was terminated, the reactor was cooled, the pressure was raised, and the substrates were removed for \textit{ex situ} materials characterization.
Figure 2.10 SEM images of InAs NWs grown with spherical seed Au particles (left) and shaped seed Au particles (right) for (a) 5 min (b) 10 min and (c) 30 min. All scale bars are 1 μm.
SEM images of InAs NWs grown with spherical and shaped Au particles are shown in Figure 2.10(a-c) for growth times of 5, 10 and 30 mins, respectively. In general, the NWs grown with either particle morphology are relatively straight and similar in diameter. TEM images of representative NWs grown with a spherical and shaped particle are shown in Figure 2.11(a) and (b), respectively, and reveal that the wires are single crystal. Analysis of the diffraction pattern by Fourier transform shows that both wires are zincblende with $<111>$ growth direction. While the structure and quality of the NWs grown with spherical and shaped Au particles appear to be comparable in regards to stacking faults (present in both samples) and tapering (none observed in both samples), a clear difference is the length of the NWs (see Figure 2.10(a-c)). To carefully quantify the wire lengths at various growth times, we performed a statistical analysis of the wires using SEM images taken from the top of the substrate. We assumed that the wires were randomly oriented, close to parallel to the substrate, such that tilting of the substrates was not necessary to measure the wire lengths. Figure 2.12(a-d) show histograms of the wire lengths after growth times of 5 min, 10 min, 30 min and 1 hr, respectively, fitted to log-normal distributions to obtain the geometric mean wire length ($L_{gm}$) and geometric standard deviation ($\sigma_g$). From these results, it is clearly evident that the wires are longer for shaped particles than spherical particles at all growth times.
Figure 2.11 HRTEM images of representative NWs grown with (a) spherical and (b) shaped Au particles. The wires were viewed down the [1-1-2] pole and the growth directions were determined to be <111> for both wires. Insets: TEM images (top right) and FFT images (bottom right) of the corresponding wires. All scale bars are 10 nm.

In Figure 2.12, L_{gm} is shown as a function of growth time and indicates that the wire length is approximately double for shaped Au particles as compared to spherical Au particles throughout the range of times explored. The growth rate, i.e. slope of the curves, is found to be significantly higher at initial times for the shaped Au particles (~4.8 μm/hr) than spherical Au particles (~2.5 μm/hr). At longer times (>30 min), the growth rate decreases to near zero for both particles, most probably due to depletion of the InAs source over time. We note that both curves in Figure 2.12 have an intercept of zero which suggests that the observed differences in wire lengths are not due to differences in the nucleation process.
Figure 2.12  Histograms of lengths of InAs NWs grown with spherical and shaped Au seed particles after (a) 5 min, (b) 10 min, (c) 30 min, and (d) 1 hr. The red lines represent a log normal fit to the length distributions. (e) Average length of InAs NWs grown with spherical and shaped Au seed particles as a function of growth time. The data points, along with error bars, represent an average of more than 30 NWs measured from SEM analysis.
Previous studies have shown that the NW growth rate is a function of many growth parameters including seed particle diameter[37], precursor concentration[38], temperature[33], catalyst density[75], and pressure[34], to name a few. Since the wires were grown under identical growth conditions with similar seed particle densities, we rule out any differences in the precursor concentration, temperature, catalyst density, and pressure as potential reasons for the difference in the initial growth rate between spherical and shaped particles. Although we attempted to keep the seed particle diameter the same, the spherical particles were slightly smaller than the shaped particles (see Figure 2.9(b)). Typically, the NW growth rate has been reported to depend inversely on diameter which confirms that the observed growth rate enhancement is due to shape and, moreover, suggests that the increase in growth rate may be even higher than reflected by our data. A few reports have shown that the NW growth rate increases with diameter, in accordance with the Gibbs-Thomson effect[77,78]. However, our estimations for the growth rates based purely on the observed diameter difference indicate that this is insignificant. To better explain the growth rate enhancement, we performed scanning TEM (STEM) energy dispersive X-ray spectroscopy (EDX). Figure 2.13(a) and (b) show line profiles of two representative wires grown with a spherical and shaped Au particle, respectively. In both cases, only In and no As was observed in the particles, consistent with previous reports for InAs NW growth. As expected, the NWs were found to contain 50:50 In:As (atomic basis), confirming an InAs crystal. We repeated this analysis on 3-4 wires at various growth times; the results for the average In:Au composition in the particle are shown in Figure 2.13(c). At short growth times (5 and 10 min), both spherical and shaped particles were found to contain 67 at% In, corresponding to the AuIn$_2$ crystal
phase. Previous reports have indicated that this is the most thermodynamically favorable phase of Au-In alloys[79]. At longer growth times (60 min or longer), the In concentration decreases to 50% in both spherical and shaped particles, indicative of the AuIn crystal phase. A striking difference is observed at an intermediate growth time of 30 min where the In concentration for shaped Au particles is significantly lower than that for spherical Au particles (54 at% vs. 67 at%). Since the supply of In during growth should be the same for both particles, the EDX analysis leads us to conclude that the In is consumed faster (and earlier) to form longer wires by shaped Au particles than spherical Au particles, which is in agreement with our SEM results.

The NWs grown with spherical and shaped Au particles were carefully imaged by TEM to evaluate the morphology of the particles after growth. Representative high-resolution TEM (HRTEM) images of NWs grown at different times are shown in Figure 2.14. At short times (10 min), the spherical particle at the tip of the wire exhibits a typical hemispherical shape (Figure 2.14(a)). In comparison, shaped particles show a highly faceted core with a thick shell after NW growth (Figure 2.14(b)). At longer times, we find similar results – spherical particles are hemispherical whereas shaped particles retain their faceting (Figure 2.14(c-f)) – albeit the thick coating on the shaped particles is no longer evident (see Figure 2.14(f)). To determine the shell material, STEM EDX line scans were carried out on the particles after growth. The line scans show that the particles are surrounded by a shell of pure In. From these results, we surmise that In preferentially adsorbs to the surface of shaped particles (see Figure 2.14(b)).
Figure 2.13. EDX line profiles of Au (green), In (blue), and As (red) in the Au seed particles after InAs NW growth with (a)spherical Au and (b)shaped Au particles. The corresponding insets indicate the approximate location and direction of the line profiles. Scale bars are 20nm. (c) Atomic concentration of In in the seed particles as a function of growth time.

This “excess” In can diffuse to a growing wire and explain the significantly higher NW growth rate for shaped particles, particularly at the earlier stages of growth (0-30 min). At longer growth times (> 30 min), the In (or InAs) vapor concentration decreases as the
solid source runs out, and the NW growth rate decreases; this is supported by the thinner In shell (see Figure 2.14).

Figure 2.14 HRTEM images of InAs NWs grown with spherical (left) and shaped (right) Au seed particles for (a)-(b) 10 min, (c)-(d) 30 min, and (e)-(f) 1 hr. All scale bars are 20 nm.

Based on EDX analysis of the particles after growth which showed the presence of Au-In alloys, we infer VLS growth. This implies that the particle melts during NW growth and the initial shape of the particle should be lost. Surprisingly, we find that the shape of the
particles still impacts NW growth, as confirmed by the enhanced the initial growth kinetics. Although it is not clear exactly what happened during growth (without in situ TEM), we propose that the shaped Au particles are only partially melted during growth and that the surface remains solid and faceted[80]; i.e. people had found particles forms partially solid to grow sapphire NWs. We note that our growth temperature is slightly lower than the bulk eutectic temperature for AuIn and AuIn$_2$[81], in support of a solid phase. This is also confirmed by ex situ TEM evidence of a highly faceted particle core after NW growth. While we cannot rule out the formation of facets during the cooling process as observed by C.-Y.Wen et al.[69], only the particles that are initially shaped exhibit clear faceting after wire growth. In addition, there is a study in VLS, they stated the catalysts shouldn’t melt throughout the NWs growth or there is no growth. Yet, the VLS mechanism detail has not been understood well. Under this scenario, the retention of flat facets on the particle surface may facilitate adsorption and wetting of In vapor as indicated by STEM EDX. Prior studies have shown that flat surfaces are easier to wet than rounded surfaces and result in thicker wetting layers[82,83]. This excess In on the surface of the particle could act as a reservoir for NW growth and explain the higher growth rate for InAs NWs with shaped particles.

In summary, the initial seed particle morphology is found to have important consequences on InAs NW growth. Our results suggest that the particles may not completely melt, even in the case of VLS growth, and the surface structure can play a role in NW growth including an enhancement of the growth kinetics. Future studies are planned to further examine the role of shape on NW nucleation/growth including a
correlation between particle shape and stacking faults, crystalline orientation, and other structural characteristics of the as-grown NWs. Previous reports have demonstrated that oriented substrates can be used to crystallographically align semiconductor NWs[84,85]. The possible preservation of the particle shape during VLS growth suggests that a similar strategy is possible to control NW structure and properties.

2.5 **Synthesis and characterization of Bi$_2$Se$_3$ nanoribbons**

Bi$_2$Se$_3$ nanoribbons were synthesized in the similar CVD system for InAs NW growth. Bare silicon (100) substrates cut to be 8 cm×1 cm in size. Before synthesis, the substrates coated with 0.1% poly-l-lysine solution and then commercial 40nm spherical (Ted Pella, Inc.), and placed at a distance of 8~10 cm from the center of the furnace. The commercial Au particles were diluted with distilled water (approx. 1 to 4 by volume). Then Bi$_2$Se$_3$ ground powder in the crucible was loaded in the center of quartz tube (furnace). The quartz tube was initially pumped down to a base pressure of 5~10 mTorr. The quartz tube was flushed and purged with a Ar gas at 900 standard cubic centimeters per minute (sccm) for three times. Then the flow rate was set to be 40 sccm and pressure at 100 Torr. Then, the temperature of the furnace was increased and maintained to 630 °C. Wires were grown by flowing the Ar gas mixture over a thermally vaporized powder Bi$_2$Se$_3$ source (Alfa Aesar) at a pressure of 100 Torr and temperature of 630 °C. The growth was maintained about 1~3 hour and then was terminated by cooling down the furnace naturally to room temperature, then the pressure was raised, and the substrates were removed for ex situ materials characterization.
Typical Bi$_2$Se$_3$ nanoribbons have thickness ranging from 50-400 nm and widths ranging from 200 nm to several µms, as shown in the scanning electron microscope (SEM) image in Figure 2.15(a). HRTEM image demonstrates that nanoribbons have smooth side walls and flat surfaces, as shown in Figure 2.15(b) for a 200nm wide nanoribbon. EDX analyses reveal uniform chemical composition with a Bi/Se atomic ratio about 2:3, indicating the stoichiometric Bi$_2$Se$_3$ HRTEM imaging and 2D Fourier transformed electron diffraction measurements in Figure 2.15(c) and (d) demonstrate that the samples are single-crystalline rhombohedral phase and grow along the [1120] direction. The upper and lower surfaces are the (0001) planes.

Figure 2.15 Morphology and crystal structure of Bi$_2$Se$_3$ nanoribbons. (a) SEM image of as-grown Bi$_2$Se$_3$ NWs and ribbons. (b) TEM image showing the shape, flat surfaces and edges of a ribbon with 200 nm width. (c) High-resolution TEM image of the edge of the Bi$_2$Se$_3$ nanoribbon showing the smooth surface. Scale bar is 5 nm. (d) The Fourier transform electron diffraction pattern indicates the single-crystalline quality of the nanoribbon. The growth direction of the nanoribbon is along [1120].

2.6 Summary

In this Chapter, besides the basic properties of InAs and Bi$_2$Se$_3$ bulk materials, we introduced the NW growth mechanism including VLS and VSS, described the VLS-based growth procedure of InAs NWs and Bi$_2$Se$_3$ nanoribbons. We are able to obtain low
mobility InAs NWs with oxygen incorporation due to small system leakage and high mobility pure InAs NWs in vacuum tight system with H₂/Ar flow. Peculiarly, the influence of the Au nanoparticles’ shape on the InAs NW growth was investigated. The growth rate was doubled by utilizing shape-controlled Au nanoparticles compared with commercial spherical ones. We attributed this enhanced growth rate to the better wetting ability of In on the flat facets of shaped Au nano-particles.
Chapter 3  NW field effect transistor: fabrication, characterization and application as gas sensor

3.1  NW field effect transistor(FET)

Field effect transistor (FET) consists of source electrode, drain electrode and a semiconducting channel controlled by a gate voltage. In FET, driven by source-drain voltage, source electrode at one end of channel supplies carriers (electrons or holes) to the channel, inducing the current through the channel. Drain electrode at the other end receives the carriers. The conductivity of the channel is modulated by a transverse electric field by means of controlling the carrier density in the channel. The electric field is induced by gate voltage. The FET can be roughly classified based on approaches by which gate voltage is applied. They are Junction FET(JFET) (Figure 3.1(a)), metal oxide semiconductor FET(MOSFET) (Figure 3.1(b)) [86]. In these FETs, the conducting channel is either n-type or p-type in which electrons or holes are majority carriers. In JFET, gate materials are of opposite type to that of conducting channel so that p-n junction is formed between gate and conducting channel. Gate voltage is applied to p-n junction in the reverse-bias mode. In MOSFET, the gate voltage is applied through the insulating metal oxide layer between gate and conducting channel. SiO₂, Al₂O₃, HfO₂, etc are typically used as gate dielectric materials.
MOSFETs, known as the most successful configuration in all type of FETs and further developed to be metal insulator semiconductor FET (MISFET) or insulator gate FET (IGFET), are widely used as building blocks of modern integrated circuits, leading to great change to the modern life. In order to enhance the FET performance and integrate more FETs into the unit area, amazing progress in top-down approaches has been made in the past five decades to reduce the size of individual FET[87]. The famous Moore’s law[88] states the number of transistors that can be placed inexpensively on an integrated circuit doubles approximately every two years, which forecasts the evolution of the typical size of MOSFET until today it is hitting its down-scaling limit. With current lithography based top down approaches, the further size shrink is faced with increasing technical bottleneck and cost.

Bottom-up synthesis of NWs offers a promising alternative to overcome the technical hurdles in the top down approach. NW FETs, one type of MOSFETs utilizing NWs as the conducting channel, take advantage of versatility of bottom-up synthesized semiconductor NWs and functionality of FET. NWs from group IV[32,36,59,89], III-V[33,38,90-93] and II-VI semiconductors[92,94-96] have been employed to fabricate NW FETs in flexible gate schematics including top gate[97,98], bottom gate[93,99] and surrounding gate[39,100].

Figure 3.1 Schematic of (a) JFET, (b) MOSFET, (c) NW FET
Figure 3.1(c) shows a schematic of NW FET with global Si back gate and top gate with metal oxide dielectric. NW FETs are demonstrated to have potential as high performance electronic devices for future advanced nanoelectronics[39,97-100].

Semiconductor NW FET not only shows great potential in the downscaling of high performance FET, but also serve as a powerful platform to investigate the unique effect of one dimensional(1D) or quasi-one-dimensional(quasi-1D) system due to lateral confinement[101-104]. It is shown the band gap can be engineered by tailoring the diameter of NWs[105]. Utilizing NW FET configuration, subband filling effect is accessible for transport. Ballistic[106,107] or diffusive transport[103,104] can be achieved by controlling the conducting channel length or impurities density. Utilizing NW FET configuration, light with tunable wavelength can be emitted or lased through sub-wavelength source[41,42], provide new possibilities for nanophotonics. Besides NW’s unique electrical and optical properties, large surface-to-volume ratio renders the NW FETs extraordinary sensitivity to the environmental perturbation, a desirable merit in the application as sensors [6,108,109].

InAs NWs possess peculiar advantages as FET candidate materials. High electron mobility guarantees the potential for high speed nanoelectronics[39,97,98]; small electron effective mass make the quantum effect more accessible at given size[110]; large intrinsic SOI is promising for application in low dimensional spintronics; surface accumulation layer[49] due to Fermi level pinned above conduction band near the surface allow the formation of Ohmic contact[98]. All these unique properties shine bright light to the application of InAs NW FETs.
In next section 3.2, we mainly focus on the fabrication and characterization of InAs NW FETs with bottom gate.

### 3.2 Photolithographic Fabrication of InAs NW FET

InAs NWs were grown by CVD as described in the Chapter 2. The as-grown NWs on silicon substrate were ultra-sonicated and suspended in ethanol alcohol which was drop-casted on the p-type silicon wafer chip capped with 300 nm silicon dioxide.

![Figure 3.2 The schematic for NW FET fabrication by photolithography](image)

As shown by Figure 3.2, the photolithography was carried out as follows: (1) spin-coat lift-off-photoresist (LOR) 3A at spinning speed 4000 rotation per minutes(rpm) on the chips with NWs on the surface for 40 s. Bake at 190ºC on hot plate for 5 minutes. (2) Spin-coat photoresist S1805 on the chips at same spinning speed and time. Then bake at 115 ºC for 3 minutes. (3) Place the photomask in contact with the chip, expose the photomask and chip under ultra-violet(UV) light for 2~3 seconds. The power of UV light at 405 nm is about 15 mW/cm². (4)Develop the chip in developer MF-CD-26 for 1 minute. Check if the pattern clearly shows up. If so, do the exposure for the rest chips. If not, adjust the exposure accordingly. (5)Etch the developed chips in 0.5% hydrofluoric
acid (HF) for 5 seconds to remove the native oxide on the surface of NW to insure Ohmic contact. (6) Evaporate 60~80nm Nickel on the chips. Nickel serves as contact metal electrode for the NWs. (7) Lift off the metal in PG remover solution for half an hour.

Figure 3.3(a) shows the electrode pattern for a typical array of two-terminal FET devices, after the lift-off process. If the NW lies between a pair of electrodes, they forms FET device which is magnified in Figure 3.3(b).

![Figure 3.3](image)

Figure 3.3 (a) Optical image of the pattern after lift-off. The electrodes are orange; the dark purple background is 300 nm SiO$_2$. The NW is located between the electrode pair in the red circle, forming the FET. (b) SEM image of a single InAs NW FET, a magnification of the device marked by the red circle in (a). S and D represent source and drain electrodes, respectively.

### 3.3 Characterization of InAs NW FETs

The performance of InAs NW FET can be characterized by electrical properties. We are able to understand the contact property between NW and metal electrode by measuring dependence of source-drain current $I_{sd}$ on the source drain voltage $V_{sd}$. Linear $V_{sd}$-$I_{sd}$ indicates the Ohmic contact, while nonlinear exponential $V_{sd}$-$I_{sd}$ indicates Schottky barrier contact.
Figure 3.4 The gate capacitance of NW FET. The dielectric SiO$_2$ layer is sandwiched between NW and Si substrate.

Metal cylinder on infinite plane model is a good approximation to determine the capacitance between NW and back gate. As shown in Figure 3.4, a single NW was placed on the surface of SiO$_2$/ p++Si substrate. The back gate capacitance $C_g$ is expressed as[111]

$$C_g = \frac{2\pi \varepsilon \varepsilon_0 L}{\cosh^{-1}(h/r)} \quad (3.1)$$

Where $\varepsilon$ is the dielectric constant of gate material (SiO$_2$, etc), $\varepsilon_0$ is the vacuum permittivity. $L$ is the channel length, $h$ is the thickness of oxide layer, $r$ is the radius of NW. In the situation where $h \gg r$, $\cosh^{-1}(h/r) = \ln(h/r + \sqrt{1+(h/r)^2}) \approx \ln(2h/r)$. So Eq. (3.1) becomes

$$C_g = \frac{2\pi \varepsilon \varepsilon_0 L}{\ln(2h/r)} \quad (3.2)$$

Theoretical simulation shows that under the condition $h \gg 6r$, the approximation gives good estimate[112].

In the case of Ohmic contact, field effect mobility $\mu_{FE}$ can be estimated by the gate voltage ($V_g$) dependence of conductance $G$ or the trans-conductance $g_m = \partial I_{sd}/\partial V_g$. The
tuning of the carrier density is due to capacitive effect of dielectric SiO$_2$ sandwiched by the NW and silicon substrate. In the linear operation region, the number of charge $\Delta Q$ induced by gate voltage $\Delta V_g$ can be expressed as

$$\Delta Q = C_g (V_g - V_{th}) = C_g \Delta V_g$$

(3.3)

The change of conductance $\Delta G$ is therefore derived as

$$\Delta G = n_0 e \mu_{FE} \frac{S}{L} - \frac{\Delta Q}{S \cdot L \cdot L} = \frac{\mu_{FE}}{L^2} \Delta Q$$

(3.4)

Where $n_0$ denotes the bulk carrier density, $e$ is electron charge, $S$ is the area of NW cross section. Combining Eq. (3.3) and (3.4), field effect mobility $\mu_{FE}$ can be derived as

$$\mu_{FE} = \frac{\Delta Q \cdot S}{S \cdot L \cdot L} = \frac{\Delta G}{\Delta Q} L^2 = \frac{\Delta G}{\Delta V_g} \frac{\Delta V_g}{\Delta Q} \frac{\Delta V_g}{\Delta Q} = \frac{\partial G}{\partial V_g} \frac{L^2}{C_g} = \frac{g_m}{V_{sd}} \frac{L^2}{C_g}$$

(3.5)

Carrier line density $n$ can be obtained by threshold voltage $V_{th}$. Based on Eq. (3.2), the line carrier density $n$ can be expressed as

$$n = \frac{|V_g - V_{th}|}{e} \frac{C_g}{L}$$

(3.6)

Now we are able to estimate the performance of InAs NW field effect transistor.
Figure 3.5 (a) Conductance $G$ as a function of back gate voltage $V_g$ of 25 nm (diameter) single NW FETs on Si substrate with 300 nm thick SiO$_2$ gate dielectric. The channel length $L=2 \mu$m. The contact metal is Nickel. The data are collected at room temperature in ambient air. Red and pink curves are for two FETs of pure InAs NWs. Blue and green curves are for two FETs of InAs NWs with O incorporation. Pure InAs NWs have larger transconductance and mobility. (b) Temperature dependence of gate effect of a single 60 nm InAs NW FET with O incorporation. The red arrow guides the gate sweep direction. The inset is $I$-$V$ dependence. The linear $I$-$V$ indicates Ohmic contact between Nickel electrode and NW.

Figure 3.5(a) shows a representative room temperature back gate effect of pure InAs NWs and O-incorporated InAs NW FETs in air. Both types of NWs have the same diameter 25 nm. Conductance $G$ increases with gate voltage, indicating the NWs are $n$-type meaning the majority and free moving carriers are electrons. Hysteresis in $G(V_g)$ is likely to stem from the defect states at the interface of NW and SiO$_2$ substrate. The hysteresis can be suppressed by lowering the temperature. Employing Eq.(3.2), (3.5) and (3.6) in which $\varepsilon=3.9$ is the dielectric constant of the SiO$_2$, $h=300$ nm is the oxide layer thickness, $L=2 \mu$m is the channel length, and $r=12.5$ nm is the NW radius for our device, we are able to estimate the field effect mobility and carrier density in the NWs. For pure InAs NWs, the mobility values of the two devices in Figure 3.5(a) are calculated to be
about 1200 and 600 cm$^2$/Vs, respectively. For O incorporated InAs NWs, the values of mobility are 120 and 90 cm$^2$/Vs. It is obvious the mobility of pure InAs NWs is larger than that of O incorporated InAs NWs. The line carrier density $n$ is estimated on the order of $1000/\mu$m, corresponding to volume density about $10^{18}$ cm$^{-3}$ for both types of NWs at $V_g=5$ V.

Figure 3.5(b) shows the temperature dependence of back gate effect of a single 60 nm O incorporated InAs NW FET. The linear $I$-$V$ dependence at various $V_g$ in the inset of the Figure demonstrates the Ohmic contact between the Nickel electrode and the NW. In the main panel, we here only show the $G(V_g)$ in one sweep direction guided by the red arrow. The room temperature mobility of the device is about 250 cm$^2$/Vs. As the temperature cools down to 200 K, the mobility also increases to 360 cm$^2$/Vs, resulting from the suppression of phonon scattering by lowering the temperature. As the temperature further goes down, the mobility is almost the constant because of weak temperature dependence of impurities scattering. As temperature goes down below 40 K, $G(V_g)$ it shows step-like conductance combined with small oscillations on the steps, which is likely to due to the filling of quasi-1D singularity subbands and Fabry-Perot interference of electrons scattered by impurities or defects (e.g. zinc blende/wurtzite crystal structure stacking faults in InAs NW). We will discuss this in details in Section 5.2. According to Eq.(3.6), the line carrier density $n$ is proportional to the threshold voltage $V_{th}$. As shown in Figure 3.5(b), $V_{th}$ shifts to the positive direction as the temperature goes down, indicating the decrease of carrier density. This is reasonable because parts of the dopant carriers are frozen at low temperature.
The carrier density $n$ below 40 K in Figure 3.5(b) is calculated to be $3.5 \times 10^9 m^{-3}$ at $V_g=5V$, the corresponding bulk density is $7.1 \times 10^{24} m^{-3}$. Utilizing 3D Fermi surface in the NW, we obtain Fermi wavelength $\lambda_F = 10.6 \text{ nm}$ and the mean free path $l=14.1 \text{ nm}$. The mean free path is larger than Fermi wavelength but smaller than the diameter $w$ of the NW. Such regime ($w>l>\lambda_F$) illustrates that NWs are in the so-called dirty metal limit in the terminology of quantum transport in disordered diffusive electron system.

### 3.4 Low temperature and low noise measurement technique

The InAs NW FETs was measured in Quantum Design Physical Properties Measurement System (PPMS) cryostat equipped with superconducting magnet. The sample stage can be cooled down to 1.8 K by pumping the liquid helium flow through the cryostat. Magnetic field up to 9 Tesla can be applied.

Low noise lock-in amplifier plus current pre-amplifier was applied to measure low frequency response of NW FETs. 1 mV or smaller AC voltage signal with low frequency (7 Hz or 13 Hz) was applied to the device, then the output current was first amplified by current amplifier and input in lock-in amplifier to read the current value.

### 3.5 A single InAs NW FETs as gas sensor

As mentioned above, rich surface states of InAs NWs lead to formation of surface accumulation layer. For the first time, we explored to utilize the surface states of as-grown InAs NW FETs as gas sensor.

NWs or carbon nanotubes (CNTs) based electronic devices are shown to be sensitive to the adsorption gaseous molecules or the binding of biomolecules in liquid[6,113], thus
enable a label-free sensing modality with high sensitivity and direct electrical readout. These merits are exceptionally attractive for applications in sensor, medicine and life sciences[114-118]. Utilizing the large surface-to-volume ratio of nanomaterials, sensors with quasi one-dimensional (Q1D) nanocomponents such as NWs and CNTs have also led to unprecedented sensitivity in biological detection, even down to single molecule level[119,120].

The principle of bimolecular detection using NW or CNT device is understood by their configuration as field-effect transistors (FETs). Similar to conventional ion-sensitive FETs[121,122], nanoscale FET bio-sensors exhibit a conductance change in response to variations in the electric field or potential at the conduction channel surface due to the binding of biomolecules[123]. The conductance change of FET is believed to be the result of the field effect induced charge transfer between the channel and electrolyte, with the aid of a reference electrode[122]. While there is much progress in the understanding of nanoscale bio-sensors[123-126], the fundamental response mechanism of NW or CNT gas molecule sensors is less clear[127]. Without a conductive ionic electrolyte surrounding the sensor, the gas sensing mechanism of CNT or NW devices has been attributed to the direct charge transfer (or doping) on surface by adsorbed molecules such as O₂, NO₂, NH₃, CO or alcoholic vapors[127-133]. For device with non-ideal metal-CNT interface contacts, gas adsorption can influence the Schottky barriers at contact area and play dominant role in the sensing response[134].
While previously studied nanoscale gas sensors are mostly based on CNT or oxide materials, III-V semiconductors with small band gaps such as InAs seem to be another promising candidate as a chemical detection material due to their high electronic transport performance and the existence of an electron surface accumulation layer which could be sensitive to molecules adsorbed on surface[49,135,136]. To the best of our knowledge, there has been no report on InAs NW gas sensing properties. Here we report the conductance response of InAs NW-FETs in the presence of different gas environment and chemical vapors (acetone, ethanol, IPA, and water). The sensing mechanism has been investigated through studying the gate response of NW conductance in different ambient conditions. Our study shows that the adsorption of gas molecules on surface of InAs NW not only induces charge transfer, but also increases the carrier mobility of NW dramatically. Our findings here may also shed light on the response mechanism of other NWs where the surface states play important role in their transport property.

On the basis of the vapor transport method reported earlier[97], InAs NWs were grown on silicon/silicon dioxide (Si/SiO$_2$) substrate in a thermal CVD system. Ultrasound sonication was used to suspend the NWs in ethanol solution that was dropped onto the SiO$_2$ surface of a highly doped n-type Si substrate that was used as a gate electrode. Photolithography and metallization were used to fabricate source and drain contact electrodes to NW, similar to a previous study[104]. The two-terminal conductance ($G$) of the NW was measured by low frequency lock-in technique within the chamber of a probe station. The source-drain voltage applied on device was 10mV. During a typical experiment, we first pumped the sample chamber to a vacuum of $1 \times 10^{-5}$ Torr to remove
the gas in chamber and residual gas molecules on NW surface. Then the chamber is filled with 1 atm Helium (or Nitrogen) gas as a background gas which has negligible (or small) influence on InAs NW conductance (see discussion later). The conductance of NW was first measured in background gas for about half an hour to confirm the stability of device. Then a cup of alcohol or water was introduced into sample chamber and the NW conductance was monitored continuously as the chemical evaporates and adsorbs on NW. For $G$ vs. gate voltage $V_g$ measurements, the data were collected after introducing the chemicals for more than half an hour.

Figure 3.6 (a) The normalized channel conductance of InAs NW FET vs. time as different chemicals are introduced into or removed from the sensing chamber. The left (right) arrow indicates the introduction (removal) of chemical vapor. Nitrogen was used as the background gas and the total pressure is one atmosphere pressure. (b) Top: SEM image of the InAs NW FET device. Scale bar is 1.2μm. Bottom: schematic of NW device in ambient N$_2$ environment with chemical vapor molecules adsorbed on surface.

Figure 3.6(a) shows the channel conductance of InAs NW FET measured in chamber filled with 1 atm nitrogen gas as different alcohols were introduced and removed sequentially. The scanning electron microscope (SEM) image of the device is shown in
the top panel of Figure 3.6(b). The bottom panel of Figure 3.6(b) shows a schematic of the adsorption of H$_2$O molecules on NW surface in N$_2$ atmosphere (not to scale). In Figure 3.6(a), it can be seen that InAs NW shows a conductance increase after the chemicals start to evaporate and molecules start to adsorb on surface. Note that it generally takes tens of minutes for the NW conductance to stabilize and saturate after the introduction of chemical solutions. We believe this time scale is associated with the evaporation and saturation of chemical vapor in the chamber, but not the intrinsic response time of NW sensor. Upon removing the chemical solution and flushing the chamber with N$_2$ gas (marked by the right arrow in Figure 3.6(a)), NW conductance dropped quickly due to the desorption of molecules from surface. For water vapor, we found that normally the conductance of NW does not fully restore its original value after water was removed, unless pumping the system to a high vacuum.

Solely based on the time dependent conductance response, one might conclude that the conductance change (increase) is due to electron transfer from adsorbed molecules to the n-type InAs NW. We will use the gate response experiment to show that the opposite is true and the conductance increase is dominated by the enhanced NW carrier mobility. Furthermore, in Figure 3.6, among the different vapors tested, water vapor seemed to have the largest effect on NW conductance and with IPA having the smallest effect. The gate response data, however, revealed that water molecule adsorption actually induces less charge transfer than alcoholic vapors.
Figure 3.7 Gate voltage dependence of InAs NW conductance in different ambient conditions: vacuum, Helium, Nitrogen, air and saturated water, IPA and ethanol vapors in helium background. The total pressure is 1 atm for all curves except the black dots which correspond to vacuum. The arrow marks the gate voltage sweep direction.

In order to understand quantitatively how much charge transfer there is after the adsorption of gas molecules, we studied the gate voltage ($V_g$) dependence of NW conductance to see how much $V_g$ is needed to deplete the carriers. Figure 3.7 presents the $G$ vs $V_g$ curves of an InAs NW in different ambient conditions: vacuum, 1 atm of He, N$_2$ or air vs saturated H$_2$O, IPA or ethanol vapor in He. First, one can see that the $G(V_g)$ curve measured in inert gas, He, does not differ much from the result in vacuum, indicating the influence of He on NW is negligible. Over the range of $V_g$ studied (-10 to +10V), 1 atm of N$_2$ generally reduces the NW conductance and increases the slope of $G(V_g)$ somewhat, compared with the response in vacuum. However, the influence of N$_2$ is much smaller than H$_2$O or alcoholic vapors, attesting to the chemical stability of N$_2$. Air also has great influence on the gate response of NW, presumably due to the water and oxygen components in air.
The carrier density \( n \) in the NW can be calculated using the Eq. (3.6). Since the conductance \( G = n e \mu / L = C_g (V_g - V_{th}) \mu / L^2 \), the mobility of carriers, \( \mu \), can be estimated from Eq.(3.5). When the NW diameter is much smaller than the gate oxide thickness, \( C_g \) can be approximated by Eq.(3.2), in which \( \varepsilon = 3.9 \) is the dielectric constant of the SiO\(_2\), \( \varepsilon_0 \) is the permittivity of free space, \( h = 290 \) nm is the oxide layer thickness, \( L = 2 \) \( \mu m \) is the channel length, and \( r = 12.5 \) nm is the NW radius for our device. From the \( G(V_g) \) traces in Figure 3.7, it is obvious to tell that there are two major consequences of H\(_2\)O, alcohol vapors and air on InAs NW. The first effect is the reduced carrier concentration, as suggested by the positive shift of threshold voltage \( V_{th} \). The second effect is the enhanced mobility, as indicated by the much steeper slope in \( G \) vs. \( V_g \) curves.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Turn on ( V_{th} ) (Volt)</th>
<th>Carrier density at ( V_g = 0 ) (( \mu m^{-1} ))</th>
<th>Carrier mobility at ( V_g = 0 ) (( cm^2/Vs ))</th>
<th>Pressure</th>
</tr>
</thead>
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<tr>
<td>Vacuum</td>
<td>ca. -50</td>
<td>ca. 2.1( \times )10^4</td>
<td>ca. 41</td>
<td>10(^3) Torr</td>
</tr>
<tr>
<td>He</td>
<td>ca. -45</td>
<td>ca. 1.9( \times )10^4</td>
<td>ca. 49</td>
<td>1 atm</td>
</tr>
<tr>
<td>N(_2)</td>
<td>ca. -21</td>
<td>ca. 9( \times )10^4</td>
<td>ca. 76</td>
<td>1 atm</td>
</tr>
<tr>
<td>H(_2)O</td>
<td>-8.7</td>
<td>3.8( \times )10^7</td>
<td>154</td>
<td>32 mBar in 1 atm He</td>
</tr>
<tr>
<td>IPA</td>
<td>-7.7</td>
<td>3.3( \times )10^7</td>
<td>181</td>
<td>108 mBar in 1 atm He</td>
</tr>
<tr>
<td>EtOH</td>
<td>-5.9</td>
<td>2.5( \times )10^3</td>
<td>226</td>
<td>78 mBar in 1 atm He</td>
</tr>
<tr>
<td>Air</td>
<td>-5.4</td>
<td>2.3( \times )10^3</td>
<td>232</td>
<td>1 atm</td>
</tr>
</tbody>
</table>

Table 1 Parameters of the InAs NW FET electronic properties measured in the presence of chemical vapors and physical properties of chemicals.

Using Eq. (3.2), (3.5) and (3.6), we extract the carrier (i.e. electron) density and mobility of InAs NW in Figure 3.7 and present the results together with the threshold voltage in Table 1. For the \( G(V_g) \) data in vacuum, He and N\(_2\), the device could not be depleted within 10V gate voltage. In these cases, the threshold voltage was estimated by extrapolating \( G(V_g) \) linearly to zero. It is quite remarkable that exposing InAs NW to alcoholic vapors or air can reduce the carrier concentration by nearly an order of
magnitude and improve the mobility by almost a factor of six. In fact, the values listed in Table 1 are obtained for the zero gate bias voltage, which is the condition we performed the conductance vs. time measurement. The peak mobility of NW in Figure 3.7 reaches ~470 cm²/Vs in water or alcoholic vapors, around ten times higher than the mobility in vacuum. Similar effect was seen in more than ten InAs NWs.

We also take water for an example to estimate how many electrons per water molecule have been transformed to InAs NW. From Table 1, there are about 1.7×10⁴ electrons transferred from per μm long InAs NW to water molecules. If we simply think the NW is fully covered by water molecules and each water molecule occupies a square area (water molecule radius is 4×10⁻¹⁰m). Then each water molecule will accept ~0.2 electrons from InAs NW.

![Figure 3.8](image)

Figure 3.8  Left: a transmission electron micrograph of InAs NW showing the amorphous oxide surface. The growth direction of NW is [111]. Right: a schematic of electron transfer between InAs NW and adsorbate molecules. The InAs NW is shown to have Fermi level pinned above the bottom of conduction band and an electron accumulation layer on surface.
The charge transfer mechanism has been used widely to explain the response of CNT or oxide NW gas sensors[127-133]. Similar charge transfer between gas molecule adsorbates and InAs NW surface is thus not surprising. However, to the best of our knowledge, it is the first report showing gas molecule adsorption can cause such a substantial increase of NW mobility. We attribute both effects to the peculiar electron accumulation and the rich defect states at InAs NW surface. It is known for long time that surfaces/interfaces involving InAs exhibit unusual properties such as negative resistivity at Schottky contacts, type III alignment at heterojunctions, and charge carrier sign-inversion in p-doped samples[137,138]. These effects have been explained as due to formation of an electron accumulation layer in the near-interface region, which is a tendency of InAs to adjust its energy bands in such a way that the Fermi level ($E_F$) becomes pinned above the conduction band minimum (CBM) [49,135,136]. In Figure 3.8, we show a schematic illustrating the band-bending and electron accumulation layer formation at InAs NW surface. The high electron concentration and rich defect states near the surface can facilitate electron transfer to adsorbate molecules. The surface defect states are known to be critical in the formation of electron accumulation layer on InAs surface. The defect states in InAs can have multiple origins: an unbalanced stochiometric ratio between In and As during the thermal evaporation of InAs source, crystal structural defects and incorporation of impurity atoms. By transmission electron microscopy (TEM), we found that the NWs studied have high concentration of oxygen, although the NW is single crystal. A few nm thick amorphous oxide was also found on surface of the InAs NWs studied, as shown in the TEM image of Figure 3.8. It is likely that incorporation of oxygen in NW has can cause many defect states in NW and the
untreated native oxide is also known to have high density of surface states. If we convert the $10^3$-$10^4/\mu$m electron concentration in NW into a surface aerial density, that corresponds to $-10^{12}$-$10^{13}$/cm$^2$, a value not far from the typical aerial density of electron accumulation layer on InAs surface[49,135,136]. The charge exchange between gas molecules and oxide surface states (e.g. oxygen vacancies) has been used to account for the oxide (e.g. SnO$_2$[130,133], ZnO[46,132]) NW gas sensor response. We believe that the rich surface states on InAs surface are responsible for the charge transfer with the adsorbed gas molecules in our case. On the other hand, the remarkable increase in electron mobility after gas molecule adsorption is surprising. With their large surface-volume ratio, electron transport in NWs should be very sensitive to the surface condition. At this moment, it is unclear how the chemisorption and charge transfer at surface reduces scattering and improves mobility. One possible explanation is that since the chemisorption reduces surface accumulation electron density and band bending, the conductance of InAs NW with adsorbates on surface would be dominated by the electrons in bulk, which has much higher mobility than the surface accumulation layer[139]. Such mechanism would open up opportunities to improve mobility of NWs through controlled surface functionalization[140].
To further elucidate the role of surface states, we compared the effect of charging surface states to the gas sensing response of InAs NWs. As shown in Figure 3.9, after charging the InAs NW FET with electron beam, the threshold voltage shifted significantly toward positive for the conductance vs. $V_g$ data in Helium (the black traces). This indicates that the electron density in the surface accumulation layer is reduced and some surface states are neutralized. What is more insightful is that we found that after electron beam exposure, the InAs NW became much less sensitive to the adsorption of molecules on surface. As shown by the dashed lines in Figure 3.9, exposing to air caused marked threshold increase and trans-conductance (mobility) increase for an untreated InAs NW. After electron beam charging of NW, exposing the NW surface to air had much smaller effect (open symbols in Figure 3.9). This experiment further corroborates the importance of surface accumulation layer and surface states in the response of InAs NW to chemisorptions.
In conclusion, we studied the response of the InAs NW FETs in the presence of various gases and alcoholic vapors. It is shown that the adsorption of most gas molecules has two major consequences on the electronic transport properties of InAs NW: it reduces electron density and enhances the electron mobility at the same time. These drastic effects are believed to be tied to the interaction between adsorbed molecules and the high density defect states and electron accumulation layer in the InAs NWs studied. The special surface properties make InAs NWs very sensitive to a broad class of chemical vapors. And the transduction mechanism can form the basis for a fast, low-power adsorption-based chemical sensor.

3.6 Summary

In this Chapter, photolithographic fabrication and characteristics of InAs NW FETs are introduced. Pure InAs NW FETs exhibit higher mobility than that of O incorporated InAs NW FETs. All the InAs NW FETs are n-type. The mean free path in the NW is larger than Fermi wavelength but smaller than the NW diameter, indicating the NWs are dirty metal diffusive system in transport. We will hereby discuss the transport behavior in the NW as disordered system in the next Chapter. The application of a single InAs NW FET as gas sensor is demonstrated. The adsorbed gas molecules on the surface of NW enhance dramatically the NW mobility while drains the carriers out of NW. We attribute this effect to the rich surface states and surface accumulation layer of InAs.
Chapter 4 Electron and spin transport in disordered low dimensional electron systems

In the thesis, quantum transport in semiconductor InAs NWs and topological insulator Bi$_2$Se$_3$ nanoribbons are the two foci. The two systems are all low dimensional disordered systems with strong spin orbit interaction (SOI). In this Chapter, in order to further understand the transport behavior in these systems, we will introduce the fundamentals of the quantum transport indicated by magneto-conductance or magneto-resistance with and without SOI.

4.1 Conductivity in the view of quantum mechanics

Classically, electrons are treated as pin balls bouncing randomly among the immobile positive ions. By introducing mean scattering time, classical conductivity is described by Drude formula $\sigma = N e^2 \tau m^*$ in which $\sigma$ is conductivity, $N$ is bulk electron density, $\tau$ is mean scattering time, $m^*$ is effective mass. Quantum mechanically, electrons are described by probabilistic wave functions. In such case the wave property of electron is important and may induce significant effect on the transport property at macroscopic level (e.g. at low temperature when electrons have long coherence time). It is instructive to examine the conductivity of disordered electronic system by considering how electron waves diffuse through the disordered medium. As illustrated in Figure 4.1(a), we consider electrons travelling from one point A to the other point B are subject to elastic
scattering by impurities, defects and phonons, therefore follow random paths. Sum of the probabilities $P_n$ of all the random paths relates to the diffusion constant and thus conductivity. In semi-classical picture (Boltzmann equation) electrons’ wave nature is ignored in transport, the overall classical probability is simply

$$P = P_1 + P_2 + \ldots + P_n$$  \hspace{1cm} (4.1)

Figure 4.1(a) Conductivity is contributed by multiple scattering paths from point A to point B in a disordered system. (b) Time reversed loop paths shown by red arrows (counter-clockwise) and black arrows (clockwise) when point A and point B coincide. Yellow solid dots represent the scattering centers. $l$ is mean free path.

At low temperature, electron phase coherence starts to take effect. Thus, we need to view the electron diffusion from quantum mechanical viewpoint where wave property of electron can have central role in the system properties (e.g. through interference effect). Based on path integral principle, electron waves scattered by scattering events are described by probability amplitudes. Square of probability amplitudes $|\psi_i|^2$ yields probability $P_i$. All the probability amplitudes associated with all the random paths from A to B interfere with one another. So the quantum interference term shows up in the total probability $P$ which is expressed
Here, $\psi^*_i$ is the complex conjugate of $\psi_i$. Due to the randomness of the paths, random phase difference between different paths results in average cancelation of the interferences contributing to conductivity among different paths. However, a pair of particular paths cannot be overseen when the two point A and B overlap with each other. In this case, a pair of loop paths is formed that are identical but opposite in travel direction. They are self-interference paths $\psi_i$ and $\psi^*_i$, also called time reversed paths, which induce electron wave back scattering. The self-interference term is quantum correction to the classical conductance. The back scattering probability $P_{\text{back}}$ can be written as

$$P_{\text{back}} = |\psi_i + \psi^*_i|^2 = 2|\psi_i|^2 + 2|\psi_i|^2 \cos 2\varphi = 2P_{\text{classical}} + 2P_{\text{quantum}} \cos 2\varphi$$  \hspace{1cm} (4.3)$$

Where $\varphi$ is phase difference between $\psi_i$ and $\psi^*_i$.

### 4.2 Electron phase coherence and weak localization

In time reversed paths shown in Figure 4.1(b), electron wave propagating clockwisely and counter-clockwisely along each of paired loop paths subjects to identical scattering events, resulting in zero phase difference $\varphi=0$ between the paired loop paths when returning to the same point. The zero phase difference leads to constructive interference and enhanced backscattering. Enhanced back scattering introduces a negative quantum correction to the classical conductivity. The decrease in conductivity due to coherent
back scattering is called weak localization\[141\]. The back scattering is enhanced by two times compared with classical probability as shown in Eq.(4.4).

\[ P_{\text{back}} = |\psi_i + \psi_i^\prime|^2 = 2P_i + 2P_i^\prime = 4P_i \quad \text{(4.4)} \]

### 4.3 Spin orbit interaction and spin relaxation

In order to understand the role of spin orbit interaction (SOI) and spin relaxation play in quantum transport in disordered system, we will introduce the concept of SOI in this Section. SOI is a relativistic effect. The static electric field is transformed into magnetic field in the rest frame of electron. Therefore, the electron spin is able to interact with this relativistic magnetic field. The Hamiltonian can be expressed as\[142\]:

\[ H_{so} = \frac{\hbar}{4m^*c^2} \vec{\sigma} \cdot (\vec{p} \times \nabla V) \quad \text{(4.5)} \]

Where \( \hbar \) is the Planck constant, \( m^* \) is the effective mass of electron, \( c \) is speed of light. \( \vec{\sigma} \) is the Pauli matrix vector. \( \vec{p} = \hbar \vec{k} \) is the electron momentum, \( \nabla V \) is potential gradient or the electric field \( \vec{E} \).

In the crystal lattice with SOI, the \( p \)-orbital energy band will split to \( P_{1/2} \) and \( P_{3/2} \) subband with total momentum \( 1/2 \) and \( 3/2 \), respectively. The energy splitting leads to formation of spin orbit energy gap \( E_{so} \) which is proportional to the fourth order of atomic number \( Z \)[142]. Generally, SOI is stronger or \( E_{so} \) is larger in materials with heavy atom elements. So SOI in InAs and Bi\(_2\)Se\(_3\) are stronger than that in Si and Ge. In the crystal with inversion symmetry like Si, Ge and Bi\(_2\)Se\(_3\), the spin degeneracy is kept so that the energy band won’t further split. However, with SOI, the absence of inversion symmetry in
crystal like InAs or InSb, breaking the spin degeneracy, results in further energy splitting of subband \( P_{1/2} \) and \( P_{3/2} \)[143]. Next, we will introduce the two types of SOI, namely Dresselhaus and Rashba SOI which broke the spin degeneracy in the crystal lacking inversion symmetry such as InAs.

In the crystal structure lacking spatial inversion symmetry, the dipolar crystal electric field serves as \( \nabla V \) term in Eq.(4.5). This bulk inversion asymmetry usually presents in zinc blende crystals as shown in Figure 2.1(a), with the Hamiltonian:

\[
H_D = \overline{\sigma} \cdot \overline{\Omega} \\
\overline{\sigma} = \sigma_x \hat{x} + \sigma_y \hat{y} + \sigma_z \hat{z} \\
\overline{\Omega} = \gamma \left[ k_z (k_y^2 - k_x^2) \hat{x} + k_y (k_z^2 - k_x^2) \hat{y} + k_x (k_z^2 - k_y^2) \hat{z} \right]
\]

\( \gamma \) is material dependent coefficient. For bulk InAs, \( \gamma \) is calculated to be \( 27 \text{ eVÅ}^3 \)[143]. \( \hat{x}, \hat{y}, \hat{z} \) are unit coordinate vectors. \( \sigma_i \) is the Pauli matrices, \( k_i \) is the wave vector. \( 2\overline{\Omega}/\hbar \) has the physical meaning of precession frequency. The effective magnetic field can be expressed as \( \overline{B}_{\text{eff}} = 2m^* \overline{\Omega}/\hbar e \). The SOI induced by bulk inversion asymmetry is named after Dresselhaus[144].

Rashba SOI[145] stems from the electric field due to asymmetry of the confining potential or external electric field. Figure 4.2 shows how the electron spin couples with the effective magnetic field transformed from electric field in an asymmetric quantum well.
Figure 4.2 Schematic of Rashba SOI in an asymmetric quantum well. The electric field $\vec{E}$ induced by asymmetric potential is transformed into effective magnetic field $\vec{B}_{\text{eff}}$ at the rest frame of moving electrons (figure cited from [146]).

The Hamiltonian can be expressed as:

$$H_r = \alpha \left( \vec{\sigma} \times \vec{k} \right) \cdot \vec{E} = \vec{\sigma} \cdot \vec{\Omega}$$  \hspace{1cm} (4.7)

$$\vec{\Omega} = \alpha \left( \vec{k} \times \vec{E} \right)$$

$$\alpha = \alpha^* eE$$  \hspace{1cm} (4.8)

$\alpha$ is the coefficient of Rashba SOI, which is proportional to electric field intensity $E$. $\vec{E}$ is the unit vector of electric field. $e$ is the electron charge. $\alpha^*$ depends on the material properties. For InAs, $\alpha^*$ is $117 \text{ eVÅ}^2$ [143].

Another electric field induced factor is due to interfacial inversion asymmetry [147] in which the different compositions of materials will induce surface electric field at the interface.

Based on the introduction above, we have brief idea of how the spin couples with the crystal field, external electric field and the electron momentum. Imagining the electrons move in the quantum well, their spins are altered by the ambient electric field associated
with crystal asymmetry or band offset. The random momentum of electrons randomizes the spins of electron ensemble, which is the idea of spin relaxation.

Spin relaxation or spin dephasing is the process for spin ensemble to reach spin equilibrium. If the spins experience a perturbation, they will take a certain time (spin relaxation time) to reach the equilibrium states. The strong tuning of spin relaxation time is favored because this allows us to tune the spins within certain distance, rendering the information encoding viable. This is one central point of spintronics[148].

The mechanisms of spin relaxation of conduction electrons are the Elliott-Yafet mechanism, D’yakonov-Perel mechanism, Bir-Aronov-Pikus mechanism.

In Elliott-Yafet(EY) mechanism[149,150], electron spins relax through ordinary momentum scattering by phonons and impurities, indicating that the spin relaxation relies on the scattering events. The more chances the electrons are scattered, the stronger the spin relaxation is. One important conclusion is:

\[ \tau_s \propto \tau_p \]

In Eq.(4.9), spin relaxation time \( \tau_s \) is proportional to the momentum relaxation time \( \tau_p \)[151].

D’yakonov-Perel(DP) mechanism[152] takes effect in systems lacking spatial inversion symmetry. The electron spins precess in momentum-related effective magnetic field generated by both Dresselhaus and Rashba SOI. The momentum-dependent spin precession leads to spin relaxation. Scattering events, no longer flip the spin, but randomize the electron momentum thus spins. The main conclusion is:
\[ \tau_s \propto 1/\tau_p \] (4.10)

The spin relaxation time \( \tau_s \) is inversely proportional to the momentum relaxation time \( \tau_p \). This is a visible distinction between EY and DP mechanism\[153\].

Bir-Aronov-Pikus mechanism\[154\] is important in \( p \)-doped semiconductors, in which the electron-hole exchange interaction gives rise to fluctuating local magnetic field flipping electron spins. Our InAs NWs is usually \( n \)-type, so we don’t take this mechanism into account.

4.4 Spin orbit interaction and weak anti-localization

In the presence of SOI in disordered system, electron spins precess and relax during or at each scattering events, bringing extra phase change to spin states. If we take the SOI into consideration in the time reversed loop paths, the phase change of spin states adds extra \( \pi \) to the total phase difference, which turns the constructive back scattering into destructive back scattering. The positive quantum correction is introduced to the classical conductivity. This is called weak anti-localization\[155,156\], opposite to weak localization.

Weak anti-localization can be interpreted by the picture as follows: the orientation of effective magnetic field \( \vec{B}_{\text{eff}} \) transformed relativistically by static electric field is always perpendicular to the electron wave vector \( \vec{k} \) and electric field vector \( \vec{E} \). The three vectors then define a local spin basis. Spin follows the local spin basis quasi-adiabatically to keep
Figure 4.3 Time reversed path loops associated with electron spin. The yellow solid dots represent the scattering center. Blue and red solid dots with arrows represent the electrons with spin. Blue and red arrows guide the clockwise and counter-clockwise scattering paths.

the relative orientation to the wave vector unchanged. In other words, spin orientation is locked with wave vector $\vec{k}$. As shown in Figure 4.3, incident electron spin rotates $\pi$ for clockwise loop path and $-\pi$ for counter-clockwise loop path after completing the time reversed loop paths. The difference between the two opposite paths is $2\pi$ rotation in real space. As we know, $\frac{1}{2}$ spin state returns to the initial state when experiencing $4\pi$ rotation in real space. So the spin rotation by $2\pi$ turns out to be $\pi$ in phase difference of the two loop paths, which leads to destructive interference and suppressed backscattering.

$$P_{\text{back}} = |\psi_i + \psi_i^*|^2 = 2P_i - 2P_i = 0$$ (4.11)

4.5 Suppression of weak localization and weak anti-localization in magnetic field
The quantum correction to classical conductivity, weak localization or weak anti-localization, depends on phase coherence, SOI and external magnetic field. Electron wave’s interferences define phase coherence length $l_\varphi$ and dephasing time $\tau_\varphi = l_\varphi^2 / D$. Here $D$ is the diffusion constant. The evolution of spin phase among scattering events is affected by the effective magnetic field induced by SOI thus defines spin relaxation length $l_{so}$ and time $\tau_{so} = l_{so}^2 / D$. Weak anti-localization (localization) dominates when $l_\varphi$ is larger (smaller) than $l_{so}$. $l_\varphi (\tau_\varphi)$ and $l_{so} (\tau_{so})$ can be extracted to indicate the strength of phase coherence and spin orbit interaction.

Upon the application of magnetic field to the time reversed loop paths, magnetic flux acts as a phase shift in the wave function. Phase shifts are same but opposite in sign to reversed loop, leading to suppression of the weak localization or weak anti-localization.

$$e^{\frac{-i}{\hbar} \oint \vec{B} d\vec{S}} = e^{\frac{-i}{\hbar} \oint \vec{A} d\vec{l}}$$  \hspace{1cm} (4.12)

Figure 4.4  Schematic of disordered NW with time reversed path loops. $l_\varphi$ is phase coherence length within which electron wave retain the capability of interference. Here, $l_\varphi > w$ and $w > l$ show the quasi-1D dirty metal diffusive system.
In the thesis, our as grown InAs NWs can be treated as quasi-1D system where $l_\varphi > w$ (NW diameter). Especially in dirty metal diffusive region $w > l$ (mean free path) shown in Figure 4.4, in the case $l_\varphi \geq l_{so}$ where weak anti-localization dominates, the magnetoconductance can be expressed as[157-159]

$$G(B) = G_0 - \frac{2e^2}{hL} \left[ \frac{3}{2} \left( \frac{1}{l_\varphi^2} + \frac{4}{3l_{so}^2} + \frac{1}{l_B^2} \right)^{-1/2} - \frac{1}{2} \left( \frac{1}{l_\varphi^2} + \frac{1}{l_B^2} \right)^{-1/2} \right]$$

(4.13)

Where $G_0$ is classical Drude conductance, $l_B$ is the magnetic relaxation length. $l_B = \frac{\sqrt{3h}}{eBw}$ for the perpendicular magnetic field; $l_B = \frac{2\pi \hbar}{eBw}$ for the parallel field. Note the Eq.(4.13) is valid when magnetic length $l_m = \sqrt{\hbar/eB} > w$.

In the case $l_\varphi < l_{so}$ where weak localization dominates, $4/3l_{so}^2$ is negligible comparing with $1/l_\varphi^2$, so Eq.(4.13) can be reduced to [160]

$$G(B) = G_0 - \frac{2e^2}{hL} \left( \frac{1}{l_\varphi^2} + \frac{1}{l_B^2} \right)^{-1/2}$$

(4.14)

Figure 4.5 shows a theoretical transverse magneto-conductance $\Delta G = G(B) - G_0$ according to Eq.(4.13). For the plotting, we set NW diameter $w = 20$ nm, channel length $L = 2$ $\mu$m, $l_\varphi = 100$ nm. Clearly, positive magneto-conductance (weak localization) dominates when $l_\varphi < l_{so}$; negative magneto-conductance (weak anti-localization)
dominates when \( l_\varphi \geq l_{so} \). The transition from weak localization to weak anti-localization occurs when \( l_{so} \) is tuned from 80 nm to 120 nm.

Figure 4.5 Theoretical quantum correction \( \Delta G \) of a quasi-1D NW conductance as a function of perpendicular magnetic field \( B \) based on Eq.(4.13) at \( l_\varphi = 100 \) nm with varying \( l_{so} \) from 80 nm to 120 nm. In the equation, \( w=20\)nm, \( L= 2 \) \( \mu \)m. The transition from weak anti-localization to weak localization occurs when \( l_{so} \) changes from the value smaller than \( l_\varphi \) to the value larger.

4.6 Summary

In this Chapter, quantum correction to classical conductance, namely weak localization and weak anti-localization in disordered system was illustrated. The key parameters or length scales to determine the strength of weak localization or anti-localization are phase coherence length and SOI induced spin relaxation length. We analyzed weak localization and anti-localization in NWs as quasi-1D disordered diffusive system. In the next Chapter, we will present and analyze the experiment data of quantum transport in InAs NWs.
Chapter 5 Magneto-transport in a single InAs nanowire

5.1 Introduction and outline

Driven by the advantages of InAs NW FET mentioned above, we will focus on the magneto-transport properties in a single InAs NW in this Chapter. For low mobility O incorporated InAs NWs due to small CVD system leakage, weak localization dominates at low temperature transport; while weak anti-localization disappears in such wires resulting from the fact that the incorporation of oxygen reduces the mobility of NWs as well as SOI effect which is supposed to be strong in pure InAs. First in Section 5.2 and 5.3, we investigate the transverse magneto-conductance in these InAs wires at low temperature, which is in good agreement with quasi-1D weak localization theory in small magnetic field. Interestingly, the step-like magneto-conductance at high magnetic field shows the possible signature of suppression of weak localization in multiple 1D subbands. The evolution of magneto-conductance from transverse to longitudinal case exhibits anisotropic property. We attribute the anisotropy to the size confinement of time reversed loop paths. Later on in Section 5.4 and 5.5, high mobility pure InAs NWs are grown and exhibit weak anti-localization effect at low temperature with improved vacuum sealing in the CVD system for NW growth. The signature of weak anti-localization indicates strong SOI in the as-grown InAs NWs. The magneto-transport study of quantum interference effect is useful in quantitative determination of the strength of SOI. We further demonstrate the application of a surrounding surface gate scheme to
tune the SOI by six fold within 1 V of gate voltage. This tunability is much stronger than ever reported in InAs NWs.

5.2 Suppression of weak localization in a single low mobility InAs NW

Recently, low temperature magneto-transport measurements were used to study the electron localization in CNTs[161,162] and silicon NWs[163]. Magneto-transport was also investigated in arrays of GaN[164] and InAs NWs[65] to extract the phase coherence length and spin-orbit relaxation length. Here, we report on low temperature magneto-conductance $G(B)$ measurements of a single gated low mobility O incorporated InAs NW in transverse magnetic field. The observed positive magneto-conductance $G(B)$ data is consistent with the 1D weak localization effect and we extract a gate voltage controllable phase coherence length $l_\phi$ in the range of 20-50 nm in our temperature range (2-30 K). The temperature dependence of the phase-coherence length $l_\phi \propto T^{-1/3}$ is consistent with the one-dimensional Nyquist phase breaking mechanism[165-170]. More interestingly, in our single NW device with gate tunable electron density $n$, we observe possible signature of suppression of weak localization associated with multiple 1D subbands as $n$ is increased.

On the basis of the vapor transport method reported earlier[97], O incorporated InAs NWs were grown on silicon/silicon dioxide (Si/SiO$_2$) substrate in a thermal chemical vapor deposition (CVD) system. Ultrasound sonication was used to suspend the NWs in isopropyl alcohol (IPA) solution that was dropped onto 290 nm thick oxide on a highly doped $n$-type Si substrate that was used as a gate electrode. Photolithography and electron
beam evaporation of Ti/Al (2 nm/60 nm) were used to establish source and drain contacts that are 2 µm apart. The sample was dipped in buffered HF solution for about 3 s before evaporation to remove any native oxide and ensure Ohmic contacts. The two-terminal conductance ($G$) of the NW was measured by low frequency lock-in technique in a Quantum Design PPMS cryostat. We have measured two InAs NWs with 20 nm diameter and both devices yielded similar results. We expect that the influence of contact resistance is small compared with the NW channel resistance in our experiment due to the low contact resistance of InAs NWs[171] and relative long (2 µm) channel in our study. This claim is further supported by the excellent consistency between our data and 1D weak localization theory discussed below.

Figure 5.1 Gate voltage ($V_g$) dependent conductance ($G$) of a 20nm InAs NW at temperatures of 20, 15, 10, 8, 4, and 2K. The dashed arrow shows the sweep direction of $V_g$. A few dominant dips in the $G(V_g)$ curves are marked by the vertical arrows, showing that the dip positions do not shift in $V_g$ while conductance decreases as $T$ reduces. The inset is the SEM image of the InAs NW device with channel length $L=2.0$ µm. Scale bar is 1µm.
Here we present representative data from one device. The conductance of the InAs NW as a function of the gate voltage \( V_g \), is shown in Figure 5.1 for different temperatures at zero-magnetic field. The scanning electron microscope image of the NW is shown in the inset of Figure 5.1. The source-drain voltage used in the measurement is 3 mV. The sample showed slight hysteresis effect in \( G(V_g) \) with respect to the sweep direction of \( V_g \).

For clarity purpose, we show the \( G(V_g) \) data for one sweep direction of \( V_g \), as indicated by the dashed arrow. There are two main features in this set of \( T \)-dependent \( G(V_g) \) traces. First, there are multiple oscillations on top of a smooth background of \( G(V_g) \). Some dominant oscillations with large \( V_g \) spacing survived even at 20 K, as marked by the solid arrows. These oscillations are likely to originate from the singularities in the density of states of the quantized 1D subbands, similar to the observations in silicon NWs with small diameter[172]. Thus multiple subbands can contribute to the transport in our system depending on the gate voltage. We also notice some oscillations have smaller spacing in \( V_g \) (~1V), these oscillations resemble the Fabry-Perot interference pattern observed in the conductance of short channel carbon nanotube devices[173]. A rough estimate excludes origin from the resonant interference between source and drain in a 2 \( \mu \text{m} \) channel. However, according to the HRTEM image our as-grown InAs NWs, stacking fault due to alternative formation of wurtzite and zinc blende is common. It is likely the resonant scatterings are caused by stacking fault planes. This is also consistent with the previous study of oscillations due to resonant scattering by defects or localized states in carbon nanotube[174,175]. Second, as temperature is lowered, device conductance decreases for all the \( V_g \)’s in this temperature range. There could be two implications for the decrease of conductance: it could be due to a decrease in either the
electron density or mobility (or both). However, the positions of the dominant dips in \( G(V_g) \) do not show any considerable displacement at different \( T \)'s.

This suggests that the position of electron Fermi energy does not shift with \( T \) and thus the electron density is constant for a given \( V_g \). Therefore, we conclude that the temperature dependent conductance in our sample is not a trivial carrier density effect.

Figure 5.2(a) shows the dependence of the magneto-conductance \( G(B) \), on the magnetic field, \( B \), for a number of temperatures at \( V_g= -4 \) V. The magnetic field was applied perpendicular to NW axis and the substrate, as shown in the inset panel of Figure 5.2(a). It is clear that the magneto-conductance is positive and the amount of conductance increase in \( B \) is larger at lower temperatures. At low temperatures, \( G(B) \) saturates around \( B=4 \) T while it increases more slowly and continuously at \( T=30 \) K. The positive magneto-conductance is due to the suppression of the weak localization by the magnetic field.
Figure 5.2 (a) Magneto-conductance \( G(B) \) at \( V_g = -4 \text{V} \), and temperature 30, 20, 15, 10, 8, 6, 4, and 2K. Solid colored curves are fits to 1D weak localization theory. The inset shows the measurement configuration. (b) Electron phase coherence length extracted from the 1D weak localization fitting to the data at various \( V_g \) and temperatures. (c) Residual Drude conductance vs. \( T \), obtained from fitting \( G(B) \) to 1D weak localization.

In one dimension, the weak localization correction to the conduction is described by Eq.(4.14) with \( l_B = \frac{\sqrt{3}h}{eBw} \), which yields

\[
G(B) = G_0 - \frac{2e^2}{hL} \left( \frac{1}{l^2} + \frac{e^2B^2w^2}{3h^2} \right)^{-1/2}
\]  

(5.1)

Here, \( L=2 \ \mu\text{m} \) is the length of the NW, \( w=20 \ \text{nm} \) is its width or diameter, and \( G_0 \) is the classical Drude conductance without the localization correction. There are only two parameters \( l_p \) and \( G_0 \) in Eq. (5.1) used to fit to our experimental data at low magnetic field. The fitting curves are indicated by the solid lines in Figure 5.2(a) and the fitted \( l_p \)'s are shown in Figure 5.2(b) for different \( V_g \)'s. Note that Eq. (5.1) is valid only for \( l_p > w \), which is satisfied from the fitting results in Figure 5.2(b). The extracted phase coherence lengths are seen to follow a power law dependence of temperature \( l_p \propto T^{-1/3} \), consistent with the dephasing mechanism being the electron-electron collisions with small energy transfers (or the “Nyquist” dephasing) as observed previously in many other systems[65,161-170]. Notably, \( l_p \) tends to saturate at \( T < 10 \ \text{K} \) in Figure 5.2(b). Similar saturation phenomenon was attributed to either the overheating of electrons[163] or other intrinsic[176] or extrinsic[170] dephasing mechanisms. It is interesting to note that unlike a previous report on arrays of InAs NWs[65], the interpretation of our data does not need to involve weak anti-localization effect that would be indicated as a negative magneto-conductance. This means that the spin flipping length is much longer than \( l_p \) in our
system, suggesting the potential use of InAs NWs in applications requiring long spin coherence length. Later on we find out the absence of weak anti-localization is due to formation of O incorporated InAs NWs. The fitted Drude conductance $G_0$ is plotted against temperature in Figure 5.2(c) for all four $V_g$’s we studied in detail. Figure 5.2(c) shows that $G_0$ still has a weak insulating behavior after correcting for the weak localization, indicating that there is additional mechanism contributing to the temperature dependence of the sample conductance. We will return to the temperature dependence of $G$ later.

With the gate voltage, we can control the electron density $n$ and thus adjust the population of different quantized 1D subband in NWs. We observed that the magneto-conductance $G(B)$ showed increased complexity related to the occupation of different 1D subbands as the electron density was increased. We studied the temperature dependence of $G(B)$ at four $V_g$’s: -8, -4, 0 and 4 V. $G(B)$ curves for the lower two electron densities ($V_g$) -8, -4 V) look qualitatively similar to Figure 5.2(a), that is, there is a positive $G(B)$ that saturates at a few Tesla. As the gate voltage is increased to $V_g$=4 V, two saturation fields appear in the $G(B)$ curve. The $G(B)$ traces for $V_g$ = 4 V are shown in Figure 5.3(a) for various temperatures, and the two saturation fields are marked in the two-step like $G(B)$ curves at low $T$. The weakening of both steps in $G(B)$ at elevated temperatures and the positive sign of magneto-conductance suggest that both are related to the suppression of weak localization effect. We also compare the $G(B)$ data for all four $V_g$’s at $T$ ) 2 K in Figure 5.3(b) to show the evolution of $G(B)$ curve as electron density is increased.
Figure 5.3 (a) Magneto-conductance $G(B)$ at $V_g=4\,V$, and $T=2, 4, 6, 8, 10, 15, 20,$ and $30\,K$. For this gate voltage where electron density is relatively high, $G(B)$ exhibits two saturation fields marked by the black arrows. This is attributed to the existence of two 1D-subbands with very different mobilities and phase coherence lengths. (b) $G(B)$ at $T=2\,K$ for four gate voltages. At low electron density ($V_g=-8, -4\,V$) there is only one saturation field for suppression of weak localization effect (marked by colored arrows). As the gate voltage is increased to $V_g=4\,V$, two saturation fields appear in the $G(B)$ curve. (c) Schematics of electron density of states in NW vs. energy above the bottom of conduction band. The bottom (top) panel shows the occupation of one (two) subband(s) for low (high) $V_g$, $\varepsilon_F$ is the Fermi energy of the occupied electrons.

We now discuss the meaning of the saturation fields in $G(B)$ in more detail. In the weak localization theory, the suppression of quantum interference becomes significant when the magnetic flux enclosed in the closed loop of electron diffusion path is on the order of flux quanta $\hbar/2e$, where $\hbar$ is the Planck’s constant and $e$ is the electron charge. This characteristic magnetic field, associated with the suppression of weak localization, can be estimated to be $B_c \sim (\hbar/2e)(w \times l_p)$ for NW with width $w < l_p$. As $B$ increases beyond $B_c$, the weak localization will be completely destroyed above a magnetic field $B_s \sim h/2e \times l^2$ with $l$ being the electron mean free path\cite{177}. If we use $w=20\,\text{nm}$ and $l_p=40\,\text{nm}$, we calculate $B_c=2.6\,\text{T}$. And alternatively, a $B_s=3\,\text{T}$ will yield an electron mean free path of $\sim$
26 nm, corresponding to an overall sample conductance scale of \((e^2/h) \times (l/L) \approx 3 \, \mu S\), which is indeed a reasonable value for the typical conductance of our NW. Therefore, the typical saturation field of 3-4 T that we observe in \(G(B)\) measurements is in good agreement with the interpretation of complete destruction of weak localization effect. The second saturation field for \(V_g = 4\) V at higher magnetic field (~7 Tesla) would be related to a subband with \(l_p\) and \(l\) at nearly half of the values we mentioned above. The gradual filling of additional subband as \(V_g\) increases from -8 to 4 V is illustrated in Figure 5.3(c).

![Conductance vs Temperature Graph](Image)

Figure 5.4 Temperature dependence of conductance \(G(T)\) for electrons in InAs NW with 20nm diameter at gate voltages of \(V_g = 4, 0, -4, \) and \(-8\) V. The magnetic field is zero. The solid lines are fits to the 1D weak localization with \(l_p \propto T^{-1/3}\). The deviation between data and fit at \(T<10K\) is due to the saturation of \(l_p\) as shown in Figure 5.2(b).

We now discuss the temperature dependence of the conductance at zero magnetic fields, which is shown in Figure 4 for all four different gate voltages. \(G(T)\) at \(B=0\) is given by setting \(B=0\) in Eq. (5.1)
\[ G(T) = G_0 - \frac{2e^2}{h} \frac{l_\varphi}{L} \]  

(5.2)

Since we have found \( l_\varphi \propto T^{-1/3} \) in \( T \)-dependent magneto-conductance analysis, we fitted the data to Eq. (5.2) with a constant Drude conductance \( G_0 \) and \( l_\varphi \propto T^{-1/3} \). The fits are shown as the solid lines. The functional form of the fits is consistent with the data, except for some deviation at \( T < 10 \) K, which is believed to be due to the saturation of \( l_\varphi \) shown in Figure 5.2(b). However, the size of \( l_\varphi \) needed to account for \( G(T) \) at \( B=0 \) by Eq. (5.2) is about two times larger than values in Figure 5.2(b). This apparent contradiction leads us to conclude that, in addition to weak localization, there is another mechanism introducing a conductance correction proportional to \( l_\varphi \) to the Drude conductance. Thus the conductance correction due to weak localization is responsible for about half of the conductance drop of the sample as the temperature decreases at \( B=0 \). This is consistent with the weak insulating-like temperature dependence of the Drude conductance obtained in the magneto-conductance analysis, as shown in Figure 5.2(c). Such residual temperature dependence in the conductance is likely due to the electron-electron interaction correction to the conductance as other researchers later found in ZnO NWs[178].

In conclusion, we studied the temperature dependence of the magneto-conductance in a single InAs NW. One dimensional weak localization theory describes our data well and is used to extract the phase coherence length at different temperatures and gate voltages. The temperature dependence of the coherence length shows that the main dephasing mechanism is the 1D electron-electron scattering mechanism. Moreover, as the electron
density is tuned by a gate voltage, we observed in the $G(B)$ data a possible signature of the suppression of weak localization in multiple 1D subbands with different mobilities. Our results illustrate the potential of chemically synthesized InAs NWs for studying coherent electron transport with tunable 1D subband fillings.

5.3 Anisotropic magneto-conductance in a single low mobility

InAs NW

Here, we present a comprehensive magneto-transport study on a single InAs NW field-effect-transistor device. An anisotropic magneto-conductance is observed with regard to the orientation between the magnetic field and the NW axis. We show that this effect can be attributed quantitatively to the suppression of 1D weak-localization correction to NW conductance. While much work to date has been focused on studying the magneto-conductance of NWs in perpendicular[65,66,104,163,164] or occasionally, parallel magnetic field[178,179], we report the first measurement of the continuous evolution between the transverse and the longitudinal magneto-conductance, and show that the anisotropic magneto-conductance is explained by the 1D weak-localization model[157,160].

InAs NWs with 20 nm diameters were grown on silicon wafer in a thermal chemical-vapor deposition system. NWs were sonicated by ultrasound sonication and suspended in ethanol which was then dropped on silicon substrate with a thermal oxide layer on the surface for device fabrication. The 600-nm-thick oxide on silicon substrate was used as the gate dielectric and the highly doped $n$-type Si substrate itself was used as the gate
electrode. Ti/Al(2 nm/60 nm) electrodes with 2 µm spacing were evaporated on both sides of NW to serve as source and drain contacts. The device was dipped in buffered hydrofluoric acid solution for about 3 seconds before metal evaporation to remove any native oxide and ensure Ohmic contacts. The two-terminal conductance $G$ of the NW was measured by low-frequency lock-in amplifier at a constant excitation voltage of 1 mV. The magneto-transport measurement on the single InAs NW was performed in a Quantum Design physical property measurement system equipped with a rotating sample stage. Prior to cool down, the device was carefully aligned to make sure the axis of NW is parallel to the fixed magnetic field direction (the misalignment is within a few degrees). Then the computer-controlled rotator was used to rotate the NW to form an arbitrary angle between NW and the magnetic field $B$ [see Figure 5.7(a)].

Figure 5.5 Dependence of conductance $G$ on gate voltage of 20nm InAs NW at temperature $T$=40 K, 30 K, 20 K, 10 K, 5 K. The arrow shows the sweep direction of $V_g$. The top inset is a schematic of the NW device. The scanning electron microscope image of the device is shown as the bottom inset. The scale bar is 1µm.

The dependence of the InAs NW conductance $G$ on the gate voltage $V_g$ at various temperatures and zero magnetic field is shown in Figure 5.5. The upper and bottom insets
show the configuration and scanning microscope image of the device, respectively. The positive slope of $G(V_g)$ in Figure 5.5 indicates that the carrier is $n$ type, similar to reports in the literature[39,97,98,180,181]. In Figure 5.5, it can be seen that the $G(V_g)$ curves shift downward as $T$ decreases. This reduction in conductance was attributed to the weak localization correction to the Drude conductance instead of a decreasing carrier density effect[104]. Below 10 K, some small amplitude oscillations start to develop on top of the quasi-linear $G(V_g)$ dependence. These oscillations are reminiscence of interference effects of electron waves undergoing multiple reflections inside the NW channel[182]. We can estimate the electron mobility by Eq.(3.5) in which $L=2 \mu m$ is the length of NW between the electrodes. We can approximate the $C_g$ by Eq.(3.2) and estimate electron mobility

$$\mu = 205 cm^2/Vs.$$  

The electron concentration can be roughly estimated by Eq.(3.6) as

$$n(V_g) = \frac{(V_g-V_{th}) C_g e L}{(V_g-V_{th}) \times 280 \mu m^{-1}}.$$  

The threshold voltage $V_{th}$ is about $-45$ V by linearly extrapolating the $G(V_g)$ curve to zero at $T=40$ K when the quantum correction to $G$ is small. Thus, for the range of gate voltage studied ($-10$ to $+10$ V), electron concentration ranges from $1.0$ to $1.5 \times 10^4 \, m^{-1}$, or equivalently, $3.1$ to $4.8 \times 10^{19} \, cm^{-3}$. For such high electron concentrations, there are many 1D subbands filled, therefore we use the three-dimensional (3D) formula to estimate the electron mean-free path $l$, $13.1$, $14.4$, and $15.2 \, nm$, for $V_g=-10, 0$ and $+10$ V. The corresponding 3D Fermi wavelength $\lambda_F$ is $6.5, 5.9,$ and $5.6 \, nm.$
Figure 5.6 (a) InAs NW conductance vs. magnetic field at temperature $T=40$, 30, 20, 10, and 5 K in $B_\perp$ (filled symbols) and $B_\parallel$ (open symbols). The gate voltage $V_g=0$ V. Solid curves show the best fits to 1D weak-localization theory. (b) The electron phase coherence length $l_\phi$ and Drude conductance $G_0$ plotted as a function of temperature. $l_\phi$ and $G_0$ are obtained by fitting the low-field $G(B_\perp)$ or $G(B_\parallel)$ data to 1D weak-localization theory.

The magneto-conductance of NW is presented in Figure 5.6(a) for both the perpendicular field and parallel field configurations at several temperatures from $T=40$ K down to 5K. There are two main qualitative observations regarding the magneto-conductance $G(B)$. 

_First_, at $B=2$ T, both the perpendicular field $B_\perp$ and parallel field $B_\parallel$ induce a positive magneto-conductance, whose magnitude increases at lower temperature. The positive magneto-conductance in $B_\perp$ has been explained by the suppression of 1D weak localization by us in an earlier paper[104]. Above 2 T, the conductance of NW tends to saturate, due to the complete suppression of weak-localization effect[104]. _Second_, at all temperatures, the increment in $G(B_\perp)$ is always larger than $G(B_\parallel)$. This difference
between the transverse magneto-conductance \( G(B_\perp) \) and the longitudinal magneto-conductance \( G(B_\parallel) \) is the main focus of this paper. Below we will show that both effects arise from the same mechanism, namely, magnetic field suppression of 1D weak localization.

Quantum correction to the Drude conductivity in weakly disordered system at low temperatures stems from weak localization, which is constructive quantum interference of time-reversed paths formed by diffusive electrons scattered elastically by impurities or defects[141]. Such weak-localization effects have been widely observed in various disordered electronic systems and received tremendous research interests[159]. One key parameter in this process is the phase coherence length of electrons \( l_\phi \), indicating the characteristic length within which the electron maintains its phase coherence for the interference to take place. Phase coherence can be destroyed by electron-electron and electron-phonon scatterings. Such inelastic scatterings are strengthened by increasing temperature, which leads to the diminishing effect of the weak-localization effect at high temperatures. Moreover, magnetic field can be applied to introduce additional phase shifts to the electron waves traveling in time-reversed paths, which suppresses the weak localization and enhances the sample conductance. The dependence of conductance on magnetic field is usually used to extract \( l_\phi \). If \( l_\phi > w \), the width of system, the localization is regarded as 1D, and the quantum correction to conductance in magnetic field is given by Eq.(4.14) with \( l_B = l_{B\perp} = \sqrt{3\hbar/(eB_\perp\sqrt{\pi(w/2)^2})} \). In these equations, \( L=2 \mu m \) is the length of the NW, \( w=20 \) nm is the width or diameter of NW, and \( G_0 \) is the classical Drude conductance. The magnetic relaxation length \( l_B = \sqrt{D\tau_B} \), with \( D \) as the
diffusion constant and $\tau_B$ as the magnetic relaxation time which depends on the field orientation, strength, and wire cross-section shape, etc. We first fitted the low-field ($B<1$ T) $G(B_\perp)$ data in Figure 5.6(a) to Eq. (4.14). Note original equation for the perpendicular field configuration was obtained for wires with square cross section in Ref.[160]. We modified the equation to account for the round cross section of the NW. $G_0$ and $l_\varphi$ are the only two fitting parameters. The fitting curves are indicated by the solid line in Figure 5.6 (a) while the fitted phase coherence lengths $l_\varphi$ and the Drude conductance $G_0$ at various temperatures are shown in Figure 5.6(b). The above expression for $l_B \perp$ is applicable in the so-called “dirty metal” regime $l \ll w$, and Eq. (4.14) requires $\sqrt{h/eB} \gg w$ (the weak field limit). These requirements are satisfied reasonably for our parameters. As shown in the Figure 5.6(b), $l_\varphi$, ranging from 41.3 to 103.6 nm, is much larger than the diameter $w$ of the NW. This justifies the use of 1D weak localization ($l_\varphi > w$) to fit our data. It is interesting to note that the extracted $G_0$ has some temperature dependence. We believe that this residual $T$ dependence in $G_0$ is due to the electron-electron interaction effects, which also give a correction to the Drude conductance[163,178]. It is well known that, unlike weak localization, electron-electron interaction induced quantum correction to conductance is independent of magnetic field[159]. Thus, ignoring the electron-electron interaction correction in our Eq. (4.14) will not affect our study of orientation-dependent $G(B)$, which is caused by the suppression of weak localization effect.

The weak-localization correction to the conductance of a wire in parallel magnetic field was also developed by Altshuler and Aronov[160]. The analytical expression is the same
as Eq. (4.14) but with \( \ell_B = \ell_B = 2\sqrt{2}h/(eB_lw) \). We fit the \( G(B_l) \) in Figure 5.6(a) to Eq. (4.14) using the expression for \( \ell_B \) and found a good agreement. The extracted \( \ell_\phi \) and \( G_0 \) are shown in Figure 5.6(b) to compare with the results from fitting \( G(B_l) \) data. There is a good agreement. Therefore, both the perpendicular and parallel field-induced magneto-conductances are consistent with the localization effect with the same parameters. Before we proceed to the angular dependence of \( G(B) \), we like to make a few comments on the physical origin of the stronger \( G(B) \) in the perpendicular configuration. Evidently, \( \ell_{B_\perp} > \ell_{B_\parallel} \), this means that a perpendicular field is more effective than a parallel field in suppressing the localization effect. This difference is related to the 1D size confinement effect of coherent electron waves (\( \ell_\phi > w \)). In the parallel field configuration, electron diffusion paths enclosing magnetic flux are well confined within the circumference of the NW while for the perpendicular field case, they are confined by NW diameter \( w \) in the radial direction and \( \ell_\phi \) in the axial direction. So the magnetic fluxes enclosed by electron diffusion paths in perpendicular field are larger in the perpendicular field configuration. Thus, the additional phase shift introduced by a small magnetic field for \( B_\perp \) is larger than \( B_\parallel \), which leads to stronger suppression of weak localization effect.
To further test the conclusion that the anisotropic magneto-conductance of our InAs NW has the same origin of magnetic field suppression of 1D weak localization, we carried out an angle-dependent experiment of the magneto-conductance at various temperatures in a fixed magnetic field of 1 T. The NW was carefully aligned to make sure it could be rotated about the axis perpendicular to both the magnetic field and the NW, as shown in Figure 5.7(a). The rotation angle $\theta$ is defined to be the angle between magnetic field and the cross-section plane of NW. The experimental data of angle dependence of magneto-conductance for $\theta=0^\circ$ to $360^\circ$ are given in Figure 5.7(b). The gate voltage $V_g$ was set to 0 V. According to our definition of $\theta$, $B$ is exactly perpendicular to the NW at $\theta=0^\circ$ and $180^\circ$, and is exactly parallel to NW at $\theta=90^\circ$ and $270^\circ$. Obviously, since that magneto-conductance in perpendicular field is larger than in parallel field, $G(\theta)$ data in Figure 5.7(b) shows peaks at $0^\circ$, $180^\circ$, and $360^\circ$, and dips at $90^\circ$ and $270^\circ$. The small deviations of the peak and dip positions from $0^\circ$, $90^\circ$, $180^\circ$, and $270^\circ$ may be caused by the
misalignment of NW relative to magnetic field. In addition, the oscillatory amplitudes of \( G(\theta) \) decrease as the temperature increases because of the weakening of the weak-localization effect at high temperatures. In order to explain the experimental data at an arbitrary angle \( \theta \), we decompose the magnetic field to a perpendicular component and a parallel component. These two orthogonal magnetic fields will both contribute to the suppression of electron localization in NW but with different magnitudes, as discussed earlier. The overall magneto-conductance is then given by

\[
G(B) = G_0 - \frac{2e^2}{hL} \left( \frac{1}{L_{\parallel}^2} + \frac{1}{L_{\perp}^2} + \frac{1}{L_{\parallel}^2} \right)^{-1/2}
\]

(5.3)

In Eq. (5.3), \( l_{\parallel} = l_{\perp} = \sqrt{3}\hbar/(eB\cos\theta\sqrt{\pi(w/2)^2}) \) and \( l_{\parallel} = l_{\parallel} = 2\sqrt{2}\hbar/(ewB\cos\theta) \) are the magnetic relaxation lengths for the perpendicular and parallel components of \( B \). The solid curves in Figure 5.7(b) indicate the theoretical fits to Eq. (5.3), with \( G_0 \), \( l_{\phi} \) fixed at the averaged values of data in Figure 5.6(b), and a misalignment angle offset was set as the only free parameter. The fitted angle offset is within 10°. This good agreement between \( G(\theta) \) data and theory further supports that the anisotropic magneto-conductance of NW is consistently explained by the 1D weak-localization theory.

In conclusion, angle dependence of the anisotropic magneto-conductance of a single InAs NW with 20 nm diameter is studied. The data are consistent with a modified model of magnetic field suppression of 1D weak localization where the magnetic field forms an arbitrary angle with the NW. Similar angle-dependent magneto-conductance measurements on 1D quantum wires may be useful to explore other effects such as spin-orbit coupling effects in semiconductor NWs\[183,184\].
5.4 Suppression of weak anti-localization in a single high mobility InAs NW

Pure high mobility InAs NWs were grown in vacuum tight CVD system as shown in Section 2.3. A single NW (25 nm in diameter) was used to fabricate FET device on Si substrate capped with 300 nm SiO$_2$ as mentioned in Section 5.2. The mobility of the device is around 500 $cm^2/Vs$. The corresponding mean free path $l$ is about 20 nm. The transport is still in dirty metal region.

The transverse magneto-conductance exhibits the signature of weak anti-localization as shown in Figure 5.8(a), corroborating the strong SOI in pure InAs NWs. The open symbols are the data; the solid lines are theoretical fitting by Eq.(4.13). As temperature rises, transition from weak anti-localization to weak localization is observed arising from the decrease of $l_\varphi$ with temperature due to electron-electron interaction. While $l_{so}$ doesn’t change much.

![Figure 5.8](image)

Figure 5.8 (a) Magneto-conductance of a single 20 nm InAs NW at various temperatures. (b) $l_\varphi$ and $l_{so}$ obtained from fitting using Eq.(4.13).
5.5 Tuning of spin orbit interaction in a single high mobility

InAs NW by floating and electrolyte surrounding gate

Manipulating electron’s spin in solid state devices has been the main target of spintronics[153,185]. A key concept in many spintronic devices is to apply gate voltage or electric field (E) to tune spin precession via the Rashba spin orbit interaction (SOI)[186]. Quasi-one dimensional (1D) indium arsenide (InAs) NWs are promising platforms in this regard due to InAs’s strong intrinsic SOI and more effective control of Rashba SOI in 1D systems with uni-directional momentum[186]. Here, we demonstrate efficient control of Rashba SOI where $E$ is created at the surface of InAs NW by solid electrolyte surrounding gate. Six-fold tuning of Rashba coefficient and 2.5 order of magnitude tuning of spin relaxation time are achieved within only 1 V of gate bias. Such a dramatic tuning of SOI paves a way towards quasi-1D NW spintronic devices with low power consumption.

Spin orbit interaction (SOI) offers us a powerful tool to manipulate electron spin in semiconductors. With SOI, motion of electrons giving rise to an effective magnetic field in the rest frame of electrons, couples to electrons spin and consequently induces spin precession and relaxation of moving electrons[143]. Moreover, Rashba SOI renders electron spin manipulation in electrical means through gate bias[145,187], which is underscored in the concept of spin field effect transistor as a hopeful paradigm. Although the first spin field effect transistor was proposed in a two-dimensional electron gas, we expect realizing spin field transistor in a quasi-one-dimensional (quasi-1D) semiconductor NW due to more effective control of Rashba SOI. In Rashba SOI,
effective magnetic field induced is proportional and perpendicular to the electron net momentum and gradient of electrical potential. Dimensional confinement results in more uni-directional net momentum in a quasi-1D NW than in a two dimensional electron gas[186]. In addition, due to large surface to volume ratio, surface gate is able to tune the electrical potential gradient efficiently. Therefore, we expect more effective control of Rashba effect in a NW. InAs NW is promising candidate with large intrinsic spin orbit interaction and significant dimensional confinement due to small effective of mass[143].

With many explorations made to take the advantage of SOI in two-dimensional electron gas[188-192], seeking for efficient control of electrons spin in quasi-1D quantum wires has set off. All the purposes are to find an effective approach to widely tune the spin orbit interaction by gate bias. Spin relaxation and electron phase coherence of InAs[65,66,103,104,193], InN NWs[194] have been investigated. However, electron spin relaxation length was tuned only by SiO$_2$ back gate in those efforts, which results in no significant tuning. The main shortcomings of SiO$_2$ back gate are the small dielectric constant (~3.9), large thickness of oxide layer (several hundred nm), and complicated distribution of electrical potential. To make better use of Rashba SOI, alternatives should be sought to overcome these hurdles.

Here, we explored two approaches of electric field tuning of Rashba SOI in InAs NWs. In the first approach, two floating gates (top and bottom gates) were used to apply electric field across the NW. Such configuration was able to accommodate $10^5$ V/cm of $E$ which in turn modulates the spin relaxation time by a factor of five. We also clarify the spin relaxation in as-grown InAs NWs to be the D’yakonov-Perel mechanism. Then we demonstrated strong (more than two orders of magnitude) tuning of SOI in a single InAs
NW gated by a solid electrolyte surrounding gate, namely mixture of polyethylene oxide (PEO) and lithium perchlorate (LiClO$_4$). We achieved strong tuning of spin relaxation time by 2.5 order of magnitude and tuning of corresponding Rashba coefficient by six-fold at 2 K. The solid electrolyte PEO/LiClO$_4$, embracing large double-layer capacitance due to large dielectric constant and short Debye length, exhibits very strong gating effect. 1V gate voltage is enough to induce large $E$ of the order $10^7$ V/cm.

The as-grown [111] NWs of diameter about 25 nm on substrate were then ultra-sonicated and suspended in ethanol alcohol solution which was drop onto $p$-type silicon substrate capped with 300nm silicon dioxide through which back gate can be applied. Photolithography and electron-beam evaporation were used to define 60 nm thick and 2 $\mu$m spaced Nickel electrodes as source and drain contacts. The sample was dipped in 0.5% HF for 3 seconds before evaporation to remove native oxide and ensure Ohmic contacts. To fabricate NW FETs with floating gate, PMMA 950C2 was spin-coated on the chips after lift-off, yielding 1 $\mu$m thick PMMA thin layer on top of the NWs. Then top gate electrode aluminum was evaporated on top on PMMA layer. To fabricate NW FETs with solid electrolyte surrounding gate, the sample was again dipped in buffer HF for 1 minute after lift-off to partly remove silicon dioxide layer beneath the NWs so that the NWs were suspended in air and a small gap was created between the NWs and substrate. Then PEO/LiClO$_4$(8:1 in weight ratio) dissolved in methanol[195] was spin-coated on the sample chip at a speed of 1500 rpm and the sample was baked at 90ºC until residual moisture and methanol were removed. Silver paste was applied on the PEO film to serve as gate electrode. The device schematic and SEM image are shown in Figure
5.10. Two-terminal NW devices were measured by low frequency lock-in technique in a Quantum Design PPMS cryostat.

As discussed in Section 4.5, Magneto-transport studies of quantum interference effects are known to be useful in the quantitative extraction of SOI in diffusive electronic systems. By analyzing weak-field magneto-conductance in diffusive InAs NWs at low temperatures, phase coherence length \( l_{\phi} \) (dephasing time \( \tau_{\phi} \)) and spin relaxation length \( l_{so} \) (spin relaxation time \( \tau_{so} \)) can be extracted to indicate the strength of phase coherence and spin orbit interaction tuned by gate-voltage-induced electric field \( E \) as shown in the following.

The schematic of floating-gated InAs NW FET is shown in the inset of Figure 5.9 (a). 300 nm SiO\(_2\) and 1 \( \mu \)m PMMA served as back and top gate dielectric respectively. Between top gate electrode and back gate silicon substrate was applied a floating gate voltage \( V_{(t-b)g} \) to induce an electric field across the NW without changing the electron line density \( n \) or Fermi velocity \( v_F \). The constant \( n \) is estimated to be 7000/\( \mu m \). Although the electron density \( n \) remains unchanged with the application of \( E \) across the nanowire under this doubly gated configuration, we note that the conductance \( G \) of InAs nanowire \( G \) decreases when sweeping \( V_{(t-b)g} \) from negative to positive voltages, as shown in Figure 5.9 (a). This indicates there is stronger scattering at the nanowire-PMMA interface when the electrons are attracted more towards the PMMA side of the dielectrics at positive \( V_{(t-b)g} \). The momentum scattering time \( \tau \), which is linearly proportional to \( G \), can be obtained by the expression \( \tau = \frac{m^*L}{ne^2}G \) where \( m^* \) is the electron effective mass, \( L \) is the
channel length, e is electron charge. Figure 5.9 (a) shows conductance $G$ and $\tau$ a function of $V_{(t-b)g}$.

Figure 5.9 (a) Conductance $G$ and corresponding momentum scattering time $\tau$ as a function of floating gate voltage $V_{(t-b)g}$. The inset is the schematic of a single InAs field effect transistor using PMMA as top gate. (b) Magneto-conductance as a function of perpendicular magnetic field as various $V_{(t-b)g}$’s. (c)spin relaxation length $l_{so}$ and phase coherence length $l_{\phi}$ obtained from fitting to the magneto-conductance data. (d) Spin relaxation time $\tau_{so}$ and elastic scattering time $\tau$ as a function of floating gate voltage.

Magneto-conductance in perpendicular magnetic field within 1 Tesla was measured at $T=2$ K at various $V_{(t-b)g}$ ’s as shown by symbols in Figure 5.9(b). Note the magneto-conductance $G(B)$ is shifted for clarity by subtracting the zero field magneto-
conductance $G(0)$ in the Figure. At positive(negative) gate voltages, the positive(negative) magneto-conductance is due to suppression of weak localization(anti-localization) due to external magnetic field. Crossover from weak localization to weak anti-localization was clearly shown when sweeping $V_{(r-b)g}$ from 20V to -30V. In our case, mean free path $l_0$ is smaller than the NW diameter $w=40$ nm. The transport is in dirty metal region where $G(B)$ in quasi-1D system can be described by Eq.(4.13) where $G_0$ is the classical Drude conductance, $L=2\mu$m is the length of NW, $h$ or $\hbar$ is Planck constant. The magnetic relaxation length is expressed as $l_e = \frac{\sqrt{3}h}{eBw}$. $G_0$, $l_e$ and $l_{so}$ are fitting parameters in Eq.(4.13). We restricted the fitting at the magnetic field smaller than 1 Tesla to fulfill small field limit $\sqrt{\hbar/eB} > w$. Solid curves in Figure 5.9(b) represent the fitting to the experimental data. Spin relaxation length $l_{so}$ and phase coherence length $l_{\varphi}$ at a number of $V_{(r-b)g}$’s are obtained as shown in Figure 5.9(c). Note Eq.(4.13) is only valid at $l_{\varphi} > w$, which is fulfilled by fitting values. We observe $l_{\varphi}$ decreases from negative to positive $V_{(r-b)g}$ due to the decrease of diffusion constant. Spin relaxation length $l_{so}$ is symmetrically tuned by $V_{(r-b)g}$. $l_{so}$ decreases by 1.5 times while increasing the absolute values of $V_{(r-b)g}$ to 30 V, indicating the SOI strength was enhanced by increasing strength of electric field across the NW, which is a distinct signature of Rashba spin orbit interaction. The Rashba SOI coefficient $\alpha = \frac{\hbar^2}{2m^*l_{so}}$ is also tuned 1.5 times by $V_{(r-b)g}$.
Although applying floating gate voltages doesn’t yield strong tuning of spin orbit interaction due to large thickness of dielectric layer, it does offer us the clue to clarify the spin relaxation mechanism in as-grown InAs NWs. Two classes of mechanism might contribute to the spin relaxation of conduction electrons. One is D’yakonov-Perel’ (DP) mechanism[152] in system lacking inversion symmetry such as InAs bulk or quantum heterostructure, originating from electron spin relaxation in a SOI-induced effective magnetic field which is perpendicular to the wave vector \( \vec{k} \) and thus fluctuates rapidly between scattering events; the other is known as Elliott-Yafet (EY) mechanism[149,150], originating from spin relaxation of conduction electrons at every scattering event due to spin orbit interaction with lattice ions, impurities and defects. The most frequently used way to distinguish the two mechanisms is to investigate the dependence of spin relaxation time \( \tau_{so} \) on the momentum scattering time \( \tau \). For the DP mechanism, \( \tau_{so} \) is inversely proportional to \( \tau \), namely, \( \tau_{so} \propto 1/\tau \); while for EY mechanism, \( \tau_{so} \) is proportional to \( \tau \), namely \( \tau_{so} \propto \tau \). Applying floating gate voltages to InAs FET gives rise to two effects: first, raising the absolute value of \( V_{(t-b)g} \) results in enhancement of electric field \( E \) imposed on the NW; Sweeping \( V_{(t-b)g} \) from negative to positive induces almost linear monotonic decrease of momentum scattering time \( \tau \). If the EY mechanism dominates, \( \tau_{so} \propto \tau \), \( \tau_{so} \) should increase with increasing \( \tau \) at negative \( V_{(t-b)g} \)’s even though the induced electric field tends to shorten the \( \tau_{so} \). This assumption contradicts with the experiment shown in Figure 5.9(d). If DP mechanism dominates, \( \tau_{so} \propto 1/\tau \). At negative \( V_{(t-b)g} \)’s, increasing \( \tau \) should shorten \( \tau_{so} \). Meanwhile, electric field induced by \( V_{(t-b)g} \) also shortens \( \tau_{so} \). Both effects strengthen the decrease of \( \tau_{so} \). At positive \( V_{(t-b)g} \)’s, decreasing
\( \tau \) lengthens \( \tau_{so} \), whereas applied electric field prefers to shorten \( \tau_{so} \), the two take effects oppositely. This explains why the tuning range of \( \tau_{so} \) is stronger in negative \( V_{(r-b)g} \)'s than in the positives. The domination of DP mechanism agrees well with the experimental data in Figure 5.9(d). Therefore, we conclude that DP mechanism still dominates in as-grown InAs NWs.

Figure 5.10 (a) Three dimensional schematic view of the suspended InAs NW field effect transistor coated with PEO/LiClO\(_4\) solid electrolyte. (b) SEM image of suspended InAs NW. (c) Conductance versus PEO/LiClO\(_4\) surrounding gate \( V_{PEO} \) and back gate \( V_{bg} \) at \( T = 200K \);

To further tune the SOI, the solid electrolyte PEO/LiClO\(_4\) was applied as surrounding gate dielectric material. The solid electrolyte is believed to be low-cost and easily processed[196], in which lithium ions driven by gate voltage diffuse in a PEO matrix and stop at the NW-electrolyte interface so that double-layer of opposing charges can be formed on the surface of NWs. It is the double-layer that possesses large capacitance due to large dielectric constant of PEO/LiClO\(_4\) and short Debye length. The strong gating effect of PEO/LiClO\(_4\) has been demonstrated in carbon nanotube[195,197] and graphene field effect transistor[198]. We suspended InAs NW and applied solid electrolyte as surrounding surface gate to induce the central symmetric \( E \) imposed on the NW. The
schematic of suspended InAs NW field effect transistor surrounding-gated by PEO/LiClO₄ is shown in Figure 5.10(a), Figure 5.10(b) is the SEM image of suspended InAs NW after removing PEO. Dependences of InAs NW conductance $G$ on PEO/LiClO₄ surrounding gate voltage $V_{PEO}$ and back gate voltage $V_{bg}$ at zero magnetic field are shown in Figure 5.10(c), respectively at 200K and zero magnetic field. The positive slope of $G(V_{PEO})$ and $G(V_{g})$ indicate the carriers are $n$-type. It can be obviously seen from transconductance that the gate effect of $V_{PEO}$ is 40 times larger than that of $V_{bg}$, indicating strong gate capacitance of solid electrolyte. We estimate the surrounding gate capacitance $C_s$ to be $1.44 \times 10^{-14} F$ by $C_s = 2\pi \varepsilon_0 \varepsilon L / \ln(1 + \lambda_D / r)$[195], where $\varepsilon_0$ is the permittivity, $\varepsilon$ is the dielectric constant of PEO to be $\sim 10$, $L=2 \mu m$ and $r=12.5$ nm are the length and radius of NW, respectively. We assume Debye length $\lambda_D$ is 1 nm[197]. According Eq.(3.5) and Eq.(3.6), one is able to estimate the carrier density $n$ to be $9000 / \mu m$ and mobility $\mu$ to be about $600 cm^2/V \cdot s$ at $V_{PEO}=0.4V$.

Figure 5.11(a) and (b) show the experimental magneto-conductance $G(B)$-$G(0)$ at $T=4$ K at various $V_{PEO}$’s and $V_{bg}$’s, respectively. By increasing $V_{PEO}$ from 0.3V to 0.9V, as shown in Figure 5.11(a) (a), magneto-conductance at $T=4$ K in perpendicular magnetic field $B$(-1 to 1 Tesla) transits gradually from pure positive to pure negative. This crossover from weak localization to weak anti-localization clearly indicates $V_{PEO}$ strongly tunes the strength of SOI over a wide range. Whereas large $V_{bg}$ up to 35V is needed to make the crossover happen and still is not large enough to tune the magneto-conductance pure negative, which indicates the weak tuning of SOI by $V_{bg}$. Parameters $l_\phi$ and $l_{so}$ are fitted by Eq.(4.13) in which $l_\phi = \sqrt{3h / eBw}$ with $L=2 \mu m$ and $w=25$ nm and extracted as a
function of $V_{\text{PEO}}$ and $V_{\text{bg}}$ at $T=4$ K shown in Figure 5.11(c). $l_\phi > w$ proves the validity of Eq.(4.13). Comparison of the gate tuning effects on SOI strength between $V_{\text{PEO}}$ and $V_{\text{bg}}$ is quantitatively shown in this figure. At $T=4$ K, $l_{so}$ is tuned from 55nm to 220nm by $V_{\text{PEO}}$, whereas $l_{so}$ can only be tuned from 65nm to 100nm by $V_{\text{bg}}$. The comparison clearly corroborates the strong tuning of SOI by surface electrolyte gate.

Figure 5.11 The magneto-conductance $G(B)-G(0)$ as a function of perpendicular magnetic field $B$ at $T=4K$ for different PEO/LiClO$_4$ gate voltages (a) and back gate voltages(b). The circles are experimental data and the solid lines are theoretical fitting by Eq.(4.13). (c) Spin relaxation length $l_{so}$ and phase coherence length $l_\phi$ obtained by theoretical fitting for both PEO/LiClO$_4$ gate and back gate.
Figure 5.12 (a) $\tau_{so}$ and $\tau_{\phi}$ as a function of PEO/LiClO$_4$ gate voltage $V_{PEO}$; (b) Temperature dependence of dephasing time $\tau_{\phi}$ follows $\tau_{\phi} \sim T^{-2/3}$ at $V_{PEO}$=0.4, 0.5, 0.7V; (c) $\tau_{so}$ versus temperature for $V_{PEO}$=0.35, 0.4, 0.5, 0.7V.

We measured all the magneto-conductance for a series of $V_{PEO}$’s at temperatures from 2 K to 15 K. All the data show the similar transition from weak localization to weak anti-localization by increasing $V_{PEO}$ as typically shown in Figure 5.11(a). The corresponding spin relaxation time $\tau_{so}$ and dephasing time $\tau_{\phi}$ at various temperatures are obtained in Figure 5.12(a) in which spin relaxation time $\tau_{so}$ decreases sharply by 2.5 orders of magnitude as $V_{PEO}$ increases at $T$=2 K. By contrast, the dephasing time $\tau_{\phi}$ shows small changes as we increase voltages at each temperature. The dependence of dephasing time $\tau_{\phi}$ on temperature shown in Figure 5.12(b) follows a power low of $T^{-2/3}$, consistent with the quasi-1D Nyquist dephasing mechanism being electron-electron interaction with small energy transfers as observed in many other systems[166,168]. Electron-electron
scattering time $\tau_{ee}$ is of the same order as $\tau_\phi$ to be $\sim 1$ ps, much larger than $\tau$ of the order $10^{-15}$ fs. This indicates electron-impurity or electron-defect scattering is dominant in momentum scattering. We further investigate the temperature dependence of $\tau_{so}$ as shown by Figure 5.12(c). For small voltage at $V_{PEO}=0.35$ and $0.4V$, $\tau_{so}$ decreases with temperature. For medium and high voltage at $V_{PEO} =0.5, 0.7V$, $\tau_{so}$ remain almost unchanged. At small gate voltages, $\tau_{so}$ decreases with increasing temperature. This might stem from the spin relaxation due to inhomogeneous broadening from DP term for degenerate electrons highly electron-impurity scattering[199]. This is consistent with the fitting data in Figure 5.12(b) where dramatic change of $\tau_{so}$ takes place at low $V_{PEO}=0.35$ V and $0.4$ V.

Figure 5.13 (a)Rashba spin orbit interaction strength $\alpha$ obtained by $\alpha = \hbar^2/2m^*l_{so}$ at $T=2, 4, 8, 15K$, our data are represented by solid symbols. We cited $\alpha$ obtained in InGaAs quantum well(hollow hexagons), InGaAs quantum wires(hollow triangles) and InAs quantum well(hollow squares) (b) Schematic of electric field $E(r)$ profile(red curve) in a single InAs NW with PEO/LiClO$_4$ surrounding gate. Blue dots represent the lithium ions and red dots represent electrons.

<table>
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<tr>
<th>$V_{PEO}$ (V)</th>
<th>$\tau_{so}$ (fs)</th>
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<tr>
<td>0.35</td>
<td>0.5</td>
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<tr>
<td>0.4</td>
<td>1.0</td>
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<td>0.5</td>
<td>2.0</td>
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<td>0.7</td>
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InAs nanowire, this work
InGaAs quantum well, $T=0.4K$, Ref.[188]
InGaAs quantum wire, $T=0.6K$, Ref [201]
InAs quantum well, $T=1.8K$, Ref [202]

E(r)
InAs nanowire
Dominant DP spin relaxation mechanism is contributed by two type of SOI including Dresselhaus and Rashba SOI. For the InAs NWs grown along [111] direction, Dresselhaus SOI due to bulk inversion asymmetry is suppressed[144]. Moreover, both theory and experiment show that Rashba spin orbit interaction dominates in InAs based quantum well[200,201]. Therefore Rashba spin orbit interaction plays the main role in DP spin relaxation mechanism. In addition, it is reported that electrons tend to accumulate near the surface of InAs due to surface Fermi level pinning[49]. Combing the surface accumulation layer with strong electrolyte surface gate effect, we expect that strong electric field can be induced near the surface of the NW as shown by schematic Figure 5.13(b). We extract the Rashba coefficient $\alpha = h^2/2m^*l_{so}$ at different temperatures as shown in Figure 5.13(a). $\alpha$ can be tuned by six fold by tuning the $V_{PEO}$ from 0.3 V to 1 V at $T=2$ K. In the figure, we compare tuning of $\alpha$ by electrolyte surface gate in our InAs NW with that obtained in InGaAs-based quantum well[189], InGaAs quantum wires[202] and InAs quantum well[203]. It is clear that we achieve stronger tuning SOI by solid electrolyte gate compared to results in Ref. [189] and [202]. A schematic of electric field profile is shown in Figure 5.13(b) in the radial direction of the NW. The larger PEO/LiClO$_4$ gate voltage is applied, the steeper is the potential gradient, and thus the stronger is the electric field induced near the surface of the NW, leading to a wide range tuning of Rashba SOI in the NW. Here, $\alpha$ is expressed in terms of average electric field by the expression $\alpha = \alpha_0 e\langle E \rangle$ in which $\alpha_0 = 117 A^2$ is Rashba constant in bulk InAs[143]. $\langle E \rangle$ is the average electric field imposed on NW. For spin relaxation length $l_{so} = 50nm$, we estimate the electric field strength $\langle E \rangle \approx 10^6 V/cm$, which is reasonable
than the peak value $10^7$ V/cm calculated by capacitance of PEO/LiClO$_4$ electrolyte gate at the electrolyte/nanowire interface toward the center of nanowire due to carrier screening.

In conclusion, we clarify DP spin relaxation mechanism dominates in our as-grown InAs NWs by investigating the magneto-conductance of NW FET with floating gate. Then we demonstrate strong tuning of Rashba SOI in a single InAs NW by applying strong solid electrolyte PEO/LiClO$_4$ surrounding gate. By means of quasi-1D magneto-transport theory, we extracted spin relaxation time and dephasing time from the fitting to experimental data. Temperature dependence of dephasing time agrees well with 1D Nyquist dephasing mechanism, which corroborates the validity of quasi-1D theory used in our analysis. More importantly, Spin relaxation time is widely tuned by 2.5 orders of magnitude within 1V of electrolyte gate. Since Rashba SOI is dominant in the as-grown InAs NW. The achievement of strong tuning of Rashba coefficient $\alpha$ by six-fold enhances the possibility to apply InAs NWs as the key components to realize nano-sized spin field effect transistor and further multi-functional spintronics devices. The flexibility of this technology also lowers the threshold to achieve electric-field-induced phenomena in other nano-structure system.

### 5.6 Summary

In this Chapter, we have investigated the quantum transport in a single InAs NW. For low mobility O incorporated InAs NWs, magneto-conductance in these InAs wires at low temperature is in good agreement with quasi-1D weak localization theory in small magnetic field. The evolution of magneto-conductance from transverse to longitudinal
case exhibits anisotropic property. We attribute the anisotropy to the size confinement of time reversed loop paths. In contrast, high mobility pure InAs NWs with correct stoichiometry were grown after improving CVD vacuum sealing and show weak anti-localization effect at low temperature due to strong SOI. The magneto-transport of such wires is useful in quantitative determination of spin relaxation length and thus the strength of SOI. Utilizing this methodology, we demonstrated the symmetric tuning of Rashba SOI in a floating double gated InAs NW and realized six-fold tuning of Rashba SOI in a surrounding electrolyte gated InAs NW, which is much stronger than ever reported in InAs NWs.
Chapter 6  Magneto-transport in
Topological Insulator Bi$_2$Se$_3$

Nanoribbons

6.1  Topological insulator

Regular insulator possesses large band gap between top of valence band and bottom of conduction band so that localized electrons cannot be excited to the conduction band and make no contribution to the current as shown in Figure 6.1(a) and (b).
Figure 6.1 (a,b) Electrons in an insulator are bound in localized orbitals and have an energy gap (right) separating the occupied valence band from the empty conduction band. (c,d) A two-dimensional quantum Hall state in a strong magnetic field has a bulk energy gap like an insulator but permits electrical conduction in one-dimensional “one way” edge states along the sample boundary. (e,f) The quantum spin Hall state at zero magnetic field also has a bulk energy gap but allows conduction in spin-filtered edge states (figures cited from [204]).

In quantum Hall effect, the sample is insulating inside but conducting along the edge. The plateaus of Hall conductance $\sigma_{xy} = m e^2/h$ (m is integer) stem from current carried by continuous circulation motion of carriers along the sample edge in high external magnetic field $B$. The edge states is topologically robust again impurities. As shown in Figure 6.1 (c), carriers circulate along the edge are able to adjust their paths according to scattering with impurities so that the whole circulation is still achieved in appearance of the impurities. The conducting edge states form energy band link the valence band and conduction band as shown in Figure 6.1(d).

In the absence of external magnetic field, strong SOI relativistically transforms local crystal field plus the momentum of charge carriers into effective magnetic field coupled with carrier spin. Analogous to the quantum Hall effect in which electrons interact with external magnetic field, the carrier spins interact with the effective magnetic field. As a result, carriers of spin-up and spin-down would undergo opposite circulation along the edge as shown in Figure 6.1(e). The spin-polarized circulation gives rise to quantized conductance plateaus $\sigma_{xy} = (m_\uparrow + m_\downarrow)e^2/h$ (where $m_\uparrow$ and $m_\downarrow$ m are integers), which is named quantum spin Hall effect[205-209]. The circulation loops is spin-dependent since spins are locked to the circulation direction or momentum as shown in Figure 6.1(f). The edge quantum spin Hall states are topologically robust against nonmagnetic impurities similar to that in quantum Hall effect. The quantum well exhibiting quantum spin Hall
effect is called 2D topological insulator, which was realized recently in HgTe quantum well[210,211].

Topological insulator in 3D is a new type of quantum materials with conducting gapless surface states in the insulating bulk band gap[212]. In the bulk, the band structure is similar to ordinary insulator. The uniqueness lies on helical Dirac cone of surface states within the band gap as shown in Figure 6.2(a). The 3D helical Dirac cone is shown in Figure 6.2(b). The surface states are induced by strong spin orbit interaction through which carrier spin is strongly coupled and locked with carrier momentum[213] as shown in Figure 6.2(c). Such a strong coupling causes the conduction-valence band edge inversion, leading to the formation of helical Dirac cone in the band gap. A special property of topological insulator in transport is that the 180° back-scattering is forbidden due to the spin-momentum locking.

![Figure 6.2](image-url)

Figure 6.2 (a) Schematic of surface state formed in the band gap for topological insulator. (b)3D view of helical Dirac cone in the band gap. (c) Top view of Dirac cone showing the spin is locked with momentum.
The history of topological insulator goes as follows. C.L.Kane and E.J.Mele invented a model for graphene with SOI, which revealed an insulating state with robust helical edge modes. This achievement laid theoretical foundation for topological insulator[214,215]. Then C.L.Kane, L.Fu, S.C.Zhang, X.Qi further developed the theory[216-220]. D.Hsieh, et al discovered the topological insulator phase in Bi0.5Sb0.1 [221]. Recently, H. Zhang et al predicted Bi2Se3, Bi2Te3 and Sb2Te3 are topological insulator[52], as shown in Figure 6.3(a). Then, topological insulator helical Dirac states were discovered in Bi2Se3[51], Bi2Te3[222] and Sb2Te3[223] and by using surface analysis method angle-resolved photo emission spectroscopy (APRES). Figure 6.3(b) and (c) are high resolution APRES measurement of Bi2Se3 surface states with Fermi level lying above the Dirac point and bottom of conduction band.

![Theoretical calculation of band structure of Bi2Se3 on (111) surface, showing surface cone in the band gap.](image1)

**Figure 6.3** (a) Theoretical calculation of band structure of Bi2Se3 on (111) surface, showing surface cone in the band gap. The red regions indicate bulk bands and the blue regions indicate bulk band gaps. The surface states can be clearly seen around the Γ point as red lines dispersing in the bulk gap for Bi2Se3 (cited from [52]). (b,c) High resolution APRES measurement of surface electron band dispersion on Bi2Se3 (111) surface (cited from [51]). The dash line represent the Fermi level above the Dirac point and bottom of conduction band.

6.2 **Coherent quantum transport in Bi2Se3 and Bi2Te3 films and nanoribbons**
Topological insulator holds great promises in the fundamental study of topological ordering in condensed matter systems and applications in spintronic devices for the spin polarized surface state[224]. The spin polarization and suppressed back scattering render 2D topological surface state an attractive platform for high mobility charge and spin transport devices. All these proposed applications of topological insulator rely on the transport properties of unique conducting surface states of massless Dirac fermions. People are interested in finding the signature of topological insulator in transport experiment. Bi$_2$Se$_3$ and Bi$_2$Te$_3$ is considered as a promising topological insulator for its relative large bulk band gap (~0.3eV) and single Dirac cone on the surface[51]. Even though both Bi$_2$Se and Bi$_2$Te$_3$ are usually $n$-type due to the bulk carrier contribution which covers the significance of surface states in transport, significant efforts have been taken to overcome this hurdle by adjusting doping or stoichiometric ratio of the materials[213,225-228] or applying large gate voltage[229-232] to tune the chemical potential or Fermi level down close to the helical Dirac point. On the other hand, even though the circumstance of $n$-type carriers which render the Fermi level above the bottom of conduction band[51,228,233], angle and thickness dependence of quantum magnetoconductance (weak anti-localization and SdH oscillation) provides an alternative to identify the topological surface states[234-237].

Since topological insulator is contributed by the presence of strong SOI, it is reasonable to observe the weak anti-localization effect in transport arising from the spin-dependent quantum interference of time reversed loop paths. $\pi$ Berry’s phase associated with the helical surface states[217] should results in weak anti-localization in electron transport. However, a problem is that both surface and bulk states contribute to the weak anti-
localization in quantum transport. Strong SOI of both bulk carriers also leads to weak anti-localization. Fortunately, the surface states don’t contribute to the magneto-conductance in the magnetic field parallel to quintuple layer planes or perpendicular to c-axis. By tilting the angle between the c-axis and magnetic field, we are able to distinguish the surface states from bulk states.

H.T.He, et al reported the study of magneto-resistance (MR) in thin layer Bi₂Te₃ film[234]. In Figure 6.4(a) for thin Bi₂Te₃ film of thickness 5 nm, they showed the MR in perpendicular field presents a sharp cusp, the signature of weak anti-localization; while MR shows parabolic $B$ dependence in parallel field, which results from Lorentz deflection obeying Kohler’s rule $R(B) / R(0) = 1 + (\mu B)^2$ [238], where $\mu$ is the mobility of the sample. The disappearance of weak anti-localization in parallel field provides strong evidence to that the weak anti-localization in perpendicular field stems from surface states. Figure 6.4(b) shows that the magneto-conductance data as a function of perpendicular component of magnetic field at various tilted angle overlap at small field, further confirming the 2D nature of weak anti-localization. In Figure 6.4(c) for thick plate with thickness of 50 nm, the weak anti-localization effect persists in parallel field, showing the coexistence of bulk contribution to the weak anti-localization. Since bulk weak anti-localization effect does not depend on tilted angle, transverse magneto-conductance as a function of perpendicular component of magnetic field can be extracted by subtracting bulk contribution, as shown in Figure 6.4(d).
Figure 6.4 Normalized MR of a 5 nm Bi$_2$Te$_3$ film (a) and a 50 nm Bi$_2$Te$_3$ film (c) measured in tilted B fields at $T=2$ K. Solid curve in (a) is a parabolic fit to the MR data measured in parallel B field. Magneto-conductance as a function of the perpendicular B component with the parallel magneto-conductance subtracted for 5 nm sample (b) and 50 nm sample (d). Solid curve in (d) is a fit to anti-localization theory in the low B-field region. (cited from Ref. [234])

The surface magnetic doping effect on the weak anti-localization also provides evidence to the topological insulator surface state origin of weak anti-localization. Introducing magnetic impurities on the surface breaks the time reversed symmetry and $\pi$ Berry phase, thus destroy the weak anti-localization effect. Figure 6.5(a) shows that deposition of 1 monolayer (ML) Fe on the surface of 5nm Bi$_2$Te$_3$ does destroy the transverse weak anti-localization. Deposition of 1 ML Au has little effect on the weak anti-localization. Deposition of Fe on the surface of 50nm sample reduces the weak anti-localization effect due to destruction of surface state as shown in Figure 6.5(b).
Figure 6.5 (a) $\Delta G$ as a function of the perpendicular $B$ component obtained in 5 nm Bi$_2$Te$_3$ film at $T=2$ K, with no metal, 0.3 ML Fe deposition, 1 ML Fe deposition, or 1 ML Au deposition at the top interface. Solid curve is a parabolic fit to red dot data by Kohler’s rule; BT data in the presence of 1 ML Fe deposition. (b) $\Delta G$ as a function of the normal $B$ component obtained in 50 nm Bi$_2$Te$_3$ films at $T=2$K with no metal or 1 ML Fe deposition at the top interface.(cited from Ref.[234])

In addition, H.Z.Lu and S.Q.Shen points out that gate tuning of Fermi energy of the topological insulator surface states is possible to transit weak anti-localization to weak localization in the magnetic doping surface doping samples[239]. Gate tuning of weak anti-localization in topological insulator Bi$_2$Se$_3$ has been reported by J.Chen, et al[229].

It is useful to utilize weak anti-localization in topological insulator to distinguish the helical surface states. However, it is sometimes obstructive to observe weak anti-localization in thick topological films or nanoribbons due to the competition of weak anti-localization with classical MR obeying Kohler rules. As shown in Figure 6.6(a) and (b), Y.S.Kim, et al reported the evolution of MR by varying the thickness of Bi$_2$Se$_3$ thin film[235]. For thin samples with several or dozen of quintuple layer, weak anti-localization dominates the MR. As the thickness increases to hundred quintuple layers, the classical $B^2$ dependence of Kohler MR[238] $R(B) / R(0) = 1 + (\mu B)^2$ dominates. Only
a tiny cusp at very small field shows a weak signature of weak anti-localization. Based on the thickness dependent mobility in Figure 6.6(c), thick sample has larger mobility so that the classical Kohler MR is more significant than weak anti-localization effect; while for thin sample with smaller mobility, Kohler MR changes slowly with magnetic field thus yield to weak anti-localization effect. In order to observe weak anti-localization, it is better to use low mobility sample.

Figure 6.6 The MR at 1.5 K of Bi$_2$Se$_3$ (a) thin film from 3 to 32 quintuple layers and (b) thick film from 60 to 3,600 quintuple layers. Deep cusp in low-field regime is characteristic of the weak anti-localization effect. Parabolic field dependence is dominant in thick films. Inset of (b): zoomed-in view of the 3,600 quintuple layers data near zero field showing signature of the weak anti-localization effect. (c) Mobility as a function of film thickness. All the figures are excerpted from Ref.[235].

In our as-grown Bi$_2$Se$_3$ nanoribbons in the next section 6.3, we didn’t observe weak anti-localization effect in our sample as shown by Figure 6.8(a). It is likely that the high mobility of our sample and a little bit high noise level of measurement cover the weak anti-localization effect.

The coherence of quantum electrons on the surface of topological insulator will manifest itself in nanostructures. With extremely high surface-to-volume ratio and thus larger surface contribution in transport, nanostructures of topological insulator's are useful to distinguish 2D surface transport from 3D bulk transport in the heated study of topological
surface state. Indeed, Aharonov-Bohm (AB) oscillations were discovered in Bi$_2$Se$_3$ nanoribbons in the parallel magnetic field induced MR. A clear modulation of the resistance with a period of 0.62 T is observed, corresponding to one flux quantum ($h=\pi$) threaded into the cross-section of the nanoribbon, proving the existence of a coherent surface conducting channel[240], as shown in Figure 6.7.

Figure 6.7 (a) Schematic diagram of 2D topological surface states of a layered Bi$_2$Se$_3$ nanoribbon in a parallel magnetic field. The red and black arrows correspond to the electric current and magnetic field lines, respectively. The two cones on the top and side surfaces illustrate the Dirac surface states. The green loops encircling the same magnetic flux stand for phase-coherent paths through which the surface electrons interfere. (b) SEM image of a Bi$_2$Se$_3$ nanoribbon, 120nm in width, contacted by four Ti/Au electrodes. The thickness of the nanoribbon is measured by AFM(a line cut in the inset) to be 55 nm. The measurement error bars from both SEM and AFM are within 5–10\%. (c) Normalized MR of the nanoribbon in radial magnetic fields at 2 K. The solid red trace (up sweep) was taken with a scan rate of 3mTs$^{-1}$ and the dashed black line (down sweep) at 10mTs$^{-1}$. (Figures and caption cited from Ref.[240])

In the following section, we will mainly focus on our discovery of linear MR of Bi$_2$Se$_3$ nanoribbons in perpendicular field.

### 6.3 Two-dimensional transport induced linear magneto-resistance(MR) in Bi$_2$Se$_3$ nanoribbons

Recently, Bi$_2$Se$_3$ and related materials have been proposed[52] and confirmed[51,213] as three-dimensional (3D) topological insulator's with a single Dirac cone for the surface state. Among these materials, Bi$_2$Se$_3$, which is a pure compound rather than an alloy like Bi$_x$Sb$_{1-x}$[221] owns a larger bulk band gap (0.3 eV), and is thought to be promising for room temperature applications. Although the existence of topological surface state in
Bi$_2$Se$_3$ has been established by surface sensitive techniques such as the angle-resolved photoemission spectroscopy, extracting the transport properties of 2D surface state in 3D topological insulator's has been plagued by the more dominating conductivity from bulk carriers [225,228,231,233,237,241,242]. With extremely high surface-to-volume ratio and thus larger surface contribution in transport, nanostructures of topological insulator's are useful to distinguish 2D surface transport from 3D bulk transport in the heated study of topological surface state. Indeed, Aharonov-Bohm (AB) oscillations were discovered in Bi$_2$Se$_3$ nanoribbons in the parallel magnetic field induced MR, proving the existence of a coherent surface conducting channel[240]. In this study, we explore the magneto-transport phenomena in Bi$_2$Se$_3$ nanoribbons in magnetic field perpendicular to the surface of nanoribbons and uncover a novel linear MR effect which is only sensitive to the perpendicular component of the magnetic field ($B$) and absent in parallel field. This 2D magneto-transport induced linear MR is weakly temperature dependent and survives at room temperature, suggesting the possibility of using 2D topological surface transport in room temperature magneto-electronic applications.

Pure Bi$_2$Se$_3$ nanoribbons are synthesized in a horizontal tube furnace via the vapor-liquid-solid mechanism with gold particles as catalysts, similar to literature[240,241]. Typical Bi$_2$Se$_3$ nanoribbons have thickness ranging from 50-400 nm and widths ranging from 200 nm to several µms, as shown in the SEM image in Figure 2.15(a). TEM image demonstrates that nanoribbons have smooth side walls and flat surfaces, as shown in Figure 2.15(b) for a 200 nm wide nanoribbon. EDX analyses reveal uniform chemical composition with a Bi/Se atomic ratio about 2:3, indicating the stoichiometric Bi$_2$Se$_3$. High-resolution TEM imaging and 2D Fourier transformed electron diffraction
measurements in Figure 2.15(c) and (d) demonstrate that the samples are single-crystalline rhombohedral phase and grow along the [1120] direction. The upper and lower surfaces are the (0001) planes. The as-grown samples are suspended in ethanol by sonication and dispersed on a heavily doped Si substrate with 300 nm SiO₂ on its surface. Photolithography is used to pattern four electrodes contacting single nanoribbon, as shown in the SEM picture in Figure 6.8(a) inset. The electrodes consist of 150 nm Pd with a 5 nm Ti adhesion layer formed via e-beam evaporation and lift-off. Ohmic contacts are obtained without annealing. The transport measurements are performed in a Quantum Design PPMS with standard low frequency lock-in technique. Four-terminal resistance of the nanoribbons is obtained by flowing a current \( I \) (typically 0.1-1 µA) through the two outer contacts and monitoring the voltage drop \( V \) between the two inner contacts (typical spacing \( \sim 2 \) µm) (Figure 6.8(a) inset).

![Figure 6.8](image_url)

Figure 6.8 (a) Resistance \( R \) vs. magnetic field \( B \) of a Bi₂Se₃ nanoribbon (sample #1) at 2 K and different angle between sample surface and the magnetic field. The grey dashed line highlights the linear MR above 1 Tesla perpendicular magnetic field. The inset shows SEM image of nanoribbon contacted by four leads in the four-probe transport measurement setup. (b) \( R \) vs. the tilt angle \( \theta \) in a fixed magnetic field of 9 Tesla and at \( T=2 \) K. The data are seen to follow the function of \(|\cos(\theta)|\) (solid red line). In both (a) and (b), the magnetic field is always perpendicular to the current when the sample is rotated with respect to \( B \) (inset).
The resistance $R$ of nanoribbon sample #1 as a function of magnetic field $B$ is shown in Figure 6.8(a) at temperature $T=2$ K. This sample was mounted on a rotating stage such that its surface (a-b plane) could be tilted in magnetic field by an arbitrary angle $\theta$ (Figure 6.8(b) inset). Using atomic force microscope (AFM), we measured a sample width of 560 nm and thickness of 100 nm for this nanoribbon. In perpendicular magnetic field ($\theta=0$), the sample exhibited the largest MR which is positive and becomes very linear above a characteristic field of 1-2 Tesla. This MR gradually decreased when sample was tilted away from perpendicular configuration and eventually became almost negligible in the parallel magnetic field configuration ($\theta=90^\circ$). It has been known for long time that regular metals exhibit quadratic MR (i.e. $\Delta R \propto B^2$) at low fields ($\omega_c \tau < 1$, with $\omega_c$, $\tau$ as the cyclotron frequency and mean scattering time) and this MR would saturate at high field $\omega_c \tau > 1$)[238,243,244]. Therefore, observing a non-saturating linear MR in strong magnetic field is interesting in both the fundamental magneto-transport phenomena and magnetic sensor applications[243,245]. Very recently, a linear MR was observed in low-doped Bi$_2$Te$_3$ crystal flakes which showed 2D surface state transport[237]. It is thus encouraging that linear MR is now also observed in topological insulator Bi$_2$Se$_3$ nanoribbons directly grown from chemical synthesis, in which case the materials can be grown and assembled in large scale for scaled up nanoelectronics applications[40]. The nature of this linear MR in Bi$_2$Se$_3$ nanoribbons is the focus of this paper.

In the electrical transport study of topological insulator Bi$_2$Se$_3$ or Bi$_2$Te$_3$, it is important to distinguish the 3D transport due to bulk carriers from the 2D surface state transport since the Se or Te vacancies may induce high electron concentration and push the Fermi
level into the bulk conduction band. One standard way to check if the magneto-transport is 2D or 3D is the angular dependence of the magneto-transport[226,228,233,236,237]. For 2D surface state transport, the magneto-transport will only respond to the perpendicular component of the magnetic field \(B \cos(\theta)\). In Figure 6.8(b), we show the resistance of sample #1 as a function of rotation angle when the sample was rotated in 9 Tesla magnetic field at 2 K (inset). The measured \(R(\theta)\) dependence is seen to have wide peaks around the perpendicular field configuration (\(\theta = 0^\circ, 180^\circ\)) and dips with cusp around the parallel field configuration (\(\theta = 90^\circ, 270^\circ\)). In fact, the experimental data have an excellent agreement with the functional form \(|\cos(\theta)|\) (red line in Figure 6.8(b)). This suggests that the sample is only responsive to the perpendicular component of \(B\) at arbitrary tilt angle and thus the linear MR is a 2D magneto-transport effect.

On the basis of results in Figure 6.8(b), we conclude that the linear MR induced by 2D magneto-transport. Due to the 2D nature of this linear MR and the associated SdH oscillations discussed later, it is likely that this linear MR is intrinsically tied to the 2D Dirac electrons occupying surface state. It would be curious if such surface state magneto-transport can be exploited at high temperature (e.g. room temperature) relevant for device applications. We found that raising temperature to room temperature does not have significant influence on the linear MR as shown in Figure 6.9. In Figure 6.9(a), where the MR of sample #2 is plotted at various \(T\) from 2 K to 300 K, one sees that the linear MR at \(B_\perp > 1\) T remains unchanged. At the same time, parallel magnetic field induces negligible MR, as shown by the data at \(T = 2, 150, 300\) K. Angular dependence of MR suggests that the MR is a 2D response over the whole temperature range from 2 K to
300 K, as illustrated by the good agreement between experimental resistance vs. tilt angle data at 9 Tesla and the $|\cos(\theta)|$ function in Figure 6.9(b).

Figure 6.9 (a) $R$ vs. $B$ of sample #2 from $T=2$ K to 300 K. The data in perpendicular field are shown as dots while the data in parallel field are shown as solid lines for $T=2$, 150 and 300 K. (b) $R$ vs. rotation angle $\theta$ at $T=2$, 150 and 300 K. The function $|\cos(\theta)|$ is shown as solid red lines. (c) Conductance of Bi$_2$Se$_3$ nanoribbon sample #2 vs. rotation angle at $T=2$, 150 and 300 K. Red solid lines represent function $-|\cos(\theta)|$ which is seen to describe the conductance data well for all the temperatures.

Since both the bulk conduction and surface conduction contribute to the total conductance in Bi$_2$Se$_3$ crystals, it is more appropriate to discuss conductance $G$ instead of resistance in a quantitative analysis. In Figure 6.9(c), we show $G$ vs. $\theta$ at 2 K, 150 K, and 300 K by inverting resistance data in Figure 6.9(b). It can be seen that the function $-|\cos(\theta)|$ also matches $G(\theta)$ very well. Therefore, the magneto-transport effect we report has 2D nature and is robust from 2 K to room temperature in the magneto-conductance
picture. However, due to the increased resistance at high temperatures from phonon scattering, the magneto-conductance at 300 K is nearly ten times smaller than 2 K. This is reasonable given that the thermal energy scale at 300 K already exceeds the maximal cyclotron energy $\hbar \omega_c$ (~100 K) at 9 Tesla, the highest magnetic field in our experiment. If we model the total resistance as $R = 1/(G_b + G_s)$ with $G_b$, $G_s$ as the bulk and surface conductance, a smaller magneto-conductance $\Delta G_s(B)$ for surface state at high temperature is compensated by the smaller value of $G_b$ and still converts into a relatively constant $\Delta R(B)$ in the measurement of total resistance at different $T$. Yet due to the striking robustness of linear MR against raising temperature and the importance of MR in magneto-electronic device applications, here we emphasize the MR instead of magneto-conductance in this paper. We suggest that this linear MR could be exploited in magneto-electronic device operation over wide temperature range and its magnitude is likely to become larger once the nanoribbon crystal quality and mobility improve.

6.4 Possible mechanism of linear magneto-resistance in Bi$_2$Se$_3$ nanoribbons

We would like to discuss the possible origins of the linear MR and its relation with other findings in materials with Dirac electronic dispersion spectrum. The well-established Kohler's rule[238] suggests that the MR of a material is a universal function of $\mu B$: $R(B)/R(0) = F(\mu B)$. It is common$^{21}$ that at low field when $\mu B < 1$, $F(\mu B) \approx 1 + (\mu B)^2$, as a result of the Lorentz force deflection of carriers. At high field condition $\mu B$, or
most materials show saturating MR and a non-saturating and linear MR is unusual. For metals with open Fermi surfaces (e.g. Au), the MR could be linear and non-saturating at high fields[238]. This is not the case here for Bi\textsubscript{2}Se\textsubscript{3}. The existence of a linear MR for small band gap semiconductor could have a quantum[244,246,247] or classical origin[243,245]. To explain the linear MR down to very low fields in silver chalcogenides[243], Abrikosov first proposed a model based on the quantum-MR[246,247] for systems with gapless linear dispersion spectrum. It is believed that such gapless linear dispersion may apply for silver chalcogenide or other small bandgap semiconductors with strong inhomogeneity[244,246]. Abrikosov's linear quantum-MR was originally developed at the extreme quantum limit $\hbar \omega_c > E_F$, when all electrons coalesce into the lowest Landau level. Another requirement is that $\omega_c \tau$ or $\mu B > 1$, i.e. Landau levels are well-formed. From the above discussions and SdH data, we see that indeed the linear MR occurs at high magnetic field regime where $\mu B > 1$ (note that our estimate of $\mu \sim 0.12 \, m^2/\text{Vs}$ is an underestimate) and SdH oscillations start to appear. However, there are clearly more than one Landau levels occupied in our sample in the field range where linear MR was observed. This does not exclude the linear quantum MR as the explanation for our data though, since previous experimental study on polycrystalline InSb shown in Figure 6.10(a) showed that the linear quantum-MR could appear at much lower field with more than one Landau levels filled[244]. Magneto-transport studies on graphite in Figure 6.10(b) and epitaxial graphene in Figure 6.10(c) also reported a linear MR persisting to room temperature similar to our observation in Bi\textsubscript{2}Se\textsubscript{3} nanoribbon and attributed the linear MR to Abrikosov's linear quantum-MR[248-
However, further theoretical study is required to understand the linear quantum-MR at temperatures so high that $k_B T > \hbar \nu_\zeta$ as in our experiment.

![Figure 6.10](image)

Figure 6.10 (a) Linear band structure induced by inhomogeneity in poly-crystalline InSb. Linear MR in (a) poly-crystalline InSb[244] (b) graphite[248] (c) multi-layer graphene[251].

Without invoking the linear dispersion spectrum, Parish and Littlewood suggested a classical origin for linear MR in which the MR is a consequence of mobility fluctuations in a strongly inhomogeneous system[245]. For our Bi$_2$Se$_3$ nanoribbons, the single crystal quality and small length scale of the device rule out the strong physical inhomogeneity of sample surface. However, we cannot exclude the possibility of strong electronic inhomogeneity being responsible for the linear MR in our Bi$_2$Se$_3$ nanoribbon. Electron and hole puddles and charge inhomogeneity have been observed in high quality graphene samples via local probe techniques[252,253], it is quite reasonable that such spatial inhomogeneity of carriers can also exist for gapless surface state on topological insulators.
6.5 **Shubnikov-de Haas oscillation in Bi$_2$Se$_3$ nanoribbon---another signature of two dimensional transport**

In the strongest perpendicular magnetic fields, $R(B)$ of sample #1 showed weak oscillations on top of the linear MR. These are Shubnikov-de-Haas (SdH) oscillations due to the formation of quantized energy levels (Landau Levels) of orbital motions of electrons. We analyze SdH oscillations in another sample (#2) in which these oscillations are more pronounced to extract the carrier density and further demonstrate the 2D nature of Fermi surface. Sample #2 had width of 300 nm and thickness of 25 nm as measured by AFM. Figure 6.11(a) shows the $R$ vs. $B$ from zero to 9 Tesla of sample #2 at various tilt angles. The sheet resistivity $\rho$ is shown on the right side of y-axis, after taking into account the ratio between the length and width of nanoribbon. Similar to sample #1, in perpendicular field, a linear MR was observed above ~1 Tesla. At high fields, the sample exhibited clear SdH oscillations which disappeared quickly once the sampled was tilted away from perpendicular configuration. In parallel field configuration, the MR is negligible, similar to the sample #1. Note that for this experiment, the sample was rotated in the way that the in-plane magnetic field was parallel to the current at $\theta = 90^\circ$ (Figure 6.11(c) inset), in contrast to the rotation scheme for sample #1. However, the magneto-transport effects are qualitatively the same. This leads us to believe that the surface state on side walls of Bi$_2$Se$_3$ nanoribbons has low mobility and do not contribute much to the observed MR.
Figure 6.11. (a) MR of sample #2 between 0 and 9 Tesla at $T=2$ K when the rotation angle $\theta$ is increased from $0^\circ$ to $90^\circ$ (inset of panel (c)). Shubnikov-de Haas (SdH) oscillations are visible on top of the linear MR background for perpendicular field ($\theta=0^\circ$) and disappear rapidly as $\theta$ increases. (b) SdH oscillations at $\theta=0^\circ \rightarrow 35^\circ$ after subtracting background MR. The black and pink arrows mark the SdH dips at Landau filling factor $\nu=7$ and 8. (c) Position of the $\nu=7$ or 8 SdH dip plotted against rotation angle. The data are consistent with the $1/\cos(\theta)$ dependence (grey lines) which is expected for 2D SdH oscillations. The inset shows the rotation configuration. (d) Landau index $\nu$ plotted against the inverse of magnetic field for the SdH oscillations in perpendicular field ($\theta=0^\circ$). The slope of linear fit gives a sheet electron density of $n\approx 1.3 \times 10^{12}/\text{cm}^2$. (e) Fitting of SdH oscillation at 2 K and $\theta=0$. A non-zero Berry’s phase ($\beta=0.81$) is obtained to match the phase of SdH oscillations. (f) SdH oscillations at different $T$. (g) Fitting the amplitudes of SdH dip/peak at $\nu=7$, 7.5 vs. $T$ yields effective electron mass $m' = 0.117$, 0.121± 0.007×$m_e$.

To analyze the SdH oscillations, we subtract the linear MR background from the raw $R(B)$ data in Figure 6.11(a) and the residual oscillatory part of the MR is shown in Figure 6.11(b) for $\theta=0^\circ$, 10°, 22°, 35° (from top to bottom curve). As can be seen, when the sample was tilted, the SdH oscillations move to higher field and eventually our magnetic field (9 T) is not large enough to track the SdH oscillations at $\theta>35^\circ$. By
comparing the magnetic field values at the SdH dips we could assign the Landau Level filling factor $\nu$ to be 7 and 8 for the two dips observed at 7.7 and 6.9 Tesla at $\theta = 0$. In Figure 6.11(c), we plot the positions of $\nu=7$ and 8 against the tilt angle $\theta$ as sample is tilted away from the perpendicular orientation. The four data points for $\nu=8$ dip are seen to follow the $1/\cos(\theta)$ function nicely. For $\nu=7$, the dip was only observable at $\theta = 0^\circ$, $10^\circ$, $22^\circ$, and moved outside our field range (9 T). However, we infer its position at $\theta = 35^\circ$ by multiplying the $\nu=8$ dip position with $8/7$. This inferred value together with the three points at lower tilt angle also obeys the $1/\cos(\theta)$ dependence. The agreement between the $1/\cos(\theta)$ function (black lines) and our angle dependent SdH position suggests that the observed SdH oscillations are likely to originate from a 2D Fermi surface.

Plotting the Landau index $\nu$ of SdH peak and dip positions vs. the inverse of magnetic field, $1/B$, we obtain a linear dependence in Figure 6.11(d). The slope of the $\nu$ vs. $1/B$ plot gives a 2D sheet carrier density of $1.3 \times 10^{12}$/cm$^2$. This carrier density corresponds to a Fermi momentum $k_F = 0.41$ nm$^{-1}$ and Fermi energy $E_F = v_F h k_F = 110$ meV. Based on ARPES results[51,228], the bottom of conduction band is 205 meV above the Dirac point in Bi$_2$Se$_3$. We thus estimate that the position of the 2D surface state Fermi energy is $\sim 95$ meV below the bottom of bulk conduction band, consistent with the fact that 3D magneto-transport is negligible in our samples. While both 2D electrons in conventional semiconductors and 2D Dirac electrons have linear $\nu$ vs. $1/B$ dependence, the different Berry's phase would make the intercept zero for regular 2D electrons and non-zero for Dirac electrons with nonzero Berry's phase$^{24}$. The linear fit of $\nu(1/B)$ in Figure 6.11(d)
produces an intercept of \(-0.2 \pm 0.2\). The relatively large uncertainty is due to the limited number of SdH oscillations observed, making it ambiguous to infer the existence of Berry's phase from a simple analysis of \( \nu(1/B) \). In such case, it is more useful to directly compare the MR data with the known SdH oscillation of 2D electrons and a non-zero Berry's phase would manifest as a phase shift in the oscillations\[254\]. The general expression for SdH oscillation is\[255\]

\[
\Delta R(B) = A \exp(-\pi/\mu B) \cos\left[2\pi \left( B_f/B + 1/2 + \beta \right) \right]
\] (6.1)

In Eq.(6.1), \( B_f \) is the frequency of SdH oscillation in \( 1/B \) and \( \beta \times 2\pi \) is the Berry's phase. The amplitude \( A \) has the following temperature dependence

\[
A \propto \frac{2\pi^2 k_B T/h \omega_r}{\sinh\left(2\pi^2 k_B T/h \omega_r\right)}
\] (6.2)

Using the \( B_f \) extracted from the slope of \( \nu(1/B) \) dependence, we fit the residual SdH oscillations in \( R(B) \) at \( T=2 \) K to Eq.(6.1) with three fitting parameters \( A, \beta, \) and \( \mu \). The fitting yielded a non-zero Berry's phase \( \beta = 0.81 \pm 0.01 \) together with \( A = 90 \pm 50 \) Ohm and \( \mu = 0.12 \pm 0.02 \) m\(^2\)/Vs, as shown by the solid red line in Figure 6.11(e). On the other hand, \( \beta = 0 \) with the same \( A \) and \( \mu \) would predict an oscillatory pattern (dashed grey line in Figure 6.11(e)) clearly shifted from experimental data. For 2D Dirac electrons, the Berry's phase of \( \pi \) should lead to \( \beta = 0.5 \). However, extracting Berry's phase from SdH of surface state in Bi compound topological insulators is complicated as the application of magnetic field itself may have influence on the value of \( \beta \)[236,237]. Therefore, a more systematic study on samples with higher mobility in higher magnetic fields (so there are more SdH oscillations) is needed to clarify the Berry's phase effect in Bi\(_2\)Se\(_3\) nanoribbons. Note that
our fitted mobility $\mu = 0.12 \text{ m}^2/\text{Vs}$ should be an underestimate since that mobility $\mu$ in Eq.(6.1) is defined with the quantum life time $\tau_q$ which is normally longer than the transport scattering time $\tau$ in the standard definition of mobility ($\mu = e\tau/m^*$) due to the dominance of small angle scattering contribution in $\tau$.

Figure 6.11(f) shows the temperature effect on SdH oscillations. As expected, SdH oscillation amplitude decreases at higher temperature due to the thermal smearing of Landau levels. Fitting the temperature dependence of SdH amplitude to Eq.(6.2) allows us to extract $m^*$, the effective mass of 2D electrons in our Bi$_2$Se$_3$ nanoribbon. The experimental amplitudes of the SdH peak/dip at $\nu = 7, 7.5$ vs. temperature are shown in Figure 6.11(g) together with fitting curves. Both fitting results yield $m^* \approx 0.12 m_e$, with $m_e$ as the free electron mass. This value is in quantitative agreement with recent magneto-transport experiment on surface state in bulk Bi$_2$Se$_3$ crystals with reduced electron densities[236].

In conclusion, we report a linear MR induced by 2D magneto-transport in chemically synthesized nanoribbons of topological insulator Bi$_2$Se$_3$. When the magnetic field is parallel to the surface of nanoribbon (a-b plane), the MR effect is negligible compared to the MR in perpendicular magnetic fields. Angular dependence of the MR showed an exact $|\cos(\theta)|$ dependence at arbitrary angle, illustrating the 2D origin of linear MR. Angle dependent SdH oscillations also suggest the existence of 2D Fermi surface. These results suggest that the linear MR is likely related to 2D surface state conduction. Furthermore, it is striking that this 2D magneto-transport induced linear MR persists at
room temperature, underscoring the potential of exploiting 2D topological surface state for magneto-electronic device application over broad temperature range.

6.6 Summary

In this Chapter, the fundamental concept of topological insulator was introduced. Quantum transport including weak anti-localization and Aharonov-Bohm effect in topological insulator ($\text{Bi}_2\text{Te}_3$ and $\text{Bi}_2\text{Se}_3$) films and nanoribbons has been discussed. We mainly focus on our discovery of linear MR persisting up to room temperature in $\text{Bi}_2\text{Se}_3$ nanoribbons. Angular dependence of the MR shows an exact $|B\cos(\theta)|$ dependence at arbitrary magnetic field configuration, illustrating the 2D origin of linear MR. Angle dependent SdH oscillations also suggest the existence of 2D Fermi surface.
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