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by

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Ph.D. Dissertation Prospectus

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ABSTRACT

TITLE HERE

by

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August 2008

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Major: Physics
Degree: Doctor of Philosophy

Abstract here

ACKNOWLEDGEMENTS

Acknowledgements here...

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Chapter 1

Introduction

1.1 Birth of Integrated Circuits

The development of microelectronics revolutionized the world in the latter half of the twentieth century. The term semiconductor, in the sense it is known today, first appears in literature in 1911 [1]. Initially, work on the subject was rather pessimistic. However, in the years following Word War II breakthroughs began shed light on the possible applications and the underlying physics involved, such as the ideas of *intrinsic* and *extrinsic* semiconductors [2, 3, 4, 5].

The history of semiconductors and transistors is a well documented subject. The first transistor was constructed at Bell Labs in 1947 using polycrystalline germanium. Shortly thereafter one was developed using silicon. Throughout the following years, these devices were improved on by replacing polycrystalline with single crystals [6]. Then Jack Kilby demonstrated the first integrated circuit (IC) in 1958, for which he would win the Nobel Prize in physics [7, 8]. The scale of ICs grew rapidly in the subsequent years. Initially only a few transistors could fit on a chip (small-scale integration), in stark contrast to moder-day chips that contains billions of transistors [9, 10]. Growth continued at a rapid pace, but eventually it was realized that some limits, material and integration based, existed in silicon and other commonly used materials [11, 12]. In part, these limitations increased the interest in alternative materials. As a result widespread and renewed interest has led to a breadth information and results on a wide range of materials and their applications.

1.2 Graphene as a New Two-dimensional Material

Layered materials have existed for billions of years, and have been studied over the last few centuries [13, 14]. In recent decades the scientific study of graphite (3D) has led to new forms materials, such as carbon nanotubes (1D) and fullerenes (0D) [15, 16, 17]. However, only more recently have scientists began to understand the potential of such layered materials and their potential technological applications. After attempting unsuccessfully to synthesize few-layer graphite during the 1960s (only around 10-50 layers were able to be synthesized) a breakthrough was finally achieved [16]. This most notably began with the synthesis of monolayer graphene [18].

1.2.1 Properties of Graphene

To date, graphene's properties have been the focus of much research, both theoretical and experimental. It has been one of the primary driving forces in study of 'relativistic' condensed matter physics due to its low dimensionality and its band structure that allows electrons to mimic relativistic particles confirming the appearance of several relativistic phenomena [19, 20, 21, 22]. In its most basic sense, graphene is

composed of a single layer of carbon atoms arranged in two-dimensional honeycomb lattice (see fig. 1.1). It has a Young's modulus of 100 GPa (several times more than steel) with a breaking force that is 13% of its Young's modulus [23, 24]. Its strength is due, in part, to its strong in-plane carbon (C) bonds. In addition, graphene can sustain elastic deformations of 20% due to its two-dimensional nature and it has high pliability [16]. These mechanical properties are of interest because graphene lies in the extreme ranges of many metrics considering its size and dimensionality.

Aside from its mechanical properties, graphene's transport properties were another reason why the material was so appealing. Graphene's mobility is several times that of silicon's. Experimental results have shown graphene mobility around $15,000 \text{ cm}^{-2}\text{V}^{-1}\text{s}^{-1}$ with a potential theoretical limit of $200,000 \text{ cm}^{-2}\text{V}^{-1}\text{s}^{-1}$ [25, 24]. The upper theoretical limit imposed on mobility is due to scattering, however, these high mobilities are achieved mainly because electrons in graphene act very much like photons in their mobility due to their lack of mass. This enables them to travel sub-micron distances without scattering [26]. In reality, there are other limiting factors that need to be considered such as the quality of graphene and scattering with the substrate, for example.

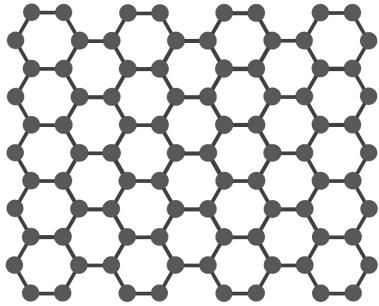


Figure 1.1: Graphene: a layer of carbon atoms in a honeycomb lattice.

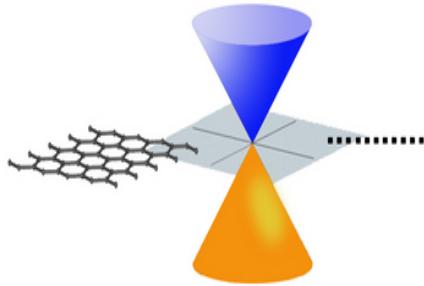


Figure 1.2: One of the most unusual features of graphene is that its conduction and valence bands meet at a point, meaning that in single-layer graphene there is no band gap (figure obtained from [27])

1.2.2 Band Structure of Graphene

Despite its impressive properties, the main drawback of graphene is its lack of bandgap. As this became known, the prospect of using graphene for the fabrication to ICs became unlikely. In graphene the conduction and valence bands touch at a single point as shown in fig. 1.2 [28]. Ultimately, the lack of a bandgap means that the current on/off ratio is low and is unappealing for logical circuit applications [29]. However, graphene exhibits some interesting properties as a result of having no bandgap, particularly as it pertains to its optical properties. The material's band structure allows for absorption of light over a large range of the electromagnetic spectrum, ranging from infrared ($< 1.65 \text{ eV}$) to ultraviolet ($> 3.2 \text{ eV}$), offering potential electronic-photonic device applications [30, 31, 32]. Since a direct use in logical circuits is not practical researchers have moved on to look for 'two-dimensional materials beyond graphene.' Several attempts at some derivatives of graphene-like materials have been studied, but for the most part they do not seem promising for use in logical circuits [33, 34]. As of late, research has been concentrated on two-dimensional materials, namely transition metal dichalcogenides, as a candidate for applications in ICs.

1.3 Two-dimensional Materials: Transition Metal Dichalcogenides

Commonly referred to two-dimensional materials beyond graphene, TMD have garnered much interest. TMDs were studied previously, however, they have gained renewed interest due to their properties [35, 36, 37, 38]. TMDs consist of hexagonal layers of metal (M) atoms in between two layers chalcogen (X) atoms (see fig. 1.4, such that the stoichiometry of the material is MX_2 [29]. The material is dependent on the type of transition metal, typically one of: molybdenum (Mo), tungsten (W), niobium (Nb), rhenium (Re), nickel (Ni), or vanadium (V), and two chalcogen atoms, typically one of: sulfur (S), selenium (Se), or tellurium (Te) [38, 39]. The most commonly studied variations of TMDs are molybdenum disulfide (MoS_2), tungsten diselenide (WSe_2), and tungsten disulfide (WS_2). These materials are commonly stacked together involving van der Waals interactions between adjacent sheets and covalent bonding within each individual sheet (see fig. 1.3) [29]. TMDs exhibit a wide variety of properties, including either being a

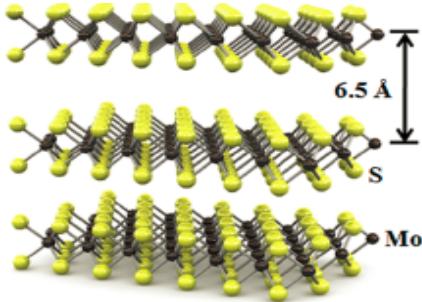


Figure 1.3: The atomic structure of a layered TMD, depicting MoS_2 . Each sheet is composed of three atoms with Mo sandwiched in between two S atoms, S-Mo-S. (Figure obtained from [40])

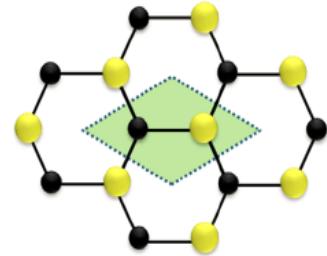


Figure 1.4: Top view of a TMD (MoS_2) lattice. (Figure obtained from [40])

metal or insulator, and displaying the topological insulator effect, superconductivity, and thermoelectricity [41, 42, 43, 44].

1.3.1 Properties of Commonly Used TMDs

As stated in sec. 1.2.2, one important property as it pertains to applications for logical circuits is the material's band structure. One of the main reasons TMDs have been so extensively studied lately is due to the fact that, unlike graphene, they do exhibit a band gap. The band gaps in some commonly used TMDs is interesting because of the transition from an indirect to a direct band gap as the layered thickness decreases. Fig. 1.5 illustrates this, for bulk and few-layer MoS_2 there is an indirect band gap while for monolayer MoS_2 there is a direct band gap. This unusual structure results in some unique optical and properties.

1.3.2 Current State of TMDs

1.4 Hall Effect

Give a brief overview of the history

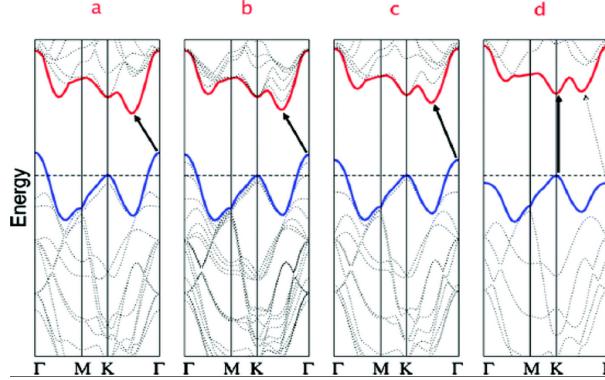


Figure 1.5: Calculated band structures of (a) bulk MoS₂, (b) four-layer MoS₂, (c) bilayer MoS₂, and (d) monolayer MoS₂. Here the solid arrows indicate the lowest energy transitions. (Taken from [45], originally appeared in [46])

2D material	theoretical E_g (eV)	experimental E_g (eV)
graphene	0	0
bilayer graphene	0	0
bulk <i>h</i> -BN	-	5.97 [47]
monolayer <i>h</i> -BN	-	6.07 [48]
few layer (2-5) <i>h</i> -BN	-	5.92 [49]
bulk MoS ₂	1.2 ^{a,b} [50, 51]	1.0-1.29 ^b [50, 51]
monolayer MoS ₂	~ 1.90 ^{a,c} [52]	~ 1.90 ^b [52]
bulk WS ₂	~ 1.30 ^{a,b} [50, 53]	~ 1.35 ^c [50, 53]
monolayer WS ₂	~ 2.10 ^{a,c} [54]	-

^a Theoretical calculations based on first-principles calculations using density functional theory (DFT).

^b Indirect band gap semiconductor.

^c Direct band gap semiconductor.

Table 1.1: Summary of the band gaps of typical monolayer, bilayer, and bulk TMDs and *h*-BN materials. Table adapted from ref. [29].

1.4.1 Overview

1.4.2 Theoretical Background

Some useful references for this section [55, 56, 57, 58, 59, 60]

Chapter 2

Experimental Details

2.1 Substrate Preparation

Using degenerately doped silicon dioxide (SiO_2) wafers that are 270 nm thick as pictured in fig. 2.1 there are several preliminary steps needed prior to device fabrication. For easy identification of locations on the substrate alignment marks are placed on the wafer using photolithography. There is a main alignment mark pictured in fig. 2.2 (REALLY ONLY WANT TO REF A,B) which allows for quicker identification during electron beam lithography, for example. The alignment marks are in a grid pattern with the coordinate $(0,0)$ at the center, stretching to $(\pm 6, \pm 6)$ in both the right and left directions. In each of these coordinate locations there smaller alignment marks evenly spaced within them as shown in fig. 2.2 (FIGURE OUT HOW TO REF SUBFIGS, HERE WE WANT TO REF (c,d)). Next, gold (Au) is deposited on the surface of the wafer, a process that will be explained in more detail in sec. 2.5.



Figure 2.1: Plain wafer

2.1.1 Substrate Cleaning

Beginning with a cut SiO_2 substrate with a deposited Au layer. To remove the Au layer, the substrate is first soaked in acetone for approximately 5-10 minutes then washed using isopropanol (IPA) and dried with nitrogen (N_2) gas. Next, the substrate is placed in acetone and sonicated for 15 minutes. Then sonicated once more but in IPA this time with a repetition of washing and drying step using IPA and N_2 as described above in between each sonication. In order to remove any remaining organic matter on the surface of the substrate, the substrate is annealed under vacuum at 600°C for 10 minutes and passing forming gas for 2 of the 10 minutes. Forming gas is a mixture of H_2 and an inert gas, usually N_2 [61]. In addition to annealing the substrate for cleanliness, in certain cases when a higher degree of cleanliness is desired the substrate can be treated with oxygen plasma cleaning.

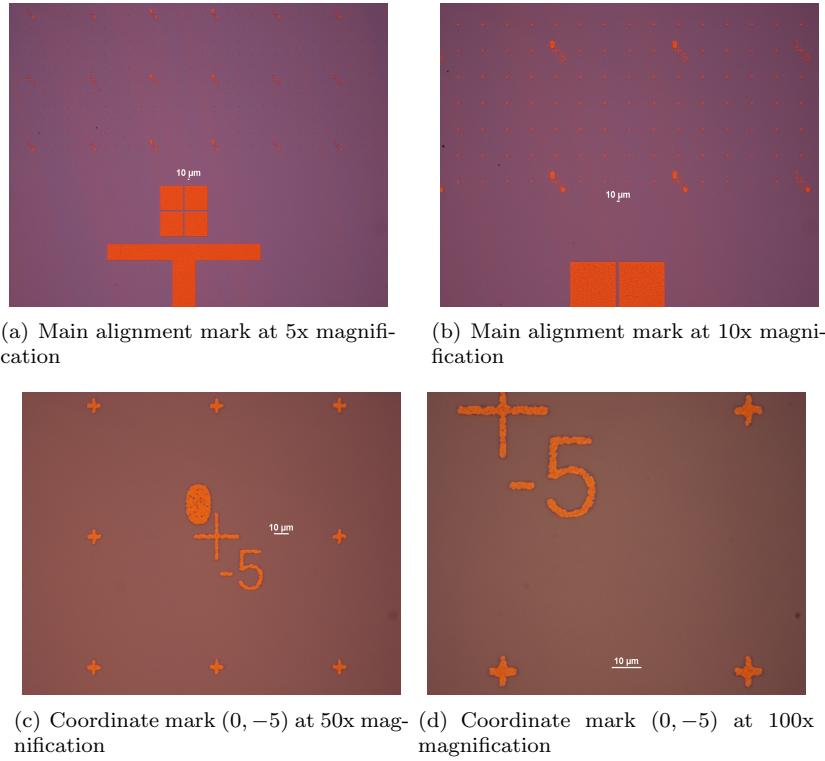


Figure 2.2: Main alignment mark and a coordinate point on a substrate at various magnifications

2.2 Exfoliation

To synthesize samples the most common and often most effective method used is mechanical exfoliation, a technique made famous by the 2004 Novoselov et al. paper. The process involves using Scotch tape to repeatedly cleave layers of MoS₂ or some other TMD. Starting with a crystal of a particular TMD, placing it on a piece of Scotch tape. Then taking another piece of tape and pressing in on the crystal that is on the first piece of tape, being sure to press hard and firm on the crystal. The tape is then lifted up and this process is repeated until the whole piece of tape is filled with small samples of the TMD. At the end of this process it is expected that there are a wide range of mixture of sample sizes in terms of area and in term of thickness as well, where thicknesses of < 3 nm are not uncommon. To better characterize the samples the optical microscope can be used to do so.

The main challenge that exists with this method is the ability to synthesize a high yield of monolayer samples. This does not seem to be much of a challenge when it comes to graphene and some other TMDs, but with regard to MoS₂ this is not so simple. Based on recently published literature in an effort to increase the yield of monolayer MoS₂ various methods and techniques were tested and modified accordingly [62]. In this modified method an additional step to cleaning the substrate is added in which it undergoes oxygen plasma cleaning for 10 minutes to ensure the cleanliness of the substrate's surface. To promote more bonding between the substrate and the samples, the substrate is first heated at 300 °C for 10 minutes without any samples on it. During this process the normal cleaving of sample on tape from crystal taking place. Once the substrate is done heating the tape containing sample is immediately placed on the substrate and pressed firmly for several minutes. Then the substrate (with the tape still on it) is placed on a glass slide and is heated at around 85 °C for five minutes. Next, the substrate (with tape) is removed from heat and the tape slowly peeled back from the substrate. The result should be a much higher yield of < 3 nm samples of larger surface area, and several trilayer, bilayer, and a few monolayer

samples. (ADD PICTURES OF EACH STEP)

Most commonly SiO_2 substrates are the main items that are exfoliated onto. However, depending on the material being synthesized, this may not always be the case. In cases where samples of hBN or in the event that the thickness of the synthesized sample is not of great importance and can be tolerated up to 20 – 30 nm, polydimethylsiloxane (PDMS) is exfoliated onto instead of SiO_2 substrates. The resulting samples are of varying thickness, on average around 20 nm. Thin samples (usually trilayer and above) can be made using this method of PDMS, however, these samples tend to have small surface area and lack uniformity which poses problems as to their usability. As such, this remains an effective method for obtaining samples in which thickness is not the main concern. Once the samples have been optically characterized, the sample(s) on PDMS must be transferred to a SiO_2 substrate.

2.3 Device Synthesis

2.3.1 Transfer

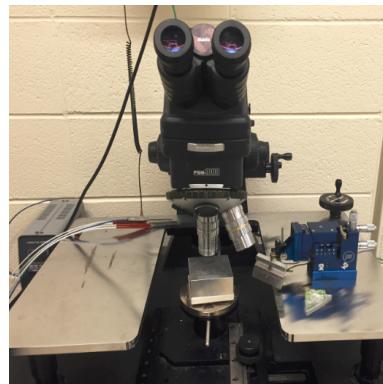


Figure 2.3: Transfer stage setup

2.3.2 Polycarbonate Pickup Method

2.4 Characterization

2.4.1 Optical Characterization



Figure 2.4: Optical microscope front view



Figure 2.5: Optical microscope side view

2.4.2 AFM Characterization



Figure 2.6: AFM front view

2.5 Device Fabrication

2.5.1 Device Design

2.5.2 Electron Beam Lithography

2.5.3 Metal Deposition

2.6 Electrical Measurements

2.6.1 Measurement Devices

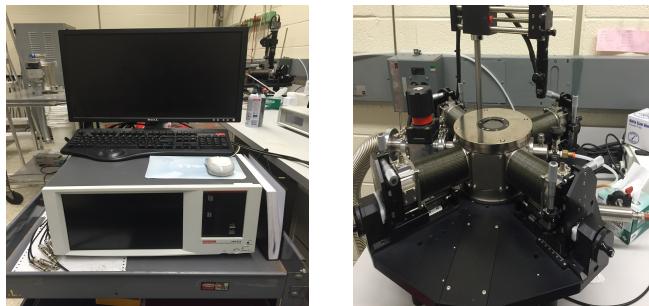


Figure 2.7: Main Caption

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Appendices

Au gold

DFT density functional theory

IC integrated circuit

IPA isopropanol

Mo molybdenum

MoS₂ molybdenum disulfide

N₂ nitrogen

Nb niobium

Ni nickel

PDMS polydimethylsiloxane

Re rhenium

S sulfur

Se selenium

SiO₂ silicon dioxide

Te tellurium

TMD transition metal dichalcogenides

V vanadium

W tungsten

WS₂ tungsten disulfide

WSe₂ tungsten diselenide