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Author
Aguilera, Juan Luis

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Electronic Transport in Strained Graphene

A Dissertation submitted in partial satisfaction of the requirements for the degree of

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in

Physics

by

Juan Luis Aguilera Servin

March 2015

Dissertation Committee:

Dr. Marc Bockrath, Chairperson
Dr. Shan-Wen Tsai
Dr. Roger Lake
The Dissertation of Juan Luis Aguilera Servin is approved:

________________________________________

Committee Chairperson

University of California, Riverside
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A mi familia.
Graphene is a single atomic layer material with exceptional electronic and mechanical properties. Graphene has formed the basis of many nanoelectromechanical and strain sensing devices. However, the ultimate limit of miniaturization of such sensors has not yet been ascertained. In this work we present the fabrication and electrical characterization of nanoscale pressure sensors realized from suspended graphene membrane devices.

We start in chapter 1 by describing the elemental electronic properties of graphene, followed by the motivation of the origin of the particular geometry used in our pressure sensors. Chapter 2 describes in detail the fabrication techniques required to make the graphene devices. In chapter 3 we show the room temperature electrical transport of our devices and find that the injected current division between counter electrodes depends on pressure and can be used to realize a nanoscale pressure sensor. Estimating various potential contributions to the resistivity change of the deflected graphene membrane including piezoresistivity, changing gate capacitance, and the valley Hall effect due to the pressure-induced synthetic magnetic field, we find that the valley Hall effect yields the largest expected contribution to the longitudinal resistivity...
modulation for accessible device parameters. Chapter 4 shows the electronic transport at low temperatures. We find conductance fluctuations as a function of gate voltage. Additionally, at fixed gate voltage, there is a clear difference in the conductance value as a function of pressure. Finally we conclude with a brief summary and potential research outlook.
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Chapter 1

Introduction

Graphene is a two dimensional (2D) material whose properties have been studied for over 10 years. The most popular among many 2D crystals whose properties are being continually studied like the transition metal dichalcogenides (MoS$_2$, MoTe$_2$, WSe$_2$) and most recently black phosphorus. Regarded as a miraculous material due to its wide range of potential applications, it is expected that graphene electronics will reach a mass production market in the long term future.

Graphene was first isolated in 2004 [50]. It gained a greater notoriety after the 2010 Physics Nobel prize was awarded to Andre Geim and Konstantin Novoselov for their ground breaking experiments.

Graphene has multiple extraordinary properties. As highlighted by Novoselov in Reviews of Modern Physics [48], it is the strongest material ever measured [36], its charge carriers behave like massless particles that move at a speed of $c/300^1$ [49], it is impermeable to standard gases [15] and it has an extremely large thermal conductivity [6].

---

$^1$where $c$ is the speed of light
An elusive achievement in the graphene community has been the use and control of its valley degree of freedom. Specifically an experimental milestone would be to achieve valley polarization in electronic transport. Because of this our original research focused on valleytronics by working with strained graphene.

1.1 The electronic properties of graphene

Graphene is a one dimensional sheet of carbon atoms arranged in a honeycomb lattice. In graphene the outer atomic orbitals of carbon are arranged in three coplanar hybridized sp\(^2\) orbitals and a perpendicular p orbital. Neighboring carbon atoms form a sigma bond between sp\(^2\) orbitals, leading to the honeycomb structure. The out of plane p orbital is responsible for the electronic properties of graphene, since it binds covalently with p orbitals of adjacent atoms, which leads to the formation of a \(\pi\) band.

![Honeycomb lattice](image)

Figure 1.1: Honeycomb lattice.

The graphene lattice can be obtained through a triangular lattice with two atoms per unit cell. The two lattice vectors are

\[
a_1 = \frac{a}{2} (3, \sqrt{3}), \quad a_2 = \frac{a}{2} (3, -\sqrt{3}).
\]  

(1.1)
where $a \approx 1.42$ Å. The reciprocal lattice vectors are

$$b_1 = \frac{2\pi}{3a}(1, \sqrt{3}), \quad b_2 = \frac{2\pi}{3a}(1, -\sqrt{3}).$$  \hspace{1cm} (1.2)

The band structure of graphene was first obtained by P.R. Wallace [61] using a tight-binding approximation. The energy bands are

$$E(k) = \pm t\sqrt{3 + f(k)} - t'f(k), \quad \text{(1.3)}$$

$$f(k) = 2 \cos(\frac{\sqrt{3}}{2}k_ya) + 4 \cos(\frac{3}{2}k_xa) \cos(\frac{3}{2}k_ya) \quad \text{(1.4)}$$

where $t$ is the nearest neighbor hopping energy and $t'$ is the next nearer neighbor hopping energy. The plus sign expression above is referred to as $\pi^*$ band while the minus sign is said to be the $\pi$ band. As can be seen from Fig. 1.2 the conduction and valence bands touch at six points. This six points are colloquially referred to as Dirac points, because the hamiltonian describing the electrons around these points is as matter of fact the Dirac equation. Two of these points are inequivalent and are labeled $K$ and $K'$. Their position in momentum space are

$$K = \left(\frac{2\pi}{3a}, \frac{2\pi}{3\sqrt{3}a}\right), \quad K' = \left(\frac{2\pi}{3a}, -\frac{2\pi}{3\sqrt{3}a}\right). \quad \text{(1.5)}$$

An interesting result is obtained when Eq. 1.3 is expanded around $K$ with $k = K + q$

$$E(q) \approx v_f q + O\left[\left(\frac{q}{K}\right)^2\right] \quad \text{(1.6)}$$

a linear dispersion, where $v_f = \frac{3t}{a}$ is the Fermi velocity. Notice that the Fermi velocity is independent of momentum and since $t = 2.7$ eV [53] and $a \approx 1.42$ Å, $v_f \approx 1 \times 10^6$ m/s.
1.2 Mobility

A highly desired quality in a device is to achieve ballistic transport. High mobility is a direct measure of electronic transport with low scattering. In this regime, the mean free path of the charge carriers is larger than the sample size. In graphene the mobility $\mu$ is proportional to the mean free path $l$ according to

$$\mu = \frac{e}{\hbar \sqrt{\pi n} l}$$  \hspace{1cm} (1.7)

### 1.2.1 Determining electron mobility

How do people actually measure mobility in graphene samples? Electronic transport in graphene is studied by fabricating a field-effect transistor (FET). When
graphene is subjected to a gate voltage $V_g$, a carrier density $n$ is induced with

$$n = \frac{V_g C}{e A} = \frac{k_s V_g}{4\pi t e}$$

where $t$ and $k_s$ are the thickness and permittivity of the substrate, respectively. The conductivity follows the dependence

$$\sigma = n e \mu$$

which allows us to determine $\mu$ experimentally from

$$\mu = \frac{4\pi t}{k_s} \frac{d\sigma}{dV_g}$$

A conductance vs gate voltage plot is shown in Fig. 1.3, where the typical ambipolar electric field effect is exhibited.

![Graph showing conductance vs gate voltage](image)

Figure 1.3: Conductance as a function of gate voltage for a graphene device.

Mobility is most commonly expressed in units of cm$^2$/Vs. Experimental work in the room temperature mobility of graphene on SiO$_2$ [18] determined a 4 × 10$^4$ cm$^2$/Vs
limit. This limit is due to remote interfacial phonon scattering by the polar optical phonons of the SiO$_2$ substrate \[30\]. However this limit is only attainable if charged impurity scattering \[33, 32\] and fabrication residues are reduced.

The mobility can be increased in suspended samples where values as high as 230,000 cm$^2$/Vs have been reported \[13\] in low temperature measurements. In such samples the substrate, below the graphene flake, has been chemically etched.

1.3 Valley degree of freedom

Since $K$ and $K'$ are independent, graphene has an extra degree of freedom, the so-called valley degree of freedom. Electronic devices manipulate the charge of the electron, a device that manipulates the valley of the electron is called by analogy a valleytronic device.

Although several theoretical valleytronic devices have been previously proposed in single layer graphene \[54, 27, 22, 23, 52\] and bilayer graphene \[43, 56\], fabricating these devices is far from simple. For instance, the proposed device in \[54\], would require a graphene strip with a sub 10 nm constriction and zigzag edges, while the device in \[27\] would require a line defect (see Fig. 1.4). Fulfilling these requirements is experimentally challenging. I should mention that tremendous progress in valleytronics has been made by Gorbachev et al.\[24\] in graphene on boron nitride samples. We would like to tackle the valleytronics challenge by proposing a feasible device on single layer graphene.

1.4 Strained Graphene and Pseudomagnetic Field

When a mechanical strain is applied to graphene, such that the Brillouin zone is deformed by moving the dirac cones $K$ and $K'$ in opposite directions, a gauge field
$A$ is generated \cite{16}. This gauge field has a different sign depending on whether the electron sits on $K$ or $K'$, that is $A_K = -A_{K'}$, since elastic deformations do not violate time-reversal symmetry of a crystal \cite{26, 25, 45}. Experimental work \cite{39} has shown Landau levels arise from pseudomagnetic field in strained graphene nanobubbles grown on a platinum (111) surface.

It is actually possible to engineer strain to produce a nearly constant pseudomagnetic field \cite{26}. When a graphene membrane covers an aperture of characteristic size $L$, as shown in the supplementary information of \cite{26}, the pseudomagnetic field can be written as

\[
B_s(x, y) = \beta \frac{\Phi_0}{aL} \left( \frac{PL}{\mu} \right)^{2/3} \tilde{B} \left( \frac{x}{L}, \frac{y}{L}, \nu \right)
\]

where $\nu$ is the Poisson ratio, $\mu$ is the shear modulus, $\Phi_0$ is the flux quantum and $\tilde{B}$ is a dimensionless function related to the membrane deformation and geometry. In the case of an equilateral triangle $\tilde{B}$ is nearly uniform, as can be seen in Fig. 1.5.
Figure 1.5: $\vec{B}$ for a graphene membrane suspended over a triangle. This image was taken from the supplementary information of [26]

### 1.5 Proposed Graphene Valley Filter

In the ballistic regime, if a beam of electrons were to pass through a constant pseudo magnetic field, $K$ and $K'$ electrons would follow divergent paths. Thus we propose to fabricate a device with the geometry shown in Fig. 1.6. Graphene sits on top of an etched equilateral triangle. A voltage is applied between the top and the bottom three electrodes. When graphene is not strained, in the ballistic regime a beam of electrons follows a straight line. On the other hand, if graphene is strained, a pseudo magnetic field of opposite sign for different valleys would split the beam into two divergent beams.

### 1.6 From Valleytronics to Pressure Sensing

The original purpose of our research was focused on realizing the graphene valley filter proposed in the previous section. Despite the challenging fabrication, due to
the tools available in the UC Riverside cleanroom, it was possible to write $\sim 50$ nm size electrodes and the required alignment step to the $\sim 200$ nm size triangle. However, we found that we were unable to obtain ballistic transport due to the relatively low mobility in our samples. Nevertheless, we investigated their transport properties under strain and found that the electronic properties could be altered by strain. Upon testing our devices under the pressure of inert gases (He and Xe), the electrical response of the graphene nanobubbles to pressure was evident under a simple experimental measurement. This fact and the timely publication of two manuscripts\cite{70, 57} on the piezoresistivity of graphene devices was a motivation to steer our research towards the application of our devices to pressure sensing.

1.7 Pressure Sensing in Graphene

As mentioned before, graphene forms a impermeable membrane \cite{15}. As such, it is an ideal material to create nanoelectromechanical systems (NEMS). I should mention that the study of graphene under strain is an interesting subject, since it has been proposed as a mechanism to open a band gap \cite{51}. Due to the miniature size of the
NEMS, the mechanical behaviour of graphene is influenced by the van der Waals force [35, 36]. The adhesion energy of monolayer graphene has been found to be $0.45 \pm 0.002 \text{ Jm}^{-2}$[35]. This adhesion allows graphene to form switchable blisters, whose shape depends on the geometry and pressure, as shown by Boddeti et al. [12, 11].

Previously, the possible application of graphene to pressure sensing technology has been experimentally studied [70, 57]. Both approaches take advantage of the piezoresistivity effect of graphene. However as we show in this work the size of the device plays an important role to determine the underlying mechanism that affects the strenght of the electrical response of the NEMS.
Chapter 2

Device Fabrication

The device fabrication can be summarized in the following three steps:

1. A triangular hole is etched in a silicon dioxide chip.

2. Graphene is directly exfoliated or transferred over the etched hole.

3. Electrodes are created.

2.1 Silicon Chips

Silicon wafers with a 290 nm surface thickness of silicon-dioxide ($\text{SiO}_2$) are cut into small chips. The chips are then cleaned in an ultrasonic bath first in acetone and then in isopropyl alcohol (IPA). Finally they are blow dried with nitrogen gas.

2.2 Etching a triangle in the silicon chip

To create a triangular hole in the silicon dioxide, a 200 nm side triangle is defined with e-beam lithography, then using polymethylmethacrylate (PMMA) resist as a mask, 150 nm of the SiO$_2$ are dried etched with CHF$_3$ in a Reactive-ion Etcher.
Finally the PMMA mask is removed using remover PG\textsuperscript{1} and the residue is burned off with oxygen at high temperature. This is to avoid introducing even more residues than the ones inherent to the eventual lithography process.

\begin{figure}[h]
\centering
\includegraphics[width=0.7\textwidth]{image.png}
\caption{Scanning electron microscope image of the etched triangles.}
\end{figure}

### 2.3 Graphene Exfoliation

Before the graphene exfoliation takes place, the silicon chip is further cleaned by ultrasonic in acetone and IPA for 10 minutes and blow dried with Nitrogen.

#### 2.3.1 Direct exfoliation

Graphene is mechanically exfoliated on a silicon dioxide wafer\textsuperscript{50} that contains multiple etched triangles and identified using an optical microscope. Due to its notable opacity\textsuperscript{10}, graphene can be visually detected when exfoliated on silicon dioxide.

\textsuperscript{1}Remover PG is a proprietary N-Methylpyrollidone based solvent stripper designed for efficient and complete removal of PMGI, PMMA, SU-8, and other resist films on Si, SiO\textsubscript{2}, GaAs, and many other substrate surfaces.
ide with a thickness of \( \sim 300 \) nm. As you can see in Fig. 2.2, there is a clear contrast between the silicon dioxide and the graphene flake. To accurately determine the number of layers in the exfoliated flake, the Raman spectra of the sample is taken. An example of such spectra is shown in Fig. 2.3. Graphene has two main peaks, the G peak at \( \sim 1580 \text{ cm}^{-1} \) and the 2D peak at \( \sim 2700 \text{ cm}^{-1} \). For single layer graphene the intensity of the 2D peak is greater than the intensity of the G peak [21]. Despite its inherent low sample yield, samples can be actually fabricated using this method. However, since it relies on random chance (in order to exfoliate graphene over the triangles), it was later substituted with a transfer technique.

Figure 2.2: Optical image of graphene on \( \text{SiO}_2 \).
2.3.2 Transfer

Alternatively, graphene can be transferred directly over the etched triangle. This is done by following a technique first described by Zomer et al. [72], but instead of transferring graphene over boron nitride, graphene is transferred over the holes. A transfer mask that contains three layers is prepared. The first layer is a transparent glass slide. The second layer is an adhesive tape (3M 600 transparent scotch tape), with the adhesive side facing the glass slide. The third layer is an spin coated layer of methyl/n-butyl methacrylate copolymer (Elvacite 2550 acrylic resin) dissolved in methyl isobutyl ketone (MIBK). The mask is baked for 5 minutes at 130°C to remove the MIBK solvent from the polymer. Graphene is then mechanically exfoliated on the mask and identified by optical microscopy. To transfer a selected flake, a silicon chip with an etched triangle is aligned with the transfer mask. The silicon chip is attached to a linear stage (Newport 460A-XYZ) that has been fitted with a heat source to promote
the adhesion of the polymer to the silicon chip. As a reference for future lab members, I should mention that during the transfer process the color of the elvacite changes as the polymer attaches to the silicon chip\textsuperscript{2}. After the transfer process is completed, I recommend baking the sample for 5 min. at $\sim 100^\circ$C. This is to decrease the chances of the flake rolling up, when the transfer polymer is dissolved. Finally the polymer is removed with acetone.

2.4 Contacting Graphene

2.4.1 Spin coating

A resist bilayer of copolymer poly(methylnethacrylate-methacrylic acid) (MMA)/PMMA is spun on the silicon chip and electrodes are defined using e-beam lithography. Both MMA and PMMA are spin coated at speed of 4000 rpm and are baked for 10 minutes at a temperature of 150$^\circ$C and 180$^\circ$C, respectively.

2.4.2 Dry transfer of MMA

Isolated single graphene flakes, that are not attached to multilayer graphene, might end up rolling up during the spin coating process. To avoid this, it is possible to dry transfer the MMA with the aid of a transfer chip and a sacrificial layer of water soluble polyvinyl alcohol (PVA). This process is adapted from [19, 20] and requires to first spin coat two layers, one of PVA and one of MMA, in a dummy silicon chip. PVA is baked for 5 min at 95$^\circ$. Then, dicing tape is used to cover the dummy chip. Afterwards, the dummy sample is immersed in water for several hours and the tape with MMA layer is released from the chip with a magnetic stirrer bar set at 750 rpm. Upon inspection

\textsuperscript{2}It goes from a semitransparent color to a dark yellow.
in an optical microscope, one can determine and select an area in the MMA that looks wrinkle free. Finally the target chip, the one that has the graphene flake, is heated up at 150° and the MMA containing tape is placed on top on our target chip and the tape can be peeled off after 5 minutes, leaving the MMA on the chip.

### 2.4.3 E-beam Lithography

E-beam lithography is a process in which a scanning electron microscope is used to define a pattern in a substrate that has been coated with a resist. This resist is exposed as the electron beam is scanned across a predetermined area. In the case of a positive resist, this exposed area can be removed with an appropriate solvent. This process defines a window in the resist, that allows us to deposit metal to create the electrodes.

In this work, when defining the electrodes, a bilayer resist is used. The bilayer is composed of a bottom layer of MMA and a top layer of polymethyl methacrylate (PMMA). Since MMA is more sensitive than PMMA this leaves an undercut that eases the lift-off process. This bilayer resist is exposed with a dose of 300 $\mu$C/cm² and developed in a MIBK/IPA (1:3) solution for 70 s.

![Nanolithography process](image)
2.4.4 Metal Deposition

After the development step, the electrodes are created by evaporating Ti (10 nm) and Au (60 nm) using an e-beam evaporator. Titanium is evaporated at a rate of 1 Å/s, while Gold is evaporated at 2 Å/s. The deposition rate along with low pressure (\(\sim 2 \times 10^{-6} \) Torr) at the beginning of the actual deposition, ensures that a typical resistance of 1kΩ (for a pair of contacts in graphene) is achieved. It is a good practice to check the appearance of the metals to be deposited, since sometimes cleanroom users load recipes that do not sweep the electron beam and end up damaging the quality of the metal by contaminating it with graphite\(^3\). Always make sure that there is enough metal in the crucible and that the evaporating recipe has not been modified.

2.4.5 Lift off

The lift off process is done by immersing the sample overnight in an acetone bath. Acetone strips off the resist, leaving the metal electrodes behind. To facilitate the lift off process the beaker, that contains the sample, is kept in a hot plate at a temperature of 65°C. To complete the lift off process, the sample is gently blown through a glass pipet. During this process the sample must be kept submerged in the acetone the entire time. In the event that some metal is still sticking to the chip, spraying the sample with a bottle can help; however the spray must not be aimed directly to the location of the graphene flake, this is to avoid tearing off parts of the graphene. Finally the sample is rinsed with IPA and blow dried with Nitrogen gas. Fig. 2.5 shows an scanning electron microscope image of a finished device.

\(^3\text{Crucibles are typically made of graphite.}\)
2.5 Experimental Setup

The experimental setup consists of a vacuum chamber, a pump and He gas cylinder. The sample is loaded in the vacuum chamber and pumped to a vacuum of \( \sim 1 \times 10^{-6} \) Torr. Whenever the vacuum chamber needs to be pressurized, speed valves can be opened and closed to control the flow of He gas from the tank to the chamber. An schematic representation of this setup is shown in Fig. 2.6.

![Figure 2.6: Experimental Setup](image.png)
2.6 Electrical Measurement

Standard dc techniques are used for the electrical measurements. Current voltage curves and gate voltage sweeps are obtained in vacuum at room temperature. A computer equipped with a National Instruments data acquisition card (DAQ), through the software Mezurit2⁴, supplies the bias voltage and the gate voltage. The bias voltage is applied to the device through current source(a 512 kΩ resistor in series with the device), such that the graphene device is probed at mV scales. Current is measured with a DL Instruments current preamplifier (model 1211), that in turn is connected to the DAQ.

![Diagram](Figure 2.7: Circuit schematic for the two terminal room temperature measurement. Adapted from [59])

⁴Available at http://www.ugcs.caltech.edu/~mezurit2/
Chapter 3

Nanoscale graphene pressure sensors

This chapter consist of the publication [1] and is reprinted with permission from AIP Publishing LLC, Copyright 2015. The formulas that are used in this chapter are derived in Appendix B.

We study the transport properties of graphene layers placed over ∼200 nm triangular holes via attached electrodes under applied pressure. We find that the injected current division between counter electrodes depends on pressure and can be used to realize a nanoscale pressure sensor. Estimating various potential contributions to the resistivity change of the deflected graphene membrane including piezoresistivity, changing gate capacitance, and the valley Hall effect due to the pressure-induced synthetic magnetic field, we find that the valley Hall effect yields the largest expected contribution to the longitudinal resistivity modulation for accessible device parameters. Such devices in the ballistic transport regime may enable the realization of tunable valley polarized electron sources.
3.1 Introduction

Graphene is a single atomic layer material with exceptional electronic and mechanical properties [16], with a band structure that consists of two inequivalent Dirac point valleys, each with dispersion corresponding to Dirac fermions [16]. Graphene has formed the basis of nanoelectromechanical and strain sensing devices [4, 60, 41, 63, 70, 5, 31, 14, 57, 17, 68], including on elastomeric substrates [63, 5] and supported by thin insulating silicon nitride membranes [70]. However, the ultimate limit of miniaturization of such sensors has not yet been ascertained, although predictions have been made towards the operation of nanoscaled pressure sensors [67, 58]. Here we report measurements on nanoscale pressure sensors realized from graphene sheets suspended over 200 nm triangular holes with a ground plane at the bottom acting as an electrostatic gate electrode. Electrical transport measurements are performed while pressurizing the graphene, which acts as an impermeable membrane [15], with an inert gas. The pressure deflects the graphene sheet into the hole, inducing lattice strain. Based on the signal to noise ratio found in our devices, we find a typical pressure sensitivity of $20 \text{kPa}/\sqrt{\text{Hz}}$. We consider a number of pressure transduction mechanisms for realistic device parameters including piezoresistivity [70, 31, 57] and deflection of the device towards the gate, and find that at these small length scales and device geometry, the largest mechanism contributing to the resistivity change for typical devices is expected to arise from a valley Hall effect [26], in which a pseudomagnetic field generated from the nonuniform strain increases the resistivity of the membrane. This may enable the fabrication of very small pressure sensors and tunable valley splitter devices.
3.2 Methods

Fig. 3.1 shows the fabrication process. Our devices were fabricated on a Si/SiO₂ substrate with 300 nm of oxide (Silicon Quest International). Using electron beam (e-beam) lithography an array of 200 nm-side equilateral triangles is defined in a polymethylmethacrylate (PMMA) resist layer and subsequently 150 nm of the oxide is etched using reactive ion etching (Surface Technology Systems) with CHF₃ (Fig. 3.1a). The PMMA is removed by dipping in acetone overnight and high temperature oxygen annealing. Graphene is exfoliated from kish graphite (Covalent Materials Corporation) and transferred mechanically over the triangular hole (Fig. 3.1b). A resist bilayer of copolymer poly(methylmethacrylate-methacrylic acid) and PMMA, [P(MMA-MAA)/PMMA] is spun on and windows are defined using e-beam lithography (Fig. 3.1c). 10 nm of Ti and 65 nm of Au are then deposited with an e-beam evaporator (Temescal) at a deposition rate of 1 Å/s and 2 Å/s, respectively. The P(MMA-MAA)/PMMA bilayer is then removed by soaking the sample in acetone overnight (Fig. 3.1d). A colorized scanning electron microscope image of a completed device showing the graphene with attached electrodes is shown in Fig. 3.2. After fabrication, devices are probed at room temperature by applying a voltage to one contact and collecting the current at two other ones. An example of the measurement geometry is shown in the inset to Fig. 2. A current is injected into the green contact and the currents at the two red contacts I₁ and I₂ are measured. The two-terminal conductance versus gate voltage for typical samples indicates a field effect mobility of ~1000-3000 cm²V⁻¹s⁻¹, and thus the electronic mean free path is expected to be tens of nanometers or less even at large doping. (This behavior may result from resist residues, which could potentially be removed in future experiments using e.g. low temperature current annealing[46]. Because our experiment
is performed at room temperature, such an annealing step was not taken.) Therefore electron transport in the triangular region can be considered to be diffusive despite its relatively small size.

![Figure 3.1](image)

Figure 3.1: (a) Triangles are created using e-beam lithography and etched with CHF$_3$ using a RIE. (b) Graphene is exfoliated over the etched triangle. (c) Electrodes are created with MMA/PMMA resist using e-beam lithography. (d) 10 nm of Ti and 65 nm of Au are deposited with an e-beam evaporator.

### 3.3 Experiment

Previously, a number of experiments have investigated the response of graphene to an applied hydrostatic pressure[41, 70, 57]. However, in these experiments, the graphene devices were much larger, and in some cases also supported by substrates[70]. In the main panel of Fig.3.3 the fractional current difference

\[
\Delta I = \frac{I_1 - I_2}{I_1 + I_2}
\]  

(3.1) for a nanoscaled device D1 (200 nm triangular region edge length) with zero gate voltage applied is monitored versus time. After maintaining vacuum for 1-3 days to enable the
trapped gas in the triangular hole to diffuse out\cite{15}, and avoid outward pressure\cite{12} at time $t\sim 120$ s, 30 kPa of He gas is let into the sample chamber to pressurize it. The fractional current difference decreases and reaches a plateau. Slightly before $t\sim 300$ s, He pump-out is initiated and $\Delta I$ returns to the previous level as the pressure drops. The resistance change acts together with the asymmetric placement of the triangular hole (or other present asymmetry) with respect to the leads to produce a differential signal dependent on the resistivity change within the strained region of the membrane. The deviation of $\Delta I$ under the pressure of 30 kPa is $\sim 5\times 10^{-3}$, as shown by the dashed lines and double arrow in Fig. 3.3. Similar behavior has been observed on 4 additional samples.
3.4 Data Analysis

Here we consider a number of possible mechanisms for resistance change of the device. The first is piezoresistivity in which a strain causes a change in resistivity due to the lattice strain. In a number of experiments[4, 63, 70, 31], the gauge factor giving the ratio of the fractional change in resistivity to the strain was found to be

$$\eta = \frac{\Delta R}{\epsilon R} \sim 2$$

(3.2)

with one experiment yielding a somewhat larger value of $\eta = 6$ [5]. The characteristic strain in a triangular membrane under pressure has been previously estimated as

$$\epsilon = \left( \frac{PL}{\mu} \right)^{2/3}$$

(3.3)
where $P$ is the pressure, $L$ is the length of the triangular hole side, and $\mu$ is the graphene shear modulus, which we take to be $\mu = 150 \text{ N/m}$. This gives for this mechanism

$$\frac{\Delta R}{R} = \eta \left( \frac{PL}{\mu} \right)^{2/3}$$

(3.4)

The resistivity change is positive. Another potential mechanism is the change in sheet resistance due to the charge density changing when the graphene membrane approaches the gate due to the applied pressure. This leads to an increase in its capacitance to the gate. Considering this mechanism and approximating the triangular hole as circular gives an estimated\[44\]

$$\frac{\Delta R}{R} \approx -\frac{R \partial G}{4 \partial V_g} \frac{V_g h}{d}$$

(3.5)

where $G$ is the conductance, $R = 1/G$ is the resistance, $V_g$ is the gate voltage, $h = 0.15L(PL/\mu)^{1/3}$ is the deflection at the center towards the gate due to the pressure, and $d$ is the initial distance of the membrane from the back gate. Note that an additional factor of 1/4 occurs compared to the parallel plate result due to the fixed boundary conditions, which results in the resistance change being concentrated near the center.

Finally, when the membrane is deflected to produce a nano-bubble shape, it has been predicted\[26\] and demonstrated experimentally\[39\] that a pseudomagnetic field develops of magnitude

$$B = 0.3 \frac{\beta hc}{aeL} \left( \frac{PL}{\mu} \right)^{2/3}$$

(3.6)

where $a$ is the C-C bond length, $\beta = \partial(\log t)/\partial(\log a) = 2 - 3$ with $t$ the nearest neighbor electron hopping parameter, $c$ is a factor of order unity, $e$ the electric
charge and $h$ Planck's constant\[40\]. Here we take $c = 1$ and $\beta = 2.5$. This results from the displacement in momentum space of the Dirac points by the local strain, which acts as an effective vector potential. A nonuniform strain then produces an effective magnetic field. However, because the strain does not break time reversal symmetry, the pseudomagnetic field has the opposite sign for the carriers with momenta near each of the two inequivalent Dirac points, which maintains overall time-reversal symmetry in the crystal\[26\]. As a result, in the diffusive transport regime, the electric field and current remain parallel. However, the longitudinal resistivity is nevertheless altered by the deflection of the carriers between scattering events. Using a Drude model\[69\] we find

$$\frac{\Delta R}{R} = (\mu_m B)^2 \tag{3.7}$$

where $\mu_m$ is the carrier mobility. The off diagonal elements of the resistivity tensor are zero because of the opposite pseudomagnetic field experienced by the carriers in the two valleys.

The contributions of these various potential resistivity modulations are summarized in table 3.1 for a particular set of device parameters for a model device denoted D2 that has similar parameters to the one studied experimentally but with somewhat higher mobility and at nonzero $V_g$. The device parameters are: $L = 200$ nm, $P = 30$ kPa, $d = 300$ nm, $V_g = 20$ V, $\mu_m = 5000$ cm$^2/(V$s), $dG/dV_g = 15$ $\mu$S/V, $a = 0.14$ nm

is the graphene nearest neighbor bond length, a typical value of the gauge factor $\eta = 2$ \[4, 63, 70, 31\] $R = 3.4$ k$\Omega$. 

27
Mechanism & $\Delta R/R$ & $\Delta R/R$ for device D2 ($\times 10^{-3}$) \\
--- & --- & --- \\
Approach of membrane to gate & $-0.00375 \frac{\partial G}{\partial V_g} \frac{V_g}{d} \left( \frac{PL}{\mu} \right)^{1/3}$ & -0.87 \\
Piezoresistivity & $2 \left( \frac{PL}{\mu} \right)^{2/3}$ & 2.3 \\
Valley Hall effect & $0.56 \left( \frac{\mu_{mh}}{aeL} \right)^2 \left( \frac{PL}{\mu} \right)^{4/3}$ & 4.2 \\

Table 3.1: General equations and specific values for a model device for the three considered resistivity modulation mechanisms of membrane approach to gate, piezoresistivity, and the valley Hall effect.

The largest contribution for this device is expected to arise from the valley Hall effect. It is interesting to study how the various contributions depend on the device size. The results for $\Delta R/R$ for a device identical to D2 except with different triangle edge lengths are plotted in Fig. 3. The crossover to the regime where the valley Hall effect dominates is $L \sim 250$ nm, and the valley Hall effect becomes yet more dominant as the device size is reduced. This indicates that for optimizing the pressure sensitivity of submicron pressure sensors, $L \sim 250$ nm leads to the least predicted sensitivity for these device parameters. The valley Hall effect also has the strongest dependence on pressure $\propto P^{4/3}$, so at larger pressures, the valley Hall effect would be expected to be even more dominant over the other mechanisms. For experimental sample D1, the change in $\Delta I$ is approximately 0.5, which suggests that the gauge factor is 4 and is somewhat larger than that typically found in larger membranes[4, 60, 41, 63, 70, 5, 31, 57]. The mobility for this device was found to be somewhat lower than for D2, and the valley hall contribution to $\Delta R/R$ is expected to be smaller but a similar order of magnitude to that expected from piezoresistivity. The gate charging mechanism is negligible since the data was taken with $V_g = 0$. Assuming that the valley Hall effect is dominant, based on the root mean square noise level, measured to be $5.7 \times 10^{-4}$ for a bandwidth of 0.1 Hz, and the value of the step in $\Delta I$ determined from Fig. 2, we estimate the pressure sensitivity for our devices under these experimental conditions to be $\sim 20 \text{kPa/}\sqrt{\text{Hz}}$ by determining the
threshold for the signal to exceed the noise floor using the results in table 3.1. Assuming
the piezoresistivity mechanism is dominant yields a similar order of magnitude result.
More experiments will be necessary to determine the origin of the resistance changes in
these devices. Nevertheless, this demonstrates that nanoscale-area graphene membranes
are capable of pressure sensitivity with a large gauge factor. Further work will explore
the low doping and ballistic transport regimes. In this regime, the cyclotron orbit radius
for a given pseudomagnetic field becomes large. Because of time-reversal symmetry, the
effective pseudomagnetic field has the opposite sign for electrons in the two valley states
of graphene[26, 40]. Thus if a ballistic electron beam were to cross a region with a
nonzero pseudomagnetic field, electrons in the two valley states would follow diverging
trajectories. This may enable the realization of devices that are able to split a charge
current by valley index to produce a tunable source of valley polarized electrons towards
realizing valleytronics[40, 54, 22, 23, 52, 43, 56, 65, 66, 24, 7].
Figure 3.4: Plot of for three different indicated mechanisms of resistivity modulation in suspended graphene devices with pressure versus the side length of the triangular suspended region, for device D2. The sign of the gate approach mechanism has been inverted to facilitate comparison of its magnitude with the other contributions. The valley Hall effect dominates for sufficiently small devices.
Chapter 4

Transport in Strained Graphene at Low Temperatures

We study single layers of graphene transferred over Cr/Au electrical contacts on oxidized Si wafers with etched triangular holes in the oxide. The layers are strained by applying pressure electrostatically using a gate voltage and hydrostatically using an external inert gas. We investigate electronic transport in this suspended variable-strain graphene system at low temperatures.

4.1 Methods

We study samples with the same fabrication procedure and geometry as described in detail in Chapter 2. The only difference lies in the metallization of the contacts. We have chosen to deposit 5 nm of Chromium and 50 nm of Gold.
4.2 Experimental Setup

Samples are measured in a liquid helium cryostat. The sample is mounted in a sample holder, that fits in the sample space of a cryostat. The sample holder contains baffles that reflect radiation from the top.

![Experimental setup for the low temperature measurements.](image)

4.3 Low Temperature electrical measurement

4.4 Current Annealing

Normally samples as fabricated will show no Dirac point due to resist residues. Samples can be cleaned using low temperature current annealing [46]. To that purpose the collimator electrodes have been slightly modified as shown in Fig. 4.3.
4.5 Experiment

The two terminal conductance of the pair of electrodes shown in green in Fig. 4.4 is studied as a function of gate voltage and pressure. The bias voltage is kept fixed while the gate is swept, this measurement is done at vacuum, 5 psi and 10 psi.

As shown in Fig. 4.5 and Fig. 4.6, we found that the conductance as a function of gate voltage exhibits aperiodic fluctuations. By sweeping up and down the back gate, we observed that these fluctuations are reproducible, and therefore not due to electronic noise. The value of the fluctuations in a $e^2/h$ range seems to suggest that the fluctuations might be universal conductance fluctuations (UCF). Additionally, at fixed gate voltage, there is a clear difference in the conductance value as a function of pressure. This might be strain-induced UCF as function of pseudomagnetic field. However, more experiments are needed to determine this. For instance we must carefully study the fluctuations as a function of increasing pressure at low temperature and experimentally show that the variance of these fluctuations decreases as the temperature is increased.
4.6 Universal Conductance Fluctuations

Universal conductance fluctuations are fluctuations of the order of the conductance quantum \( e^2/h \), that are found in mesoscopic samples. They are typically observed when the conductance is measured as a function of parameters like the magnetic field or the gate voltage. The conductance depends on the disorder configuration and presents fluctuations which are characteristic of the phase coherence of the system [2]. The amplitude of the fluctuations is independent of the sample size and degree of disorder as long as the temperature is low enough so that \( k_B T \) and the inelastic-scattering rate are less than the inverse time to diffuse across the sample [38]. It is important to remark that these aperiodic fluctuations are not noise, since they are reproducible with respect to the inversion of the external parameter for which they have been observed [29].

\[^1\text{where } k_B \text{ is Boltzmann constant} \]
Figure 4.4: Schematic of the device. A voltage is applied between the two green electrodes.

In 1985, UCF were experimentally observed in nanometer-size metallic samples by Webb et al. [64] confirming earlier theoretical predictions by Altshuler and Khmelnitskii [3] and Lee, Stone and Fukuyama [37].

4.7 Universal Conductance Fluctuations in Graphene

UCF in graphene have been studied numerically, analytically and reported in several experiments.

Numerically, the mesoscopic fluctuations of the conductance of a graphene strip have been studied by Pereira et al. [55]. They found that the variance of the conductance depends on whether there is strong or weak disorder in the graphene strip. For weak disorder, the variance is greatly enhanced if the potential is smooth on the scale of the atomic separation. On the other hand, for strong disorder, the value of the conductance approximates the value predicted by the Altshuler-Lee-Stone theory of universal conductance fluctuations.

Analytically UCF in graphene samples at energies not very close to the Dirac point were studied by Kharitonov and Efetov [34]. They demonstrated that in the absence of elastic scattering, that breaks the valley symmetry, the variance is four times
greater than in conventional metals, in agreement with the numerical study described above. These variance is however reduced by strong intervalley scattering and warping effects.

Experimentally UCF have been observed both as a function of magnetic field [45, 9] and gate voltage [28]. Furthermore, it is possible to image such fluctuations with the use of a liquid-He-cooled scanning probe microscope, as shown by Berezovsky et al. [7, 8]
Figure 4.6: Conductance as a function of gate voltage for 0 psi, 5 psi and 10 psi for device 2.
Chapter 5

Conclusion and Future Work

5.1 Conclusion

We have developed nanoscale graphene pressure sensors, determined the possible mechanisms that explain the observed resistivity change under pressure and have shown that the size of the device determines the dominant effect in such devices.

We have characterized the electronic transport of our strained graphene devices at low temperatures. We found conductance fluctuations as a function of gate voltage and changes in conductance as function of pressure at fixed gate voltage.

5.2 Future Work

To be able to realize a graphene valley polarizer under the proposed scheme the mobility of the fabricated devices must be improved. Since the fabrication methods have been well established, a way to improve the mobility of the samples would be to use a boron nitride substrate. Our fabrication layout should be readily applicable, since instead of etching a triangle in the SiO$_2$, boron nitride can be dry etched just as is done
for 1D electrical contacts in BN/Graphene/BN hetero structures [62]. Another interesting aspect that can be explored further is to systematically study the low temperature and pressure dependence of the observed conductance fluctuations.
Appendix A

Writing an E-beam Pattern

Successfully writing a pattern with e-beam lithography is skill that takes several
sessions to master. This appendix covers subtle details and pointers that will allow a
new user to master quickly the e-beam lithography art in the UC Riverside cleanroom.

For a helpful guide about e-beam lithography systems integrated to scanning electron
microscopes I recommend going over reference [47].

A.1 The Nanometer Pattern Generation System

The software used to write patterns in the e-beam lithography system of the UC
Riverside cleanroom is called the Nanometer Pattern Generation System (NPGS). The
pattern of electrodes must be created using DesignCAD. In DesignCAD, the electrodes
are normally drawn using a dashed line. A dashed line indicates to NPGS that the
entire area inside the dashed electrode must be scanned by the beam and therefore in
this area the resist will be exposed. This exposed resist can be subsequently removed
during the development process.
A.2 Focusing

To be able to write a sharp pattern, it is necessary to focus the electron beam on the surface of the resist. This can be done by focusing on residues that are deposited when scratching the resist with a diamond scribe. It is important to select features at the ends of the scratch. Ideally you want to focus at a scale smaller than the width of the thinnest electrode. Fig. A.1 is an example of how this scratch-made features look like. Notice the small gap indicated by the arrow. The scale bar tells us that this gap has a dimension of less than 50 nm. At high magnification like the one shown in Fig. A.1 I suggest scanning the image with line average at a scanning speed of 5.

Figure A.1: SEM image of a resist residue. The arrow points at a small gap

A.3 Aligment

Samples are align using two alignment steps, a broad alignment and a fine alignment. The crosses in the broad and fine alignment step are separated a distance
of 50 \( \mu m \) and 30 \( \mu m \), respectively. The alignment marks are etched in the SiO\(_2\) and are created in the same step as the triangles.

When running the alignment step in NPGS the following hot keys come in handy:

- ”A” Automatically adjust the contrast in the alignment windows.
- ”C” Increases the scanning speed of the alignment windows
- ”P” Increases the magnification

The samples fabricated in this project required electrodes of width as small as 50 nm. To write an electrode of this size is necessary to select the appropriate parameters in the ”NPGS Run File Editor”. The thin electrodes were exposed with a line dose of 2.5 nC/cm at a Magnification of 1000 with a Center-to-Center Distance and Line Spacing as close to 10 nm as possible. Bigger features were exposed with an area dose of 300 \( \mu C/cm^2 \). Notice that this recipe corresponds to an accelerating voltage of 20 kV with a 20 \( \mu \) aperture for a MMA/PMMA resist bilayer where each layer was baked for 10 minutes.
Appendix B

Mechanism for Resistance Change

In this appendix, the equations, that estimate the three possible mechanism for resistance change of the device in [1], are derived.

B.1 Piezoresistivity

In several experiments[4, 63, 70, 31], the gauge factor was found to be

\[ \eta = \frac{\Delta R}{\varepsilon R} \sim 2 \] (B.1)

The strain in a triangular membrane under pressure has been previously estimated\(^1\) as

\[ \varepsilon = \left( \frac{PL}{\mu} \right)^{2/3} \] (B.2)

where \( P \) is the pressure, \( L \) is the length of the triangular hole side and \( \mu = 150 N/m \) is the graphene shear modulus.

\(^1\)Guinea et al., Nat. Phys., 6, 2009.
Thus

\[
\frac{\Delta R}{R} = \eta \left( \frac{PL}{\mu} \right)^{2/3} \sim 2 \left( \frac{PL}{\mu} \right)^{2/3}
\]  
(B.3)

**B.2 Approach of Membrane to Gate**

We start with a parallel plate approximation. If the membrane gets a distance \( h \) closer to the gate, and the charge density in the membrane \( \rho \) changes by an amount \( \delta \rho \), the resistance change \( \Delta R \) will be approximately

\[
\Delta R = \frac{\partial R}{\partial \rho} \delta \rho
\]  
(B.4)

But \( \rho = C_g V_g / A \), where \( C_g = \epsilon_0 A / d \) is the gate capacitance with \( A \) the membrane area and \( \epsilon_0 \) is the vacuum dielectric constant. Therefore since the gate voltage is fixed \( \delta \rho = V_g / A \delta C_g \).

Under the applied pressure \( P \), the deflection at the center of the triangle is

\[
h = 0.15L \left( \frac{PL}{\mu} \right)^{1/3}
\]  
(B.5)

We then find

\[
\delta \rho = \frac{V_g}{A} \frac{\partial C_g}{\partial d} h = \frac{V_g}{A} \frac{\partial (\epsilon_0 A / d)}{\partial d} h
\]  
(B.6)

Thus

\[
\Delta R = \frac{\partial R}{\partial \rho} \delta \rho = \frac{\partial R V_g}{\partial \rho} \frac{\partial (\epsilon_0 A)}{\partial d} h
\]  
(B.7)

We can rewrite this in terms of the transconductance \( T = \partial G / \partial \rho \). We have

\[
T = \frac{\partial G}{\partial \rho} = \frac{\partial (1/R)}{\partial \rho} = -\frac{1}{R^2} \frac{\partial R}{\partial \rho}
\]  
(B.8)

using the chain rule we also find

\[
\frac{\partial G}{\partial \rho} = \frac{\partial G}{\partial V_g} \frac{\partial V_g}{\partial \rho} = \frac{\partial G}{\partial V_g} \frac{A}{C_g}
\]  
(B.9)
Putting this together gives

\[ \frac{\Delta R}{R} = -\eta R \frac{\partial G}{\partial V_g} \frac{V_g h}{d} \]  \tag{B.10}

where we added a factor \( \eta \) as a geometric factor which corrects for the true non-parallel plate geometry. We expect \( \eta \) will be about 1/4: one factor 1/2 since the average deflections is \( \sim h/2 \) and another 1/2 from the fact that the resistance change does not occur over the whole sheet.

### B.3 Valley Hall Effect

From [26] on strained graphene the pseudomagnetic field \( B \) is

\[ B = 0.5 \frac{\beta h c}{aeL} \left( \frac{PL}{\mu} \right)^{2/3} \] \tag{B.11}

where \( a = 2.4 \times 10^{-10} \) m is the graphene lattice constant ... The conductivity tensor components for each valley are

\[ \sigma_{xx} = \frac{\sigma_0}{1 + (\omega_c \tau)^2} \] \tag{B.12}

and

\[ \sigma_{xy} = -\frac{\sigma_0 \omega_c \tau}{1 + (\omega_c \tau)^2} \] \tag{B.13}

where \( \tau \) is the carrier momentum relaxation time, \( \sigma_0 \) is the dc conductivity for each valley, and the cyclotron frequency \( \omega_c \) is given by where \( v_F \sim 10^6 \) m/s is the fermi velocity and \( E_F \) is the Fermi energy. From [69] the conductivity and the relaxation time are related by

\[ \sigma_0 = \frac{e^2 E_F \tau}{2\pi \hbar^2} \] \tag{B.14}

while the Fermi energy is given by

\[ E_F = \hbar v_F \sqrt{\pi n} \] \tag{B.15}
where \( n \) is the carrier density. Thus we can write \( \omega_c \tau \) as

\[
\omega_c \tau = \frac{2AB\sigma_0}{C_gV_g} = 2\mu B
\]  

(B.16)

The conductivity tensor for the charge is the sum of that for the two valley [71]. Since \( B \) is the opposite sing for the two different valleys, the off-diagonal terms cancel and the conductivity is

\[
\sigma = \frac{\sigma_{dc}}{1 + (\omega_c \tau)^2}
\]  

(B.17)

where \( \sigma_{dc} \) is the total dc conductivity of the sheet. Finally

\[
\frac{\Delta R}{R} = \frac{\Delta \sigma}{\sigma} \approx (\mu B)^2
\]  

(B.18)
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