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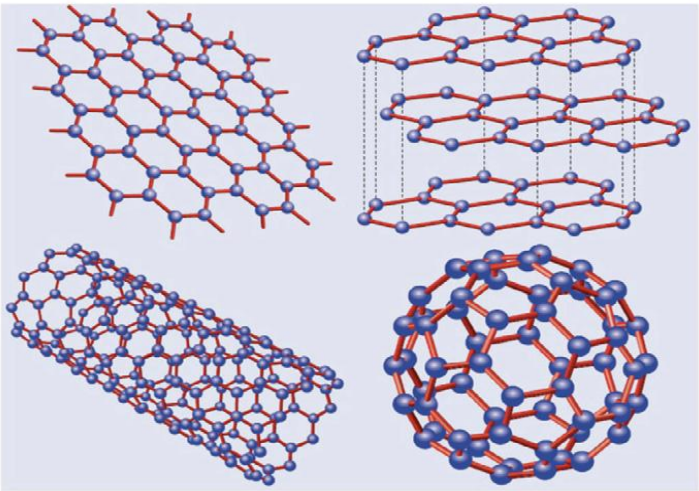
Multi-wavelength Raman characterization of back-gated monolayer and bilayer graphene

Master's Thesis in Electrical Engineering

Maedeh Arvani

Mahdi Aghajanloo



School of Information Science, Computer and Electrical Engineering
Halmstad University

Multi-wavelength Raman characterization of back-gated monolayer and bilayer graphene

Thesis submitted to the
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For the degree of Masters of Science
In
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By

Maedeh Arvani

Mahdi Aghajanloo

Department of physics

Freie University of Berlin

School of Information
Science,ComputerandElectrical Engineering

Halmstad University

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This diploma work is dedicated to our mothers.

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Abstract

In this work, we investigate the Raman spectrum of gated monolayer and bilayer graphene devices. We used Raman spectroscopy with three different excitation wavelengths: (488nm, 514nm and 633nm).For monolayer devices we observed the expected behavior for doped devices. For bilayer devices, we present a comparison between the theoretical model and our experimental results.

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1-Introduction of Graphene

1.1-Graphene

Graphene is an allotrope of carbon. It has a two dimensional crystal structure, that is, just one-atom thick. The atoms are arranged tightly in a honeycomb lattice. Graphene is considered the basic building form of all other allotropes of carbon. Graphite can be thought as stacked sheets of graphene one on top of each other, nanotubes are sheets of graphene which are rolled and fullerenes are wrapped sheets of graphene. Figure 1.1 presents this illustration in graphical form.

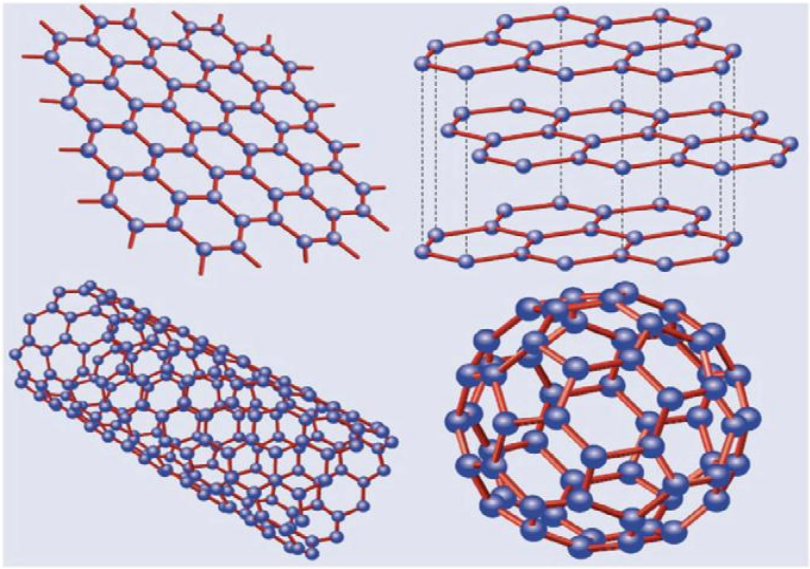


Fig1.1-Carbon allotropes: Clockwise from top left: 2D graphene, 3D graphite, 0D and 1D fullerens[4].

The length of the bond between two atoms of carbon is approximately 0.142 nm, and the distance between two sheets of graphene in graphite is 0.335 nm.

In 1962, Hans-Peter Boehm first used the term graphene, which is a combination of graphite together with the suffix - ene, to name a monolayer of carbon. For decades, scientists believed that graphene was an unstable material

and did not exist. There was a theory, the Mermin-Wagener theorem, that the melting temperature of the layers of crystal lattices with low thickness would decrease very fast, if their thickness decreased and the crystal would become unstable. Therefore, it was considered a material of theoretical interest. However, in 2004, a freestanding monolayer of graphene was isolated [5].

In 2010, Andre Geim and Konstantin Novoselov received the Nobel Prize in Physics for their fundamental experiments with graphene. Graphene research is a very young field and is one of the quickest growing areas of science. Because it is a completely new field, there are many new opportunities for research. This is because graphene has remarkable properties. For example, electrons in graphene present a Dirac-like electronic structure, something not observed in solids before. Furthermore, graphene has extraordinary mechanical stability [7] and optical properties. Thus, this area presents a rich platform for scientists to explore and industries to explore applications [4].

The atomic structure of graphene can be probed by TEM (Transmission electron microscope) where the diffraction pattern of electrons shows the hexagonal lattice of graphene [17]. It is possible to collect an atomically resolved image of isolated graphene on a substrate by STM (Scanning tunneling microscopy) [17]. Raman spectroscopy has proved an invaluable tool in graphene research. It can unambiguously and non-destructively determines its monolayer structure, determined by the shape of the 2D peak. Furthermore, it can provide insights regarding the doping level, the amount of disorder and the edge geometry [9].

Intrinsic graphene is a zero gap semiconductor or a semi-metal. In order to calculate the band structure of graphene, one calculates the band structure of one layer of carbon atoms, isolated from graphite. P.R.Wallace showed that the E-k relationis the following, $E = \hbar v_F \sqrt{k_x^2 + k_y^2}$, where the Fermi velocity is$v_F \sim$

10^6 m/s near the six corners of the hexagonal Brillouin Zone for low energies [17]. Due to the linear dispersion relation, electrons and holes in these six points behave as relativistic particles that can be described by the Dirac equation [17]. Therefore, they are called Dirac fermions and these corners are called Dirac points. Figure 1.2 shows monolayer and bilayer's valence and conduction band. As we can see here, the valence and conduction band of graphene are not separated. That is, graphene does not have a band gap.

1.2-Electronic properties of graphene

Graphene does not have a band gap, which is necessary for use in transistors. However, one can artificially introduce a band by cutting graphene in nanoribbons: the smaller width of the ribbons, the higher the gap. The other way to open band gap in graphene is by chemical modification. Graphene as a one atom thick allotrope of carbon, exhibit unusual two-dimensional Dirac-like electronic excitations. The Dirac electrons can be controlled by application of external electric and magnetic fields [20]. Raman spectroscopy probes phonons as well as electronic states. If the electronic dispersion changes, the Raman spectrum will also change. The electronic structure of graphene changes when moving from monolayer to bilayer, trilayer and so on up to about 10 layers. Above 10 layers, the electronic dispersion is the same as in graphite.

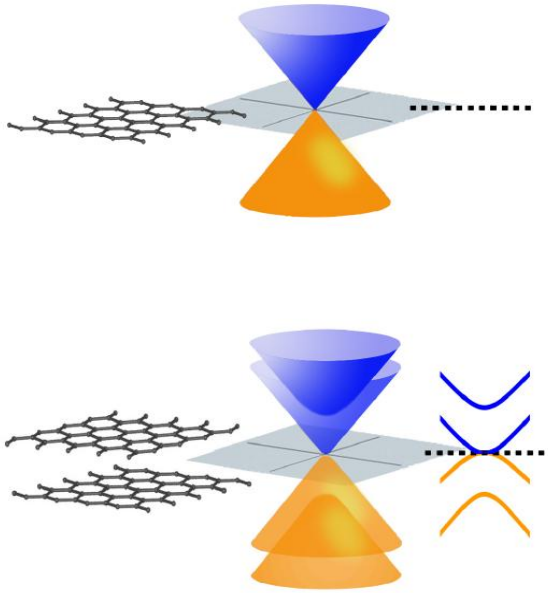


Fig1.2- Valence and conduction band of monolayer graphene (top) and bilayer graphene (bottom) [12].

1.3-Potential applications

Due to its special electronic behavior and its structural properties, it seems that graphene surpasses many metals and other conventional semiconductors. Today, silicon is by far the most important electronic material with applications in e.g. super computers, cell phones and GPS devices.

Many graphene-based applications are envisioned. The main theme is of course in electronics and it is expected that "graphenium" microprocessors will appear in the next 20 years. [13]

However, the most realizable application at the time being is the use of graphene as a composite material. Graphene´s extraordinary strength will be at the core of making a new generation of ultra-light and super-strong composite materials. Graphene's optical and electrical properties are also expected to find applications in flexible displays. Graphene also offers advantages in being a very good material for solid state gas sensors.

Graphene might also be used in solar industries, for biological sensing, and in chemical industries as a transparent conductor. It can be used in medical applications e.g. producing nanopores for DNA sequencing because it is very thin (less than 1nm) [13]. It can conduct electricity and heat better than copper.

2- Raman spectroscopy

2.1-Introduction of Raman spectroscopy

Raman spectroscopy is based on the Raman Effect, i.e on the inelastic scattering of light. Monochromatic light should be sent on the sample so a laser source is used, usually operating in the visible part of the spectrum. The light interacts with phonons, molecular vibration or other excitation in the sample system, and the energy of the photons will be shifted up, or down. Thus, a very small portion of the scattered light does not have $f_s = f_0$ where f_s is the frequency of the scattered light and f_0 is the laser frequency. The wave vector and frequency of the scattered light is shifted by a small amount $\mathbf{q}$ and f_m, respectively. Because of the momentum conservation we have: $\mathbf{k_s}=\mathbf{k_0}\pm\mathbf{q_m}$ and $f=f_0\pm f_m$.$\mathbf{k_s}$, is the momentum of the scattered light and $\mathbf{k_0}$is the momentum of the laser light. The energy shift gives information about the vibration modes of the material. Figure 2.1 shows the schematics of a Raman spectroscopy setup.

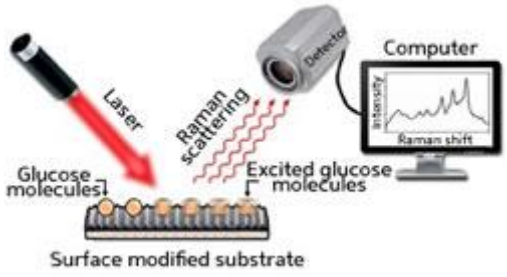


Fig2.1- The schematic diagram of the Raman spectroscopy setup [11].

2.2-Raman spectroscopy of monolayer and bilayer graphene

Raman spectroscopy is one of the best methods to characterize graphene because it is fast and nondestructive [9].High resolution TEM is the most direct tool for identification but it is time consuming and destructive. It is suitable only for fundamental studies [7].

The Raman spectra of graphene show features in 800-2000 cm^{-1} [9]. The G peak, which corresponds to the E_{2g} phonon at the central zone of the Brillouin zone, lies at 1580 cm^{-1} [9].The D peak, which is a breathing mode for sp^2 atoms, is a defect activated peak [9]. Usually, as there are not enough structural defects in pristine graphene, the D peak is not present. It can however be found at the edges of flakes. The second order of the D peak, the so-called 2D peak, is always present, even when there are no defects. The 2D peak lies at 2700cm^{-1} [9].The shape of the 2D peak gives us information about the number of layers. Monolayer graphene has a sharp 2D peak, while few-layers and thick graphite displays a broader 2D peak which consists of several unresolved peaks. Figure 2.2 shows the Raman spectrum of graphene and few-layer graphene. This figure shows that the 2D peak is a single sharp peak in graphene, while in graphite it contains a shoulder.

The Raman spectra of doped monolayer graphene is characterized by a doubly degenerate *G* peak at around 1580 cm^{-1}. This mode shows a strong electron-phonon coupling, which induces a phonon renormalization when *n*(carried concentration)is varied [3]. Therefore, Raman can be used to measure the total electron concentration.

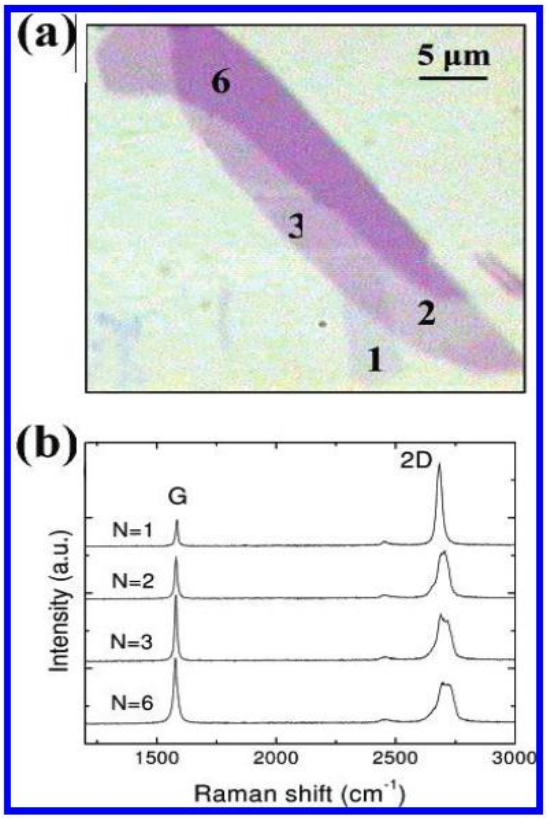


Fig2.2- Evolution of the Raman spectrum of graphene as a function of layer thickness. Monolayer graphene has a single sharp peak [7]

The G peak position of undoped graphene is at 1580 cm^{-1} and for increasing doping the position increases. The G shifting is due to the non-adiabatic removal of the Kohn anomaly at Γ [14].The Kohn anomaly is an anomaly, which describes the relation of a phonon branch in a metal[17].

2.3- Raman fitting parameters

Raman spectra were recorded with a WiTec Raman spectrometer under three different wavelengths: 488nm, 514nm and 633nm. The laser power that we used was 0.5mW. The peaks of the spectra were fitted with one single Lorentzian function using Origin Pro and peak-o-mat. The fitting parameters are used to establish the peak position and full-width at half-maximum. We refer to the position of the G peak as POS (G), the full-width at half-maximum FWHM(G).

3- Experiment

3.1-Fabrication of graphene (Scotch tape technique)

The experimental part of the thesis project has been down in the Freie University of Berlin in AG Casiraghi's laboratory. In this thesis we used the method originally used to isolate graphene. A piece of graphite is peeled off by scotch tape and then the flakes are transferred onto a substrate. Graphene was deposited on a silicon substrate covered with a layer of silicon oxide. This process is called micromechanical cleavage (the Scotch-tape method as it has been colloquially known). This is used to isolated graphene to provide optical enhancement that makes graphene visible under the optical microscope. The silicon, which is under SiO_2, can be used as a "back gate" electrode. The scotch tape technique process is based on 14 steps:

1. Cut wafer in pieces of about 1cm x1cm, oxidized layer always on top.
2. Place the wafer pieces on a hot plate at 100 C for 10 minutes.
3. Prepare sticky tape of 3cmx11cm, cleave a piece of graphite and make sure good coverage avoid dust falling down on it.
4. Wash the wafer in heated acetone to boiling point (56C) for approx 10 minutes.
5. Place beaker in sonicator for 10 min.
6. Continue cleaning in iso-propanol for 5 minutes.
7. Dry the wafer with nitrogen (again this has to happen quickly so the isopropanol cannot evaporate as well). You may repeat the cleaning procedure a second or third time for bigger flakes on the pieces of substrate.
8. Put the cleaned and dried pieces of wafer in a Petri dish and glue the prepared sticky tape on the pieces and press.
9. Cut the pieces.

10. Dissolve the glue in acetone.
11. Transfer to isopropanol.
12. Dry with nitrogen.
13. Heat samples up to 100C for 5-10 minutes.
14. Maybe cleave once again with tape if there are a lot thick pieces of graphite to be seen on the sample.

By this method, we can fabricate pristine graphene. Graphene can also be transferred so called wedging technique [19].The procedure is as follows: the graphene flake is first encapsulated in a polymer. Then the sample is immersed in deionized water. The water intercalates at the substrate-polymer interface, since the former is hydrophilic and the latter hydrophobic. This causes the graphene flakes to be lifted off the substrate, while still attached to the polymer film. Subsequently, the graphene–encapsulated polymer is deposited onto a new piece of substrate. The sample is then heated to70^{o}C in order to evaporate any water residuals on the surface. The results show that the doping can be removed by transferring the flakes on a clean silicon substrate. This could indicate that the doping is related to the oxygen and/or water trapped at the silicon surface after plasma cleaning. However it is also possible that the polymer used to encapsulate graphene could dope graphene, compensating the existing charges.

Our substrate consists of 550-micron thick silicon covered with 90nm of thermally grown silicon oxide.

3.2-Method of contacting

Electron-beam lithography is the conventional method used for extremely precise electrical contacting of nanostructures. Although having excellent resolution, the method is expensive, complex and time consuming. In addition,

the polymer resists and solvents which are used in this method can leave residues that contaminate our samples and contacts.

Another less accurate method for contacting nanoscale films e.g. grapheme is submicron soldering. We used this method to fabricate a gate contact to the graphene to be able to apply a high electric field between the graphene and the silicon back contact. This kind of samples allowed us to carry out Raman spectroscopy of gated pristine monolayer- as well as bi-layer graphene. The submicron soldering set up consists of an optical microscope, a sample heater and micromanipulator. [6] Figure 3.1 shows the set up.

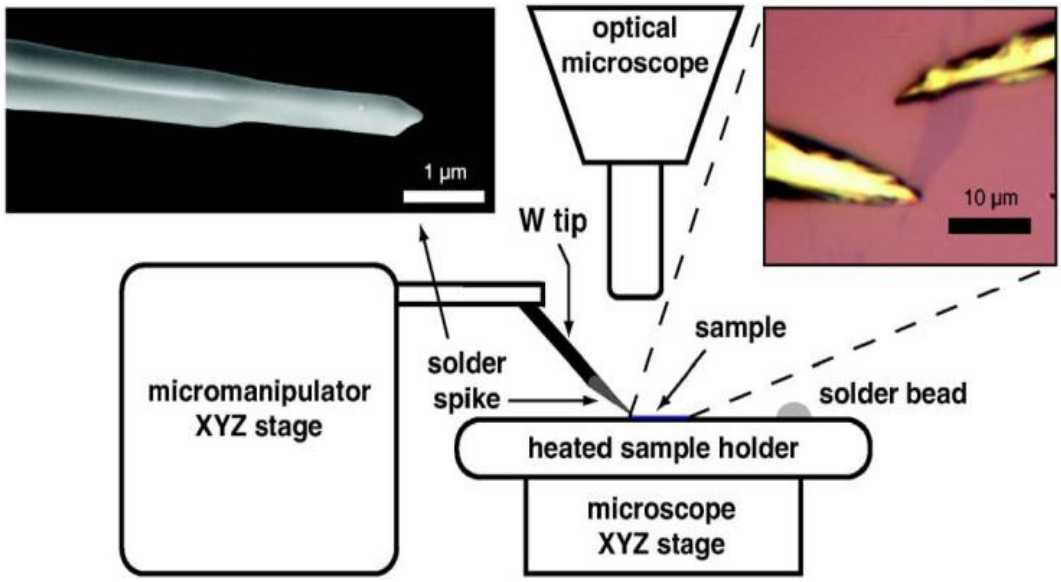


Fig3.1-Set up of submicron soldering, Upper left: Indium soldering pike ending
Upper right: Contacted graphene device [6].

3.3-Identification

The first step in working with graphene or its bilayer is identifying it on a substrate using a microscope. Our graphene monolayer and bilayer samples are

provided by Dr. Casiraghi's group at the University of Manchester. We selected only flakes that look thin (based on their color). Afterwards, we measured the Raman spectrum. From Figure 3.2, we observe that the shape of the 2D peak is a single, sharp Lorentzian peak. This is characteristic for monolayer graphene.

Figure 3.3 shows an optical micrograph of a graphene flake attached to a bilayer and few-layer. Graphene is the light violet part.

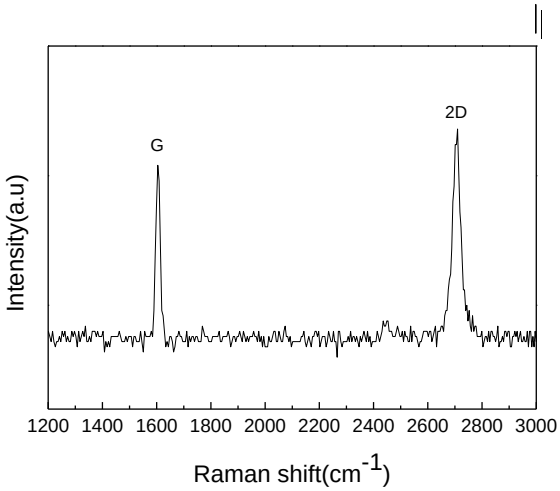


Fig3.2-Raman spectra of monolayer grapheneFig3.3-A graphene sample

Figure 3.4 shows bilayer graphene, as determined by the shape of the 2D peak (Figure 2.2).

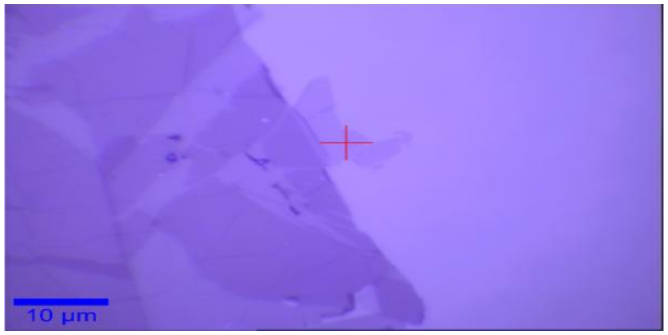


Fig3.4 - Bilayer graphene.

However, we can see a split in the G peak of the bilayer, In order to investigate the nature of this splitting, we applied a gate voltage to the sample. Figure 3.5 shows a fitting of Lorentzian functions to the 2D peak. Fig 3.6 and

3.7 show corresponding fittings of the G peak for pristine monolayer and bilayer graphene, respectively.

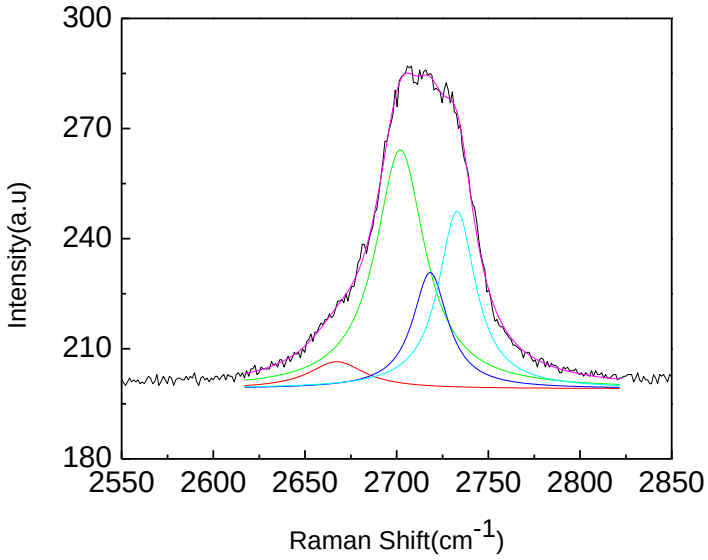


Fig3.5-2D peak is fitted by 4 Lorentzian curves.

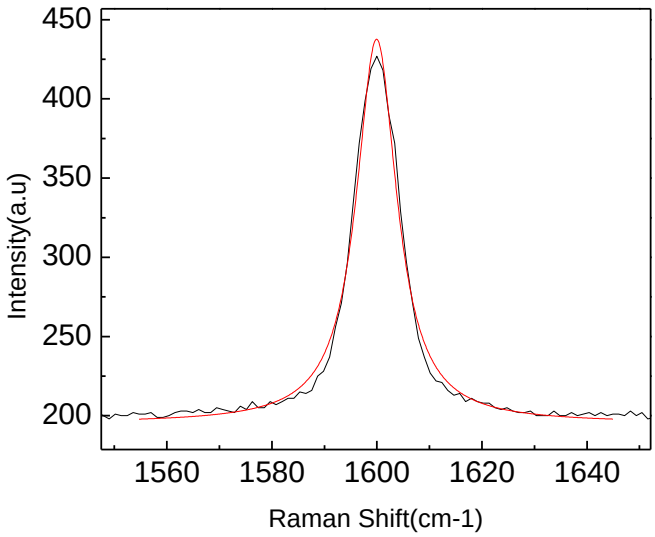


Fig3.6- G peak is fitted by 1 Lorentzian function.

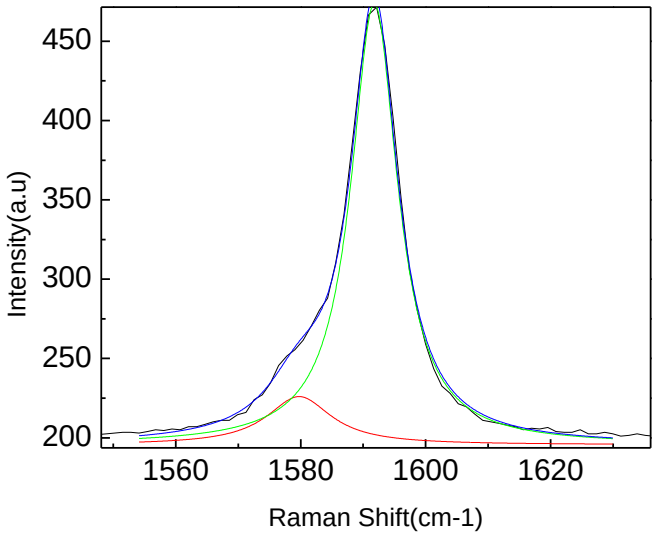


Fig3.7-G peak is fitted by two Lorentzian functions. Pristine bilayer graphene, the gate voltage is 12V.

We applied a gate voltage to the monolayer and bilayer graphene and the positions of the G and 2D peaks versus gate voltage were recorded under different wavelength. The G peak in bilayer graphene shows a single G component with a spectral position similar to monolayer graphene. When we apply gate voltage, due to the external electric field, the Fermi level position will change. The changes in the Fermi level make charge transfer split the G peak for the E_{2g} phonon mode. This mode shows very strong coupling between electrons and phonons [3]. The Raman G peak will have two components, which are associated with symmetric (S) and antisymmetric (AS) vibration of the atoms in the two sheets of bilayer graphene. In the bilayer, usually a single peak is detected that can split if the two sheets are doped differently [18]. When we have a doped bilayer, we can find two components in the G peak because the symmetry is broken by doping due to different n_{top} and n_{bottom} [3]. Figure 3.8 shows these two symmetric and antisymmetric vibrations.

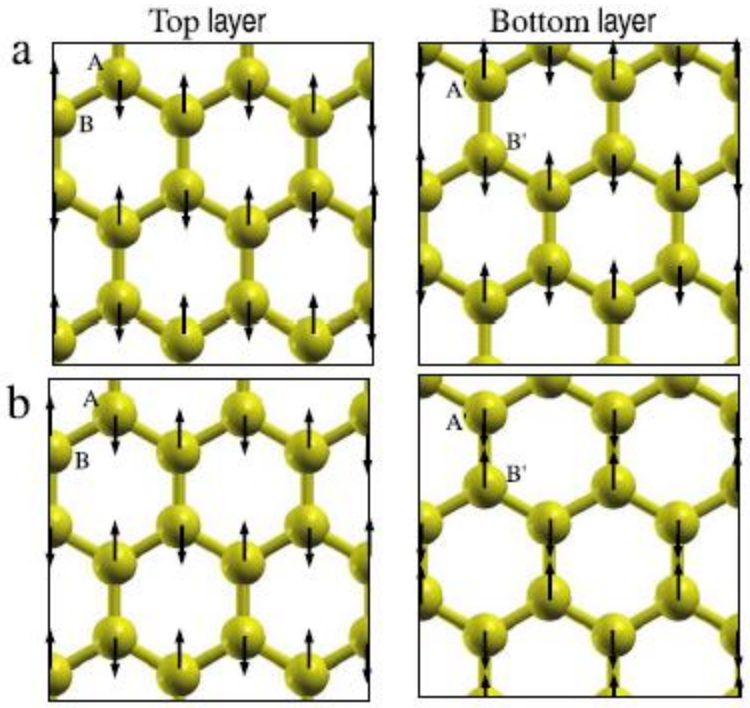


Fig3.8Schematic picture of (a) Symmetric(S) vibrations and (b)antisymmetric (AS) vibrations in bilayer graphene. [3]

The self-energy of the S and AS phonons for different Fermi energies is calculated by Ando [3]. He found a hardening of the symmetric and antisymmetric phonons, strongly influenced by carrier doping [3].

In this work, our experimental results confirm the theory and we can see that the G peak of the bilayer graphene at around 1580 cm^{-1} has two components that exhibit opposite dependence as the Fermi level of energy is tuned. The results are in agreement with Ando calculations. The splitting of the G peak in bilayer graphene can be used to find the electron concentration, n. [3]

3.4-Charge concentration in gated graphene

The gate voltage changes the Fermi energy ε_f and the Fermi surface of graphene. The following equation relates the gate voltage to amount of charge:

$$n=\eta\ (V_g - V_n)$$

Where V_g is applied gate voltage, V_n is the gate voltage required to reach the charge neutrality point, and η is the capacitive coupling η can be calculated as:

$$\eta = (\varepsilon_r . \varepsilon_0)/(t_{ox} e)$$

Where ε_0 is vacuum permittivity, $\varepsilon_r$is permittivity of silicon, $t_{ox}$is the thickness of the silicon oxide and e is electron charge. In our case ,ε_r=3.9 andε_0=8.85*10^-12 Fm^{-1} and t_{ox}=90nm therefore η=2.39*10^11 $cm^{-1}V^{-1}$. If the sample is undoped, at V_n=0 we have n=η.V_g.

When the sample is doped (i.e. the G peak position > 1582 cm^{-1}) then we have to add an extra voltage to have graphene at the charge neutrality point.

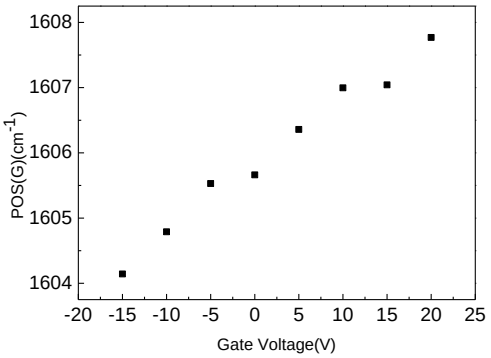
4- Results

4.1-Gated Monolayer graphene

When we apply gate voltage the Fermi level will change. This results in changes in the positions of the G peak and the 2D peak. We measure G peak and 2D peak shifts versus gate voltage from-30V up to +30V by voltage step 1V at three different wavelengths (633nm, 514nm and 488nm). Figures 4.1 and 4.2 show G peak and 2D peak shift versus gate voltage from -20V up to +20V. Our data show a linear relationship between gate voltage and G peak position, and similar relationship between gate voltage and 2D peak position. As we can see in Figure 4.1, for both wavelengths, the position of the G peak shifts to higher energy for increasing whole doping and to lower energy for electron doping.

Figure 4.2 shows the 2D peak position.

a)



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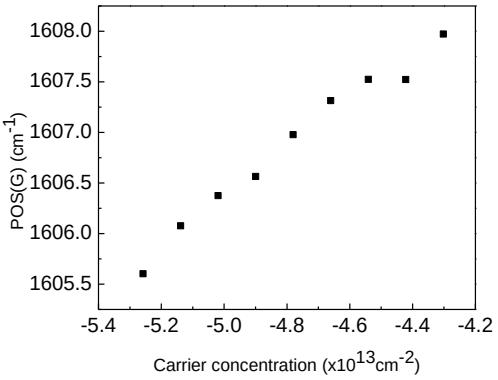

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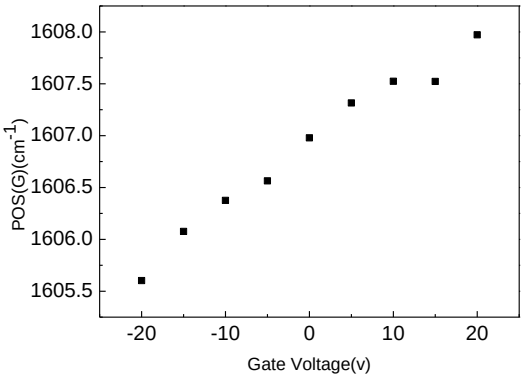


d)

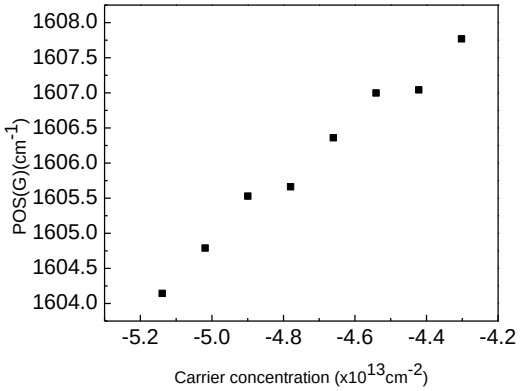
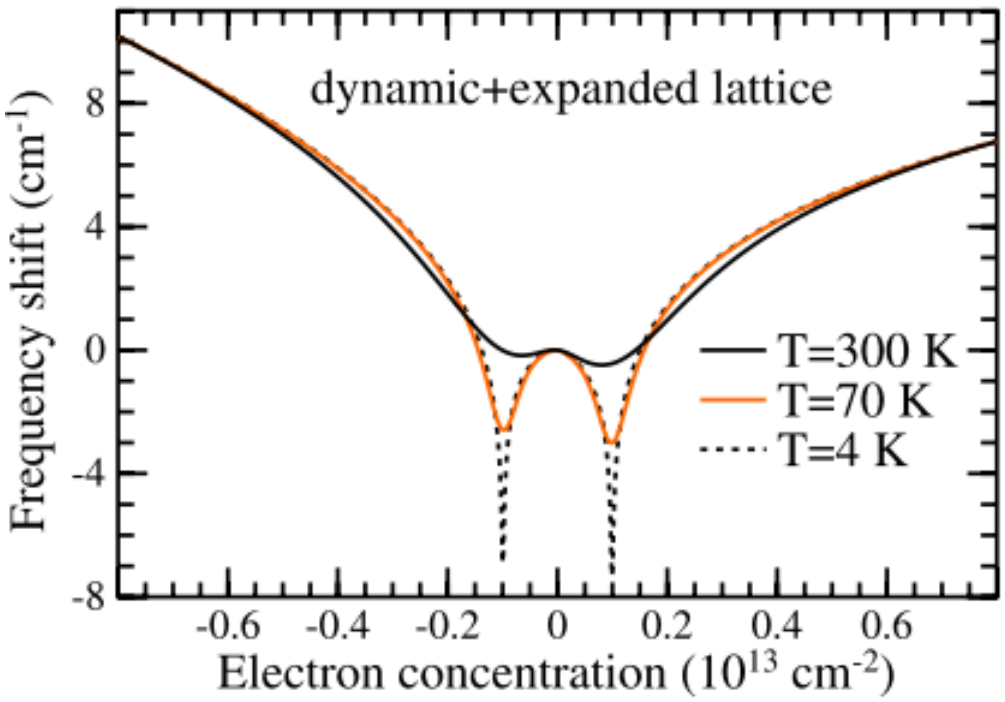


Fig4.1- POS(G) as a function of : a) applied gate voltage $\lambda = 633$,b) carrier concentration, $\lambda = 633$. c) applied gate voltage $\lambda = 488$,d) carrier concentration, $\lambda = 488$.

According to literature [8, 14], the G peak position shifts to higher energy for increasing doping, no matter the sign of the charges (see Fig. 4.2). In our doped samples, an applied positive gate voltage causes an increase in doping and a shift in G peak position to higher energy. An applied negative gate voltage results in a decrease in doping and a shift in G peak position to lower energy. Even though these results at a first glance seem contradictory to theory, they are still in agreement with [8].Since the samples are doped; the charge neutrality point is far away from zero gate voltage. That means we have to apply a very large voltage to observe the trend reported in [8]. However, at high voltage the devices become unstable producing large hysteresis or they actually break .So, because the sample is doped, the graphs don't have the expected V-shape behavior, we are only probing half of it.



4.2-Frequency shift as a function of electron concentration[14].

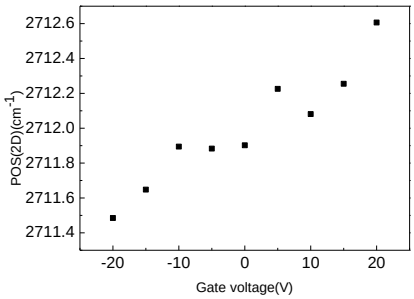


Fig 4.3- POS(2D) under apply gate voltage (-20V, +20V) in monolayer graphene,λ =488nm.

Figure 4.3 shows POS(2D)as a function of gate voltage. We do not notice any significant shifts, if we consider the resolution of our Raman setup. This is in agreement with existing literature, since our back gating approach only allows concentration slower than 10^{13}cm^{-1}. This concentration is not enough to observe changes in POS (2D).

4.2-Gated Bilayer graphene

Bilayer graphene has a tunable band gap**.** When we apply gate voltage, the Fermi level will be shifted and we can observe POS (G) changing. Our experimental results are consistent with theory. Figure 4.4 shows the Raman spectra taken at different values of Vg. We observe that both the position and the shape of the G band depend on Vg, in agreement with previous Raman studies of gated bilayer graphene [15, 1]. However, in our experiments we were not able to observe the initial softening of the G band for$|\varepsilon_F| < \hbar\omega_G/2$, as reported by Yan et al. [15]. This result can be explained by the presence of a nonhomogeneous charge distribution in our sample in the submicron range and by the fact that our experiment was done at room temperature [1].

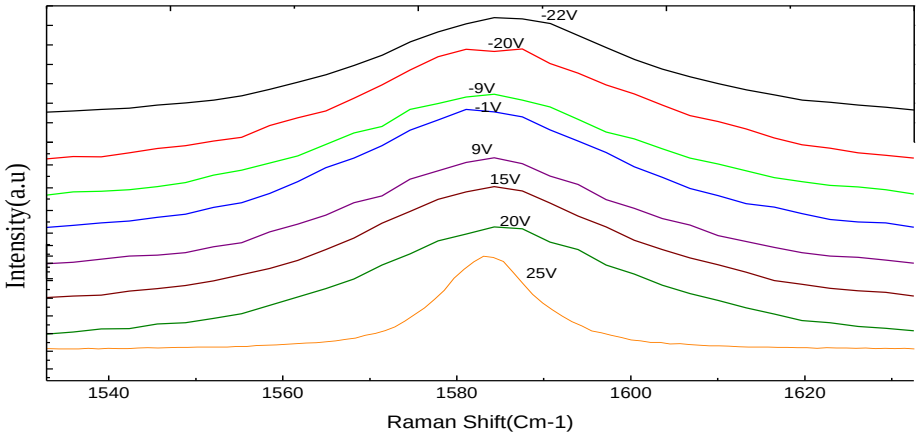
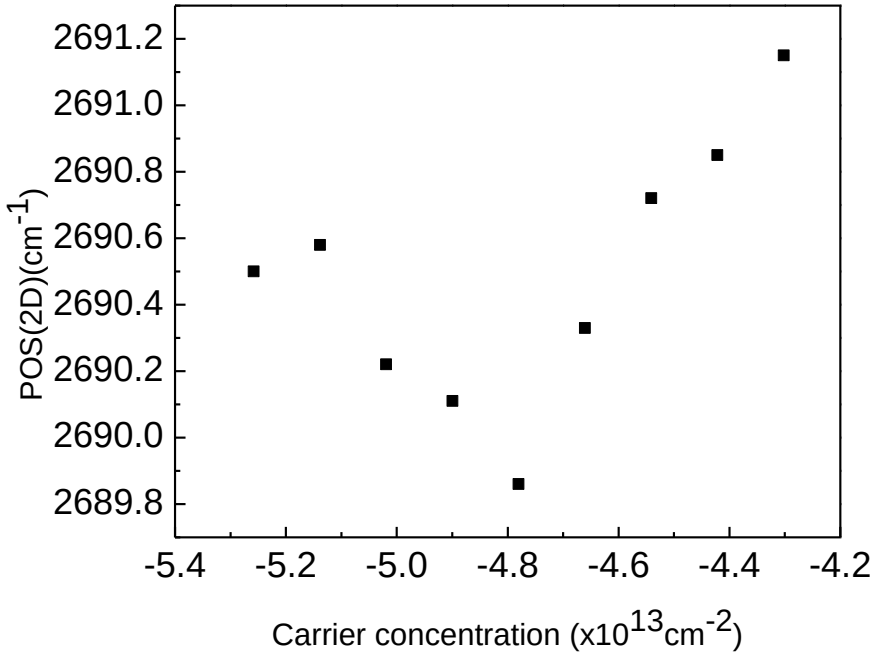


Fig4.4- G peak Raman spectra at different applied gate voltage

Figure 4.5shows the 2D peak shifts of pristine and transferred graphene as a function of carrier concentration. The G peak and 2D peak positions increase in energy for increase doping concentration (applying gate voltage), no matter the sign of the charges.

a)



b)

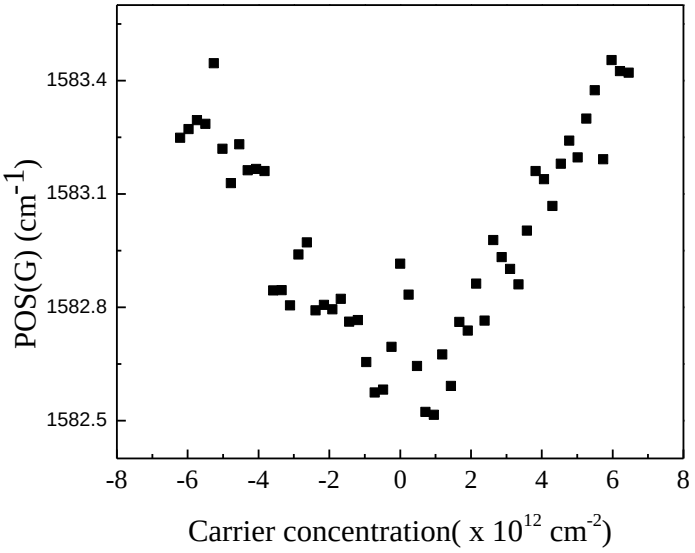


Fig4.5-POS (2D) as a function of carrier concentration: a) bilayer pristine graphene,λ =514nm, b) bilayer transferred graphene λ =514nm,(applying gate voltage (-30V, +30V) Step: 1V).

By applying positive or negative gate voltage, we will have positive or negative carrier concentration and we observe that the G peak position increase

in energy for increasing concentration, in agreement with theory[16]. Figure 4.5, shows that pristine bilayer graphene and transferred bilayer graphene have different carrier concentrations (n=C (V-Vo)), i.e. different V_0..

5-Conclusion and future work

In conclusion, we have studied the Raman spectrum of mono and bi-layer graphene as a function of gate voltage. Raman spectroscopy is particularly useful for characterizing flakes since it is a fast, non-destructive method.

By applying gate voltage to graphene and its bilayer, we shift the position of the Fermi level and tune the carrier concentration. These changes appear as shifts in the G and 2D peaks and can be used to extract the carrier concentration. Our experimental results are in agreement with Ando calculations [2].

Graphene research is still in its infancy. The work can be extended by calculating the carrier concentration on the top and bottom layer separately. The two will have different values (as expected) since the doping from the bottom layer will largely be due to the substrate, while doping for the top layer is due to expose in the atmosphere, for example, water adsorption, in addition, the experiment part can be down in different temperature especially high temperature results will be interesting. The investigation can follow by working on folded graphene of other few layer graphene.

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