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## Graphene Transfer and Characterization

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### Abstract

The aim of this project was to determine whether or not it was possible to develop a reliable method for transferring carbon vapor deposition graphene samples from nickel and copper to other substrates in order to take optical measurements on the graphene. Samples were characterized by Raman Spectroscopy, Fourier Transform Infrared Spectroscopy, and Hall effect measurements in order to determine: if the methods of transfer were successful; and to ascertain the carrier density and carrier mobility of the samples. The most successful method of transfer of samples involved pieces of graphene grown on nickel by carbon vapor deposition and used a silicon dioxide etchant and a nickel etchant before applying the graphene to other substrates. Good quality pieces of graphene with carrier densities typical of carbon deposition vapor graphene films grown on nickel were successfully transferred to other substrates. However a reliable method for the transfer of graphene films grown on copper was not completed.

### Cover Page Footnote

It was a pleasure to work on this research project and therefore I would like to thank Professor James Heyman for the opportunity and all the knowledge and experience he has imparted upon me as a result of my participation in this project. Further, generous gratitude is also expressed to the other collaborators directly relevant to the results obtained in this project: Yilikal Ayino and Minh Nguyen who helped take Hall effect measurements; and Jacob Stein who took the majority of Raman spectra.

# **Graphene Transfer and Characterization**

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**Professor: James Heyman**

Collaborators: Yilikal Ayino, Minh Nguyen, Jacob Stein

## **Abstract**

The aim of this project was to determine whether or not it was possible to develop a reliable method for transferring carbon vapor deposition graphene samples from nickel and copper to other substrates in order to take optical measurements on the graphene. Samples were characterized by Raman Spectroscopy, Fourier Transform Infrared Spectroscopy, and Hall effect measurements in order to determine: if the methods of transfer were successful; and to ascertain the carrier density and carrier mobility of the samples. The most successful method of transfer of samples involved pieces of graphene grown on nickel by carbon vapor deposition and used a silicon dioxide etchant and a nickel etchant before applying the graphene to other substrates. Good quality pieces of graphene with carrier densities typical of carbon deposition vapor graphene films grown on nickel were successfully transferred to other substrates. However a reliable method for the transfer of graphene films grown on copper was not completed.

## Introduction

Graphene is recognized as one of the strongest and thinnest materials ever. Defined as an allotrope of carbon measuring one atom thick in a two dimensional lattice, graphene has been the focus of many research pieces within the past decade. Graphene flakes were first identified in 2004, when they were stripped and split from graphite by the use of adhesive tape[1]; however, the interest in the material arose due to its peculiar properties. Many of these unique characteristics concern the charge carriers in graphene, which are known to sometimes showcase: high mobilities (on the order of  $15,000\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ ), no effective mass, and relatively large scattering times[2]. However, as a material, graphene is known to have some useful properties as well; it is semi-conductor able to withstand huge current densities and is also impervious to gases. Just as impressive is the fact that graphene is known to be contrastingly stiff and malleable. Due to the distinct combination of properties demonstrated by graphene, it is expected that the material will feature in many electrical devices, such as transistors, developed within the next twenty years.

In the Optics Laboratory at Macalester College, Saint Paul, MN Professor James Heyman utilizes time-resolved infrared spectroscopy to conduct research pertaining to electronic materials, bulk semiconductors, and quantum wells. With the current relevance of graphene and its relation to Professor Heyman's expertise, the material is a subject of a variety of analyses in his laboratory. Given the fascinating nature of graphene and the potential it holds for the advancement of electronic devices, the ultimate goal of the project was to understand the transport and scattering of energetic electrons in graphene by conducting transient photoconductivity measurements on samples of graphene.

This aspect of the project focused on preparing and characterizing samples for these ultrafast photoconductivity measurements. It was important to create a reliable method for transferring samples from the (opaque) metals on which they were grown to substrates on which optical measurements could be taken. As such, techniques were developed for transferring films grown on nickel (Ni) and copper (Cu) substrates by chemical vapor deposition (CVD) onto: silicon dioxide ( $\text{SiO}_2$ ) on silicon (Si) substrates; pure Si substrates; and sapphire ( $\text{Al}_2\text{O}_3$ ) substrates[3]. The films were then characterized using Raman Spectroscopy, which is a probe of the vibrational modes of graphene; Hall effect measurements, which sought to determine the carrier concentration and mobility of free electrons; and infrared spectroscopy, which offered a non-contact probe of the electron density and mobility.

## Methodology

The graphene samples used in the research project had previously been grown a metal by the CVD of methane. There were samples grown on Ni on top of  $\text{SiO}_2$ <sup>1</sup> on Si and samples grown on a sheet of Cu. Due to the nature of the growth of graphene on Ni and Cu, it was understood that the films on Ni were not completely monolayer graphene films while the films on Cu were. The available literature revealed that the most common methods of CVD graphene transfer were conducted by the use of: thermal release tape and a metal etchant; only a metal etchant; a  $\text{SiO}_2$  etchant and a metal etchant; or by the use of a thermoplastic and a metal etchant[3][4][5].

The graphene samples were purchased from Graphene Supermarket. The first task of the research was to transfer graphene from the Gr/Ni/ $\text{SiO}_2$  samples to other substrates (such as:

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<sup>1</sup> Further referred to as Gr/Ni/ $\text{SiO}_2$  /Si

SiO<sub>2</sub>/Si, Si, gold (Au) gated SiO<sub>2</sub>/Si, Al<sub>2</sub>O<sub>3</sub>) while the second duty of the research was to transfer graphene from Cu sheets to the relevant substrates. Roughly seven different methods of transfer were attempted with the success of each being summarized in Table 1. Below is an elaboration on what was found to be the most successful method graphene of transfer.

### **Most Successful Method of Transfer**

This method of graphene transfer utilized buffered oxide etchant (BOE) to remove the SiO<sub>2</sub>, ferric chloride (FeCl<sub>3</sub>) to etch the Ni layer, and deionized water to clean the sample. First, the Gr/Ni/SiO<sub>2</sub>/Si was left in BOE while the SiO<sub>2</sub> etched. As this completed the Gr/Ni would float at the top of the BOE while the Si would be fully submerged and eventually sink to the bottom of the BOE when the process was complete. This process generally took less than an hour to complete. Next the floating Gr/Ni would be removed from the BOE and placed in an aqueous solution of FeCl<sub>3</sub>. This Ni etch was an extremely quick process and usually took less than 10 minutes. Thus, only a 1cm x 1cm piece of graphene was left floating on top the FeCl<sub>3</sub> as shown in Figure 1. Next, the graphene would be removed from the FeCl<sub>3</sub> and washed by being placed in a beaker of deionized water before being scooped up on a substrate and left to air dry. Please see Figure 7.

### **Methods of Characterization**

After transfer, various methods of characterization were performed on each sample. Raman Spectroscopy is a technique commonly used to measure the vibrational frequencies of carbon atoms. Raman Spectroscopy is performed by hitting a sample with light of a certain frequency (green light) and measuring the shift in the frequency of the light emitted from the

sample. Raman Spectroscopy is immensely useful in characterizing graphene samples because the vibrational transitions of graphene are not infrared allowed. Hence, Raman Spectroscopy was performed on the samples created in order to determine whether or not each method resulted in the successful transfer of graphene.

The remaining results pertain to the transfer of graphene from the Gr/Ni/SiO<sub>2</sub>/Si samples as the method of transfer of graphene from the sheet of Cu by the use of the thermoplastic did not feature in any of the other types of analyses as the transfers involving the thermoplastic were conducted very close to the end of the research period.

FTIR readings provide the transmission of spectra of a sample measured as a percentage of the transmission spectra of a control for light at varying infrared frequencies. Hence, measurements were conducted on clean substrates (as controls) and the created samples in order to produce transmission spectra for the samples. Readings were taken in both the near-infrared and far-infrared and combined to produce graphs such as Figure 10 on which a Drude conductivity approximation could be made in order to find the carrier densities and carrier mobilities of samples.

By putting samples with In contacts in a magnetic field, an electric current was produced perpendicular to the magnetic field creating a voltage difference known as the Hall Voltage. LabView programs were utilized to measure and record the Hall Voltage before using it to calculate the carrier mobilities and concentrations of the samples.

## **Results and Discussion**

### **Results of Raman Spectroscopy**

The results of the Raman Spectroscopy indicate that there was indeed successful graphene transfer for at least one of the samples from each of the methods of graphene transfer reported in Table 1, and for the vast majority of samples on which Raman Spectroscopy was conducted. Figure 9 shows the Raman spectrum for sample 0728E (Graphene/SiO<sub>2</sub>/Si), the figure has three prominent features: a D peak at approximately 1400cm<sup>-1</sup>, a G peak at approximately 1600cm<sup>-1</sup>, and a 2D peak approximately 2700cm<sup>-1</sup>. The G and 2D peaks of the Raman spectrum are expected characteristic features of graphene samples, the relative heights of which are known to be related to the carrier concentration of the sample[6]. The D peak is recognized as a sign of disorder in a graphene sample; as such, it is not present in the Raman shifts of perfect graphene structures, and is known to only appear at grain boundaries, edges, and defects. Nevertheless, the presence of the G and 2D peaks confirmed the successful transfer of graphene from the initial substrate to SiO<sub>2</sub>/Si.

## Results of FTIR

The Drude theory of conductivity, shown in equation (1), predicts current as a response to a time-dependent electric field and was used to model the results obtained from the FTIR readings by giving the direct current conductivity as a function of the scattering time.

$$\sigma = \frac{\sigma_0}{1 - i\omega\tau} \quad (1)$$

Where  $\sigma_0$  is the (initial) alternating current conductivity,  $\omega$  is the angular frequency, and  $\tau$  is the scattering time. It was known that the transmission amplitude  $t$  obtained by the FTIR is a function of the angular frequency such that:

$$t(w) = \frac{n_s + 1}{(n_s + 1) + \mu_0 c \sigma} \quad (2)$$

Where the constant  $n_s$  is the index of the sample substrate (which for Si, one of the most commonly used substrates was taken to be 3.5) and the value of  $\mu_0 c$  was known to be  $120\pi$ .

However equation (2) could be rewritten as:

$$t = \frac{1}{1 + \alpha \sigma} \quad (3)$$

Where  $\alpha = \mu_0 c / (n_s + 1)$ . Knowing that the power transmission  $T$  is a function of  $t$ :

$$T = |t(\omega)|^2 \quad (4)$$

And applying a bit of algebra to equation (2):

$$\frac{1}{T} = (1 + \alpha \sigma)(1 + \alpha \sigma^*) \quad (5)$$

$$\frac{1}{T} = 1 + \frac{2 \alpha \sigma_0}{1 + \omega^2 \tau^2} + \frac{2 \alpha^2 \sigma_0^2}{1 + \omega^2 \tau^2} \quad (6)$$

However, for a small transmission charge,  $\alpha \sigma_0 \ll 1$  and

$$\frac{1}{T} = 1 - dT \quad (7)$$

So that the scattering time can be calculated from the transmission change by:

$$dT \approx - \frac{2 \alpha \sigma_0}{1 + \omega^2 \tau^2} \quad (8)$$

With the scattering time, it is possible to apply the following equations in order to determine the carrier density and carrier mobility of the samples of graphene, where the fermi-velocity of graphene,  $v_f = 1.09 \times 10^6 \text{ms}^{-1}$ :

$$n = \left( \frac{\sigma h}{2e^2 v_f \langle \tau \rangle} \right)^2 \frac{1}{\pi} \quad (9)$$

$$\mu = \left( \frac{2e v_f}{h} \right) \sqrt{\frac{\pi}{n}} \langle \tau \rangle \quad (10)$$

The FTIR results are presented in Table 2 and show that the values for carrier mobilities ranged from approximately  $280\text{cm}^2\text{V}^{-1}\text{s}^{-1}$  to  $1820\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ . Additionally, the FTIR results place the range of carrier densities to stretch from  $2.58 \times 10^{12}$  charge carriers per  $\text{cm}^2$  to  $5.0 \times 10^{13}$  charge carriers per  $\text{cm}^2$ . A graph of the FTIR results is presented in Figure 11.

### Results of Hall Effect Measurements

The results of the Hall effect measurements show lower mobilities for each sample, ranging from  $142\text{cm}^2\text{V}^{-1}\text{s}^{-1}$  to  $223\text{cm}^2\text{V}^{-1}\text{s}^{-1}$ , and a mixture of carrier concentrations, ranging from  $2.43 \times 10^{13}$  charge carriers per  $\text{cm}^2$  to  $9.97 \times 10^{15}$  charge carriers per  $\text{cm}^2$ . The Hall effect results are presented in Table 3, with a comparison of FTIR and Hall effect readings for particular samples presented in Table 4 and graphed in Figure 13. Hall effect readings were only successfully conducted on a few samples as some had been contaminated or continuous films were too small for In contacts to be attached.

### Discussion

From the results described above it is important to appreciate a number of successes. First, the techniques developed by this research allowed for the reliable transfer of  $1\text{cm} \times 1\text{cm}$  CVD graphene pieces grown on Ni to various substrates. Next, the carrier densities recorded by both FTIR and Hall effect measurements were on the order of  $10^{12}\text{cm}^{-1}$  and  $10^{13}\text{cm}^{-1}$ , typical of CVD graphene films grown on Ni. Furthermore, the measurements from Raman Spectroscopy indicated good quality graphene films as they displayed narrow line widths and relatively small D peaks, while the ratios of the G/2D peak reported high carrier densities consistent with the other measurements. Additionally, the FTIR results displayed an inverse correlation between

carrier density and carrier mobility (see Figure 12 for the samples of graphene on Si) that has also been observed by other groups.

There were also two phenomena in the results which are worth mentioning along with possible explanations for them. Firstly, (see Figure 12) the samples of graphene transferred to SiO<sub>2</sub> on Si substrates showed higher carrier concentrations and lower carrier mobilities than those transferred onto pure Si substrates (with samples transferred onto Al<sub>2</sub>O<sub>3</sub> registering between the two). It is believed that this is due to polar atoms on the surface of the graphene acting as donors, acceptors, and scattering centers; therefore graphene films on non-polar substrates such as pure Si would show lower carrier densities and higher carrier mobilities. Third, the results from the noncontact FTIR measurements consistently displayed higher carrier mobilities than those retrieved by the Hall effect measurements. It seems that this discrepancy can be explained by large scale cracks and defects in film of graphene which would influence the Hall effect measurements (which need continuous layers of material to work perfectly) but not the FTIR results which just produce transmission spectra. Another possible cause for this discrepancy could have been air exposure as the Hall Effect readings were taken a few weeks after the FTIR measurements.

In the related project, some of these transferred graphene films were successfully analyzed by transient photoconductivity measurements and showed positive photoconductivity with a photoconductivity lifetime of  $\approx 3ps$  favorable to the development of ultrafast transistors and electro-optic devices. These results are similar to those reported by other groups in exfoliated graphene and the results previously determined in this laboratory on epitaxial graphene  $\approx 5ps$ .

## Summary and Conclusion

In conclusion, reliable techniques for the transfer of 1cm x 1cm CVD graphene pieces grown on Ni were developed. Raman Spectroscopy indicated that the pieces transferred were of good quality and high carrier densities consistent with the other measurements. Moreover FTIR and Hall effect measurements confirmed that the samples transferred displayed carrier densities within the range of  $10^{12}\text{cm}^{-1}$  and  $10^{13}\text{cm}^{-1}$  which are typical of CVD graphene films grown on Ni. Additionally, the FTIR results displayed an inverse correlation between carrier density and carrier mobility (see Figure 12 for the samples of graphene on Si) that has also been observed by other groups. However, due to time constraints, this project did not develop a reliable method for the transfer of graphene films grown on Cu. Although, this may be an interesting direction in which to proceed in terms of research it is not of current significance to this project as samples which can be characterized by optical methods are currently being bought from a supplier. Nevertheless, some of the samples produced in this research we characterized by photoconductivity measurements and showed positive photoconductivity with a photoconductivity lifetime of  $\approx 3\text{ps}$

## Acknowledgements

It was a pleasure to work on this research project and therefore I would like to thank Professor James Heyman for the opportunity and all the knowledge and experience he has imparted upon me as a result of my participation in this project. Further, generous gratitude is also expressed to the other collaborators directly relevant to the results obtained in this project:

Yilikal Ayino and Minh Nguyen who helped take Hall effect measurements; and Jacob Stein who took the majority of Raman spectra.

## References

1. Geim, A.K. Novoselov, K.S. Nature Materials 6 (2007)
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3. Kim, K.S. Nature 457 (2009)
4. Li, X. et al. Nano Letters 12 (2009)
5. Reagan, W. Applied Physics Letters 96 (2010)
6. Cadwell, J.D. et al. ACS (2010)

**Table 1**

Brief Description of Method	Results
<ul style="list-style-type: none"> <li>• Thermal Release Tape was applied to Gr/Ni/SiO<sub>2</sub>/Si sample.</li> <li>• Ni layer of sample was etched in FeCl<sub>3</sub>.</li> <li>• Tape and graphene were: washed; left to dry; and placed on a SiO<sub>2</sub>/Si substrate.</li> <li>• The materials were clamped together.</li> <li>• The sample was heated to remove the tape.</li> <li>• Organic solvents were applied to remove left over tape. (Figure 2)</li> </ul>	<ul style="list-style-type: none"> <li>• Raman Spectroscopy identified sparse graphene transfer.</li> <li>• From inspection (under a microscope) there seemed to be impurities on the sample.</li> </ul>
<ul style="list-style-type: none"> <li>• The Gr/Ni/SiO<sub>2</sub>/Si was left to float on an aqueous solution of FeCl<sub>3</sub>.</li> <li>• The graphene was removed on a substrate.</li> <li>• The graphene was washed by being dipped in a beaker of deionized water and removed on a substrate. (Figure 3)</li> </ul>	<ul style="list-style-type: none"> <li>• Raman Spectroscopy confirmed graphene transfer.</li> <li>• Graphene samples tended to break into pieces during washing.</li> </ul>
<ul style="list-style-type: none"> <li>• The Gr/Ni/SiO<sub>2</sub>/Si was left to float on an aqueous solution of FeCl<sub>3</sub>.</li> <li>• The graphene was washed by using syringes to dilute the FeCl<sub>3</sub> before being removed on a substrate. (Figure 4)</li> </ul>	<ul style="list-style-type: none"> <li>• Raman Spectroscopy confirmed graphene transfer.</li> <li>• Graphene sample did not break into as many pieces as in the previous method during washing.</li> <li>• Sample showed signs of FeCl<sub>3</sub> impurities.</li> </ul>
<ul style="list-style-type: none"> <li>• The Gr/Ni/SiO<sub>2</sub>/Si was left to float on an aqueous solution of FeCl<sub>3</sub>.</li> <li>• The graphene was removed on a substrate.</li> <li>• The graphene was washed by being dipped in a beaker of deionized water with a few drops of surfactant and removed on a substrate. (Figure 5)</li> </ul>	<ul style="list-style-type: none"> <li>• Raman Spectroscopy confirmed graphene transfer</li> <li>• Excellent coverage of graphene achieved.</li> <li>• Some pieces had a few ruptures or fractures.</li> </ul>
<ul style="list-style-type: none"> <li>• SiO<sub>2</sub> layer of Gr/Ni/SiO<sub>2</sub>/Si was etched in BOE.</li> <li>• The Gr/Ni put to float on solution of FeCl<sub>3</sub>.</li> <li>• The graphene was removed on a substrate.</li> <li>• The graphene was washed by being dipped in a beaker of deionized water with a few drops of surfactant and removed on a substrate. (Figure 6)</li> </ul>	<ul style="list-style-type: none"> <li>• Raman Spectroscopy confirmed graphene transfer.</li> <li>• Excellent coverage of graphene achieved.</li> <li>• Some pieces had a few ruptures or fractures.</li> <li>• This was quicker than the previous method.</li> </ul>

Described in the methodology. (Figure 7)	<ul style="list-style-type: none"> <li>• Raman Spectroscopy confirmed graphene transfer.</li> <li>• Excellent coverage of graphene achieved.</li> <li>• Some pieces had a few ruptures or fractures.</li> <li>• As quick as the previous method and reduced the likelihood of introducing impurities from the surfactant.</li> </ul>
<ul style="list-style-type: none"> <li>• Graphene on Cu foil sample was spin coated with poly(methyl 2-methylpropanoate) (PMMA) in electronic spinner.</li> <li>• Sample was baked at approximately 180°C.</li> <li>• Sample was allowed to float on top of an aqueous solution of FeCl<sub>3</sub>.</li> <li>• The sample was washed and removed on a substrate.</li> <li>• Finally, the PMMA was removed from the sample with an organic solvent. (Figure 8)</li> </ul>	<ul style="list-style-type: none"> <li>• Raman Spectroscopy confirmed graphene transfer.</li> <li>• Some areas of good coverage of graphene achieved.</li> <li>• Poly-carbon may not have been completely removed by organic solvent.</li> </ul>

Table detailing various methods of graphene transfer.

**Table 2**

<b>Sample</b>	<b>FTIR Carrier Concentration (cm<sup>-2</sup>)</b>	<b>FTIR Mobility (cm<sup>2</sup>V<sup>-1</sup>s<sup>-1</sup>)</b>
0616A	1.0252E+13	602.693321
0616B	1.4307E+13	385.788464
0616C	1.2489E+13	619.111743
0726C	5.0006E+12	849.076728
0826A	1.354E+13	280.575766
0826B	1.0551E+13	330.947936
0826C	2.8231E+12	1820.63836
0916 .	2.5801E+12	1498.38272
0826C	6.497E+13	148.973355
JR12-02	4.7271E+12	1410.72803
JR12-03	7.7455E+12	1387.80856
JR12-04	4.4036E+12	1429.77763

Table showing the FTIR results obtained from different samples.

**Table 3**

<b>Sample</b>	<b>Hall Effect Carrier Concentration (<math>\text{cm}^{-2}</math>)</b>	<b>Hall Effect Mobility (<math>\text{cm}^2\text{V}^{-1}\text{s}^{-1}</math>)</b>
0616B	9.97E+15	142
0726C	2.43E+13	223
0826B	3.15E+13	169
0826C	2.78E+13	190

Table showing the Hall effect results obtained from different samples.

**Table 4**

<b>Sample</b>	<b>Hall Effect Carrier Concentration (cm<sup>-2</sup>)</b>	<b>Hall Effect Mobility (cm<sup>2</sup>V<sup>-1</sup>s<sup>-1</sup>)</b>	<b>FTIR Carrier Concentration (cm<sup>-2</sup>)</b>	<b>FTIR Mobility (cm<sup>2</sup>V<sup>-1</sup>s<sup>-1</sup>)</b>
0616B	9.97E+15	142	1.43E+13	386
0726C	2.43E+13	223	5.00E+13	849
0826B	3.15E+13	169	1.06E+13	331
0826C	2.78E+13	190	2.82E+12	1821

Comparison of FTIR and Hall effect readings.

## Figure Captions

Figure 1. A 1cm x 1cm piece of graphene floating in  $\text{FeCl}_3$  after the Ni layer has been etched

Figure 2. Sample 0607, created by the first method of transfer.

Figure 3. Sample 0610B, created by the second method of transfer.

Figure 4. Sample 0614A, created by the third method of transfer.

Figure 5. Sample 0621, created by the fourth method of transfer.

Figure 6. Sample 0720B, created by the fifth method of transfer.

Figure 7. Sample 0728E, created by the sixth method of transfer.

Figure 8. Sample 1102, created by the seventh method of transfer.

Figure 9. Raman spectrum of a Graphene/ $\text{SiO}_2$ /Si sample created by the sixth method of transfer.

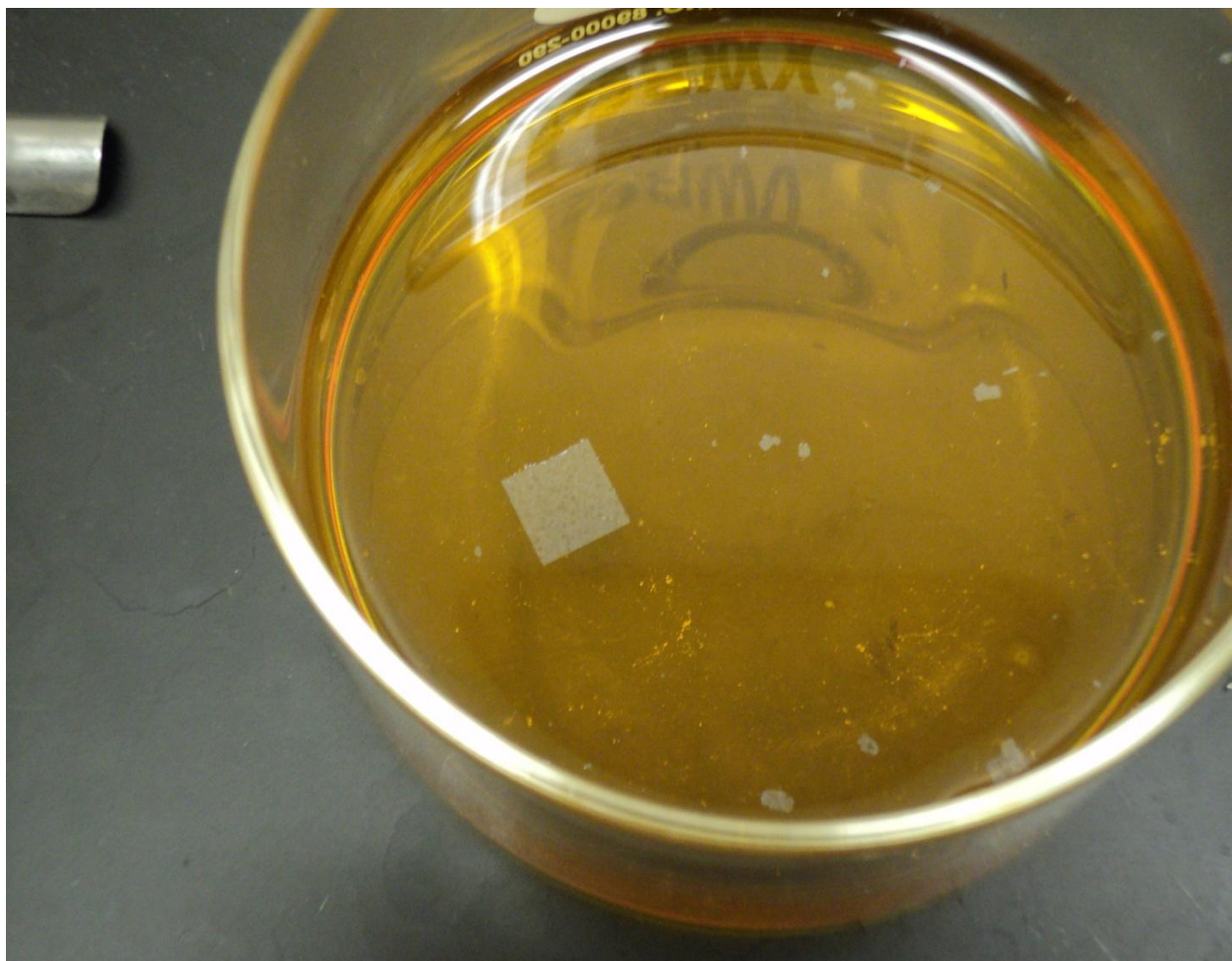
Figure 10. FTIR measurements with a Drude conductivity approximation.

Figure 11. Drude Fit to FTIR Data

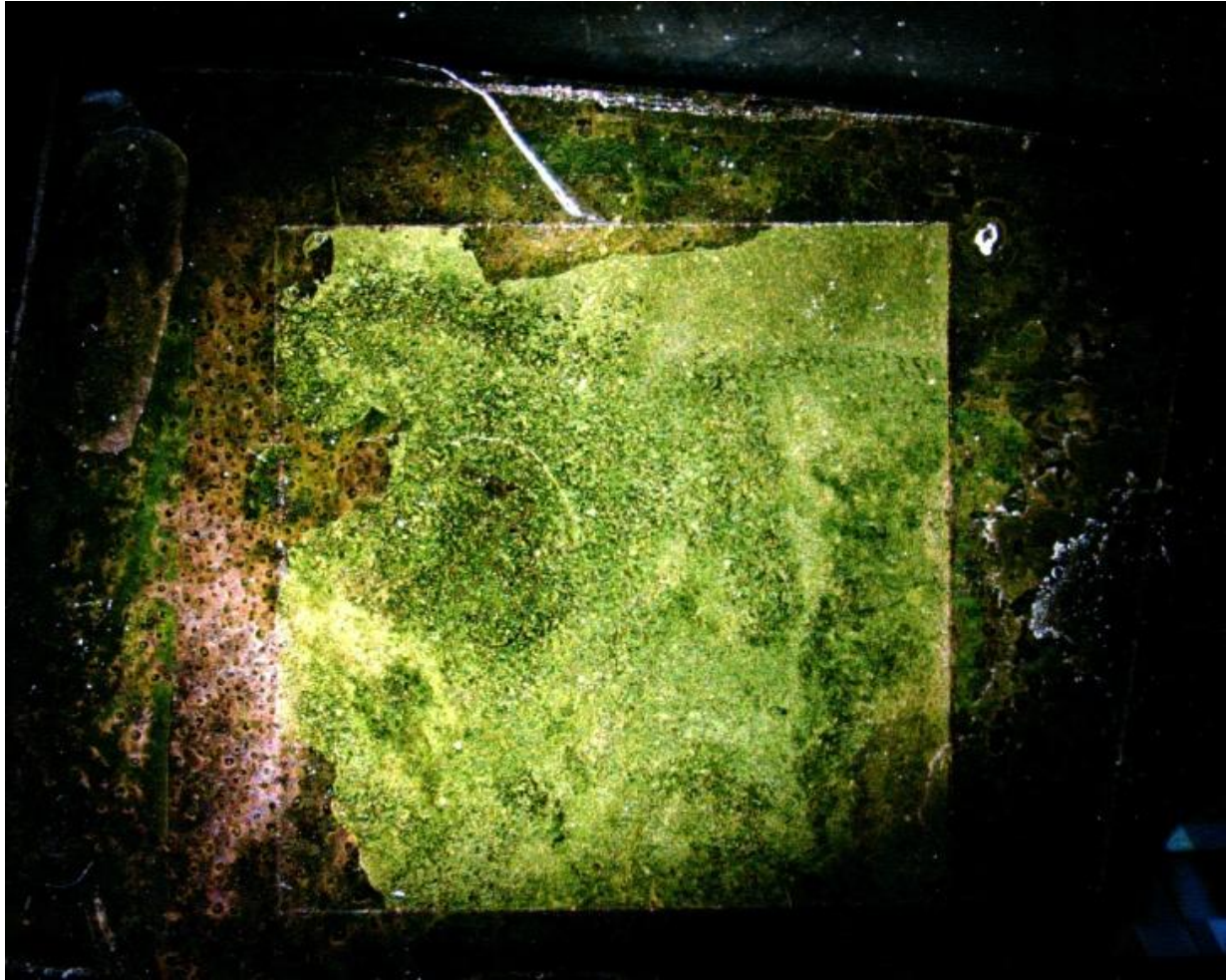
Figure 12. FTIR results showing Graphene on different substrates.

Figure 13. Comparison of FTIR and Hall effect Results.

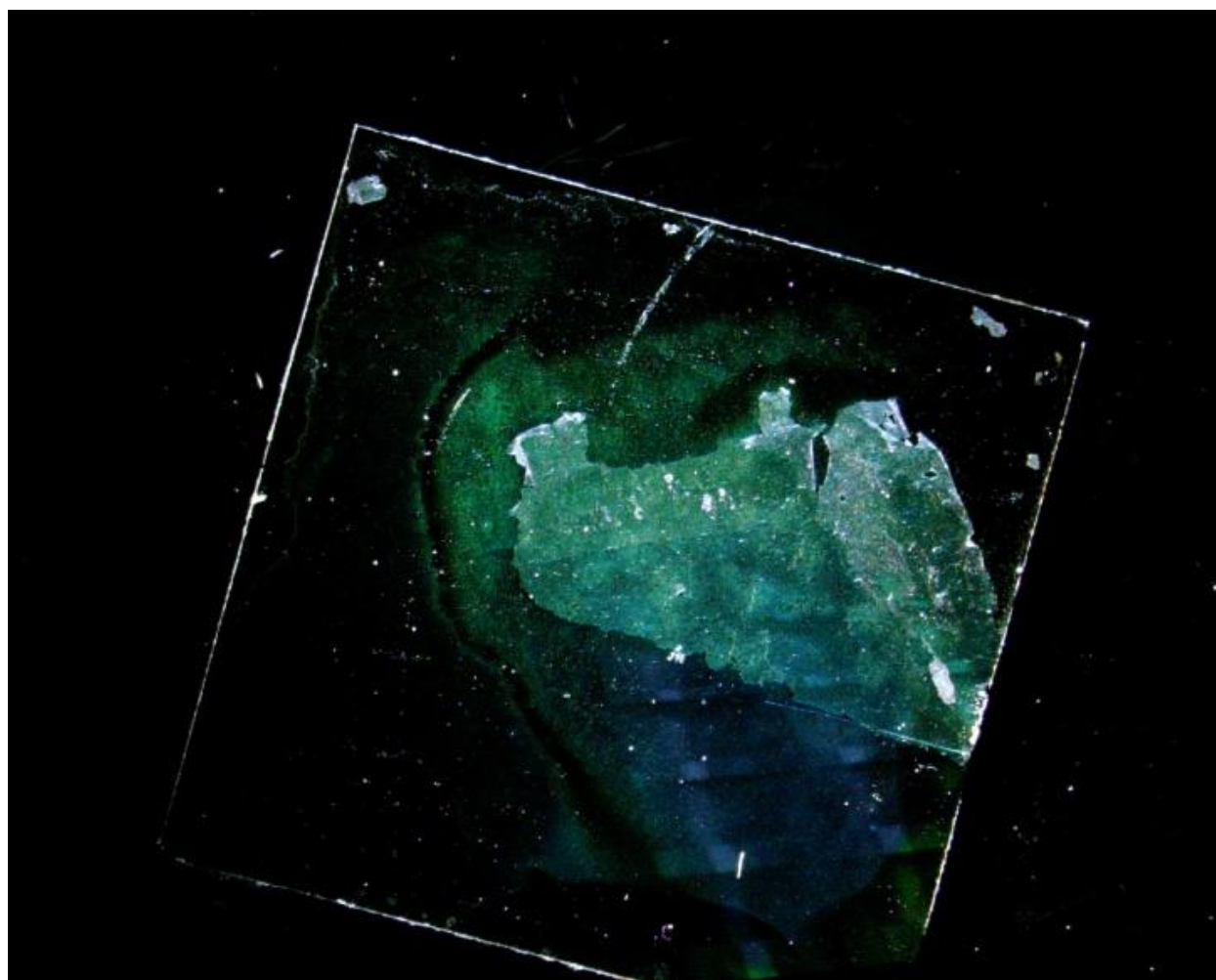
**Figure 1**



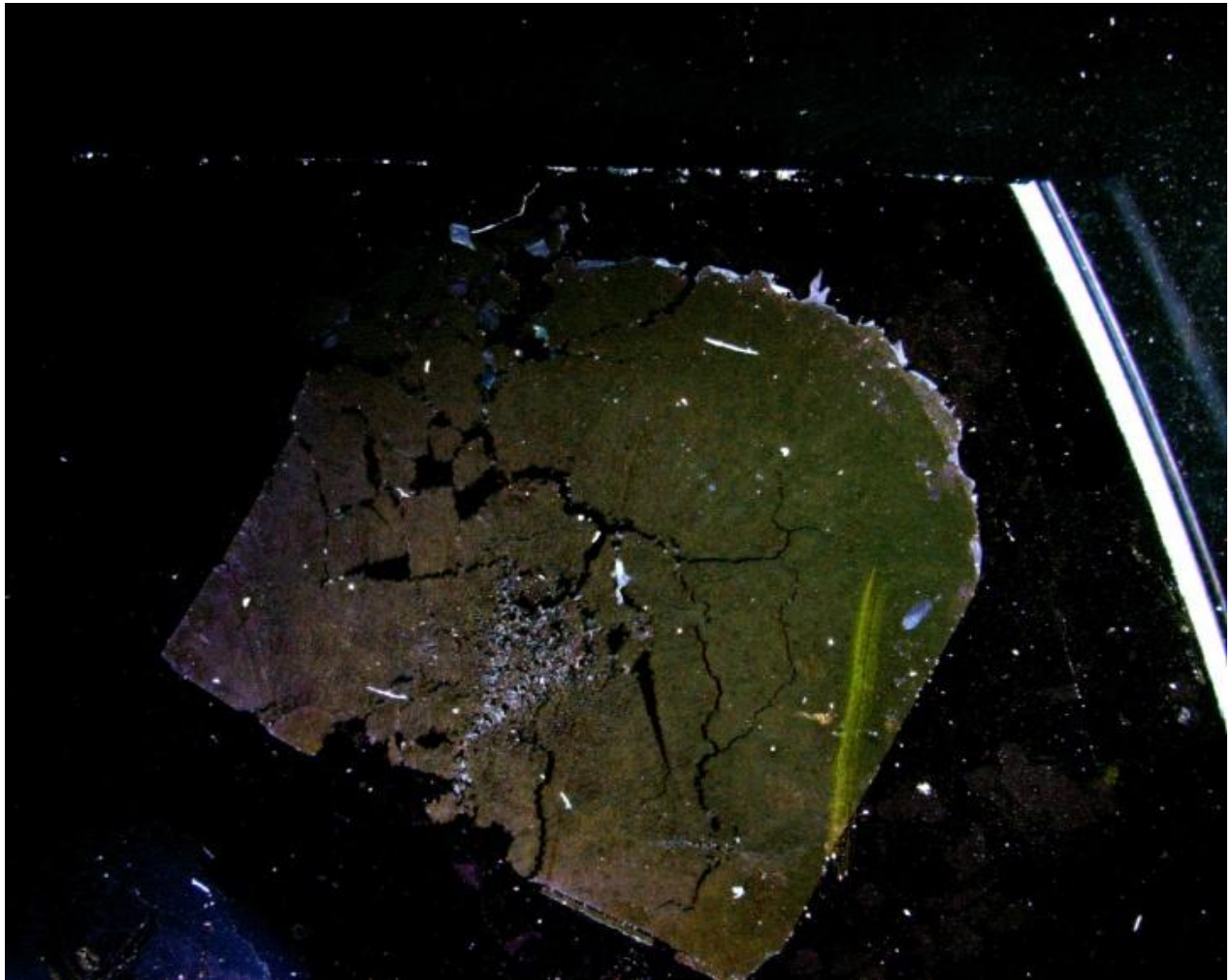
**Figure 2**



**Figure 3**



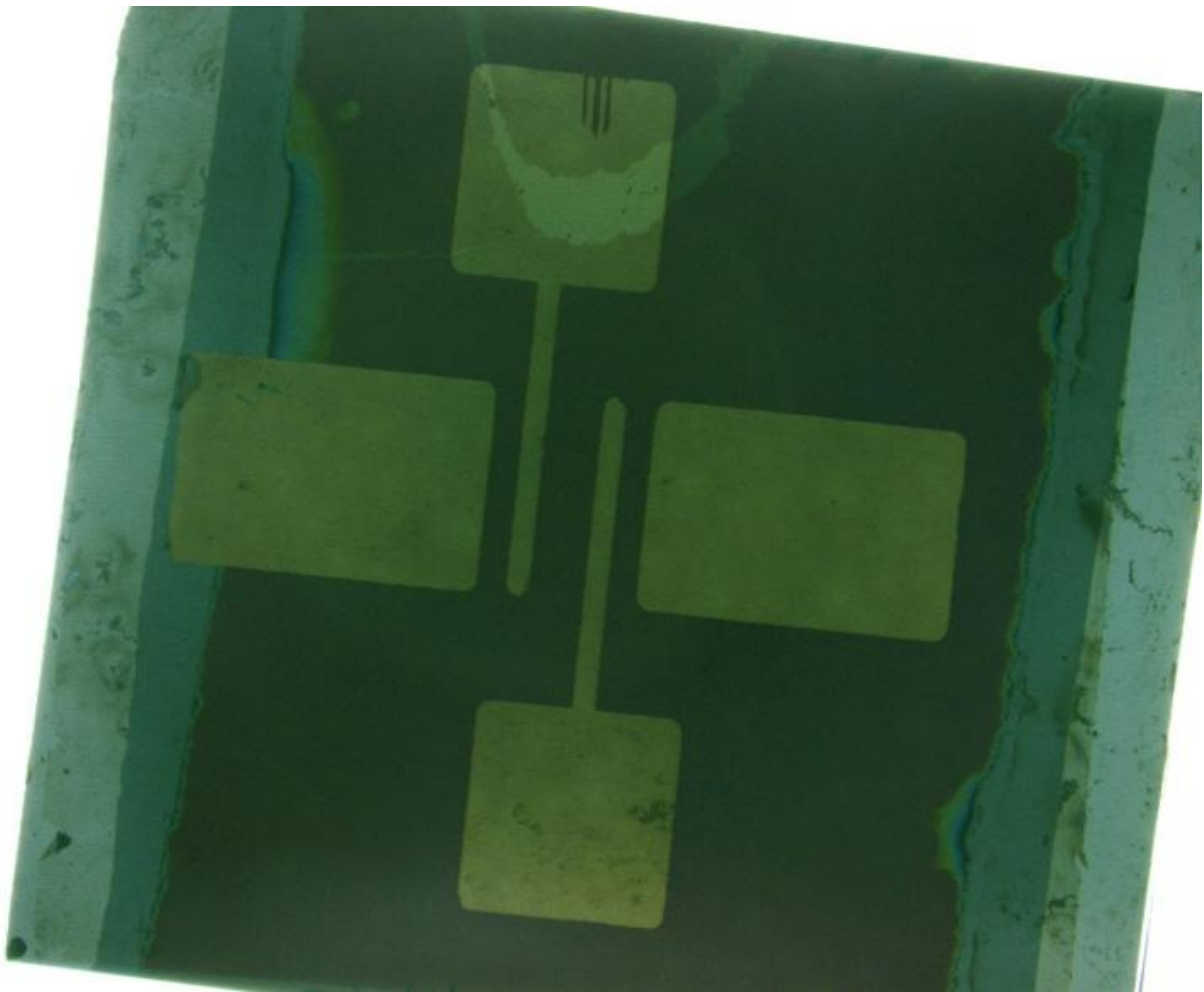
**Figure 4**



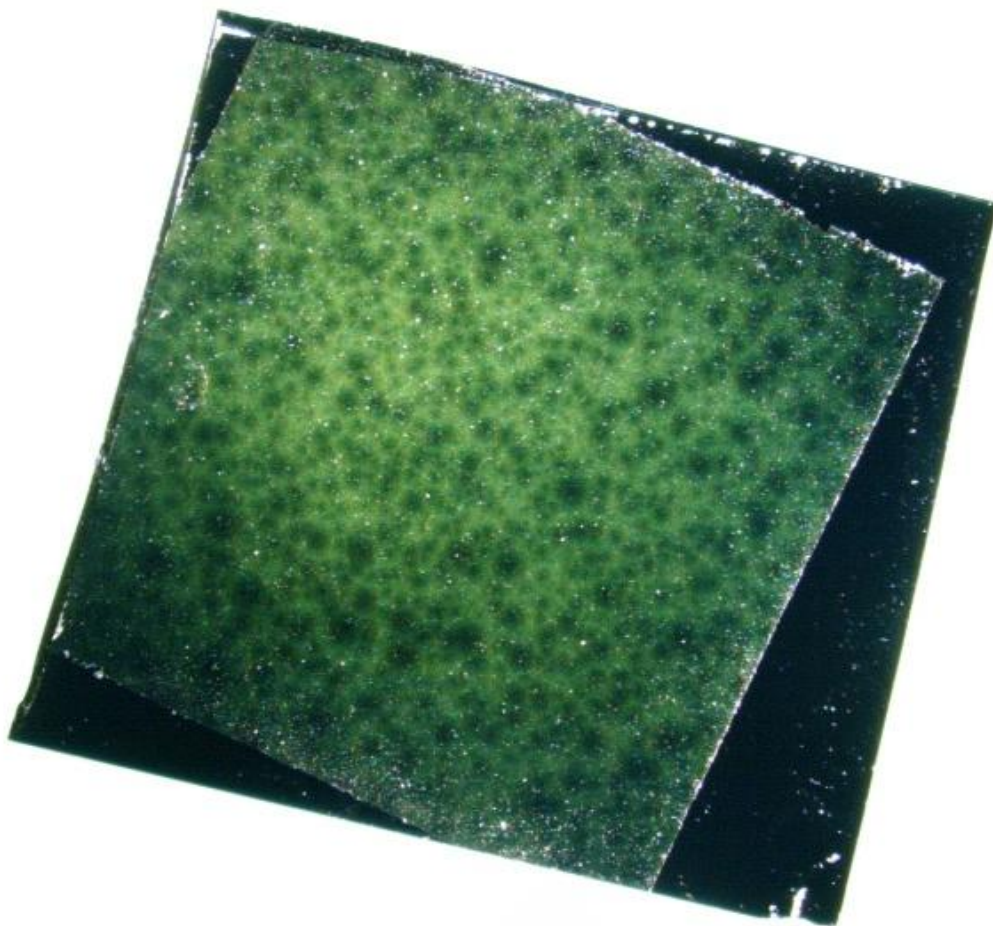
**Figure 5**



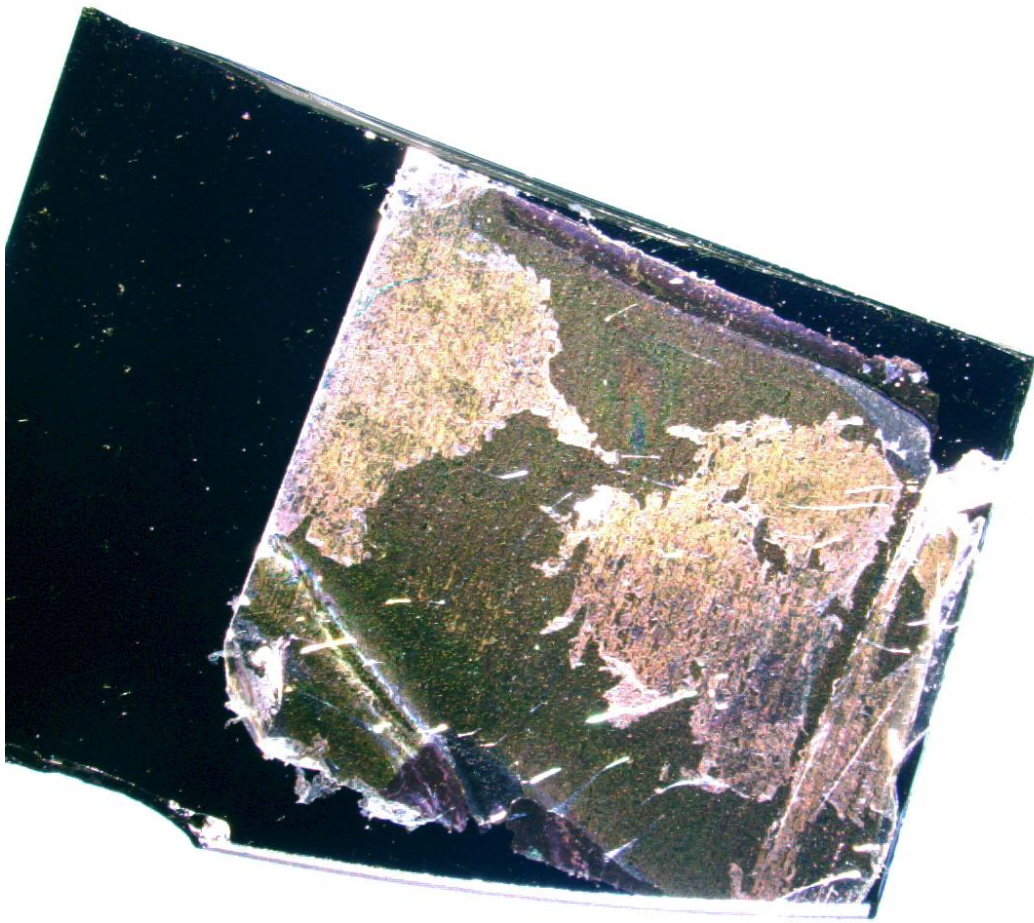
**Figure 6**

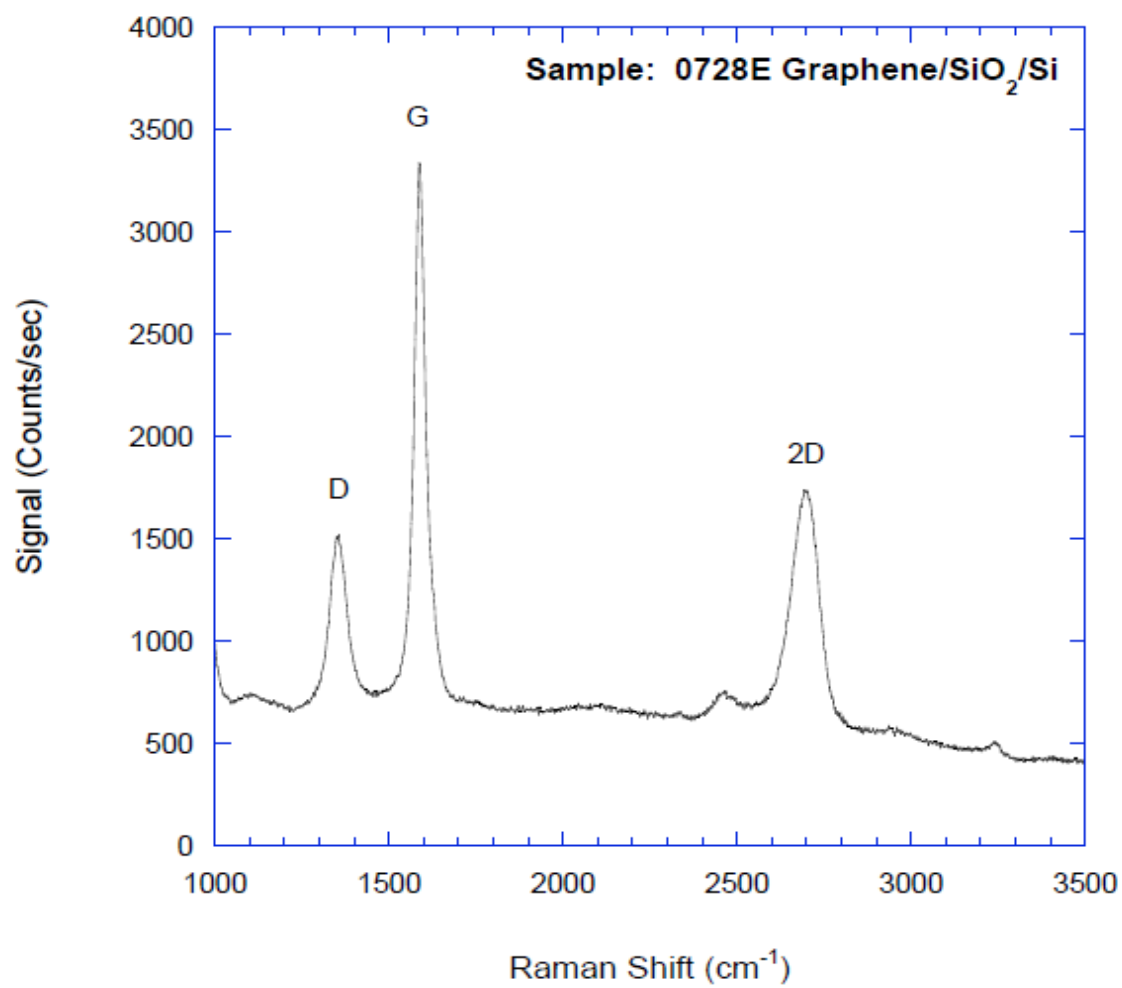


**Figure 7**

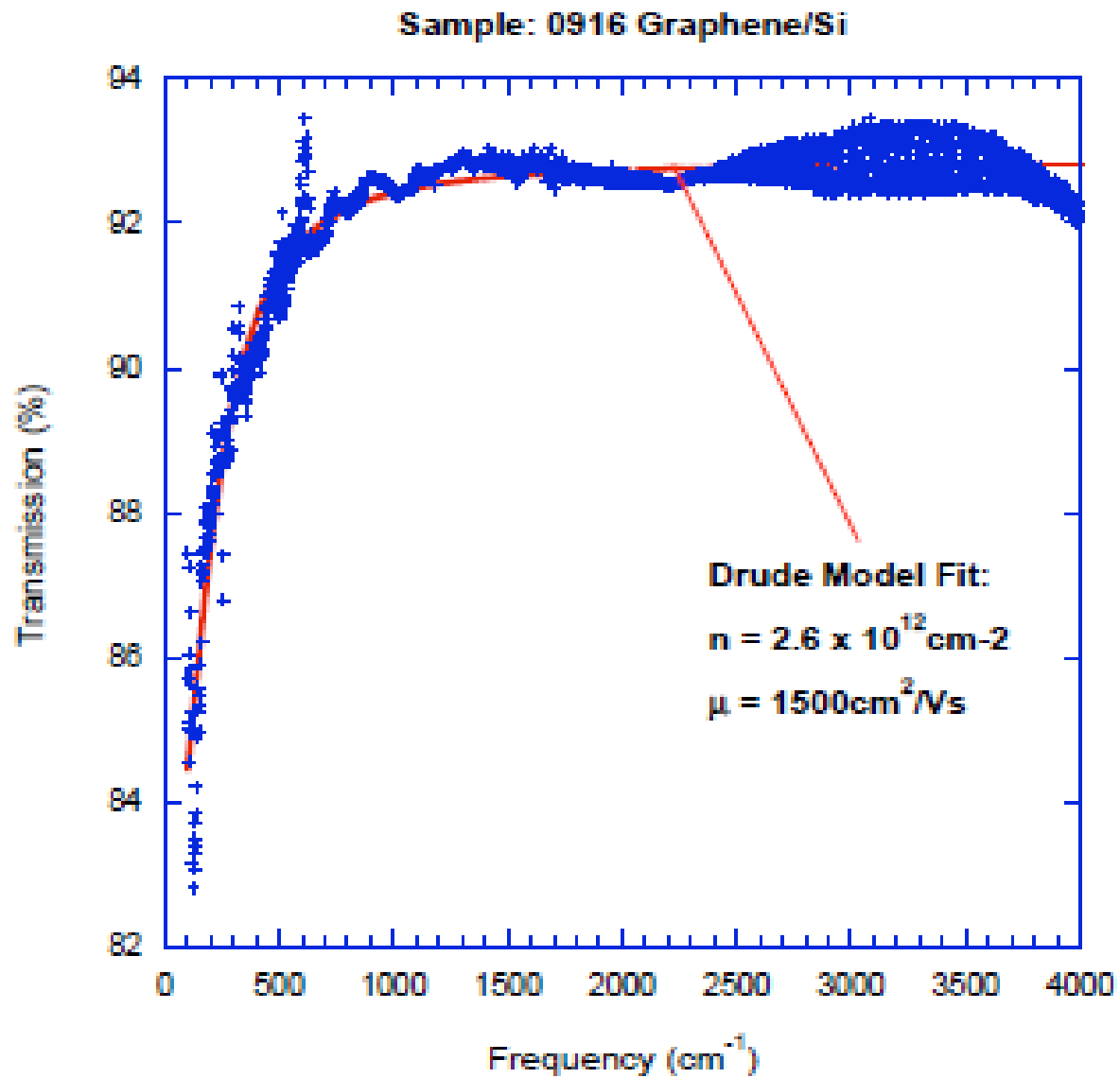


**Figure 8**



**Figure 9**

**Figure 10**



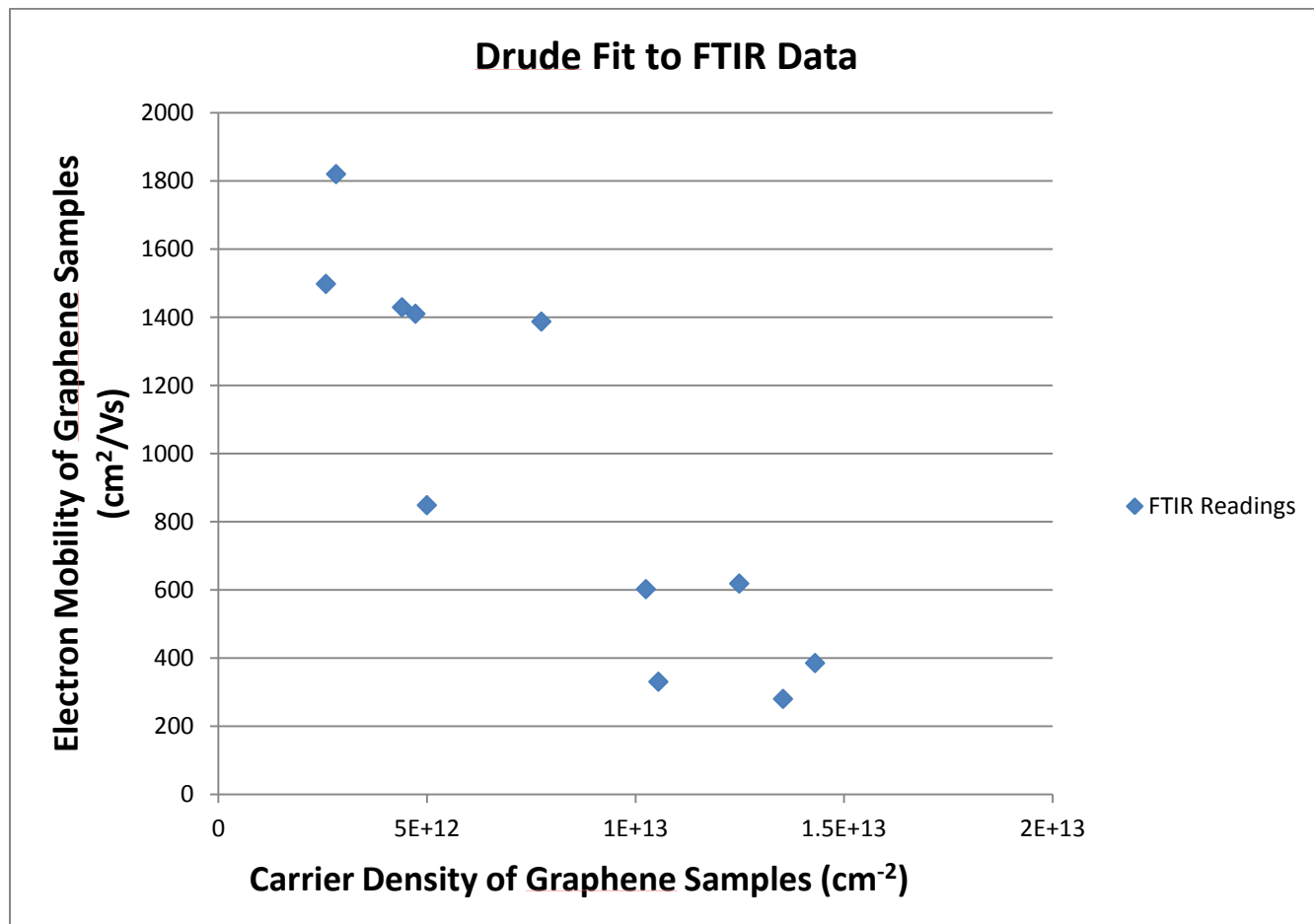
**Figure 11**

Figure 12

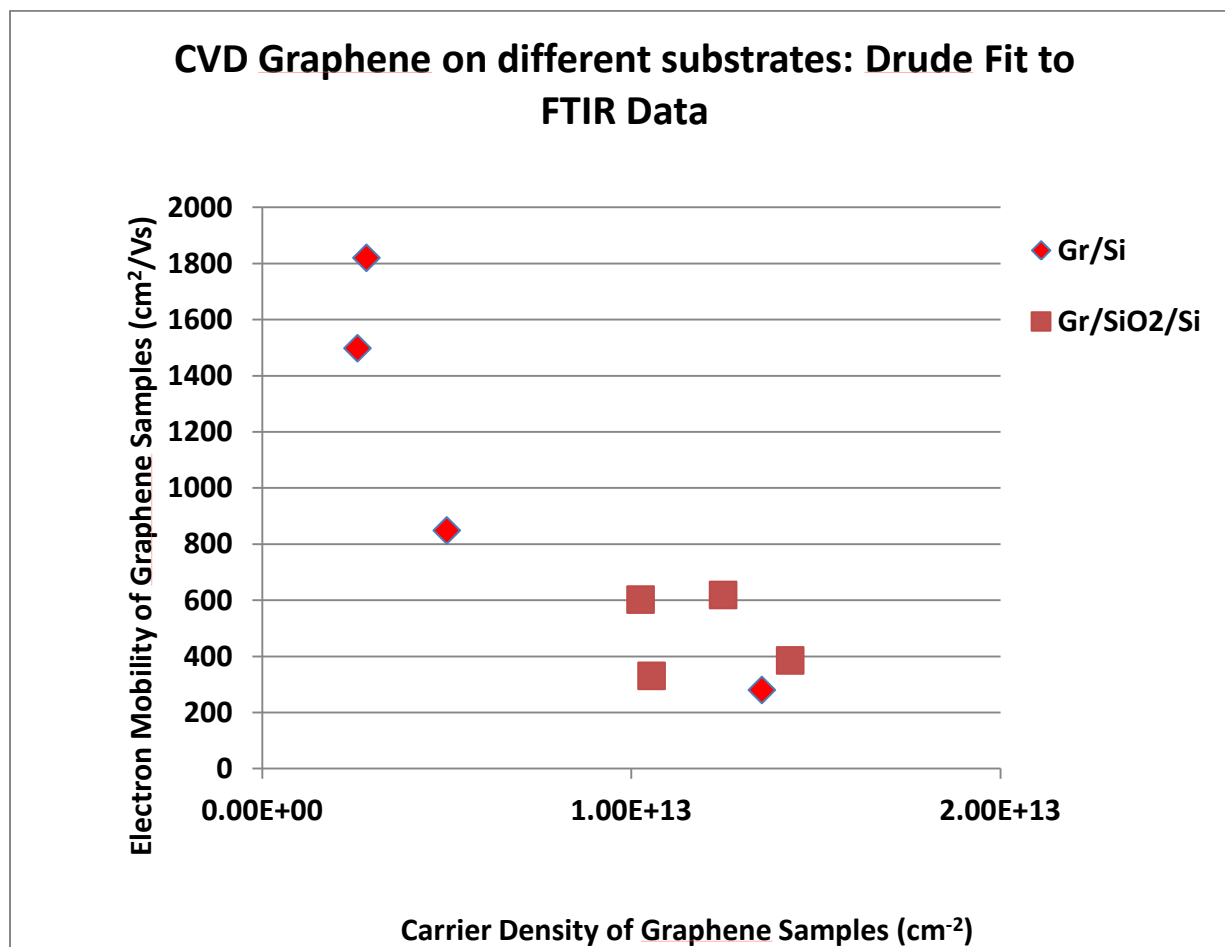


Figure 13

