National Aeronautics and Space Administration

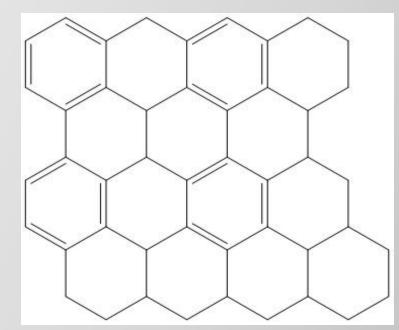






#### Introduction

Graphene, a single atomic layer of sp<sup>2</sup> carbon atoms, has gotten a lot of attention from the scientific community because of its excellent mechanical and electronic properties. The mechanical exfoliation methods used to obtain graphene nanosheets are not effective for large scale manufacturing, so scalable synthesis approaches from structurally similar compounds are of great scientific interest. Graphene oxide is one such compound which is synthesized through the oxidation of graphite powder. The oxidation was performed using a modification of the previous experimental procedure. After synthesis, the powder was fabricated into graphene oxide films through vacuum filtration and transfer to desired substrates. Both the powder and the films were then characterized using Raman spectroscopy and a four point probe measurement.



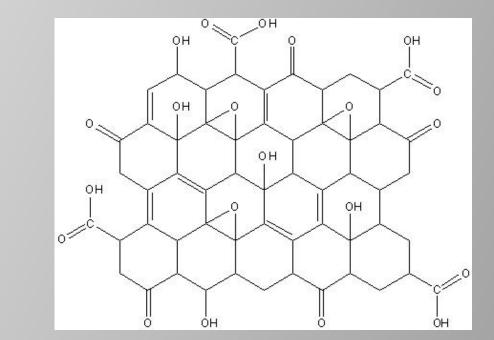


Figure 1: Structures for graphene nanosheets (left) and graphene oxide (right). Graphene oxide has a large number of oxygen functional groups substituted onto the starting structure.

# Synthesis and Characterization of Graphene Oxide

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## Synthesis

A 9:1 mixture of concentrated H<sub>2</sub>SO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub> (90:10 mL) was added to a mixture of graphite flakes (1.0 g) and KMnO<sub>4</sub> (6.0 g) producing an exothermic reaction. This reaction was then heated to 50°C and stirred for 12 hours. The reaction was then cooled overnight and poured onto ice (around 200mL) with 30% H<sub>2</sub>O<sub>2</sub> (1mL). The resulting mixture was then centrifuged (4000rpm for 1 hour) and the supernatant decanted away. The remaining solid material was then washed with 100mL of HCI followed by centrifuging (4000 rpm for 1 hour) and the supernatant decanted away. Finally the solid material was washed twice with 100mL of ethanol, centrifuged at 4000 rpm for 30 minutes each time, and the supernatant decanted away. The material from the centrifuge tubes remaining after the multiple wash process was then dissolved into water, collected by vacuum filtration, and ground with a mortar and pestle..

## **Sheet Resistance**

After depositing graphene films onto glass using vacuum filtration, the sheet resistance was determined using a four point probe with the Van Der Pauw method. Multiple square. The average partial resistance from the IV graph was found to be  $85.8\pm4.4~\mathrm{k}\Omega$  (see figure 2 below). Using the calculation for sheet resistance gives 408 k $\Omega$  per Square.

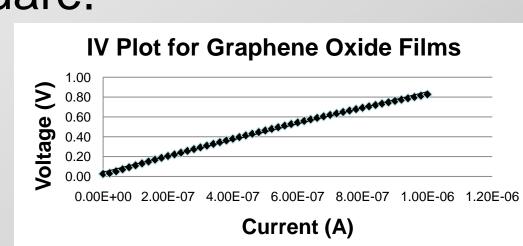


Figure 2: An IV curve for graphene oxide film on glass.

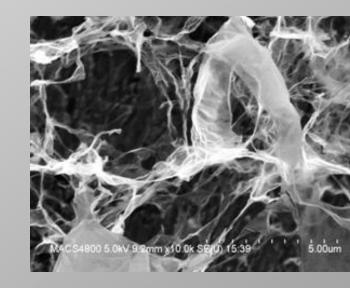


Figure 3: A graphene oxide thin film.

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## Raman Spectroscopy

Figure 5 below shows a typical Raman spectra of graphene oxide. The G peak is the result of in plane optical vibrations and has a literature value of 1580 cm<sup>-1</sup>. The D peak is located at 1350 cm<sup>-1</sup> and is due to first order resonance. This peak is absent in defect free graphene nanosheets as shown in figure 4 below.

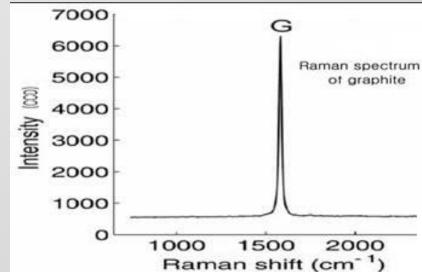


Figure 4: The Raman spectra of graphite powder has a strong G peak and almost no D peak...

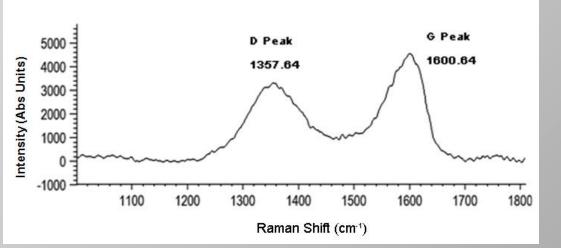


Figure 5: The Raman spectra of graphene oxide shows both the D and G peaks.

#### **Future Work**

Graphene oxide can be reduced through the use of several methods including chemical reaction with sodium borohydride to synthesize reduced graphene oxide. The reduced graphene oxide nanosheets will be characterized and compared to graphene oxide nanosheets with the expectation that reduced graphene oxide will have a smaller D peak than graphene oxide

### References

Marcano et al. "Improved Synthesis of Graphene Oxide" ACS Nano Vol 4. No 8 (2010): 4806-4814 Zhu et al. "Graphene and Graphene Oxide: Synthesis, Properties, and Applications" Adv. Mater. 2010, 22, 3906-3924