# Mechanical Exfoliation to Make Graphene and Visualization

## Instructor Preparation and Discussion

Authors: Angela Jones, Ph.D.[1, 2] and Nathaniel Safron[2, 3]
[1]Institute for Chemical Education, [2]Nanoscale Science and Engineering Center, [3]Department of Materials Science and Engineering
University of Wisconsin – Madison

**Age:** This activity is most appropriate for a high school science class rather than a take home project.

**Background:** In 2004, Andre Geim and Konstantin Novoselov of University of Manchester, UK won a Nobel Prize in Physics for isolating 1-carbon atom thick graphene sheets. To separate the graphene sheets from graphite flakes they used Scotch Tape. To visualize the graphene, they stuck the tape on a silicon wafer and examined the wafer under an optical microscope. Thin films of graphene are transparent to the naked eye which is why it is believed they will play an important role in the next generation of extremely thin electronics. However, when stuck to the wafer, the added graphene layers interfere with the light causing a shift in colors that allow you to distinguish wafer from graphene, and few-layer graphene from multi-layer graphene. In this activity students use natural graphite to duplicate the mechanical exfoliation technique used to separate graphene sheets from graphite. Ideally this would coincide with a trip to an electron microscope facility.

**Instructor Notes:** Preparation for the experiment will take approximately 30 minutes of instructor time. You'll want to prepare the graphite flakes adhered to the tape and cleave the silicon wafer into ~1 $cm^2$ pieces, once piece for each student. Handle the wafers using wafer tweezers as fingerprints will be clearly visible on the wafer when examined using the microscope. Instruct your students to not touch the wafer surface as well. That being said, examining fingerprints on wafers using an electron microscope is pretty interesting!

**Safety:** Graphite flakes are small and could easily form dust when first adhering to the Scotch Tape. It is best if you provide the students with graphite flakes already on the tape prepared in advance. This will also reduce the waste that could result from spilling the graphite flakes.

### Supplies

- Scotch tape
- Natural graphite flakes (Sigma Aldrich, 332461-2.5KG)
- $SiO_2$/Si wafers , 90 nm oxide thickness, N type/Antimony dopant, <100> orientation, SSP finish (Addison Engineering, (408) 956-5127)
- Diamond scribe
- Wafer tweezers
- Ziploc bags
- Access to an SEM and/or an optical microscope with at least 100X objective.

### Preparing graphite flake and tape:

1. Gently tap your bottle of graphite flakes on the end quarter of a 2 inch piece of Scotch tape. You'll want approximately 10-15 flakes for each sample.
2. Take another piece of tape, and stick it on top of the graphite flakes, perpendicular to the initial piece of tape. The sticky-sides of both pieces of tape are now together at a 90 degree angle. It might be best to fold over the remaining exposed tape to prevent the samples from adhering to each other and to provide a nice tab for the students to hold.

### Cleaving the silicon wafers:

1. The wafer has a flat edge. It will cleave perpendicular and parallel to the flat edge.
2. Place your silicon wafer on a clean surface shiny side up.
3. Press the diamond scribe firmly down at the flat edge of the wafer. The wafer will cleave perpendicular to the flat edge.
4. Continue to cleave along the flat edge, making strips ~1 cm wide.
5. Cleave the strips by pressing the diamond scribe firmly down either flat edge every centimeter. With very careful cleaving, you could get 50 1-$cm^2$ squares from one 100 mm wafer.
6. Use tweezers to move the small squares and put into individual Ziploc bags.

# Mechanical Exfoliation to Make Graphene and Visualization

## Student Instructions

### Background

#### *History*

Graphene is a flat sheet of carbon just one atom thick. Strong bonds link each carbon atom to three other carbon atoms and form hexagonal (six-sided) shapes. Graphene sheets resemble a honeycomb or chicken wire. Graphene sheets are so thin that it takes more than a million layers stacked on top of each other to make the graphite in a typical pencil "lead". Pencils work because the individual graphene sheets are not strongly bonded to each other. As you write, some of these layers peel off the pencil and stay on the paper. Individual graphene sheets were first separated from graphite in 2004 when scientists Andre Geim and Konstantin Novoselov of University of Manchester, UK stuck Scotch Tape to graphite and peeled off many layers. Next, they used a second piece of tape to peel a few graphite layers off of the first piece of tape. They continued this process about a dozen times. When they stuck the last piece of tape to a flat silicon wafer and peeled it away, some of the layers remaining on the wafer were a single atom thick– graphene!

### Purpose

In this activity, you get to duplicate the mechanical exfoliation technique using natural graphite flakes.

### Procedure

#### *Mechanical Exfoliation*

1. Take the top piece of tape and gently pull it apart from the bottom.
2. Re-adhere the top piece to the bottom and pull apart again. Repeat this 4-5 more times with the same piece of tape.
3. Throw away the top piece of tape.
4. Get a fresh piece of tape, and repeat step 2. As you repeat this step, you'll note that the bottom piece of tape is gradually covered with a shiny-gray graphite "film."
5. Repeat step 4, until the "film" is a dull gray. This will likely take 4-5 pieces of tape, depending on the initial amount of graphite.
6. Next, adhere the sticky side of the bottom piece of tape to the shiny purple side of the silicon wafer piece. Press the tape firmly yet gently onto the wafer piece.
7. The silicon wafer can now be viewed under an optical microscope with at least a 100X objective.
8. The color and graphene thickness will vary depending on the microscope and thickness of the silicon dioxide, but Figure 1 represents an example sample.
9. The wafer can also be viewed under an electron microscope or atomic force microscope if one is readily available.

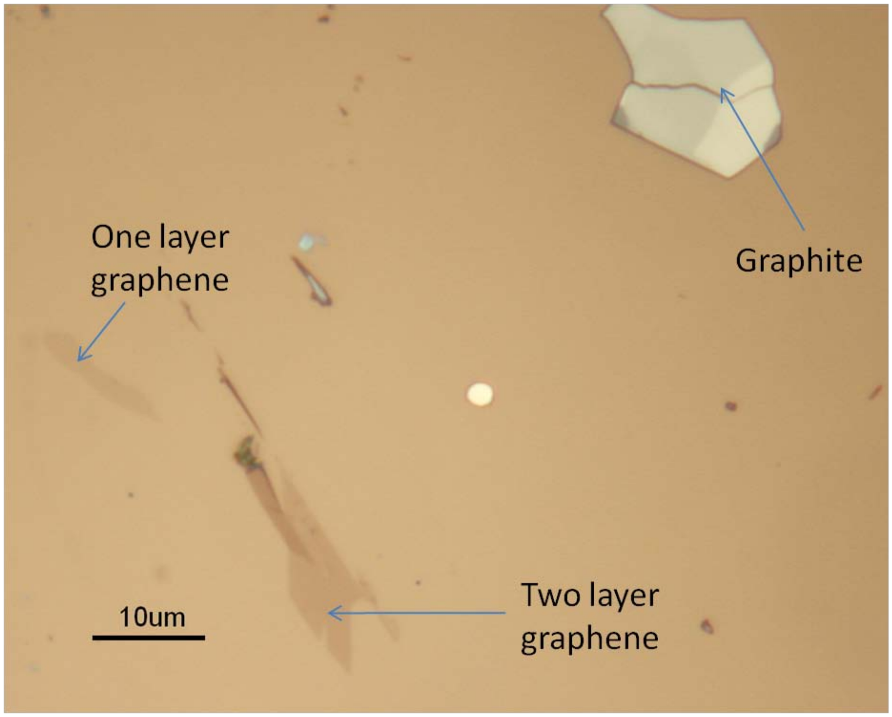


**Figure 1: Mechanically exfoliated graphene on $SiO_2$ imaged with white light using an optical microscope (Photo credit: Nathaniel Safron).**

## References

1. "The Nobel Prize in Physics 2010". Nobelprize.org. 31 Oct 2011 http://www.nobelprize.org/nobel_prizes/physics/laureates/2010/
2. Novoselov, K. S.; Geim, A. K.; Morozov, S. V.; Jiang, D.; Zhang, Y.; Dubonos, S. V.; Grigorieva, I. V.; Firsov, A. A., Electric field effect in atomically thin carbon films. *Science* **2004**, *306* (5696), 666-669.

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