

Graphene Resonators for Mass and Charge Sensing

Reyu Sakakibara

Chemical Biology, University of California, Berkeley

NNIN REU Site: Cornell NanoScale Science and Technology Facility, Cornell University, Ithaca, NY

NNIN REU Principal Investigator: Professor Jeevak Parpia, Department of Physics, Cornell University

NNIN REU Mentor: Robert A. Barton, Jr., Department of Applied and Engineering Physics, Cornell University

Contact: rsakakibara@berkeley.edu, jmp9@cornell.edu, rab375@cornell.edu

Abstract:

Nanoelectromechanical systems (NEMS) mechanical resonators are capable of detecting single atoms or molecules [1-3] and can potentially be used as compact, sensitive, and minimally destructive alternatives to traditional analytical chemistry techniques. Graphene, with its low mass, large area, and electrical conductivity, is an even better sensor of mass per unit area that can detect both mass and charge. The purpose of this project was to fabricate an electrically contacted graphene resonator from graphene grown by chemical vapor deposition (CVD). The device consisted of graphene suspended over a circular trench and contacted with platinum electrodes. Graphene electromechanical resonators have potential applications in biological sensing, gas chromatography, and mass spectrometry.

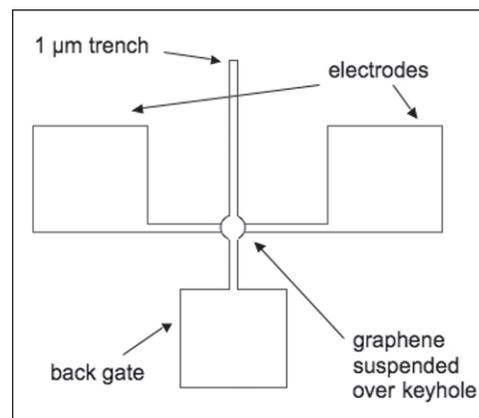


Figure 1: Device layout, consists of trench over which to suspend graphene and electrodes with which to contact the graphene.

Introduction:

The graphene resonator senses mass through its resonant frequency: adsorption of particles onto a resonator changes its mass and resonant frequency. The graphene is actuated in two separate ways. One is to apply an AC voltage across the back gate, which drives graphene resonance by applying a periodic attractive force. The other method is to heat the device at its resonant frequency with a laser. Light absorption and the associated temperature change result in a change in tension in the graphene that mechanically actuates the device. The motion can be detected using optical interferometry, in which a laser illuminates the graphene and the interference pattern of the light reflected from the graphene and the light reflected from the back gate is measured.

Measurements of conductivity give information about the effective doping and the charge on the graphene. Applying a voltage across the electrodes induces a current through the graphene, which can be measured to infer conductance. Additionally, applying a voltage on the back gate varies the Fermi level of the graphene and thus the concentration of charge carriers of the graphene. Variation in the Fermi level can be detected as a change in conductivity, which is proportional to the product of mobility and number of charge carriers.

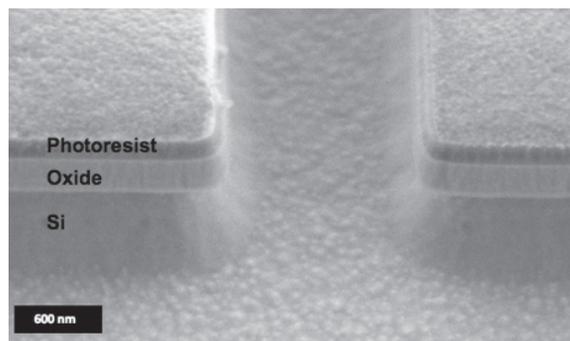


Figure 2: SEM image of desired undercut profile in the silicon, taken after the silicon etch.

Experimental Procedure:

Fabrication was based on that described by a previous paper [4]. Fabrication began by growing about 240 nm of oxide on a 10 kΩ•cm silicon (Si) wafer. The first level of features, trenches over which to suspend the graphene, was exposed using a deep ultraviolet stepper, which required spinning both photoresist and anti-reflective coating, or ARC. Following the lithography, trenches were etched into the wafer using deep reactive ion etching. An ARC descum

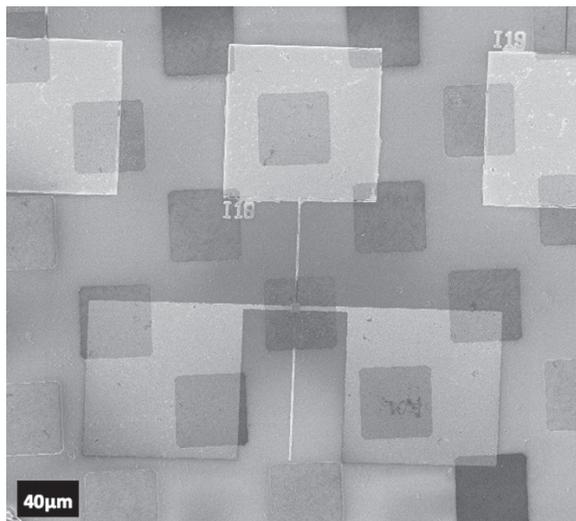
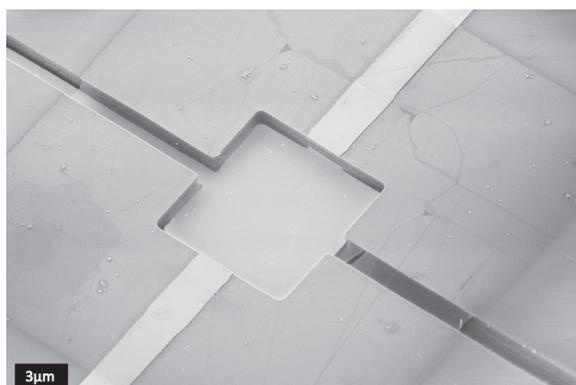


Figure 3, above: SEM image after graphene transfer; dark squares are graphene.

Figure 4, below: SEM image of a completed device with suspended graphene.



was performed with oxygen and argon plasma. The silicon oxide was etched through with either CHF_3/O_2 or CHF_3/O_2 followed by CF_4 . Silicon was etched with SF_6/O_2 , first anisotropically and then isotropically to create an undercut profile. An undercut profile was desired to prevent metal from evaporating onto the sidewalls, which would short the device. The desired trench depth was about a micron.

After stripping the photoresist and ARC, an additional 220 nm of oxide was grown. Next, the gate, source, and drain electrodes were patterned using photolithography, followed by an evaporation of metal (5 nm titanium and 25 nm platinum). The resist and ARC were removed in a liftoff process using sonicating n-methyl-pyrrolidone.

Graphene was grown via CVD on copper foil and transferred using a method cited previously [5]. First, graphene on copper foil was patterned with a checkerboard pattern of $50 \mu\text{m}$ by $50 \mu\text{m}$ squares and etched using oxygen plasma. Then poly(methyl methacrylate) (PMMA) was spun onto the surface of the copper and the copper was etched in ferric chloride. The graphene was rinsed in water and transferred

onto the wafer. The graphene squares landed randomly when transferred. The PMMA was removed and the devices were critical point dried to prevent stiction.

Results and Conclusions:

Profilometry and interferometry were used to characterize the etches and the oxidation. The initial oxidation yielded 243 nm of oxide. The SF_6/O_2 etched 1170 nm of Si with less than 0.1% nonuniformity across the wafer. Scanning electron microscopy (SEM) confirmed an undercut profile in the Si. Roughly 210 nm of oxide grew within the trenches, while outside, the oxide grew to 372 nm. Optical microscopy confirmed correct alignment of second layer exposure. SEM was used to find working devices: devices that were not shorted and with graphene that was not ruptured or stuck to the bottom of the trench. Raman spectroscopy confirmed that graphene was successfully grown and transferred.

The largest devices had a diameter of $12 \mu\text{m}$. The resonant frequencies of the devices were on the order of megahertz (MHz); for example, the $6 \mu\text{m}$ devices had a frequency range of 30 to 40 MHz.

Future Work:

Future experiments will include using both the mechanical resonance and the conductance of these devices to study biological and chemical analytes adsorbed to graphene.

Acknowledgments:

This work was supported via the National Nanotechnology Infrastructure Network Research Experience for Undergraduates (NNIN REU) Program by the National Science Foundation Grant No. ECS-0335765. This work was performed at the Cornell NanoScale Science and Technology Facility (CNF). Special thanks to Professors Jeevak Parpia and Harold Craighead, mentor Robert Barton, Program Coordinators Melanie-Claire Mallison and Rob Ilic, the CNF staff, and the Craighead Group.

References:

- [1] Jensen, K., et al. An atomic-resolution nanomechanical mass sensor. *Nat. Nanotechnol.* 3, 533-537 (2008).
- [2] Schedin, F., et al. Detection of individual gas molecules adsorbed on graphene. *Nat. Mater.* 6, 652-655 (2007).
- [3] Naik, A.K., et al. Towards single-molecule nanomechanical mass spectrometry. *Nat. Nanotechnol.* 4, 445-450 (2009).
- [4] van der Zande, A.M., et al. Large-Scale Arrays of Single-Layer Graphene Resonators. *Nano Lett.* 10, 4869-4873 (2010).
- [5] Li, X., et al. Large-Area Synthesis of High-Quality and Uniform Graphene Films on Copper Foils. *Science* 324, 1312-1314 (2009).
- [6] Reina, A., et al. Large Area, Few-Layer Graphene Films on Arbitrary Substrates by CVD. *Nano Lett.* 9, 30-35 (2009).