

AFM-Assisted Etching and Electrical Characterization of Graphene

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Abstract:

A pressure sensor, consisting of graphene transferred onto a chromium/ gold (Cr/Au) electrode and a silicon nitride (Si_3N_4) membrane, exhibits ultra high sensitivity. The device's high piezoresistivity was explored, and further measurements on the fabricated devices were taken for optimal design. Where typical, medical-grade pressure transducers have a sensitivity of 10 mV/V/mmHg, our device more than quadrupled this with as much as 50 mV/V/mmHg [1]. The transferred graphene was analyzed, employing scanning transmission electron microscopy (STEM), and both Raman and electron energy loss spectroscopy (EELS).

Introduction:

In the current era of electronics, the rate of increase in device density has decreased. Graphene, exhibiting exceedingly high electron mobility, has become a promising supplemental material to carry electrons for device operation, and its piezoresistivity has made it a candidate for high sensitivity strain transduction. Perfectly structured, low-area regions of mono-layered graphene have been produced through exfoliation, but larger areas are required for manufacturing. The process widely used for large-area synthesis involves chemical vapor deposition (CVD) on a copper substrate, and transferal via polymer. This commonly introduces impurities and defects within the graphene. Quality-assurance in the films can be verified through Raman spectroscopy and EELS, which respectively assure the number of layers and the composition of the film. STEM allows direct imaging of the film to verify the hexagonal crystal lattice.

Device Fabrication:

The device fabrication started with a double-sided polished (DSP), <100>, p-type silicon wafer, with 500 nm of low stress, low pressure CVD (LPCVD) silicon-nitride. After removing photoresist with an oxygen dry etch, an anisotropic KOH wet etch terminated on the nitride layer, forming the membrane. Electron-beam evaporated electrodes, consisting of 140 nm of gold on top of a 10 nm chromium adhesion layer, were patterned through optical lithography and lift-off in warm acetone. Copper (99.8%) foil was cleaned in chrome etchant, dried, and placed in a nanotube furnace, optimized to grow graphene through low pressure chemical vapor

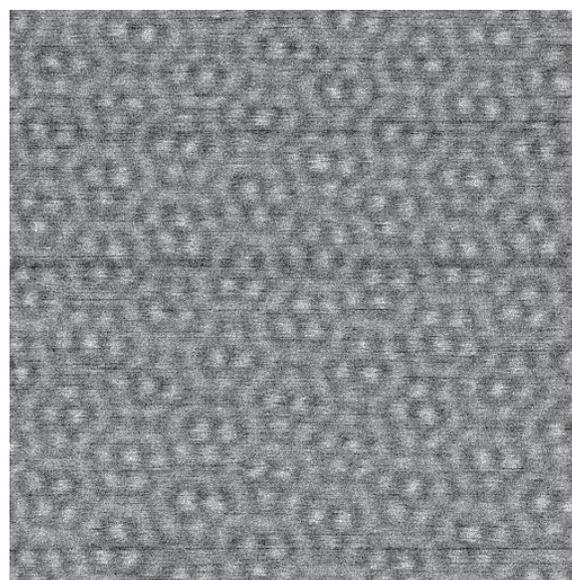
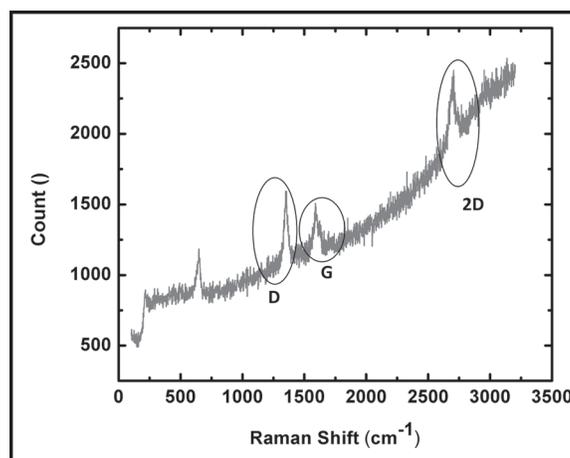


Figure 1, top: Raman spectroscopy of graphene on copper substrate, where the D, G, and 2D peaks characteristic of graphene are shown. The D peak, which shows defects within the lattice, is very high in this particular sample. The G peak verifies the sp^2 -bonds of graphene. The 2D peak can be used for identifying the number of layers. In this case, it is likely mono or bilayer graphene.

Figure 2, bottom: Nion STEM image of multi-layer graphene. Honeycomb atomic structure visible.

deposition (LPCVD). Poly(methyl methacrylate) (PMMA) was spun onto each sample at 4000 rpm for one minute, and the copper was removed with iron (III) chloride etchant. After transferal through five baths of deionized water, each sample was placed onto the device membrane and left to dry. Lastly, the PMMA was ashed in a 325°C air ambient for three hours.

Graphene Quality Measurements:

High-quality graphene consists of planar, sp^2 -bound carbon atoms in a crystalline, honeycomb lattice. Raman spectroscopy was used to characterize the thickness of the carbon films. For our pressure sensor, multilayered graphene was suitable. Figure 1 shows a Raman spectrogram of graphene on Cu-foil. The background noise is due to the copper, but the D (defect), G (graphitic), and 2D peaks of graphene are clearly visible. STEM imaging of suspended graphene sheets is shown in Figure 2: an image of the atomic structure of a multi-layered film. The large defect peak of the sample shown is not necessarily representative of the graphene on our devices, as the growth process was improved. However, machinery issues hindered the acquisition of a better plot.

An unexpected finding of our film characterization was iron residue from the Cu-etching process. EELS confirmed a considerable amount of iron was found on our sample, but the existence of this iron has not shown to interfere with the efficacy of the device (Figure 3).

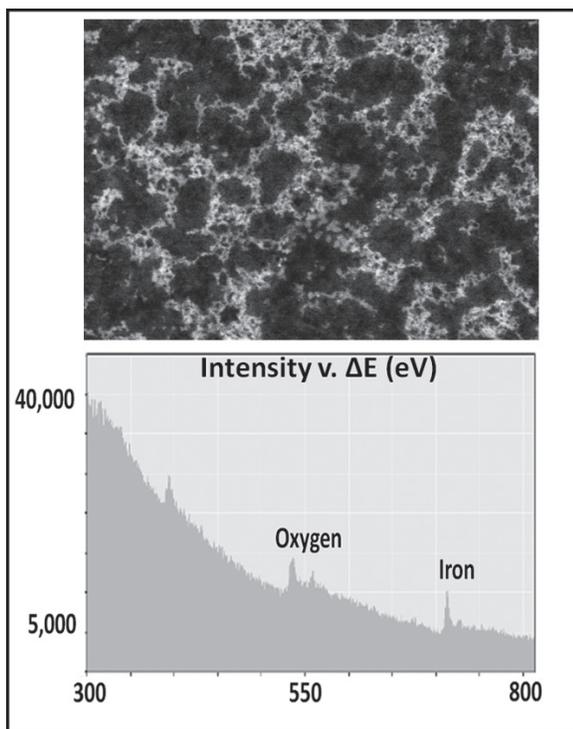


Figure 3: Nion STEM image of graphene on 50 nm silicon nitride (top). The amorphous, high-contrast material confirmed to be iron through EELS, coming from the copper etchant used in the graphene transfer protocol (bottom).

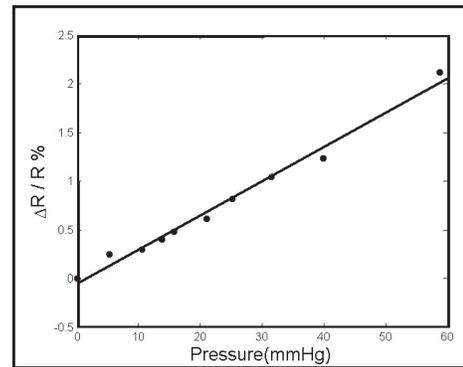


Figure 4: Fractional change in resistance vs. the applied pressure (mmHg). The current device, exhibiting 25% change at 5 mmHg, may be more sensitive for small pressures than its predecessor, which reached 15% at 6 mmHg. More measurements must be done for verification.

Pressure Sensitivity Measurements:

The set of four electrodes on each device were used for four-point probe measurements, using an applied pressure drop and measuring the corresponding change in resistance (Figure 4). The applied pressure causes strain in the graphene film, changing its resistance. As stated in the original paper for this device, the pressure sensor should be highly non-linear [2]. However, the thicker membrane used in this study did not rupture and reached significantly higher pressures than expected from the first study. It is therefore possible that the results displayed here are all within a relatively linear regime. Further measurements were not able to be taken to confirm this hypothesis.

Conclusions:

The current device is a viable microelectromechanical systems (MEMS) sensor, suitable for high sensitivity applications without requiring linearity. Further measurements will have to be taken. Hall effect measurements through the Van der Pauw method will have to be done to obtain the electron mobility of our graphene samples. Ascertaining piezoresistive non-linearity in the sensor via applied pressure, and actuating the membrane at resonance and using optical interferometry to determine strain, will be the likely next steps.

References:

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