Electrical Measurements on Graphene Inside Scanning Electron Microscope Using Four Point Probes

M. B. Olesen, B. Malm, D. H. Petersen, P. Bøggild

DTU Nanotech, Technical University of Denmark, Build. 345E, DK-2800 Kgs. Lyngby, Denmark mikkel.olesen@nanotech.dtu.dk

Since the discovery of the experimental technique for fabricating graphene by cleaving Highly Oriented Pyrolytic Graphite (HOPG)^[1], the interest of graphene as a electronic component has increased dramatically^[2]. Graphene is a two dimensional, single-layer carbon material arranged in a hexagonal grid. It is a semi-metal, meaning it has discrete K points where conduction band and valence band intersect^[3]. The interest in graphene as a electronic device comes from fact that the electrons acts as massless Dirac fermions^[4], which gives rise to the high electron mobility and long mean free path^[2] even at room temperature.

Graphene fabricated from HOPG can be placed on 90nm or 300nm silicon dioxide (SiO₂) for highest optical contrast between the substrate and the graphene^[5]. In this manner, pieces of graphene, multilayered graphene and graphite attach to the surface, and the contrast increases with the number of layers^[5]. Another, albeit more time consuming method, is to use Raman spectroscopy, which gives rise to a peak at 2600 cm^{-1[6]}. We here present a characterization method for graphene, using scanning electron microscopy (SEM). The contrast between a graphene sample and the substrate depends on the tilting angle of sample, ie. the angle between the electron beam and the normal of the sample surface.

Since graphene is a two dimensional material, the surface to volume ratio is very high, and the electrical properties are highly influenced by environmental variations. Theoretical calculations show electron mobilities of 100.000 cm²/Vs^[2], and experimental measurements on graphene on top of SiO₂, shows mobilities of 44.000 cm²/Vs^[7]. Characterization of graphene on an atomic level has been performed inside TEM^[8], and it has been shown that the electron induces damage on the graphene^[9], but not to what extend it effects the electrical properties. To better understand the electrical properties of graphene under the influence of an electron beam, four point probe (4pp) measurements are performed inside a SEM.

SEM Contrast on Graphene

Graphene cleaved from HOPG is placed on a wafer with 90nm of SiO_2 . The sample are examined inside SEM. Using acceleration voltages ranging from 1keV to 10keV, and tilting the sample in angles of 0 (zero) to 55 degrees, a contrast difference between the substrate and the graphene is observed. Higher angles gives results in higher contrast. The contrast is calculated from,

$$contrast = \frac{I_{substrate} - I_{graphene}}{I_{substrate}}$$

where I_i is the brightness intensity of material *i*. Fig. 2 shows the visible contrast difference on a sample at zero degrees and 55 degrees.

The tilting measurements are done on graphene samples with 1,2,3,>5 layers, and the result shows that the fewer layers, the less contrast difference when tilting. By measuring the contrast difference on graphene samples, the number of layers can be calculated. The output from the SE2 detector increases as the sample is tilted towards the detector. The intensities are related to the SEM brightness and contrast settings, in order to evaluate the data.

Electrical Measurements Using Four Point Probes

Cleaved graphene placed on SiO_2 , are placed inside a SEM on a SmarAct manipulator arm. On another arm, a 4pp is placed and electrical connections are made, so that the manipulator and the 4pp can be controlled from outside the chamber. Using this structure of setup, electrical measurements can be performed while the sample is exposed to the electron beam. By performing the measurements in vacuum, the effects from the air is eliminated. When the 4pp has electrical contact to the graphene, the electron beam can be turned on and off to measure the conductance with and without the beam affecting the sample. Thereby, the contamination induced by the beam on the graphene sample is estimated.

Measurements are done on different number of layers of graphene, on graphene on top of SiO_2 and on suspended graphene. Fig. 2 shows a four point probe contacted to a graphene sheet suspended inside SEM. Our findings suggests a difference in conductance over time, when the graphene sample is exposed to an electron beam.

References

[1] K. S. Novoselov, A. K. Geim, S. V. Morozov, D. Jiang, Y. Zhang, S. V. Dubonos, I. V. Grigorieva, A. A. Firsov, Science 306, (2004) 666

[2] A. K. Geim, K. S. Novoselov, Nature Mat. 6, (2007) 183-191

[3] C. H. Park, L. Yang, Y. W. Son, M. L. Cohen, S. G. Louie, Nature phy., vol 4 (2007) 213-217

[4] K. S. Novoselov, A. K. Geim, S. V. Morozov, D. Jiang, M. I. Katsnelson, I. V. Grigorieva, S. V. Dubonos, A. A. Firsov, Nature Lett. Vol 438, 10, (2005) 04233

[5] P. Blake, E. W. Hill, A. H. Castro Neto, K. S. Novoselov, D. Jiang, R. Yang, T. J. Booth, A. K. Geim, App. Phys. Lett. 91 (2007) 063124

[6] A. Gupta, G. Chen, P. Joshi, S. Tadigadapa, P. C. Eklund, Nano Lett. Vol. 6, No. 12 (2006) 2667-2673

- [7] R. S. Shishir, D. K. Ferry, J. Phys.: Condens. Matter 21 (2009) 232204
- [8] H. Zhang, P. X. Feng, Elsevier, 48 (2010) 359-364
- [9] D. Teweldebrhan, A. A. Balandin, App. Phys. Lett. 94 (2009) 013101

Figures



Fig. 1: Multilayered graphene at different tilting angles. (A) zero degrees, (B) 55 degrees. The contrast difference between substrate and graphene is increasing with increasing tilting angle.



Fig. 2: (Left) Micro four point probes in contact with graphene suspended over ridges. (Right) Four point probe measurements performed on graphene in air. The curves are for different probe pitches.