

Correlative Microscopy of a 2D Graphene Flake via Scanning Probe Microscopy

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Project location: Lancaster University

Lay Summary:

Atomic force microscopy (AFM) is a powerful technique often used to study surfaces at the nanoscale, helping researchers to better understand the structure and properties of materials. This project focused on harnessing the adaptability of the simple AFM to perform correlative microscopy on a sample of graphene, allowing the properties of the heavily researched material to be presented through visual images. I began by looking at the standard topography of the graphene flake to determine the thickness relative to the surrounding substrate. I followed this on by looking at the effect of friction when the probe was in contact with the sample. To go beyond surface imaging, I experimented with Kelvin probe force microscopy (KPFM) to map electrical potential under different bias voltages, before then utilising ultrasonic force microscopy (UFM) to reveal variations in surface elasticity. I implemented a thermal probe to analyse the thermal conductivity through scanning thermal microscopy (SThM), and tested the novel technique of scanning thermal gate microscopy (STGM) to investigate the Seebeck effect. By combining all these approaches, I built a detailed portfolio of complementary measurements, showing how AFM can provide a comprehensive picture of graphene's physical, electrical, and thermal properties.

Main Report

Project Aim

The aim of this project was to perform correlative microscopy on a conductive graphene flake using scanning probe microscopy (SPM) to comprehensively investigate its structural, electrical, mechanical, and thermal characteristics.

How did you address the aim?

To address this aim, I applied a series of scanning probe microscopy techniques, starting with basic imaging and then extending to more advanced modes. The most basic of these tests involved using the atomic force microscopy (AFM) to perform simple topography scans through both contact and tapping mode, allowing mapping of the graphene flake and determining its thickness relative to the substrate.

Contact mode also enabled measurements of surface friction and probe deflection. I then used Kelvin force probe microscopy (KPFM) to explore the difference in surface potential between the sample surface and the probe tip under varying bias voltages. To examine mechanical properties, I employed ultrasonic force microscopy (UFM), exploiting the extreme stiffness of the cantilever to generate images that reflect variations in elasticity. Next, to measure the thermal conductivity of the material, I utilised a thermal probe to perform scanning thermal microscopy (SThM). Varying AC and DC voltages enabled the heating of the cantilever tip which was then placed onto the sample in contact mode, allowing heat to be conducted onto the surface. Finally, I implemented the novel technique of scanning thermal gate microscopy

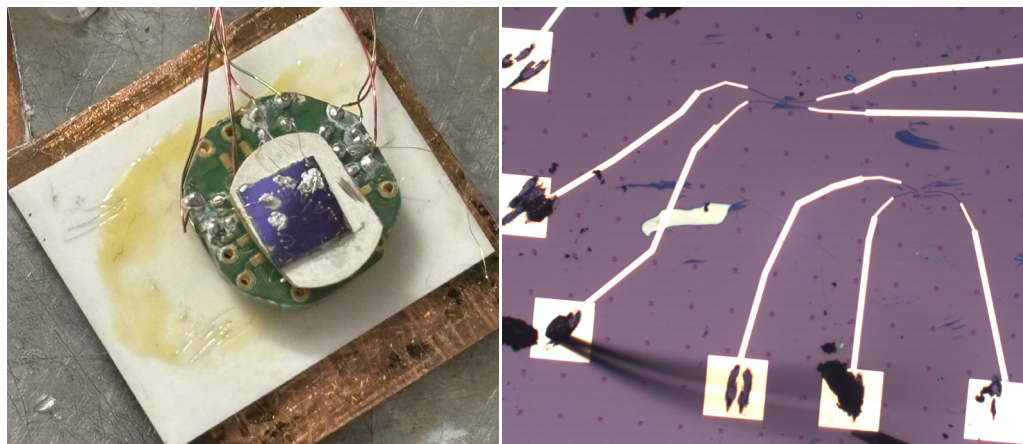


Figure 1. Image of the selected sample for testing. The sample consists of exfoliated graphene with two contacts and a backgate. The left image shows an overhead view of the sample mounted on a piezoelectric material and circuit board, while the right image is an optical microscope view at 5 $\times$ magnification, highlighting the overlap between the wires and the graphene flakes, which appear as dark blue shards.

(STGM), which combines SThM with scanning gate microscopy to allow the visualisation of the gradient of the Seebeck coefficient through thermal-electric interaction. Both SThM and STGM were performed under ambient and vacuum conditions to determine how restricting thermal conduction to the sample influenced image quality.

What did you find out?

For consistency, all tests were completed at the same section of the flake using 25 μ m wide scans at a 2:1 scan ratio and a scan rate of 0.2 Hz. The first analysis was of the surface topography of the sample. Due to surface debris (visible as white dots in figure 2a), the precise height difference between the flake and substrate was uncertain. However, in several areas the separation was less than 1 nm, consistent with the claim of a two-dimensional surface. In contact mode it was also possible to view the effect of friction on the probe as it was dragged over the surface. While there was only a small change in voltage (-100 mV to 50 mV), there was a clear contrast between the substrate and flake, with the substrate appearing slightly darker as seen in figure 2b. Tests of the surface potential were completed via KPFM at DC bias voltages ranging from -2 V to +2 V, with the most distinct images created at ± 1 V. The resulting image at +1 V DC can be seen in figure 2c,

with the graphene flake being substantially brighter, indicating a lower work function than the substrate. Attempts to measure the intrinsic potential difference without applied bias were completed, but the electrostatic coupling from the large cantilever dominated over the probe tip. UFM was utilised to investigate the elastic properties, with both a doped silicon probe and a thermal probe. The doped silicon probe produced higher quality images, possibly due to increased cantilever stiffness. The resulting image can be seen in figure 2d showing that the graphene layer had a lower lock-in output and therefore was more compliant than the substrate. Thermal properties were probed using SThM. Figure 2e shows the resulting image conducted inside a vacuum with a 4 V DC input and 1 V peak-to-peak modulation. The substrate appeared substantially darker than the graphene, indicating higher thermal conductivity. While placing the sample into the vacuum chamber the wire connections broke, not allowing images of the same region of the flake. Instead, figure 2f shows the visible change in the gradient of the Seebeck coefficient in air with an input of 7 V DC. The bright and dark spots correspond to the polarity of the contacts which is relative to the thermoelectric response, simply swapping the polarity flips the sign of the Seebeck-induced voltage.

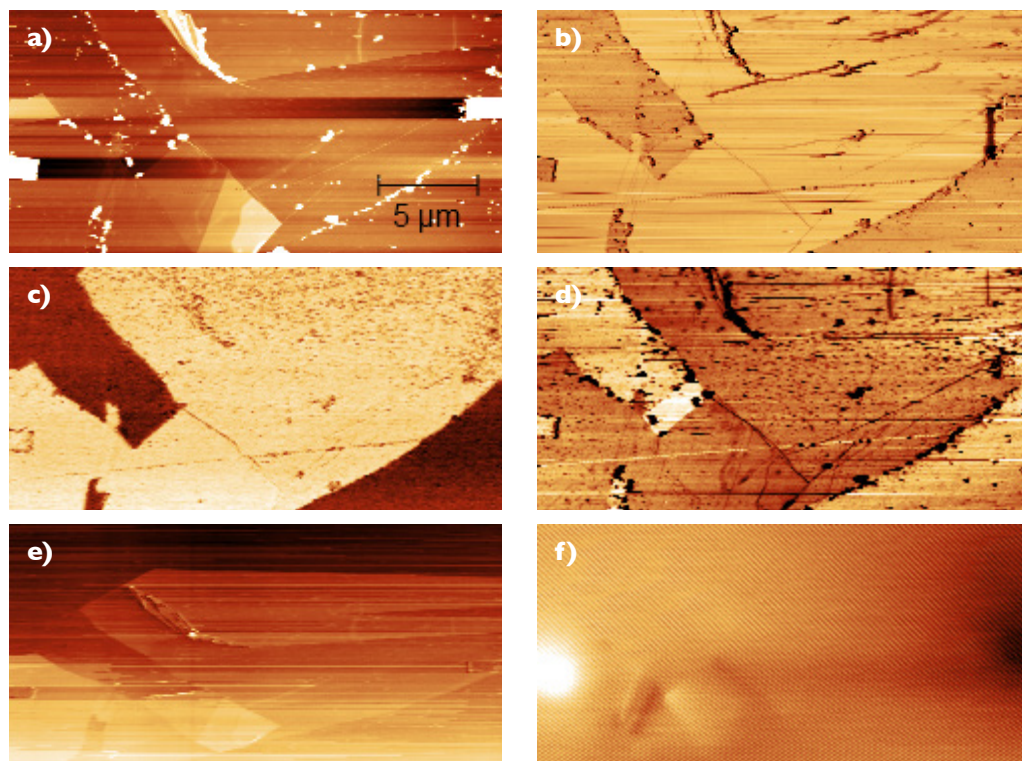


Figure 2. Correlative microscopy of the same section of graphene: (a) Topography, (b) Friction, (c) KPFM at +1 V DC bias, (d) UFM, (e) SThM in vacuum, (f) STGM in air.

What did you learn from participating in this project?

Before taking part in this project I had absolutely no experience in the field of microscopy! This summer project unveiled a whole new area of research and fascinating physics that I never even considered exploring. Learning about the characteristics of graphene and gaining experience in using an AFM was incredibly rewarding. My favourite part of this project was performing ultrasonic force microscopy. I enjoyed understanding the use of piezoelectric materials and how using them to vibrate samples at such incredibly high frequencies can uncovering detailed information about their elasticity.

How has this project affected your long-term goals?

This project resonated with me and has inspired me to complete my masters degree in microscopy, focusing on the creation and characterisation of energy-efficient atomic scale organic materials at

Lancaster University. After completing this, I plan on pursuing a PhD in materials science, potentially continuing in utilising AFM or branching out into electron microscopy. I can confidently say that this project has reinforced my commitment to continuing through academia and into research.

