Mapping of nanoscale mechanical and thermal properties of graphene using UFM and SThM

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Motivation

Graphene is an one-atom-thick material made of carbon. The last few years have uncovered the potential of graphene to be used for many applications given its remarkable properties. Most of the recent research is focus on the electronics properties of graphene. Largely unexplored remain its mechanical and thermal properties. In this work we report experimental results of mechanical and thermal properties of thin graphene layers using **Ultrasonic Force Microscopy (UFM)** and **Scanning Thermal Microscopy (SThM)**.

SEM image of the cantilevers with integrated heater for SThM

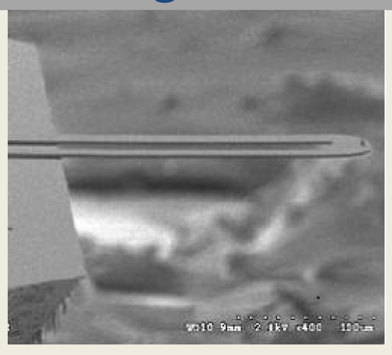


Figure 1: The 1 μm cantilever is u-shaped, having the heater integrated at the free end.

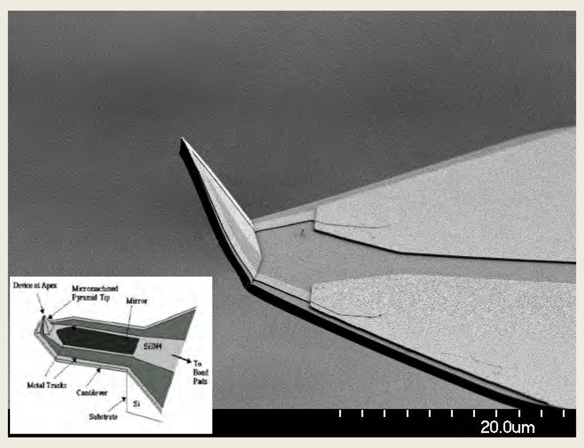


Figure 2: Micromachined Pd/ SiO_2 tip, *device at apex.*

Modelling: SThM response to the thermal conductivity of the sample

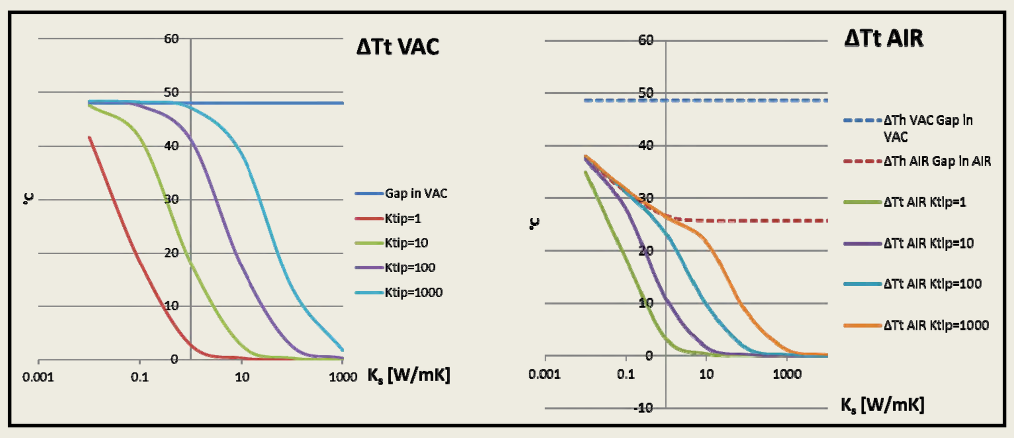


Figure 3: Tip T as a function of heat conductivity of the sample - vacuum and ambient air conditions. *As viewed from the tip.*

- Thermal response of any 'normal' tips with heat conductivity between 1 and 200 W/mK is very low in vacuum and almost non-existent in air.
- In order to sense highly conductive materials, highly conductive tip is needed.

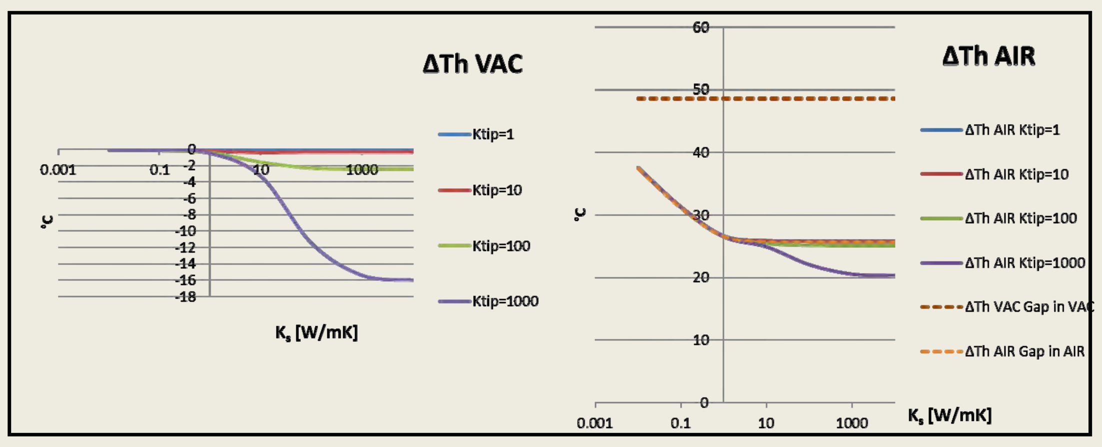


Figure 4: Sensor (heater) T as a function of heat conductivity of the sample. *As viewed from the sensor.*

- In vacuum: predominantly end of the tip heats the material
- In ambient environment: heat transfer from the whole cantilever leads to significantly distorted results and decreased spatial resolution.

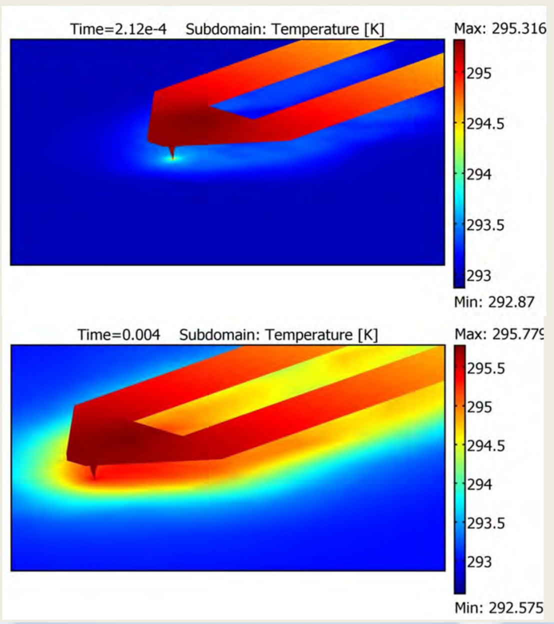


Figure 5: Modelling of the heated SThM probe in Vacuum and air.

SThM setup integration & Calibration

The vacuum system includes a HV vacuum chamber, turbo-molecular pump (TMP), ion pump, and has a oil-free fore pump with a working pressure ~10^{-7} Torr.

Figure 6: Vacuum multimode SPM; NT-MDT microscope with magnetic damping, and electronics for detection of DC and AC thermal signals, UFM.

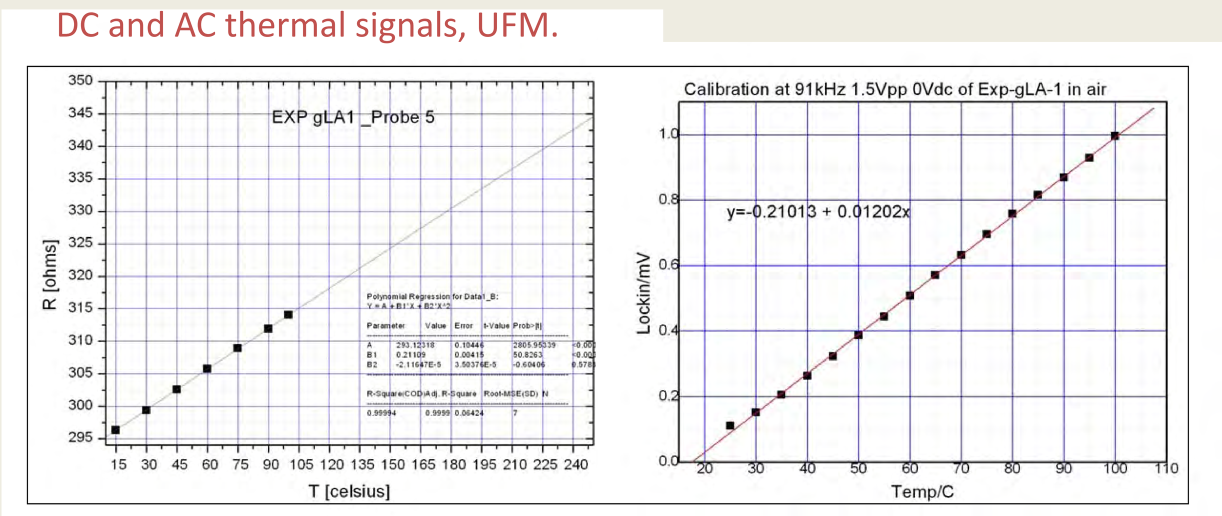


Figure 7: Calibration and Temperature sensitivity of multimode SThM.

AC detection of tip resistance is a very helpful approach eliminating multiple sources of error. The calibration then becomes a two-stage process:

- First, the resistance of the heater/sensor as a function of T is calibrated via DC measurements
- Second, an AC response of a balanced bridge with the sensor is measured via lock-in amplifier and linked with the T and resistance of the tip

High Vacuum SThM scans of a multilayer graphene

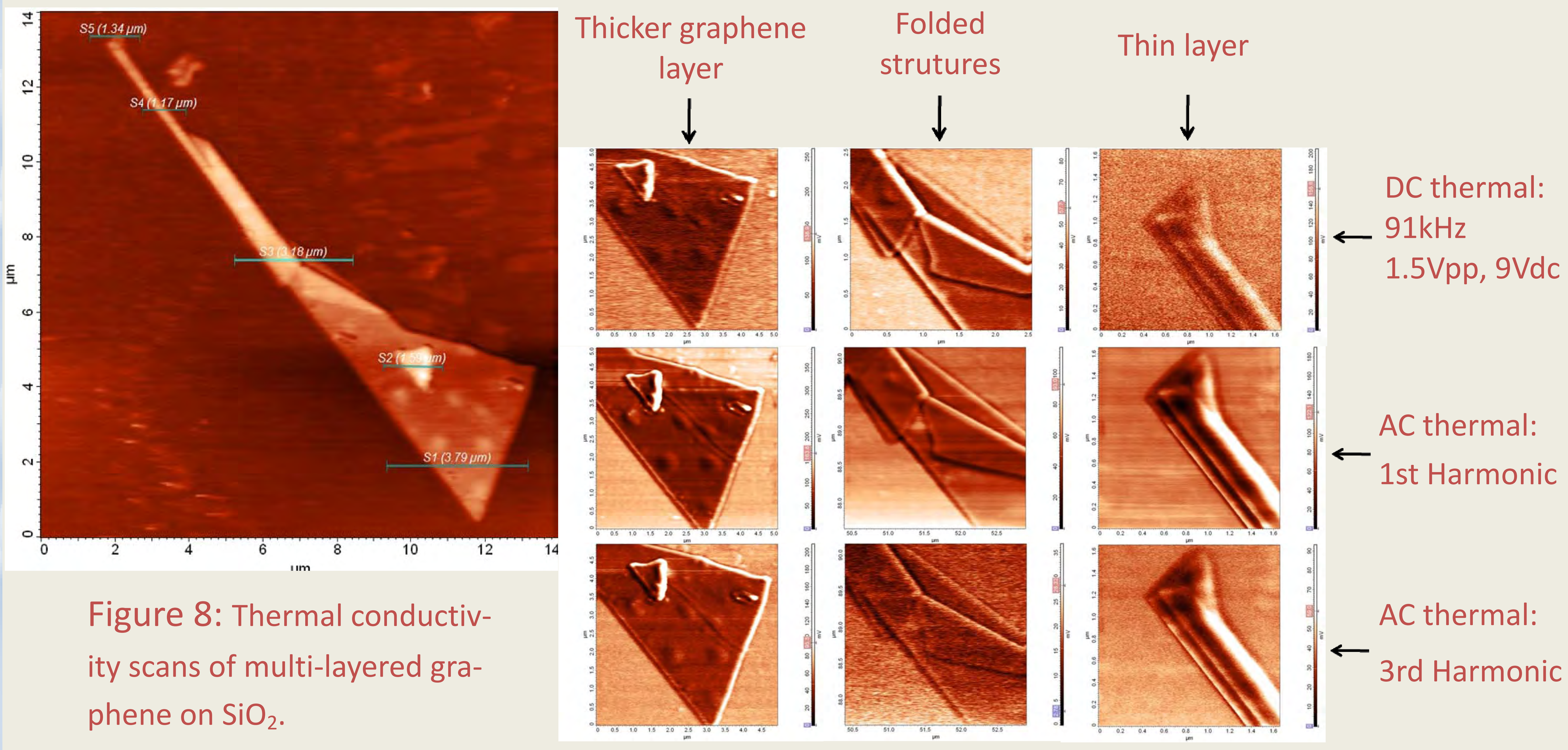
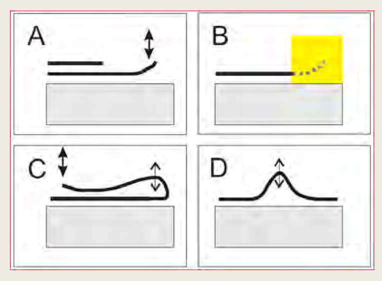


Figure 8: Thermal conductivity scans of multi-layered graphene on SiO_2.



Types of graphene folds

- 'Ultimate SThM' needs (at least in the present stage)
  - Vacuum environment,
  - High thermal conductivity of apex of the tip.
  - Shortening the path between the tip and the thermal sensor.
- SThM can be further tuned up with optimised sensitivity of high thermal conductivity materials (*i.e.* traditional semiconductor industry materials) via tapping mode, but vacuum is essential!

UFM setup & Scans of suspended and supported graphene

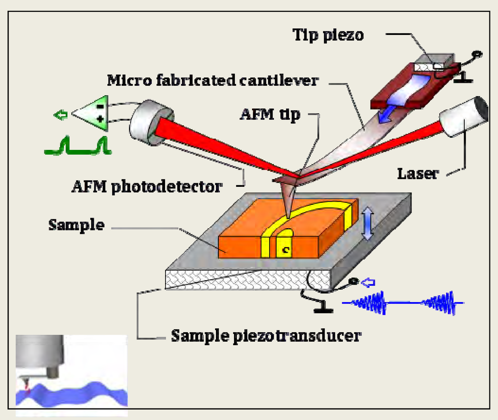


Figure 9: An ultrasonic force microscope. *Kolosov et al, PRL, 81, 1046 (1998)*

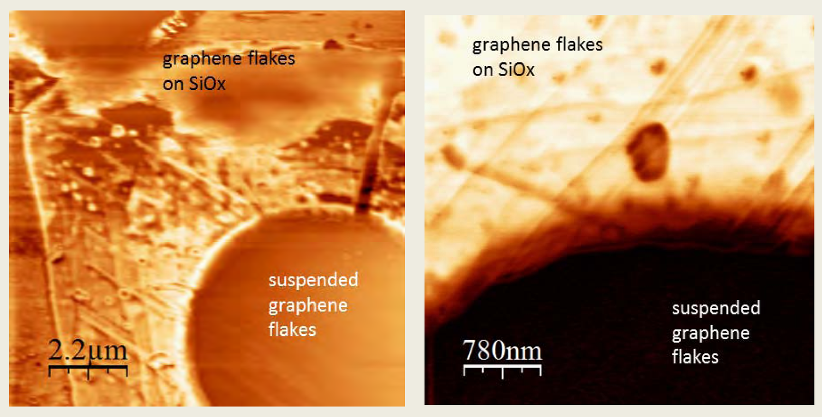


Figure 10: UFM images of HOPG graphene flakes overhanging holes. The apparent structure of the graphene flakes is clearly different to the corresponding pure India graphene flakes

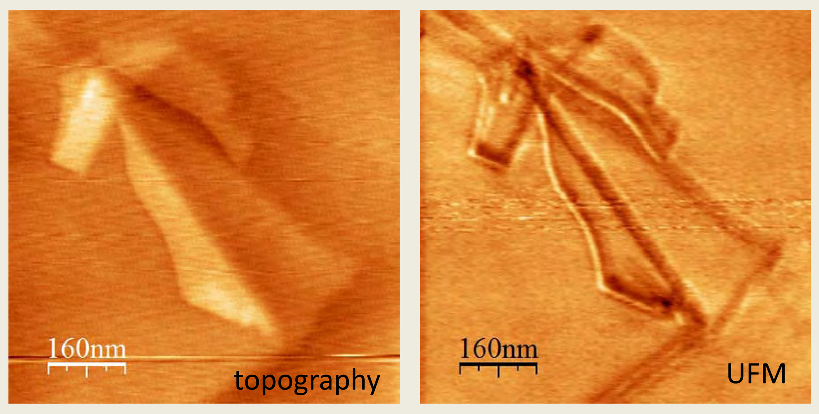
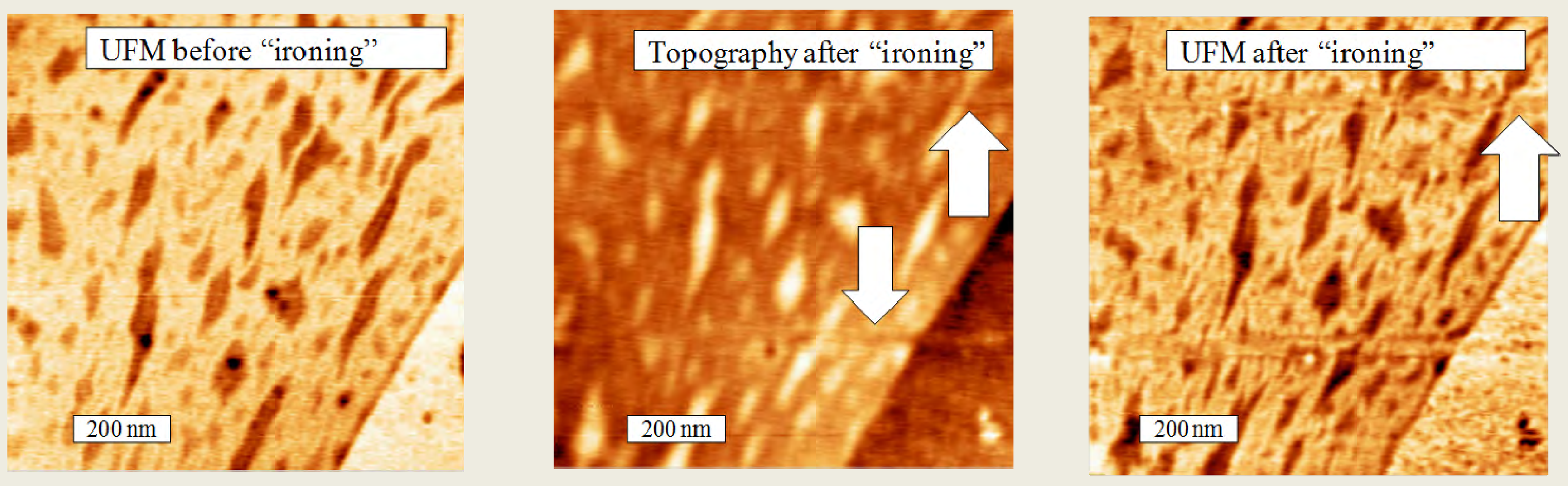


Figure 11: Graphene folds on bulk graphite. The presence of well-defined dark stripes is in stark contrast to the topographic view.

'Nano-ironing' of corrugations on as-deposited graphene



UFM images before and after 'ironing' with ~100nN normal force shows that the mechanical contact between the graphene and substrate in the area of 'nanodomes' was restored.

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