

Exploring Low-dimensional Carbon Materials by High-resolution Electron and Scanned Probe Microscopy.

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The microscopic characterization of low-dimensional carbon materials such as graphene or carbon nanotubes by high-resolution electron microscopy is a particular challenge owing to their intrinsically low contrast and high susceptibility to radiation damage. However, the recent developments in aberration-corrected electron optics opened a route to atomically-resolved studies of these materials at reduced electron energies, below the knock-on threshold of ca. 80kV for carbon atoms in graphene [1]. Current results are presented for 60kV studies of graphene and under ultra-high vacuum conditions using the Nion UltraSTEM100. In these conditions, graphene and related samples remain stable up to extremely high doses on the order of $10^{10} \text{ e}^-/\text{nm}^2$. The use of very high doses, enabled by high sample stability, opens the door to new types of measurements such as an analysis of charge redistribution at single point defects [2], which provides only a very weak signal, or the analysis of electrons scattered to high angles [3] from single atoms, which has a very small cross section.

Besides the use of low voltages, we have recently developed a new approach to use low doses and automated image acquisition on large areas of the sample. The theoretical description is given in [4], using simulated data. For the experimental realization we have developed automated low-dose acquisition where images are recorded at atomic or near-atomic resolution from a fresh area of the sample, and in particular, without depositing any dose for focusing into the region of interest. The focus is set for reference only at the corners of the selected region. Fig. 1 shows an example of a large area map of graphene where all sample regions, except for those at the corners, have not been exposed to the primary beam at all prior to data acquisition. At the current status we reliably obtain 2 Angstrom resolution by this approach, shown by the first set of reflections in graphene, whereas 1 Angstrom would be possible with perfectly tuned conditions.

The second part of the presentation describes scanned probe studies of free-standing graphene membranes using a novel two-probe scanning tunneling microscope (STM). In this experiment, two STM probes are brought into contact with the graphene membrane from opposing sides, and at the closest point, the two probes are separated only by the thickness of the membrane. In this way, we have studied the tip-induced deformations in the graphene membrane and have revealed different regimes of stability of few-layer graphene. Fig. 2 shows results demonstrating the working principle of the setup and further results are shown in Ref. [5].

References:

- [1] J. C. Meyer *et al*, Phys. Rev. Lett. **108** (2012), p. 196102.
- [2] J. C. Meyer *et al*, Nature materials **10** (2011), p. 209.
- [3] G. Argentero, *et al*, Ultramicroscopy, in press (2014) (DOI:10.1016/j.ultramic.2014.11.031)
- [4] J. C. Meyer, J. Kotakoski, C. Mangler, Ultramicroscopy **145** (2014), p. 13.
- [5] F. Eder *et al*, Nano Letters **13** (2013), p. 1934.

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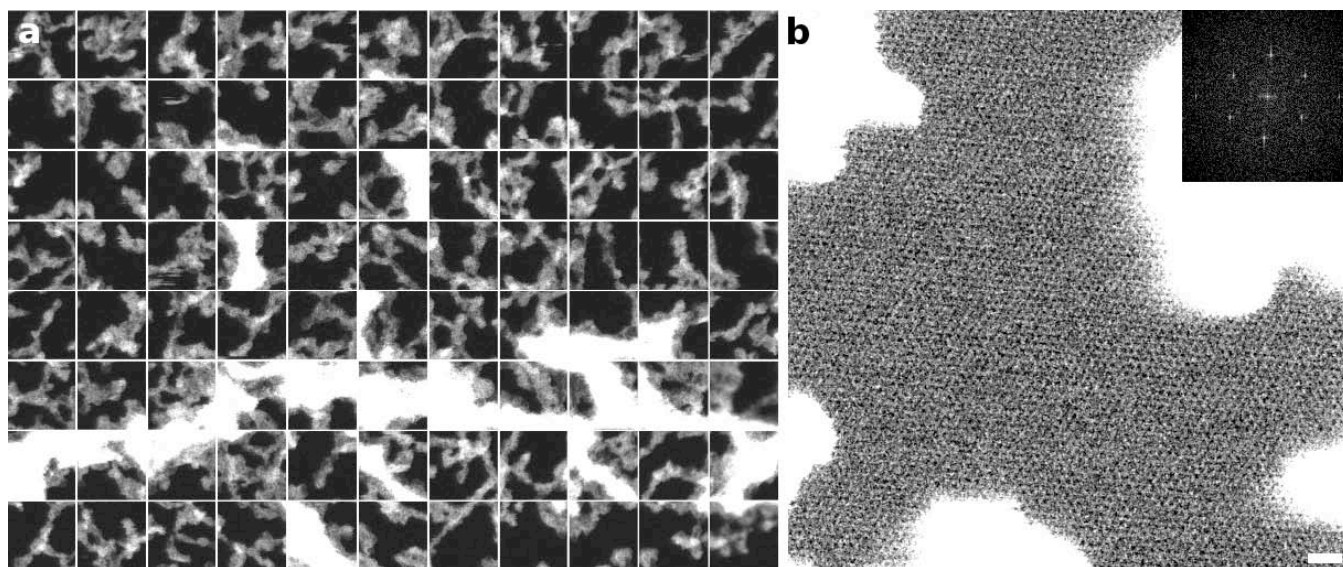


Figure 1. (a) Montage of 88 images obtained automatically on a ca. 200nm x 150nm area of the sample. A focus reference was set only at the corners. (b) Example of one image showing the graphene lattice. Scale bar is 1nm.

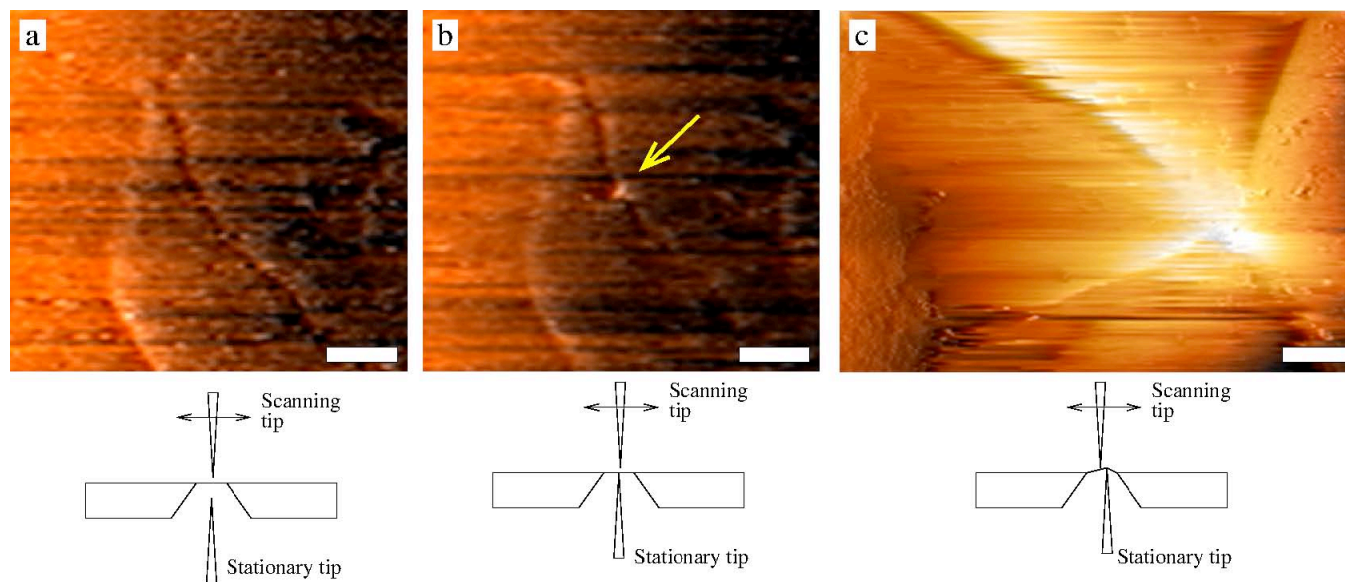


Figure 2. Initial results from the 2-tip test setup. (a) Suspended few-layer graphene, with only one tip (second tip is retracted). (b) STM image observed from one side, with the other tip placed into contact with the graphene membrane (curved lines in a-b are likely due to grain boundaries, and show that in fact the same area is imaged in both cases). (c) Observation of graphene membrane deformations, where the second tip is strongly pushing into the membrane. Scale bars are 200nm (a+b), 500nm (c). Height color scale is 20nm (a+b), 100nm (c).