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Adventures in Atomic Resolution *in situ* STEM

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Advances in transmission electron microscopy have enabled an increasing range of *in situ* experiments to study the effects of external perturbations including heating, electrical biasing, and controlled residual vacuum on a range of different samples. Although some trade-offs in imaging resolution and sample stability may be inevitable, with advanced instrumentation it is in many cases possible to conduct such experiments while retaining atomic resolution. *In situ* approaches make it relatively easy to obtain atomically clean surfaces, but can also reveal surprising effects from the residual vacuum composition as well as the thermal diffusion of surface adatoms.

The NionUltraSTEM 100 microscope in Vienna has been modified to enable a wide range of *in situ* experiments without compromising its excellent imaging capabilities [1]. These customizations include a viewport with a line of sight to the sample, which has allowed us to aim a laser at the specimen sitting in the column via telescope optics. This makes it possible to effectively remove amorphous carbon contamination by heating [2] as well as to mitigate mobile contamination that occasionally builds up under the beam. The laser can be precisely aligned to irradiate a ca. 560 μm^2 spot of the sample at the optical axis, minimizing thermal drift and localizing heating to the area of interest.

In terms of sample chemistry, the ultra-high vacuum (UHV) base pressure ensures that no unwanted chemical interactions can influence precise quantitative measurements of electron irradiation effects [3], revealing for example that pristine hexagonal boron nitride is remarkably stable at electron acceleration voltages below 80 keV. Further, a gas line connected into the column at the sample stage via a leak valve allows experiments at controlled atmospheres up to 10^{-6} mbar to be conducted without affecting imaging. These have revealed that oxygen is the active gas for the etching of pores in graphene, and that its armchair edges are indeed more stable than zigzag when chemical etching is not active [4].

Finally, heating experiments can be performed either using a Protochips heating insert in the standard Nion electrical cartridge, or via electrical biasing using conducting leads contacted with the sample. Using the latter, Joule heating of graphene-MoS_2 heterostructures up to estimated temperatures exceeding 2000 K has allowed us to observe the structural transformation of 2D MoS_2 into separated 3D nanocrystals of hexagonal shapes with the 2H and hybrid polytypes [5].

On the other hand, heating a monolayer graphene sample deposited on a SiN chip to temperatures between 300–1073 K using resistive heating with the Protochips Fusion chip remarkably increases its radiation hardness against otherwise damaging 90 keV irradiation [6]. This has further allowed us to estimate the carbon adatom migration barrier by quantifying how electron knock-on damage is reduced at elevated temperatures due to adatom migration and the healing of vacancies [7].

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