

In-situ HRTEM electrical experiments on graphene at high temperatures

B Westenfelder¹, T Amende¹, J Biskupek², S Kurasch², F Scholz¹ and U Kaiser²

1. Institute of Optoelectronics, Ulm University, Ulm, Germany
2. Central Facility of Electron Microscopy, Ulm University, Ulm, Germany

benedikt.westenfelder@uni-ulm.de

Keywords: Graphene, HRTEM, in-situ Hall measurements

Recently, we have realized an approach to study free-standing graphene and its adsorbates at high temperatures (300 K - 2000 K) in a transmission electron microscope (TEM) with atomic resolution. We showed the effect of in-situ Joule heating on graphene membranes in HRTEM images at temperatures until 2000 K [1] and observed the transformation of physisorbed hydrocarbon adsorbates via amorphous carbon monolayers (at 1000K) into polycrystalline graphene (at 2000K) [2], however the electronic properties could not be studied yet.

Here we investigate the electronic properties of graphene after modifying a conventional Gatan TEM specimen heating holder (Model 628) such that 4 contacts are practically available for electrical measurements whereas 4 other contacts remain for heating and temperature sensing at one and the same small specimen volume (3 mm in diameter). This extension enables electrical measurements and well-controlled specimen heating at the same time. The magnetic field of the microscope's objective lens was determined to be 1.2 T at 80 kV applying a custom-made Hall probe as TEM specimen (Figure 1). This one-time-only calibration procedure allows us to carry out Hall measurements at different temperatures.

In first experiments, graphene membranes have been suspended freely between four electrodes deposited on suitable TEM specimens. Simple characteristics on bi-layer graphene were measured inside the TEM for temperatures up to 500 K (Figure 2, left). The reliability of the obtained carrier concentration and mobility values could be assured by their temperature dependence. The first measured carrier mobility value of 314 cm²/Vs could be increased reaching 1580 cm²/Vs after 30 min pre-annealing at 500 K. This happened strictly without exposing the sample to the electron beam. Now, the first combined TEM images and electrical measurements have been recorded which revealed a large amount of hydrocarbon residues (Figure 2, right) and a mobility, which continuously drops during TEM imaging. This may originate from the interaction between electron beam and contaminants which was found to initialize localized etching processes to the graphene membrane. This preliminary result demonstrates the importance of a clean surface in order to obtain high mobilities for free-standing graphene. It also confirms previous observations that this requires annealing temperatures of at least 700 K [3]. Further work will be addressed (1) to reduce contamination on graphene and (2) to measure the mobility of graphene and its adsorbates at room temperature and at elevated temperatures while imaging.

References

- [1] B Westenfelder, J C Meyer, J Biskupek, G Algara-Siller, L G Lechner, J Kusterer, U Kaiser, C E Krill III, E Kohn and F Scholz, *J Phys D: Appl. Phys.* **44** (2011), 055502.
- [2] B Westenfelder, J C Meyer, J Biskupek, S Kurasch, F Scholz, C E Krill III and U Kaiser; *Nano Lett.* **11** (2011), p. 5123.
- [3] Z Cheng, Q Zhou, C Wang, Q Li, C Wang and Y; *Nano Lett.* **11** (2011), p.767.
- [4] We acknowledge the financial support of the German Research Foundation (DFG) and the state of Baden-Württemberg within the frame of the Sub-Ångström Low Voltage Electron Microscopy (SALVE) project.

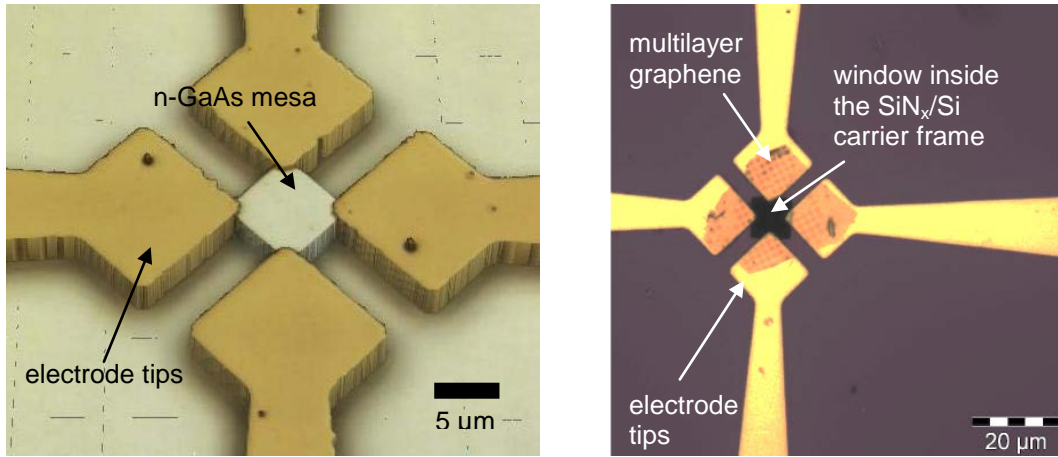


Figure 1. Left: Confocal image of a custom-made GaAs Hall probe. Right: Optical micrograph of the measuring electrode design. For first experiments arbitrary shaped multilayer graphene sheets were placed on the electrode tips.

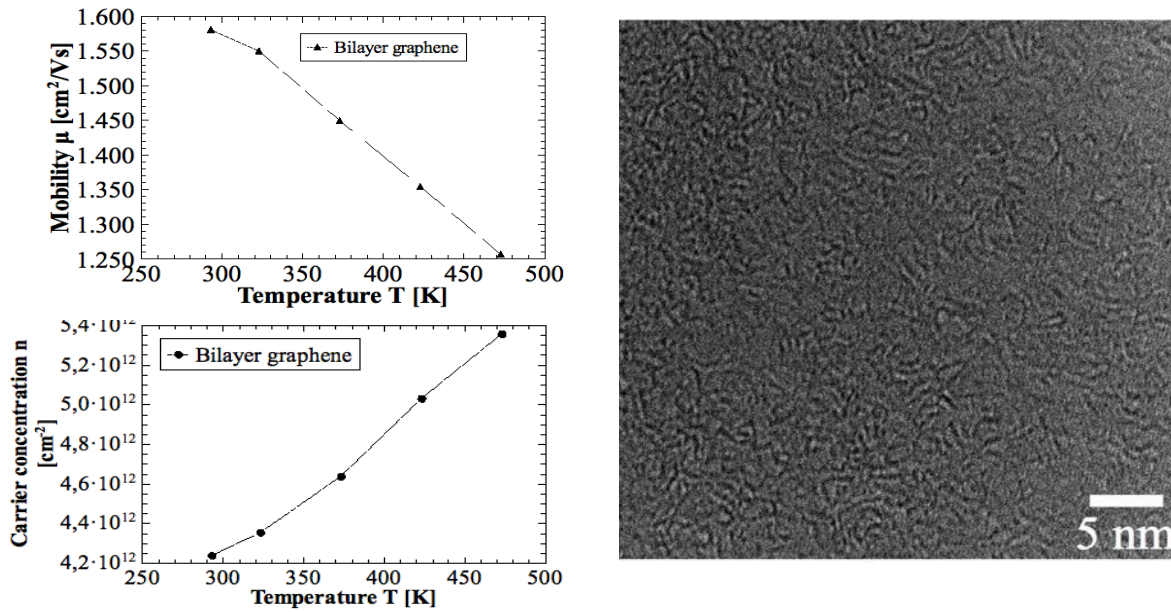


Figure 2. Preliminary Hall measurements performed on bilayer graphene after pre-annealing at 500 K (left). Even though the mobility increased by a factor of 5 after annealing it is still drastically reduced by hydrocarbon residues as shown by the corresponding TEM image (right). This confirms previous observations that annealing temperatures of at least 700 K are required.