# Open Topics

## MIM.6.P099
## Rocking curves of few layer graphene by evaluation of TEM dark field tilt series

C. Dolle[1], B. Butz[1], F. Niekiel[1], D. Waldmann[2], H. Weber[2], E. Spiecker[1]

[1]University, CENEM, Erlangen, Germany
[2]University, Lehrstuhl für Angewandte Physik, Erlangen, Germany

christian.dolle@ww.uni-erlangen.de

Graphene is one of the most promising materials of the new century; besides its peculiar behavior in electronic applications it is also the mechanically most robust material known so far with a breaking strength of 42 N/m [1]. Not only the mechanical strength but also the potential for developing devices is challenging at the moment. Especially membranes of mono or bilayer graphene can be utilized to engineer chemical sensors, resonators or for a recently developed DNA sequencing procedure employing small holes in a sheet of graphene [2]. To produce such free-standing membranes different approaches can be followed while in most cases a transfer of the layers is part of the preparation. We employ a recently developed method to grow epitaxial graphene on SiC [3] and remove the substrate in an additional processing step [4]. The preparation route allows the production of TEM conform samples by employing 3 mm disks of 6H-SiC, a mechanical thinning, the growth of graphene under reduced inert gas atmosphere, the definition of a metal mask by Lithography and the etching of the substrate via an electrochemical treatment in KOH (see figure 1). The Lithography allows the definition of arbitrary shapes of the resulting membranes with a size up to 500 $\mu m^2$. With the chosen growth parameters we obtain membranes consisting of mostly bi- and tri-layer graphene which can be readily investigated with complementary microscopic techniques to probe the local distribution of the number of layers, study the interaction of substrate and graphene and evaluate the microstructure of the membranes in detail accounting eg. for stacking order and defects. Dark-field TEM (DFTEM) tilt series and the evaluation of intensity variations during tilting allows to reassemble the projected structure amplitude of graphene and makes a precise evaluation of local number of layers possible. A much finer lateral resolution compared to mappings acquired by e.g. Raman Microscopy can be achieved. Complementary to a method suggested by Meyer et *al*. [5] who conducted tilting experiments on diffraction patterns of graphene, not only the number of layers in a given field of view can be investigated but the microstructure of the layers can be observed at the same time (not shown). The rocking curves measured from the tilt series are compared to theoretical curves calculated from kinematic diffraction theory. The theoretical rocking curves not only account for partially constructive/destructive interference due to deviation from Bragg condition but also for damping resulting from the angular dependence of the atomic scattering amplitude. The atomic scattering amplitude has been used in the form parameterized by Kirkland [6] and thermal vibrations (Debye-Waller factor) contribute only in a minor effect to the damping. Thus for comparison of experiment and calculation the Debye-Waller factor is not included and an excellent agreement between calculations and experiment is achieved (Figure 2). By utilizing DFTEM tilting series with a conventional sample holder it is possible to precisely evaluate the local number of layers in bi- tri- and four-layer graphene. No advanced analytical procedure has to be applied while the evaluation gives not only qualitative but quantitative results of the local distribution of layers with a resolution limited only by the magnification of the microscope. In addition the inter-layer spacing, stacking order and the topography of inclined surfaces can be examined with this approach.

1. C. Lee et *al*., Science 321, 385 (2008)
2. S. Garaj et *al*., Nature 467, 190 (2010)
3. K.V. Emtsev et *al*., Nature Mater. 8, 203 (2009)
4. D. Waldmann et *al*, submitted (2012)
5. J. C. Meyer et *al*., Solid State Communications 143, 101 (2007)
6. E. J. Kirkland: "Advanced Computing in Electron Microscopy" (2nd ed.), Springer, New York (2010)
Financial support from the German Research Foundation (DFG) via the SFB 953 “Synthetic Carbon Allotropes” and the Erlangen Cluster of Excellence EXC 315 “Engineering of Advanced Materials” is gratefully acknowledged

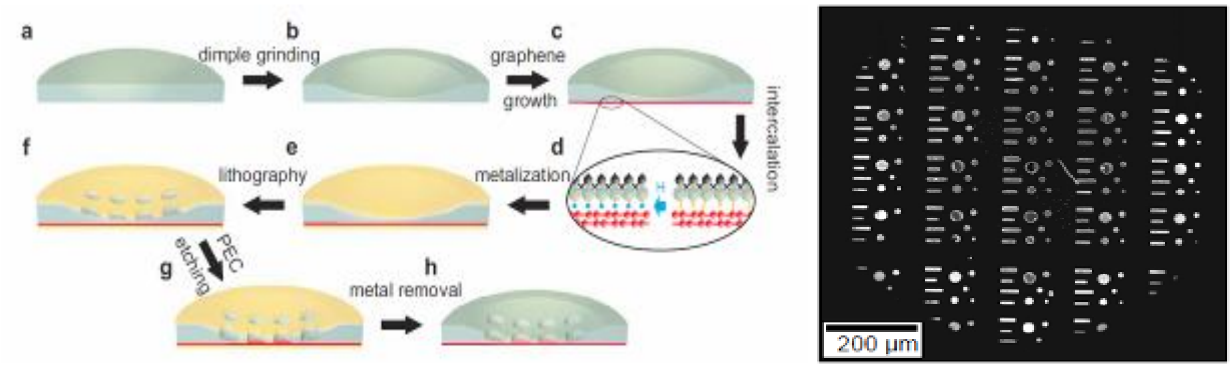


**Figure 1.** Preparation route for free-standing graphene membranes and low-kV STEM overview of prepared sample.

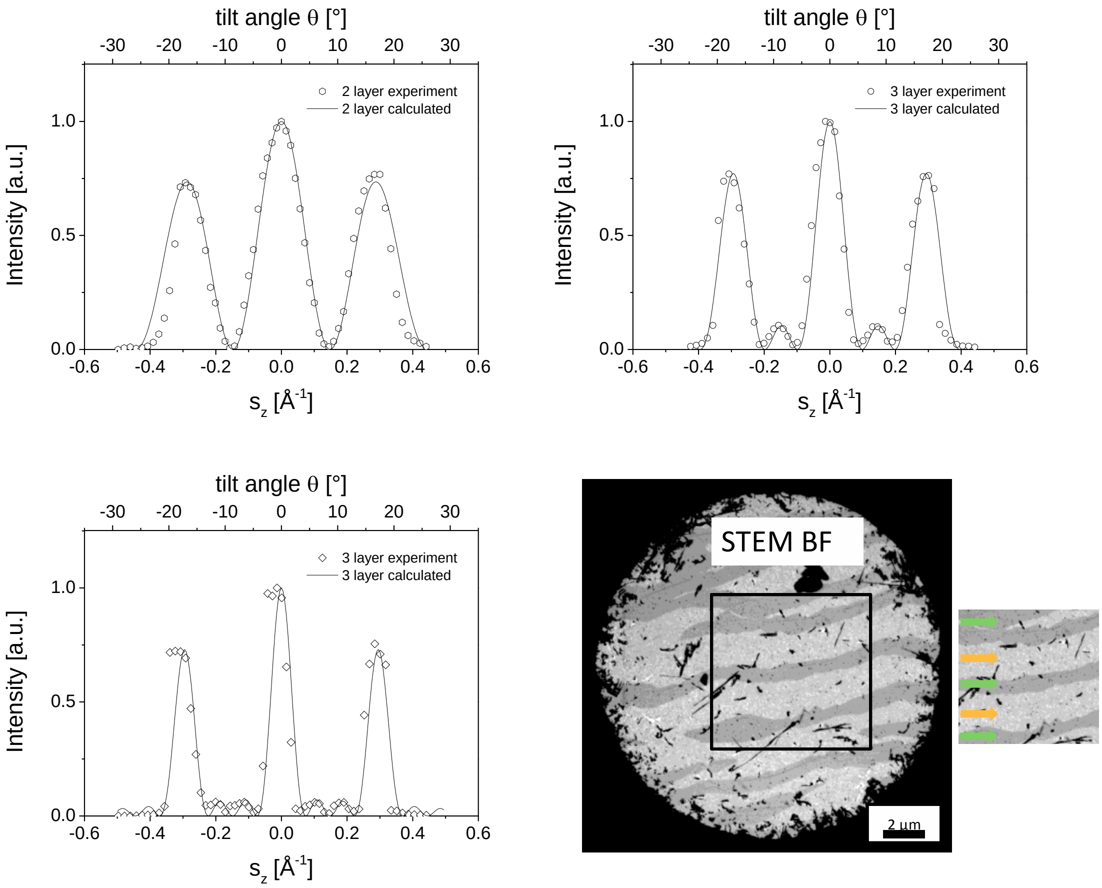


**Figure 2.** Normalized rocking curves of 2-, 3- and 4-layer graphene (open symbols experiment, solid lines calculated) and STEM BF image of representative round free-standing membrane showing the distribution of 2- (light grey, yellow arrows) and 3- (dark gray, green arrows) layer areas.