Study of Low-Angle boundaries in Graphene-like materials: an example of optimizing imaging conditions in a Cs-corrected S/TEM

D. Delille[1], S. Lopatin[1] and A. Chuvilin[2]*

[1*] FEI Company, Achtseweg Noord 5, 5600KA Eindhoven, The Netherlands
[2] CIC nanoGUNE, Tolosa Hiribidea 76, E-20018, Donostia - San Sebastian, Spain
* Corresponding author Dominique.delille@fei.com

Recent advances in spherical aberration (Cs) correction for transmission electron microscopes (TEM) in combination with electron sources of very low energy spread (use of monochromator) enabled imaging of single- and bi-layer graphene with atomic resolution [1]. Newly developed TEM techniques such as single atom or single atomic column spectroscopy [2, 3] and atomic resolution electron tomography [4] recently drove the need for increased electron radiation doses to be applied to samples, while radiation damage started to be the key limitation factor for high resolution TEM [5].

For graphene-like (light element) materials [6] the radiation dose limitation is shown to be particularly severe through several explanations. First, the knock-on damage cross section is higher for low atomic number elements [7]. Second, light elements like Carbon produce less contrast than heavier elements, so that even higher doses are needed to obtain a sufficient signal-to-noise ratio (SNR). Finally, graphene-like materials appear in the form of low dimensional allotropes that have only one or a few atoms in the typical projection of a high-resolution TEM image. While resolution-wise we are not limited any more by modern Transmission Electron Microscopes, a big question mark remains about the sample stability under the beam during the image acquisition.

Optimization of the acquisition parameters of TEM systems allows minimizing electron dose and thus reducing possible sample damage. Here we present an extensive study of TEM tuning to obtain high quality HRTEM images of graphene. FEI Titan Transmission Electron Microscope was used, equipped with a Cs image corrector, a high brightness gun and a monochromator (energy spread of the electron beam better than 0.2eV). Special attention was paid to optimize the settings of the Cs corrector.

Tuning of Cs corrector is based on measurement of image defocus (df) and astigmatism while recording so-called Zemlin tableau [8]. It will be demonstrated here that accounting for Cs of 3rd and 5th order (C3 and C5 correspondingly) and systematic error of C3 measurement results in more than 2 times increase in contrast, i.e. more than 4 times less electron dose to obtain the same SNR (Fig.1) in HRTEM pictures.

The optimal settings found using this novel technique will be used in the context of the study of low angle boundaries (LAB) in graphene. LAB is typically a row of edge dislocations, separation of those defining the boundary angle. LABs are not directly observable on the HRTEM images but can be identified using dedicated methods like geometrical phase analysis (GPA), see Fig.2. Physically speaking, LABs are of strong interest, as they represent a perfect discontinuous layer with periodically spaced singularities.

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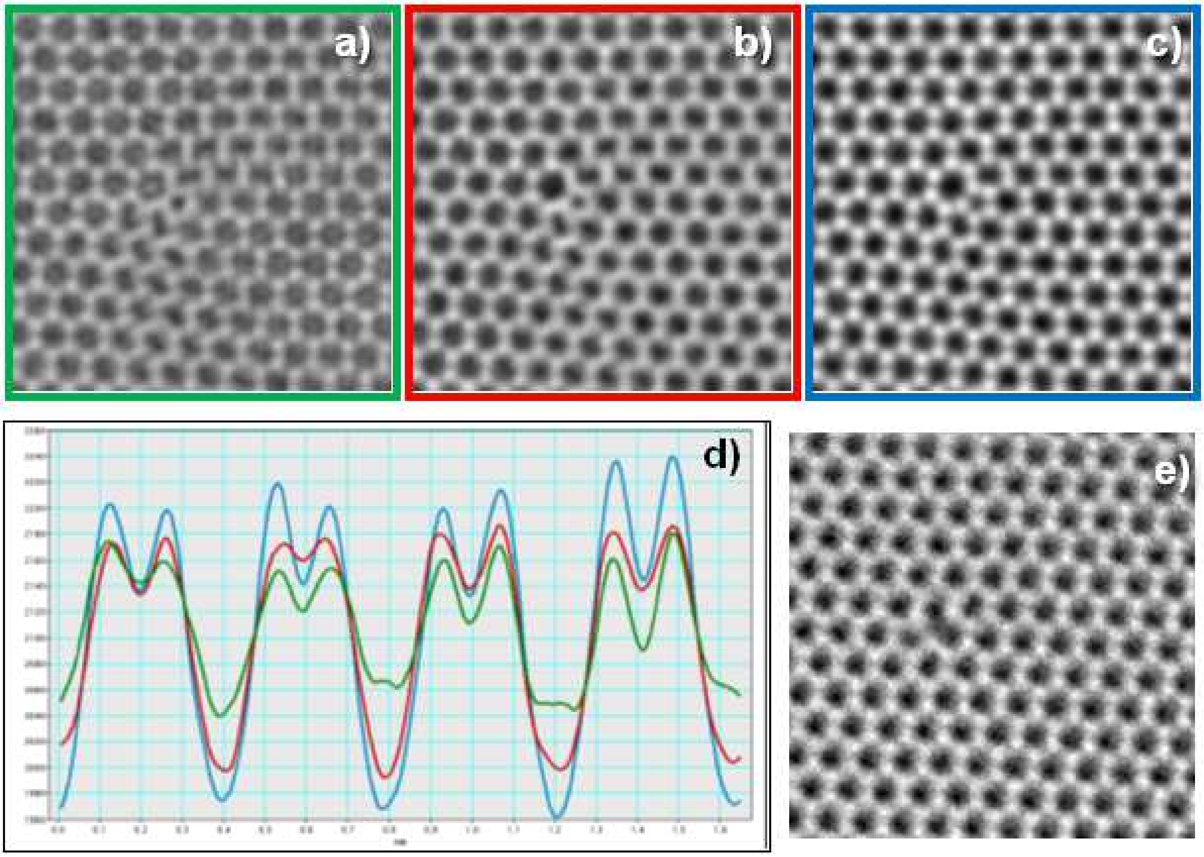

Fig.1. Simulation verification of the impact of optimum conditions: a) Scherzer conditions optimized for 0.1nm transfer; b) C5+C3+df conditions optimized for 0.1nm transfer; c) C5+C3+df conditions optimized for 0.1nm transfer and systematic error from Zemlin tableau is accounted; d) the intensity profiles across simulated images; e) an experimental image acquired at approximately optimum conditions.

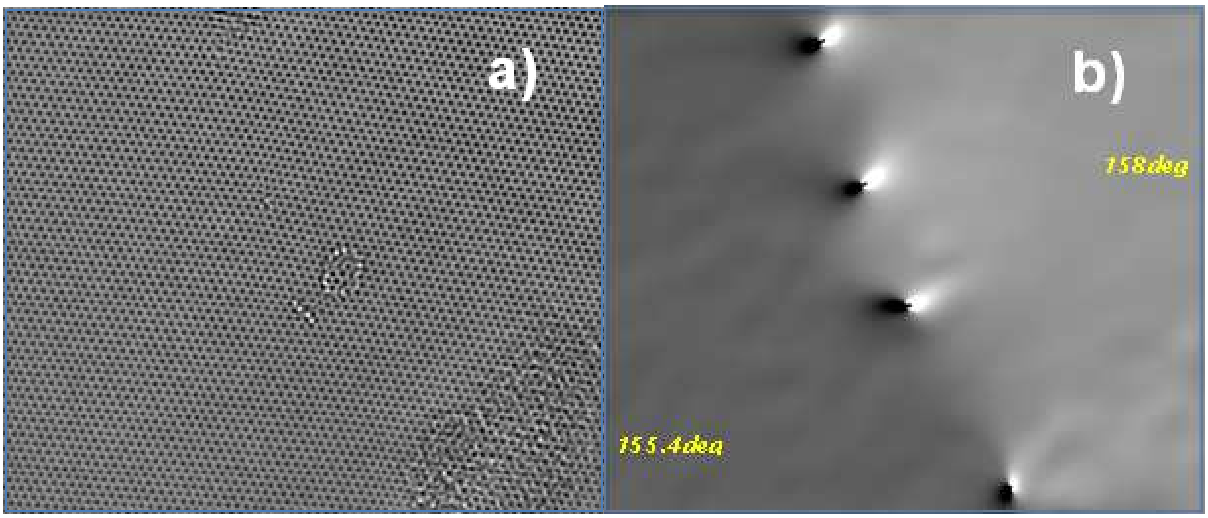

Fig.2. LAB in graphene: a) original HRTEM image; b) dislocations identification by GPA (rotation map).