Optimization of imaging conditions for atomic resolution in Titan TEM to minimize radiation damage and to study low angle boundaries in graphene-like materials

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Recent advances in spherical aberration (Cs) correction for transmission electron microscopes (TEM) in a combination with electron sources of very low energy spread (use of monochromator) enabled imaging of single and bilayer graphene with atomic resolution [1]. Newly developed TEM techniques such as a single atom or single-atomic-column spectroscopy [2, 3] and atomic resolution electron tomography [4] drive the need for increased electron radiation doses to be applied to samples. The 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 particularly severe for several reasons. First, the knock-on damage cross section is higher for low atomic number elements [7]. Second, the light elements produce less contrast than heavier elements, so that even higher doses are needed to obtain a sufficient signal-to-noise ratio (SNR). Finally, the graphene-like materials appear in the form of low dimensional allotropes that have only one or a few atoms in a typical projection of a high-resolution image. While resolution-wise we are not limited any more by modern TEM systems, there is still a big question about stability of the sample under the beam during the image acquisition.

The optimization of acquisition parameters of TEM systems allows to minimize electron dose and thus reduce possible sample damage. Here we present an extensive study of TEM tuning to obtain high quality HRTEM images of graphene. We used Titan transmission electron microscope (FEI Co) equipped with Cs image corrector, a super-high brightness gun and a monocromator (energy spread of the electron beam better than 0.2eV). Special attention was paid to optimize setting 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 was demonstrated 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, meaning more than 4 times decrease in dose for obtaining the same SNR (Fig.1).

The optimal settings found were applied to study low angle boundaries (LAB) in graphene. LAB is a row of edge dislocations, separation of those defining the boundary angle. LABs are not visible directly on the image but can be identified by methods such as geometrical phase analysis (GPA), see Fig.2. Physically LAB may be interesting as they represent a perfect discontinuous layer with periodically spaced singularities.

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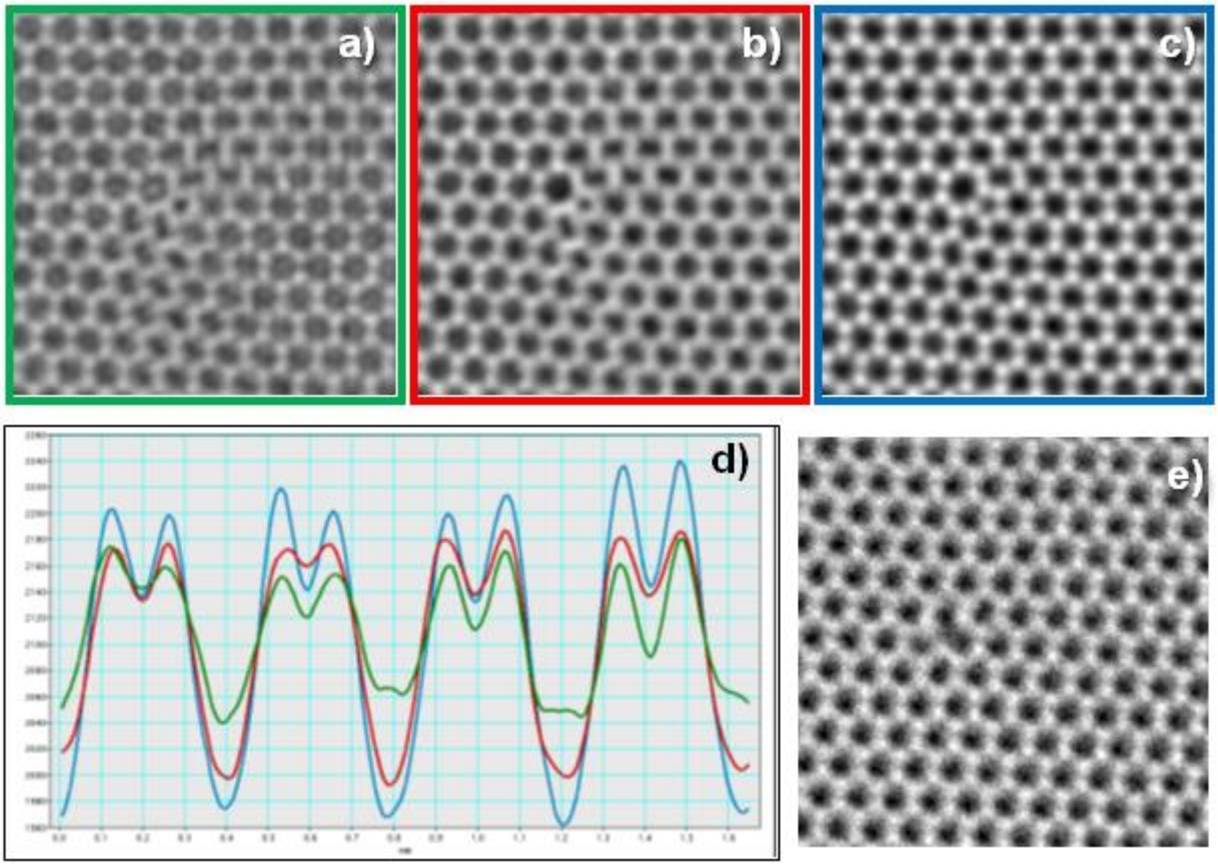

Fig.1. Simulation verification of the impact of optimum conditions: a) Scherzer conditions optimized for 0.1nm transfer; b) C5+C3+df conditions optimized for 0.1nm transfer; c) C5+C3+df conditions optimized for 0.1nm transfer and systematic error from Zemlin tableau is accounted; d) the intensity profiles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

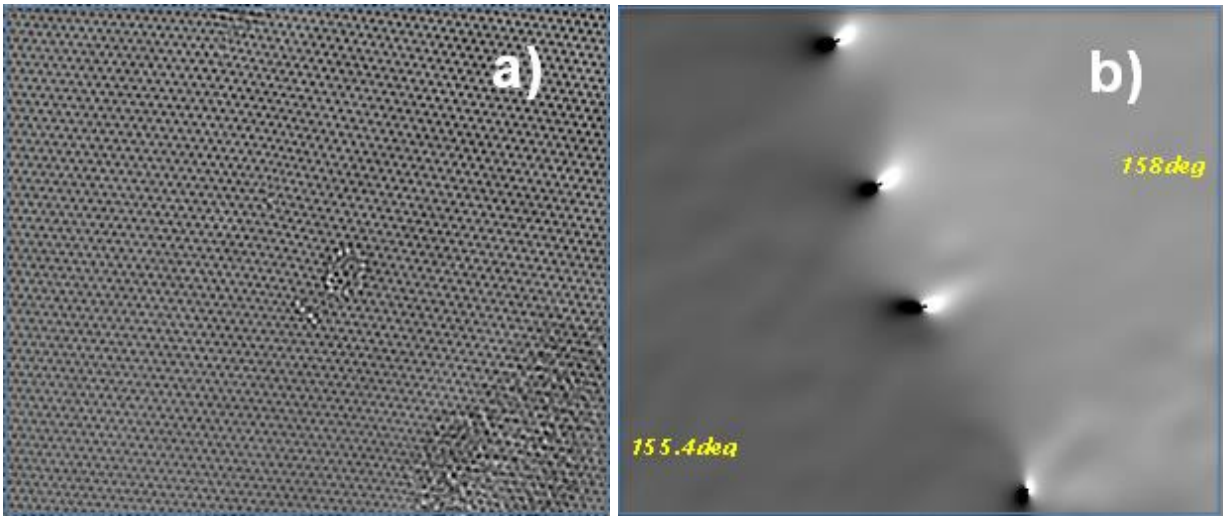

Fig.2. LAB in graphene: a) original HRTEM image; b) dislocations identification by GPA (rotation map).