doi:10.1017/S1431927611008336
Microsc. Microanal. 17 (Suppl 2), 2011
© Microscopy Society of America 2011

The Effect of Dose and Accelerating Voltage on Graphene Imaging in TEM

H. A. Calderon*,**, C. Song***, B. Barton**, C F. Kisielowski**,***

* Escuela Superior de Física y Matemáticas, IPN, Ed. 9 UPALM Zacatenco, Mexico D.F. 07738.
** Lawrence Berkeley National Laboratory, Berkeley CA
*** National Center for Electron Microscopy, LBNL, Berkeley CA 94720

Low dose electron microscopy is of prime importance to beam sensitive materials. Interaction between the highly energetic electron beam and low Z elements often alters the original structure of materials in a short time. Lately there has been a tendency to decrease the electron beam energy as a means to reduce beam-sample interaction but it is well known that an important parameter is the total electron dose deposited on the sample. Since the interaction between the beam and the sample often translates into lattice vibrations or heat, high-resolution images can deteriorate if recorded in high dose conditions. Thus low dose electron microscopy can also turn out to be important for metallic and semiconductor materials that are made of mostly high Z elements. Additionally interfaces joining hard and soft matter can be imaged in identical low-dose conditions. In the present investigation, graphene is used to vary the electron dose as a function of beam energy. The TEAM 05 microscope is used in TEM mode with the monochromator excited to provide an energy spread of 0.1 eV for imaging. Beam energies of 300, 80 and 50 kV are used.

Figure 1 shows direct images of graphene at different defocus settings for a dose rate of 60 electrons/$Å^2$s in average. The accelerating voltage is 80 kV and 1 s of exposure time. Depending on the defocus, some broad features can be noticed but all details of the structure are hidden. However the corresponding Fourier transforms do show crystalline reflections up to 1.2 Å. A total of 40 images are used for exit wave reconstruction, the starting defocus is 16 nm and the final one -23 nm with a ΔF of 1 nm. The reconstructed image phase is given in Fig. 2a where a monolayer, double layer and a triple layer can be identified. Because of the low dose imaging, the monolayer shows a relatively low contrast but the atom positions can be still recognized. Nevertheless, the rim of the monolayer can be readily interpreted in most of its sections. As for the surrounding triple layer, all the expected variations of intensity are found as can be seen by comparing to the corresponding simulation (Fig. 2 b-c). Figure 2 also shows phase line profiles (see Figs 2d-f) as a function of distance (see lines in Fig. 2a for exact location). As for the experimental phase image, the recovered phase values for scattering at a single C atom exceed (~ 20 %) recently published values [1] for the same beam energy but a considerably higher dose. This is unexpected because in previous investigations, a higher magnification has been also used which in turn enhances the signal strength because of the present signal damping by the modulation transfer function of the camera. Currently the project advances towards collecting more information from large image series while keeping the dose rate at a minimum. This is necessary to enhance signal to noise ratios for elements of low scattering power. Further the effect of voltage variation will be discussed.

References
[1] J. Jinscheck, E. Yucelen, H. A. Calderon, B. Freitag. *Carbon* 49 (2011) 556.
[2] This research is supported by CONACYT, IPN (COFAA, SIP) and Helios SERC–LBNL. HAC is on sabbatical leave at LBNL.

https://doi.org/10.1017/S1431927611008336 Published online by Cambridge University Press

Microscopy was performed at NCEM, which is supported by the Office of Science, Office of Basic Energy Sciences of the U.S. Department of Energy under Contract No. DE-AC02—05CH11231.

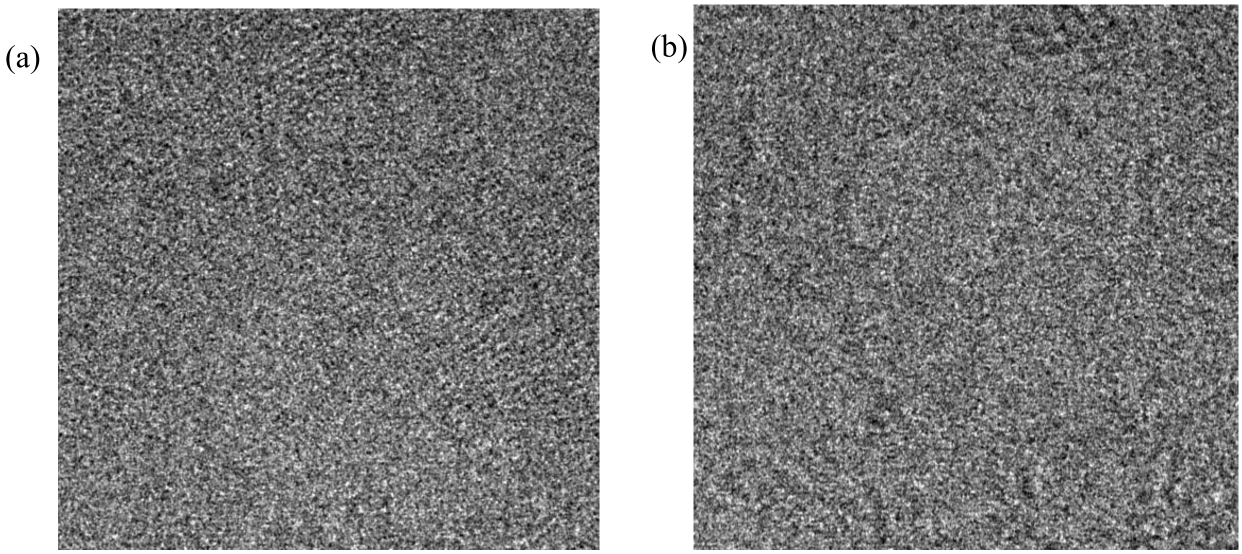

Figure 1. Low dose images of graphene at 80 KV as a function of defocus. (a) Df = 0 nm , (b) Df = 10 nm.

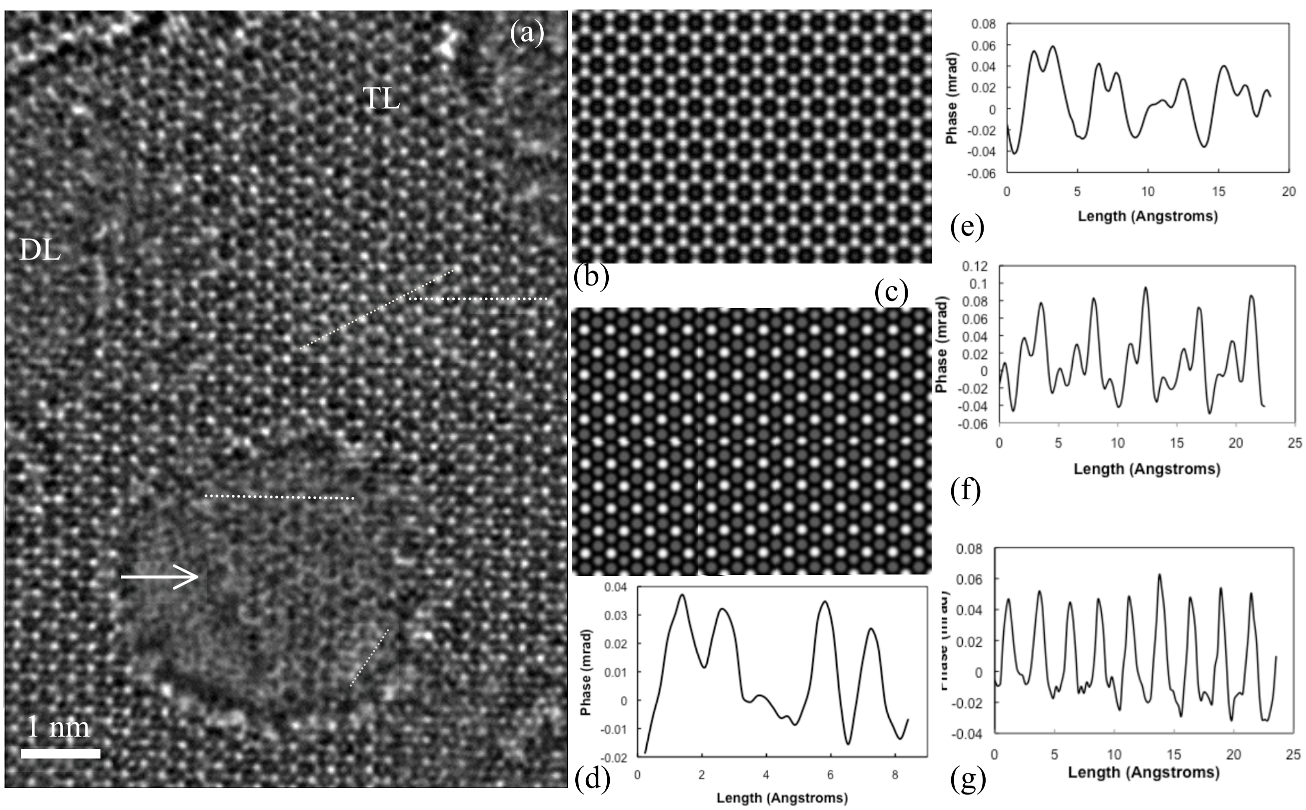

Figure 2. Results of Exit Wave Reconstruction and image simulation using 40 images. (a) Phase image, an arrow indicates a monolayer of C atoms, the triple and double layer are identified by TL and DL, respectively. Simulated phase image of a monolayer (b) and triple layer (c). Line profiles of recovered phase as a function of distance from regions indicated in (a), (d, e) single layer, (f, g) triple layer.

https://doi.org/10.1017/S1431927611008336 Published online by Cambridge University Press