Low voltage electron holography as a technique for mapping the number of graphenes in flakes

Castro Celia, Masseboeuf Aurélien, Monthioux Marc

CNRS-CEMES, 29 rue Jeanne Marvig BP 94347 31055, Toulouse Cedex 4, France
celia.castro@gmail.com

Abstract

Ideal graphene with desired properties is an utopian goal to reach without developing efficient analyzing method to support fabrication techniques. Since optical and electrical properties of few-layer graphene (FLG) are related to the number of layers and the stacking configuration [1-3] one challenge is to allow accurate numbering at nanoscale over flake area.

Recently, thanks to aberration-corrected transmission electron microscopy (AC-TEM), low voltage is now synonym of high resolution observation for carbon-based materials which are sensitive to irradiation damage. [4-5]

For configurations with uniform graphene layers, counting edges or peeling layer by layer the flake under the electron beam until a hole is opened provide basic information on HREM images. This method is analogue to drilling. It gives local information and cannot easily apply to a large number of flakes. Quantitative thickness mapping can be obtained by combining HAADF and electron diffraction. HAADF intensity is thickness-related and electron diffraction provides a calibration by determining the signal of a monolayer depending on the TEM settings [6-8].

Another way for mapping the number of graphene layers is low voltage transmission electron holography. The phase shift of electrons induced by the surface electrostatic potential is proportional to the thickness. This phase shift is intrinsic to the mean inner potential of the individual graphene layer. It directly represents the local number of layers. [9]

In the present study, this technique is transposed in the I2TEM, a new AC-TEM dedicated to electron holography developed between CEMES and Hitachi. We take here advantage of three characteristics of the I2TEM: the double biprisme configuration, the second stage unit located upper in the column (lorentz configuration), and the low voltage (80 KeV). By this way the hologram is larger than in regular TEM with no fresnel franges and irradiation damages are limited. First results of mapping the layer numbering over a flake scale and with nanometer spatial resolution by electron holography will be presented. An example taken from a graphite flake is provided as figure 1, In which the number of graphene is large. The method is however sensitive enough for mapping FLG flakes with thickness variations related to single graphenes. Also the influence of contamination - a recurrent issue in graphene synthesis – in the phase shift measurement will be discussed.

References

[1] Koshino M., New J. Phys. **15** (2013) 015010.
[2] Nair R.R., Blake P., Grigorenko A.N., Novoselov K.S., Booth T.J., Stauber T., et al., Science, **320** (2008) 1308.
[3] Castro Neto A.H., Guinea F., Peres N.M.R., Novoselov K.S., Geim A.K., Rev. Mod. Phys., **81** (2009) 109.
[4] Sasaki T., Sawada H., Hosokawa F., Kohno Y., Tomita T., Kaneyama T., et al., J. Electr. Microsc. **59 (**2010) S7.
[5] Suenaga K., Koshino M., Nature, **468** (2010) 1088.
[6] Gass M.H., Bangert U., Bleloch A.L., Wang P., Nair R.R., Geim A.K., Nature Nanotechnol. **11** (2008) 676.
[7] Li Z.Y., Young N.P., Di Vece M., Palomba S., Palmer R.E., Bleloch A.L., et al., Nature. **7174** (2008) 46.
[8] Meyer J.C., Geim A.K., Katsnelson M.I., Novoselov K.S., Obergfell D., Roth S., et al., Solid State Comm., **1–2** (2007) 101.
[9] Ortolani L., Houdellier F., Monthioux M., Snoeck E., Morandi V., Carbon, **49** (2011) 1423.

Figures

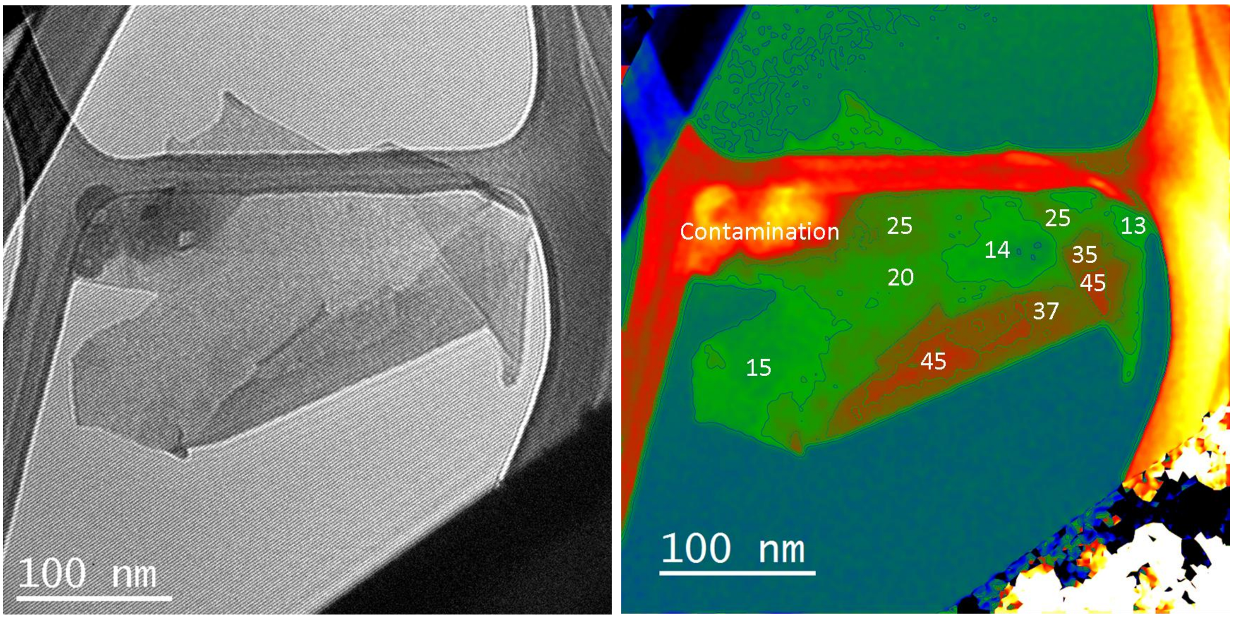

Figure 1: a) Electron hologram of a multi-graphene flake, b) Phase contour map every 10 graphene layers. Reported values represent local measurements.