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IT-11-O-2018 Mapping the number of graphenes for whole micron-size flakes by mean of low voltage electron holography

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Synthesis of graphene with controlled properties is a utopian goal to reach if a multiscale analyzing method is not developed. One of the challenges is to allow accurate counting of monoatomic layers at the nanoscale over a flake area. Therefore, the number of layers as well as their stacking configuration have been related to optical and electrical properties of few-layer graphene.[1]
The recent developments of aberration-corrected transmission electron microscopes (AC-TEM) working at low-voltage (LV) conditions, which limit the knock-on damage, make possible to obtain atomic-resolution information on carbon-based materials.[2-3]
Counting edges of graphene stacks or peeling them under the electron beam provide very local information and cannot be applied to thick stacks. Quantitative thickness mapping can be obtained by combining high angle annular dark field imaging (HAADF) and electron diffraction. HAADF intensity is thickness-dependent and electron diffraction provides a calibration tool by determining the signal of a monolayer related to the TEM settings.[4]
Another way for mapping the number of graphene layers is LV 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 and directly represents the local number of layers.[5]
In the present study, this method is emphasised in the I2TEM machine, a new AC-TEM dedicated to electron holography developed by Hitachi with CEMES Lab. We take advantage of much larger holograms, free of Fresnel fringes keeping irradiation damages limited and still achieving nanometer scale resolution thanks to the unique combination of a double biprism configuration, a second stage unit located upper in the column (Lorentz mode), and the LV with cold field emission. First maps of quantized graphene layers over micronic field of view will be presented. A variety of graphene flakes obtained from CVD or exfoliated graphitewill be analyzed through parameters including stacking type, sample preparation and artifacts of carbon based materials on TEM. Two examples taken from graphite flakes are provided as figure 1 and 2, in which the number of graphenes is large. The method is however sensitive enough for mapping thickness variations related to single graphene.

1 Koshino M. New J. Phys. 15 **2013** 15010
2 Sasaki T., et al. J. Electr. Microsc. 59 **2010** S7
3 Suenaga K. et al. Nature, 468 **2010** 1088
4 Meyer J.C. et al. Solid State Comm., 1–2 **2007** 10
5 Ortolani L. et al. Carbon, 49 **2011** 1423

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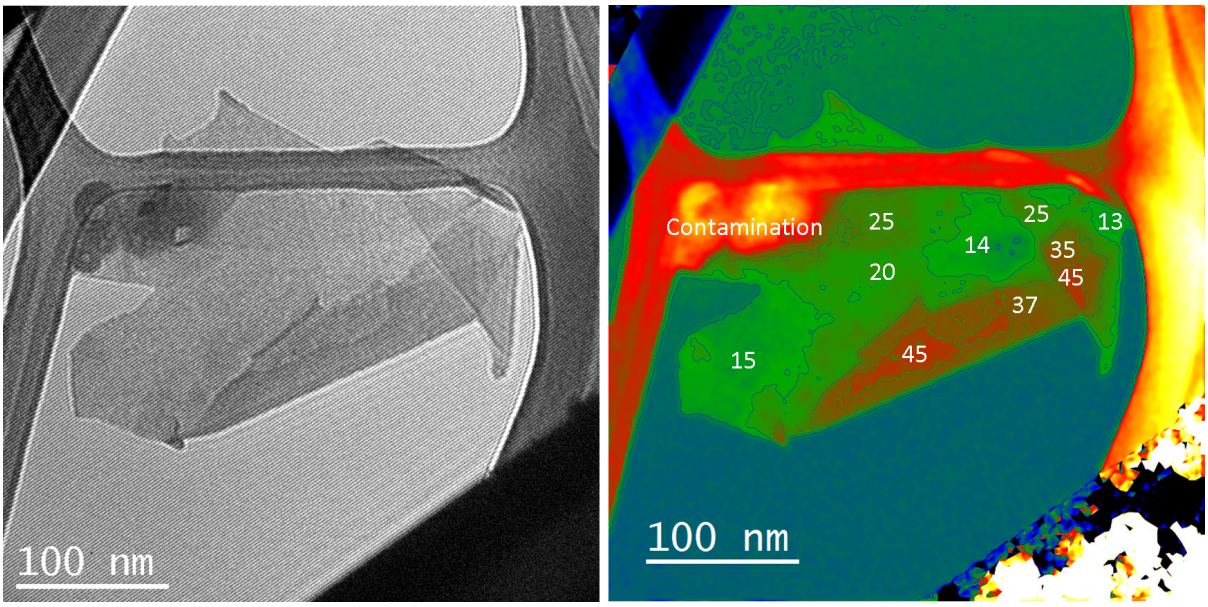

Fig. 1: a. Electron hologram of a multi-graphene and folded flake b. Phase contour map every 10 graphene layers. Reported values represent local measurements.

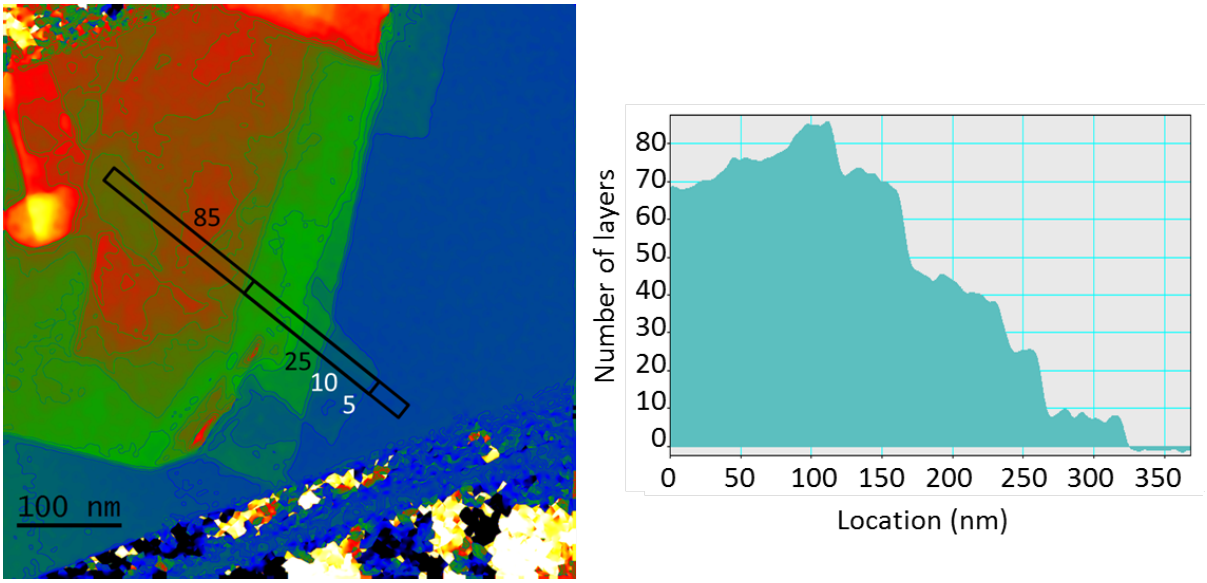

Fig. 2: a. Phase contour map every 10 graphene layers. Reported values represent local measurements. b. Profile of number of layers extracted from figure 2a.