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A High-Resolution Electron Microscopy Study of Dislocations in Graphene Nano-Flakes Produced by Chemical Ultrasonic Exfoliation

Dwight Acosta, Natasha Kramsky, Francisco Hernández and Carlos Magaña

Instituto de Física, Universidad Nacional Autónoma de México, CDMX, A.P.

The presence of edge dislocation in crystalline materials plays an important role in physical properties, mainly in those related to plastic deformation [1]. In graphene and graphene-like materials, and because its 2D nature, dislocations acting as charge scattering and trapping sites might influence their electrical, magnetic and optical properties, which in turn may affect the efficiency of devices developed for practical applications [2].

In this work, we report the synthesis, characterization and study of micro and nano graphene and graphene oxide laminates, obtained by chemical exfoliation assisted by systematic ultrasonic agitation processes. A commercial graphite powder was exhaustively grounded in an agate mortar with a pestle. Small amounts of finely grounded powder were dispersed and sonicated in a surfactant ionic solution. After a centrifugation process, the solution was sprayed onto 200 mesh copper grids covered with a holey carbon film. Raman spectroscopy results let us detect graphene and graphene oxides in our samples. A High-Resolution Electron Microscopy study of graphene materials was performed with a FEG JEOL 2010 electron microscope on some selected samples. HREM micrographs were processed and analyzed with the Digital Micrograph (by GATAN) computing program.

Figure 1a is a typical HREM micrograph of our samples, where individual and overlapped graphene layers, with Moiré fringes and edges, can simultaneously be observed. From a general view of the image it is not easy to detect changes in geometrical order, lattice distance variations, etc. In order to perform a systematic structural study, the micrograph was divided in twelve zones, marked from A to L, of equal size; then, for each zone and in a first instance, Fourier Transform and Inverse Fourier Transform were obtained and analyzed.

Figure 1b, in the upper right side, corresponds to the zone labelled with E in Figure 1a, with its corresponding FT. Figure 1c is a processed HREM micrograph of zone E using the mask displayed in the inset. Figures 2a-c, correspond to IFT images derived from Zone E; the figure display images corresponding to different crystallographic orientations showed in each inset. Lattice resolution images reveal coexistence of a well-ordered lattice together with lattice dislocations. An analysis performed on the 12 zones marked in figure 1a, let us determine an average density of dislocations of 0.218 per square nanometer. This alternative method to determine structural defects number in grapheme flakes might be an important result to be considered for dislocation migration phenomena studies [3,4].

References:

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[3] H.Terrones et al, Rep. Prog. Phys. **75** (2012) p. 062501.
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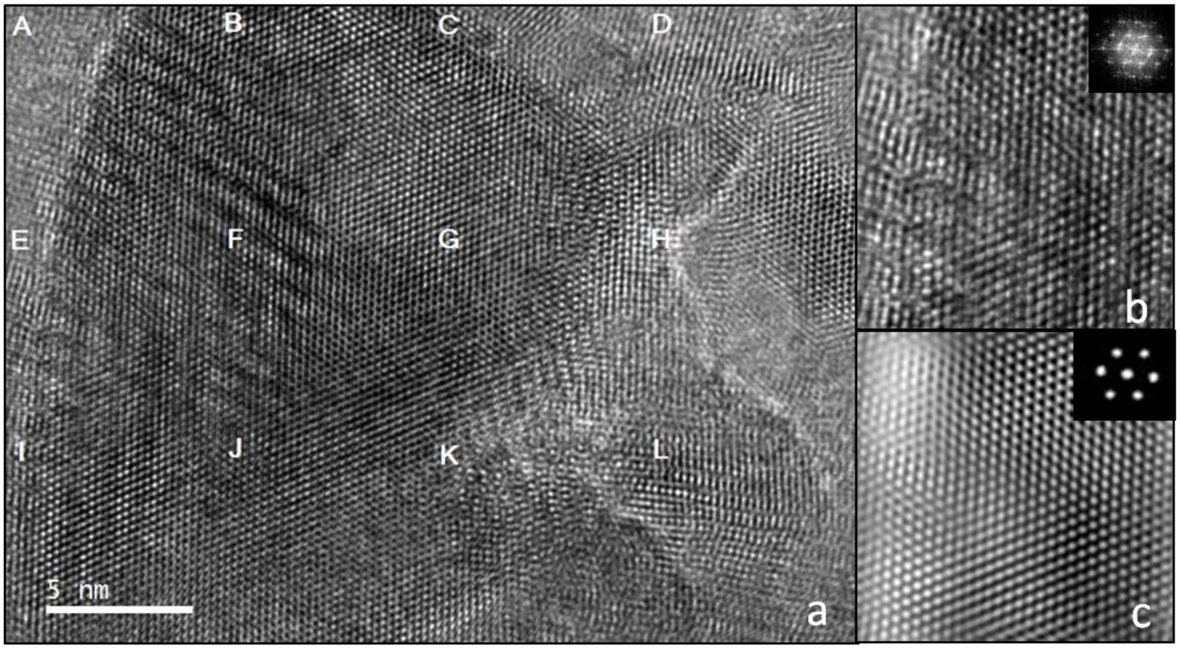
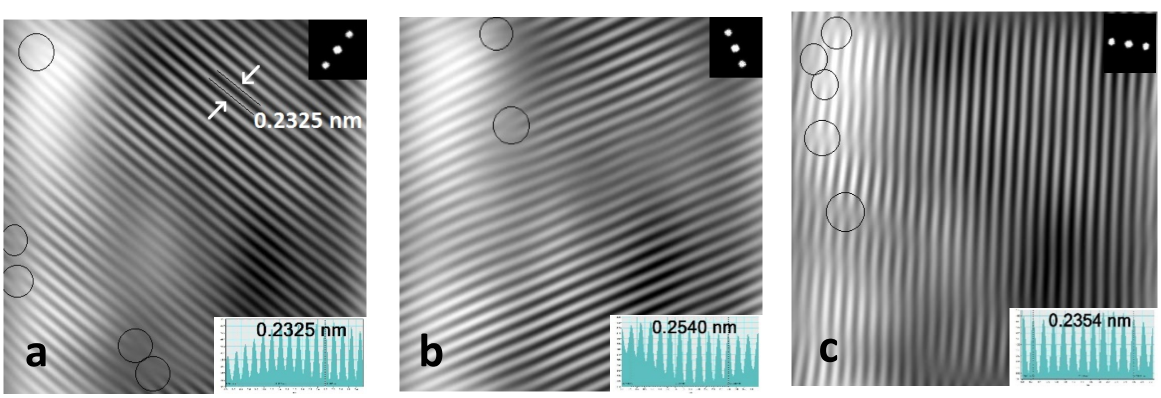


Figures 1a-c. In Fig. 1a, an overlapped graphene laminates array, divided in 12 zones, is displayed in this HREM micrograph. Ordered and distorted configurations can be appreciated along the image. In Fig. 1b the FT in the inset and the IFT from zone marked with E are presented. In Fig. 1c, the IFT obtained from the mask showed in the inset reveals an orderly configuration, together with several structural defects.



Figures 2a-c. In this sequence, we present three IFT micrographs of zone marked with E in Figure 1a with its corresponding FT in each inset. Edge dislocations in black circled zones, planes distortion and ripples can be clearly appreciated along each micrograph.

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