3D reconstruction at the nanoscale of graphene undulations

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Graphene has increasingly attracted interest in the scientific community due to the extraordinary electronic, mechanical and optical properties. These properties are tightly bounded to the electronic structure and strongly modified in the presence of local undulations and/or bending in the structure itself. TEM investigation of freely suspended graphene membranes showed that their surfaces are continuously undulated, over distances of ~ 5 nm, with height variations of less than 1 nm [1]. Moreover, theoretical studies showed that this local curvature of the crystal lattice induces valence charges redistribution, and modulates the electronic and transport properties of the flake [2]. For all there reasons, methods for resolving the 3D structure of graphene flakes are therefore mandatory.

To date, only few experimental results indirectly provide information on the three-dimensional (3D) atomic structure in graphene from the investigation of peaks intensities in electron diffraction patterns [1].

Here we present a novel method to reconstruct the 3D waviness at the nanoscale of 2D crystal (i.e. graphene) from high-resolution transmission electron microscopy (HREM) images. Differently from other approaches, such as electron tomography, our method requires only one HREM micrograph of a crystal flake and relies on the geometrical analysis of the spatial frequencies composing the image to recover 3D information [3].

HREM images contain information on the z-projection of the crystal atomic structure, where the arrangement of crystal planes is imaged as modulated interference fringes in space according to interplanar distances. In Fig. 1 is reported an HREM image of the border of a Few Graphene Crystal (FGC) flake. The membrane is folded over itself on two sides, exposing (0002) fringes, which makes possible to determine the number of layers in the membrane as 3. Moreover from the disposition of the intensity peaks of the folded zone it is possible to determine that the flake has a ABAB stacking sequence. But no information about the height variation of the folded flake is directly provided.

Nevertheless, the sample is observed using an electron beam almost perpendicular to flake surface and the projected atomic positions will appear compressed in the regions where the flake is bended in the z-direction, as shown in the scheme of Fig. 2. Using Geometric Phase Analysis (GPA) [3], a technique analyzing the geometric distortions in the HREM micrograph of a crystal lattice by means of the Fourier analysis of the image, it is possible to achieve a measurement of this compression. The measurement of the apparent compressions will provide the determination of the slope and the local height variations of the FGC flake.

In Fig. 3 are reported the results of the GPA deformation reconstruction for the graphene flake previously shown. Fig. 3 b) shows the reconstructed phase map for the [10-10] direction in false colors. Three regions of significant phase displacement are aligned over the border and indicated by black arrows. The one on the right, partially covered by the indicated region of interest (ROI), is the larger and the most intense, and the line profile of the phase displacement over this region is reported in Fig. 3 c). From this phase displacement map it is possible to easily calculate the associated strain maps, and from the strain profile over the same region the slope of the flake. The final result is reported in Fig. 3 d) where a

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model of the reconstructed structure of the graphene flake in the ROI is reported. The slope of the flake and the height of each stacked layer are calculated accordingly with the described experimental procedure.

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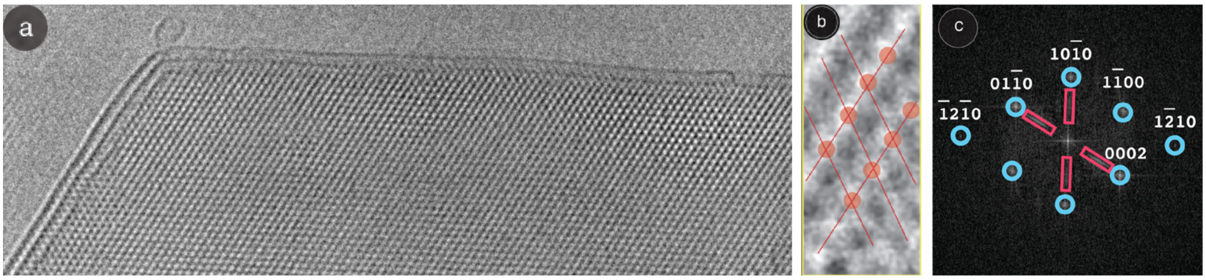

Figure 1. (a) HREM image of the border of a FGC flake. (b) Close-up of the (0002) folded zone, from which it is possible to determine that the flake has a ABAB stacking sequence. (c) Fast Fourier Transform (FFT) of the HREM image, where are clearly visible the graphite reflections (blue circles) and the 0002 reflections of the border (red rectangles).

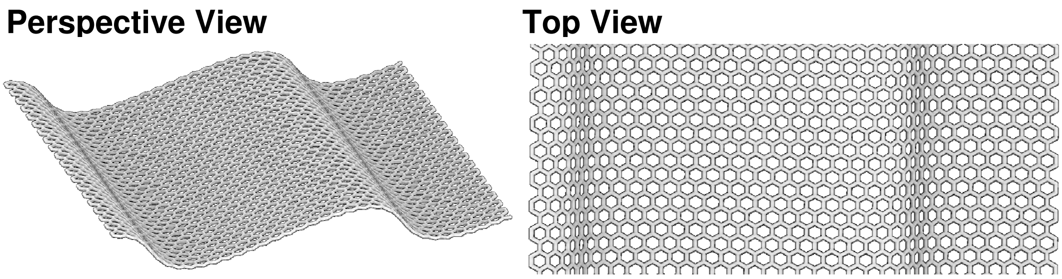

Figure 2. Scheme of a simplified graphene flake with undulations. On the left Perspective View showing the three-dimensional structure, and on the right the two-dimensional Top View where the sloped regions appear compressed.

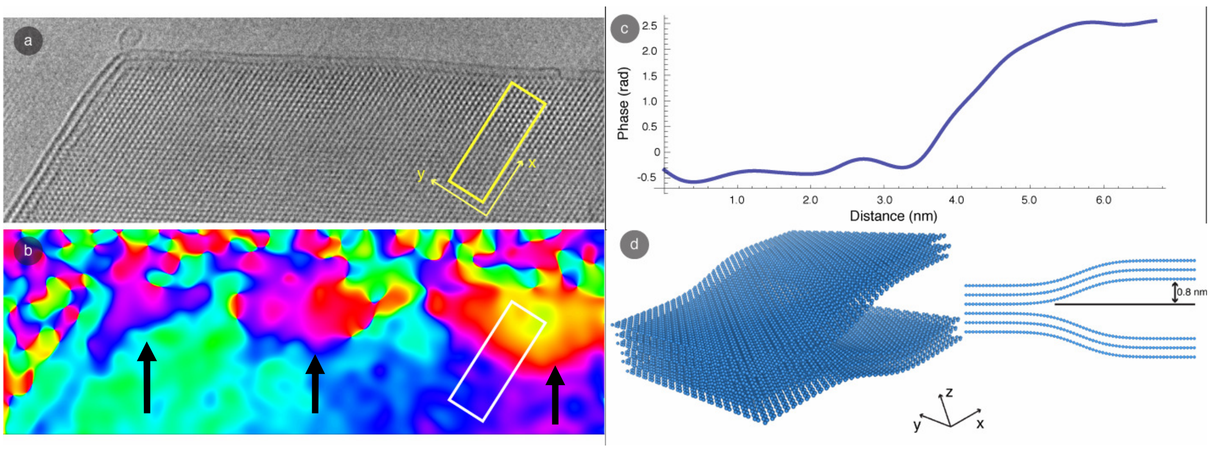

Figure 3. Results of the GPA reconstruction. (a) HREM image of the FGC graphene flake. (b) reconstructed phase map for the [10-10] direction. (c) phase profile in the region marked by the rectangle. (d) schematics of the reconstructed structure of the flake in perspective and lateral view.

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