

Digital pulsed force mode (DPFM) used to probe local mechanical properties of graphene layers

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Abstract – DPFM extends the capabilities of the AFM far beyond the topographical images, allowing to map some mechanical properties in parallel with sample topography. It is a nonresonant mode designed to allow approach curves to be recorded along the scanning path. It thereby provides the topography of the sample and a direct and simple local characterization of adhesion and stiffness. In this work, DPFM was used to probe the mechanical properties of graphene layers. The sample presents a great variety of graphenes with different number of layers. The mechanical properties were analysed according to the number of graphene layers present.

The measurement of nanomechanical properties of materials with AFM has become possible due to the precise control of the microscope tip position and forces applied between the tip and the studied surface [1]. Several AFM operational modes have been developed in the last few years giving new information regarding the materials properties like: phase imaging in tapping mode, force modulation technique and nanoindentation using the AFM tip [2]. The Digital Pulsed Force Mode (DPFM) extends the capabilities of the AFM far beyond the topographical images, allowing to map the local stiffness, adhesion, viscosity and many other mechanical properties in parallel with sample topography [3]. In this mode of operation, the AFM is set to work as in contact mode and a sinusoidal voltage is used to modulate the Z piezo of the AFM. The amplitude of the signal is adjusted such that the tip snaps in and out of contact during each period. Therefore, a complete force-distance curve is measured during each cycle. Initially, the AFM tip is well above the sample surface. Moving closer to the surface, the tip snaps into contact due to the attractive force between tip and surface. As the piezo pushes the tip further toward the sample, the repulsive force reaches a maximum. Then, the piezo pulls back, the repulsive force decreases and the force signal changes from repulsive to attractive. Finally the tip loses contact to the surface when the force on the cantilever is larger than the attractive force between tip and sample (adhesion peak). The subsequent free oscillation of the cantilever is damped and the cantilever oscillates around the baseline. The cycle repeats then. The local adhesion force is determined by the adhesion peak. The local stiffness of the sample is related with the rising slope of the repulsive force signal. In parallel with the topography image, an adhesion map and a local stiffness map are recorded in DPFM operation.

In this work, the DPFM system has been tested using a sample of a multilayer graphene. The sample was obtained by means of mechanical cleavage of HOPG graphite and further deposition on a 300 nm SiO₂ film on top of a silicon wafer. The sample presents a great variety of graphenes with different number of layers whose identification is done both with an optical microscope and Raman spectroscopy. The stiffness and adhesion map of the tested sample were recorded together with the topographical image. The mechanical properties were analysed according to the number of graphene layers present.

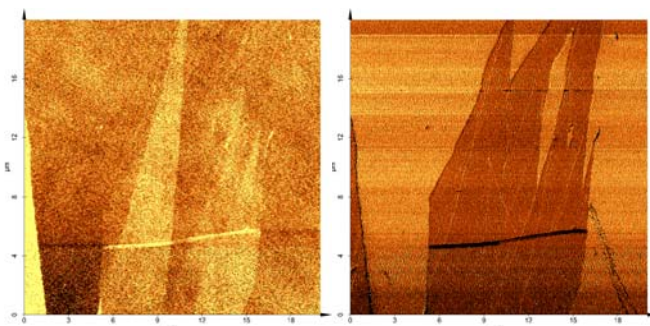


Figure 1: (a) Topographical image of graphene with different number of layers (z scale from 0 to 0.02 μm) and (b) the stiffness map of the same region.

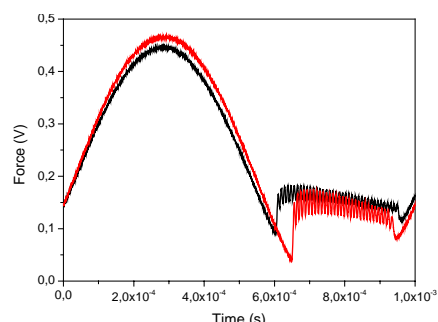


Figure 2: Force curves of a dark and a bright region in figure 1 (b).

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