Measuring the correct thickness of graphene layers by intermittent contact atomic force microscopy

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Since its successful preparation in 2004 [1], research into the properties of graphene has produced a large spectrum of exiting new results in the fields of materials research and condensed matter physics [2]. Right after its preparation, atomic force microscopy (AFM) was employed to check the thickness of the graphene and few layer graphite (FLG) flakes obtained by mechanical exfoliation [1].

AFM is a very effective and easy to use research tool in nanoscience. Primarily in its tapping or intermittent contact mode, AFM is a quick and easy method to check the thickness of individual FLG and graphene crystals. However, it is well known that AFM images are not purely topographic. Depending on the imaging parameters, anomalies may arise in the height measurements of nanosized structures. This effect is not negligible when examining a heterogeneous sample in which subnanometer height differences have to be measured precisely. Heterogeneity is understood in the sense that the sample has more than one type of material in its composition, or the surface has variations in mechanical properties. For example in the case of WO_3 nanoparticles supported on graphite and mica [3] and single walled carbon nanotubes dispersed on a SiO_2 support [4]. Considering that AFM is such a widely used tool in nanoscience and particularly in graphene research, to evaluate the thickness of FLG samples, a more detailed investigation of the anomalies that may arise during the thickness measurement of FLG samples is of great importance.

FLG samples were prepared by the mechanical exfoliation of HOPG onto Si wafers, having a 300 nm layer of SiO_2. Graphene and FLG crystals were selected by optical microscopy and AFM measurements were performed under ambient conditions, using a Veeco Nanoscope IIIA instrument. Following the discovery of graphene, other methods have been developed to confirm the presence of graphene and bilayer graphite, for example Raman spectroscopy. Still, AFM measurements show a very large deviation from the expected value of 0.35 nm thickness for graphene, variations even as large as 1 nm have been reported [1]. We have successfully reproduced such errors in our measurements and have found that the measured thickness is strongly dependent on the free amplitude of the tapping tip and the amplitude setpoint at which the measurement is performed [5]. A discontinuity in the amplitude response of the cantilever is responsible for such a behavior. This itself is a function of measurement parameters and occurs when the tip – sample forces change from net attractive to repulsive. Measurements in the attractive regime regularly give erroneous results and this artifact can be responsible for an increase in the thickness of graphene by about 1 nm, which is consistent with the measurement results reported in the literature.

When carrying out intermittent contact AFM measurements on graphene, great care should be taken in choosing the free amplitude and amplitude setpoint of the measurement. Our work sheds some light on how a more precise estimate of the number of layers in a FLG

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crystal can be obtained, giving significant insight into how AFM can be used to characterize graphene on SiO_2 substrates and other heterogeneous samples.

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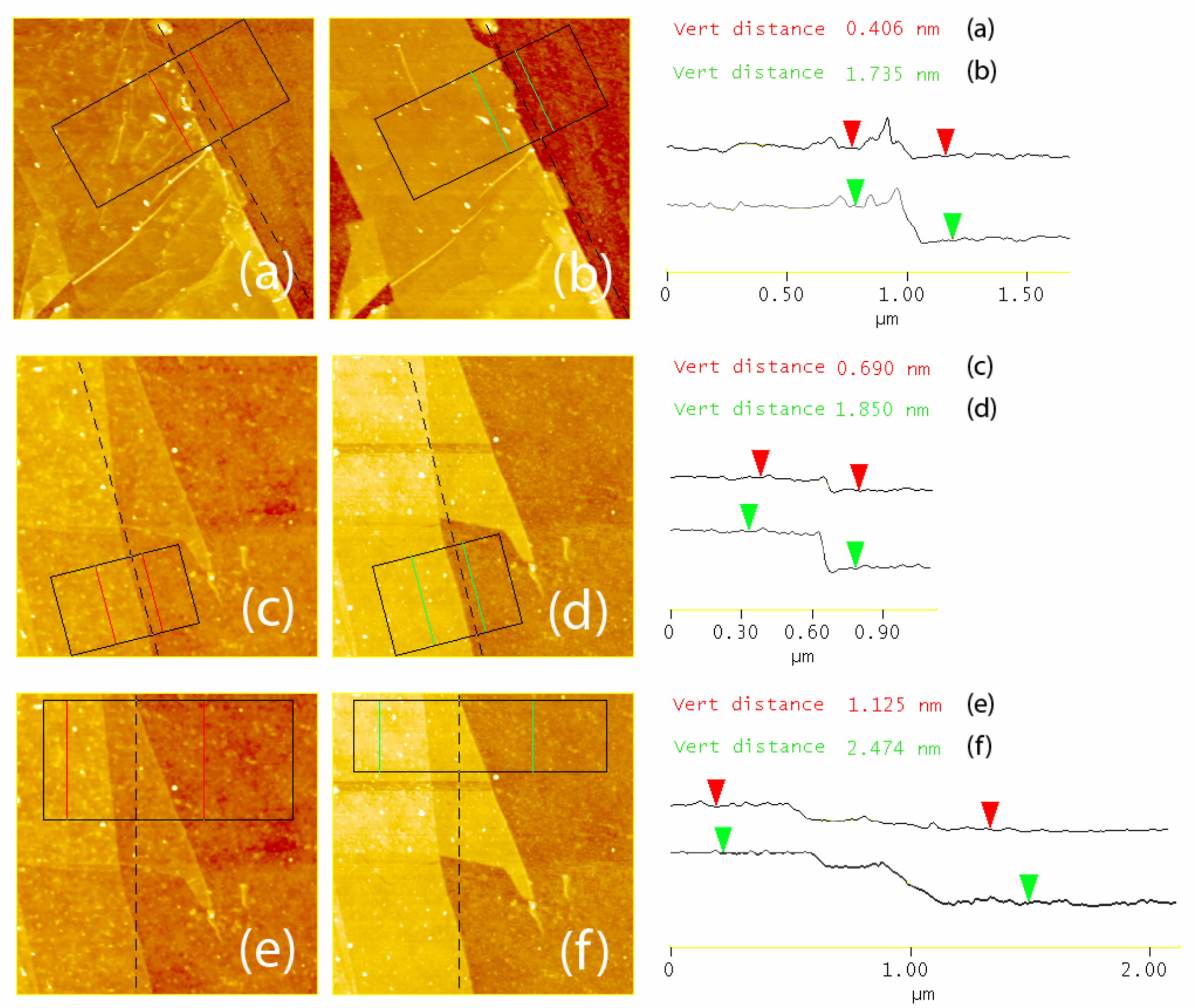

Figure. AFM measurements on graphene (a, b), bilayer (c, d) and three layer graphite (e, f) at large (a, c, e) and small (b, d, f) free amplitudes of the cantilever. The thickness of the layers was also checked by Raman spectroscopy. The thickness measured by AFM changes with changing free amplitude and it can be seen that at large free amplitudes a much better correlation can be found with the expected thickness of the respective layers.