Application Note
AN.KFM.092017

High Definiton Kelvin Force Microscopy

BIMODAL HD-KFM

INTRODUCTION

Since the invention of the Atomic Force Microscope in 1986, many different measurement modes have been proposed to characterize other interaction forces or surface properties in addition to the topography.
One of the most interesting applications of AFM is nano-electric characterization, this is, the capability to measure electric properties with nanometric lateral resolution. This is of great interest for the development of so called nano-materials. The emergence of 2-D materials like graphene, molybdenum disulphide or black phosphorous has created an interest in characterizing and understanding the electrical properties of flakes with one or multiple layers. In the field of solar cells and piezoelectric materials, there is also a growing interest in perovskite type materials as they show promising percentages of light efficiency conversion. There are also other fields of materials science or biology, where nano-electrical properties are of high interest.
One of the most popular electrical modes of AFM is the Kelvin Force Mode (KFM). This technique allows measurement of the surface potential between the tip of the AFM and the surface which can be related to material properties like the work function or the bandgap:

$$V_{POTENTIAL} = \frac{\Delta\varphi}{e} = \frac{\varphi_{tip} - \varphi_{sample}}{e}$$

HD-KFM.THE MOST ADVANCED SINGLE-PASS KFM MODE

On the contrary to other single-pass setups, Bimodal HD-KFM is characterized because the electric feedback is tuned to the second eigenmode frequency of the cantilever. Figure 1 shows the concept on which HD-KFM is based: the first flexural eigenmode of the cantilever is excited mechanically and the second flexural mode is excited electrically. In the past decade, multi-frequency approach has become one of the front-end topics amongst the AFM community as it has shown many potential applications.

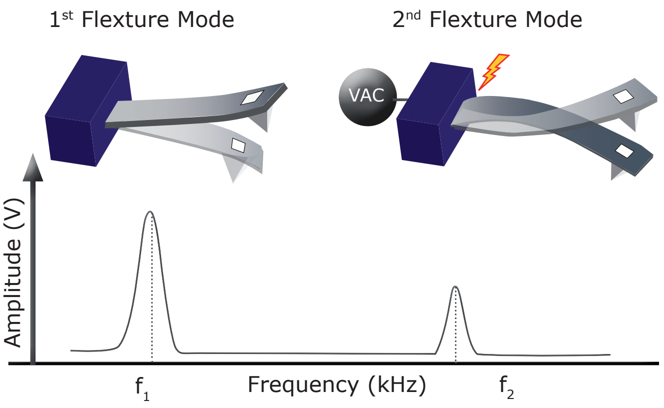

Figure 1 : Concept of Bimodal HD-KFM: first flexural mode (or eigenmode) frequency is excited mechanically to measure topography, while the second flexural mode frequency is excited electrically to measure surface potential.

The advantages of tuning the VAC bias used for the electric feedback to the second eigenmode of the cantilever is that the signal is amplified by the Q factor of the second eigenmode. This effect provides the possibility to use smaller VAC values to obtain an oscillation amplitude with an acceptable signal to noise ratio as compared to other implementations not-based on the second eigenmode amplification. Additionally, stiffer effective spring constant of the second eigenmode provides more stability of the oscillation during the scanning of the surface.

BIMODAL HD-KFM. HIGH SENSITIVITY AND ROBUSTNESS

Graphene has emerged as a nanomaterial for the future due to its bidimensional structure and electronic properties. However, synthesis and transfer processes are not easy to implement especially on large areas where a single layer of graphene may have impurities or flakes of multiple layers of graphene. Although Raman spectroscopy is most commonly used to characterize monolayers of graphene, KFM measurements have also proved to be useful to distinguish between single monolayer and multiple layers of graphene.

Figure 2 shows both topography (Fig. 2a) and HD-KFM surface potential images (Fig 2b) of a monolayer of graphene transferred onto a Si/SiO2 substrate. In the topographic image, the presence of two flakes with 1 and 2 additional layers of graphene can be seen; however the presence of contaminants in the transfer process makes it difficult to clearly resolve the height of the flakes over the surface. In the corresponding surface potential image (2b) the flakes are clearly resolved against the monolayer substrate. The areas with an additional layer have a potential difference of 90 mV while the bilayer areas (brighter zones in the centre of the flakes) have a potential difference of 120 mV respect the graphene monolayer substrate.

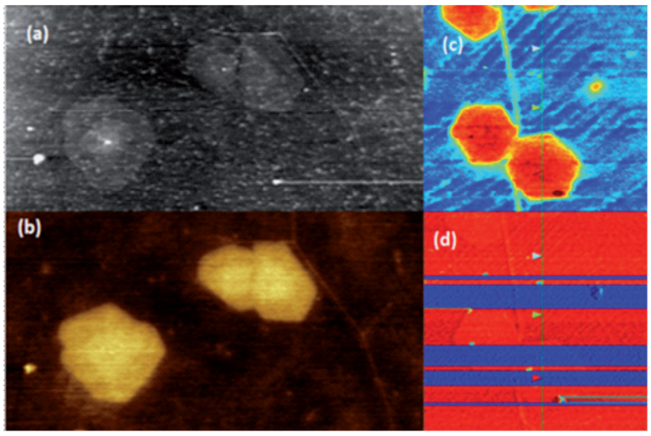

Figure 2 : Topgography (a) and surface potential (b) of a monolayer substrate with some bilayer and trilayer flakes. Surface potential provides a better contrast than topography to localize the flakes. Figs 4(c) and 4(d), surface potential and phase images of the same graphene sample on a different area. Red areas in the phase image corresponds to the cantilever oscillating in an attractive regime, while blue areas correspond to an oscillation in a repulsive regime.

Another important feature of HD-KFM is the robustness of the electric feedback loop which is enhanced by the coupling with the second eigenmode resonance of the cantilever. In figure (2c) and (2d) are depicted both surface potential and phase image of the same graphene sample described above. Phase image is related to the first eigenmode of the cantilever used for the acquisition of the topographic image and it is related to the dissipated energy per cycle of the cantilever when interacting with the substrate. In the case of hard samples, the phase image shows a discontinuous jump in the phase values when there is a transition between the non-contact (nc) regime to the intermittent contact (ic) regime. This can clearly be seen on the phase image as the red areas correspond to the cantilever oscillating in the nc-regime and the blue areas to the ic-regime. In the image shown, ic-regime was tuned by decreasing the setpoint amplitude of the cantilever; the nc-regime was recovered by increasing the setpoint amplitude. However, this situation can occur spontaneously if some contaminate adheres to the cantilever tip while imaging a surface. In the ic-regime areas it can clearly be seen on the surface potential image that the surface potential image keeps the stability although some resolution is lost due to the mechanical contact. The high resolution is recovered when the nc-regime is tuned again.

BIMODAL HD-KFM. HIGH ACCURACY AND SENSITIVITY TO DIFFERENT MATERIALS

In order to test the accuracy of the surface potential values obtained with Bimodal HD-KFM, a standard sample test for KFM was imaged (see Figure 3). This sample consists of a silicon substrate with two different metals (Gold and Aluminum) regions of 100 nm height defined by lithography. It is easy to find a region where three different materials are exposed (Gold-Silicon-Aluminum) thus, defining clear regions for the surface potential image. Figure 3 shows both topography (upper) and SP images and a cross-section (middle and bottom). It is important to notice that the SP cross-section (white line) shows no bending due to capacitive influences of tip and cantilever or averaging with the surrounding areas (Au and Al regions are 100 nm high). Even more, HD-KFM shows its high sensitivity as an intermediate region between silicon and Aluminum can be resolved. This region might be an effect of the lithography process or the fabrication of the sample as the topography reveals that the Aluminum area is not as flat as the gold area. The differences in the SP values between the Au-Si-Al areas are consistent with standard values of the work function for each material.

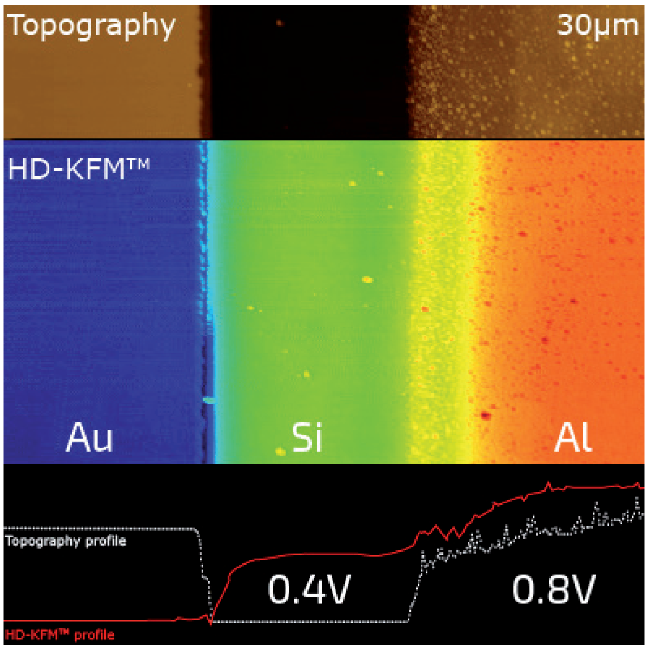

Figure 3 : Topography (top), surface potential (middle) and cross-section (bottom) High sensitivity of bimodal HD-KFM reveals a transition region between silicon and aluminum. Values are in good agreement with the bulk values of work functions.

As mentioned above, Bimodal HD-KFM has a higher sensitivity due to the capability to probe the electric field very close to the surface. This is of vital importance when probing the SP of small objects like carbon nanotubes. In the example shown in Figure 4, a sample of carbon nanotubes was deposited on a silicon substrate by dropcasting from a diluted solution. The length of the carbon nanotubes ranges from 1-10 microns randomly oriented over the surface. The SP corresponding to the silicon surface is 295 mV, while for the corresponding nanotubes it drops to 39 mV as shown in Figure 4(right). The apparent width of the carbon nanotubes in the SP image is 30 nm, which is due to a convolution effect of the conductive tip. Additionally it can be seen the presence of small contaminants on top of the carbon nanotubes as shown in Figure 4(left). This can obscure the SP signal for high concentration (red arrows) but not for small concentrations (blue arrows). Even in the worst case, with Bimodal HD-KFM the signal is not completely lost. This signal could not be probed by using a double-pass setup as the signal would be lost with the lift separation.

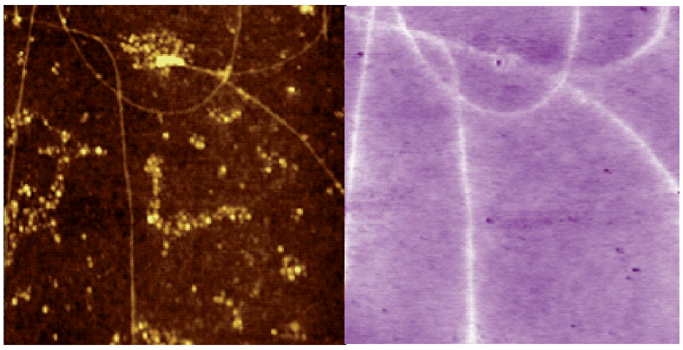

Figure 4 : Topography (left) and surface potential (right) of a dropcasted sample of nanotubes. HD-KFM is sensitive enough to probe the surface potential signal of the nanotube even with the presence of contaminants

Organic molecules like fluoroalkanes F14H20 can also be characterized by HD-KFM. A sample of these polar molecules was prepared by spin cast of a small droplet dissolved in perfluorodecalin on a silicon substrate. By controlling the concentration, they are adsorbed onto the surface forming small toroids of few nanometres diameter. Figure 5 shows both topography (left) and SP (right) images. HD-KFM show a difference in the surface potential of -0.2 volts respect to the silicon substrate, which results from its well-defined dipole between the fluorinated parts with negative charge and carbon parts with positive charge. Although this value has been reported higher in the literature, it can be attributed to a deformation of the structure of the chain, so that the effective dipoles are tilted. A negative value indicates that the fluorinated parts are facing the tip. Also, the difference in lateral resolution of the individual molecules in both topography and SP images can be seen. The apparent size in topography is about 49.3 nm average, while in the SP image it is 64.7 nm. This can be attributed to the fact that the tip radius of the cantilever used in the experiments is about 20-30 nm. However, the topography image is based on the surface interaction which is more localized than the electric interaction. That is why the inner part of the donut-shaped molecules is resolved in topography, while in the SP image it is not resolved. Also in the molecules that are closer, there is a small increase of the signal in the borders (tiny white lines along the contour). This is due to the addition of the electric signal of dipoles of two adjacent molecules (see the screen in the inset of Figure 5(b).

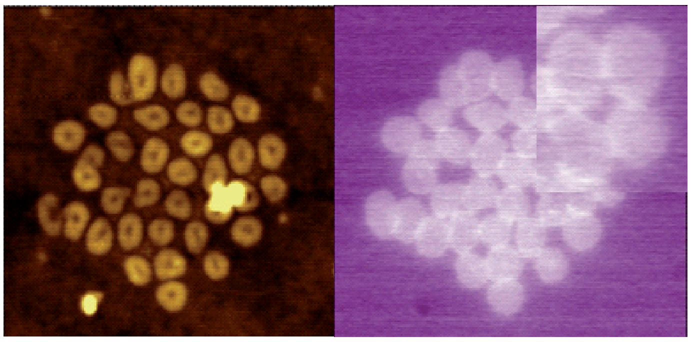

Figure 5 : (left) topography and (right) surface potential of fluoroalkenes molecules. Topography shows higher lateral resolution due to capacitive effects of the tip which enlarge the effective interaction area.

BIMODAL HD-KFM ON ELECTRONIC DEVICES AND SOLAR CELLS

Figure 6 shows an electric characterization of a perovskite solar cell made with HD-KFM mode that has not been polished before measurement. Figures 6 (left and right) shows a 3D image of the cleaved SC with a color overlay of surface potential values (more details can be obtained in reference 5) .To our knowledge no other publication has shown a surface potential profile of the active layers in this conditions (not polished). Bimodal HD-KFM is the only commercially available mode robust and sensitive enough to provide reliable surface potential measurements even on a sample with high topographic variations.

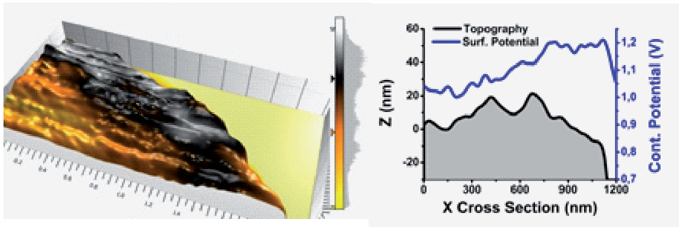

Figure 6 : (left) lateral view of the positioning of the cantilever on the edge of the solar cell. (middle) 3D reconstruction of the edge of the solar cell with coloured overlay of the surface potential. (right) cross-section of the edge of the solar cell.

It is also interesting to measure a solar cell that has been polished as shown in Figure 7 where both topography (Fig.7-top) and surface potential (Fig 7-middle) are shown. Some different layers can be seen on topography despite the polishing process, however, surface potential shows the actual position of the active layers. Moreover, on the right side of the surface potential image it can be seen some defects in the active layers that could be due to the polishing process (red arrows in the surface potential image).This small defects are about 50-70 nm size, showing the high resolution provided by the bimodal HD-KFM. It is interesting to compare the cross-section in Fig. 6(right) with the cross-section in Fig. 7 (bottom): the process of polishing allows to obtain a smoothed signal, however the overall behavior is not that different, thus showing that HD-KFM can be reliable even if there are high roughness variations.

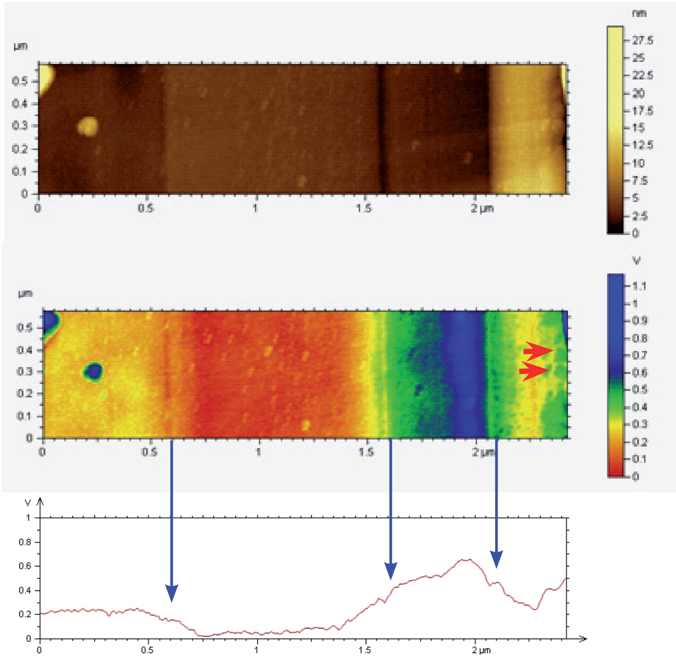

Figure 7 : Topography(top) and surface potential(middle) of a polished solar cell.(bottom) SP cross-section.

BIBLIOGRAPHY

1.D.W. Abraham, C. Williams, J. Slinkman, H.K. Wickramasinghe, J. Vac. Sci. Technol. B 9,703 (1991)

2. T.R. Albrecht, P. Gr¨utter, D. Horne, D. Rugar, J. Appl. Phys. 69, 668 (1991)

3. T. Glatzel, S. Sadewasser, R. Shikler, Y. Rosenwaks, and M. C. Lux-Steiner, Mater. Sci. Eng. B 102(1–3), 138–142 (2003).

4. J. Colchero, A. Gil, A.M. Bar´o, Phys. Rev. B 64, 245403 (2001).

5. A. Liscio, V. Palermo, and P. Samorı, Adv. Funct. Mater. 18(6), 907–914 (2008).

2017 CSInstruments Corporation. All rights reserved.
Nano-Observer AFM, HD-KFM mode,
HD-KFM is trademarks of CSInstruments Corporation.