

Keysight Technologies

Quantitative Surface Potential Measurement Using KFM: Effects of Imaging Parameters and Experimental Conditions

Application Note

Scanning kelvin force microscopy (KFM) has been widely used in mapping surface potential (SP) distribution at the nanoscale[1]. There are two different approaches in KFM imaging, i.e., Lift Mode and Single-Pass. In Lift Mode, topography and SP images are acquired in two different scans: the topography is obtained first and the tip then lifted to a certain distance above the topography for a second scan to measure SP. In Single-Pass, topography and SP are acquired simultaneously using a multi-frequency technique: the cantilever is excited simultaneously with two ac modulation signals at different frequencies, the first one is used for mechanical excitation to measure topography and the second one, usually with a much lower frequency, is for electric modulation to measure SP. Single-Pass KFM is implemented in Keysight Technologies, Inc. AFM systems using a high performance triple lock-in AC Mode controller. Detailed instrumentation and discussion of Single-Pass KFM techniques can be found in other Keysight application notes[2,3]. KFM contrast reveals important information about surface charging, molecular

dipole orientation in organic thin films, band bending and dopant concentration in semiconductor materials, etc. There are also reports that use KFM to measure the work function of a conducting material, e.g., the change in work function with film thickness of few-layer graphene[4-6]. However, the reported values measured by KFM are not always consistent in literature. This note will discuss a number of experimental parameters that have significant effect on the accuracy and resolution of SP measurements.

The principle of KFM is based on the measurement of electrostatic forces between the tip and the surface. When a dc bias (V_{dc}) and a small ac modulation signal $V_{ac}\sin \omega t$ are applied between the tip and the sample, the induced capacitive force is

$$\begin{aligned} F(z) &= F_{dc} + F_{\omega} = F_{2\omega} \\ &= -\frac{1}{2} \frac{\partial C}{\partial z} [(V_{dc} - \phi)^2 + \frac{1}{2} V_{ac}^2] \\ &\quad - \frac{\partial C}{\partial z} (V_{dc} - \phi) V_{ac} \sin \omega t \\ &\quad + \frac{1}{4} \frac{\partial C}{\partial z} V_{ac}^2 \cos 2\omega t \end{aligned} \quad (1)$$

where ϕ is the contact potential difference (CPD) between the tip and the sample. It is evident from the F_{ω} term in Equation (1) that F_{ω} depends linearly on V_{dc} and becomes zero when $V_{dc} = \phi$. It seems that SP can be measured directly by nullifying F_{ω} , independent of tip-sample distance and V_{ac} . Since SP is measured here by nullifying the amplitude of the F_{ω} , it is named AM-KFM, meaning amplitude sensitive. Alternatively, SP can be measured by nullifying the resonance frequency shift, Δf_{ω} , caused by the ac modulation (FM-KFM),

$$\Delta f_{\omega} = -\frac{f_0}{2k} F'_{\omega} = -\frac{f_0}{2k} \frac{\partial^2 C}{\partial z^2} (V_{dc} - \phi) V_{ac} \sin \omega t, \quad (2)$$

where f_0 and k are the resonance frequency and spring constant of the cantilever, respectively. A plot of F_{ω} measured against the sample bias is presented in Figure 1. It shows that the force approaches to zero as the bias approaches the CPD. It also indicates the curve is linear in a certain range of bias, as predicted by the formula in Equation 1. The linearity break down when the bias is large enough to cause the servo to pull the cantilever away from the surface. A careful examination of the plot will reveal, however, that the force never actually

reaches zero, instead it reaches a certain minimum value δ at a bias close to CPD. Consequently, the SP measured now has an error that depends on z and V_{ac} :

$$V_{dc} = \phi - \delta_{AM} / \left(\frac{\partial C}{\partial z} V_{ac} \right) = \phi - \phi_{error, AM}(z, V_{ac}) \quad (3)$$

for AM-KFM, and similarly

$$V_{dc} = \phi - \delta_{FM} / \left(\frac{d_0}{2k} \frac{\partial^2 C}{\partial z^2} V_{ac} \right) = \phi - \phi_{error, FM}(z, V_{ac}) \quad (4)$$

for FM-KFM. Figure 2 presents the surface potential measured on HOPG as a function of tip-sample distance. It shows the measured SP increases monotonically as the tip moves closer to the sample surface. There have been a number of efforts to understand the effects of experimental parameters on the errors involved in KFM measurement, based on simulations from sophisticated capacitance models and experimental measurements [7-11]. A set of significant factors that affect the resolution and accuracy of KFM measurement are discussed below.

FM-KFM vs AM-KFM

As shown by Equation 1, AM-KFM serves on the electrostatic force between the cantilever and the sample. The total force includes contributions, however, from the tip apex, the tip cone, and the cantilever beam [7]. Since the cone and the lever are 'seeing' over a relatively large area on the surface, they sense an averaged 'global' surface potential that is different from the local SP sensed by the tip apex. Unfortunately, the overall force becomes dominated by the cone and lever and remains almost 'constant' when the tip is only a couple of nm away. Consequently, SP measured with AM-KFM are often averaged and have a large error below the true contact potential difference of the tip-sample interface.

On the other hand, FM-KFM monitors the gradient of the electrostatic force, as shown by Equation 2. By using the force gradient, most of the effects of the cantilever beam and the tip cone are derived away, the measurement is thus determined by the interaction between the tip apex and the sample surface. Therefore, FM-KFM measures more accurately the 'local' surface potential than the averaged 'global' surface potential. In other words, FM-

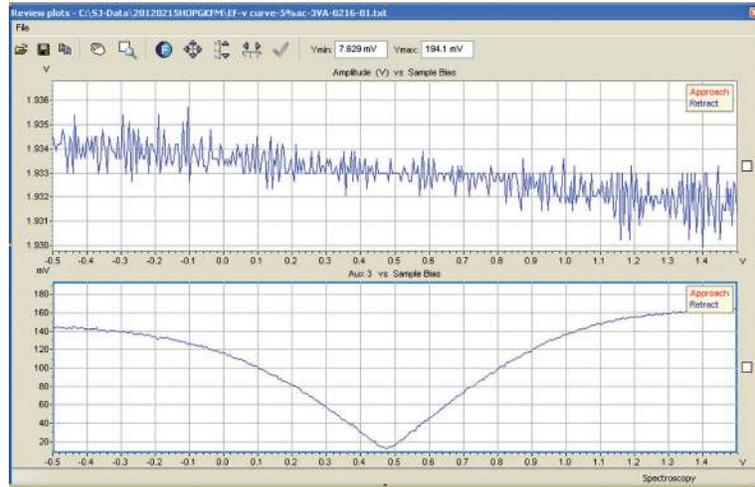


Figure 1. Mechanical oscillation and EFM F_{ω} amplitudes plotted against sample bias.

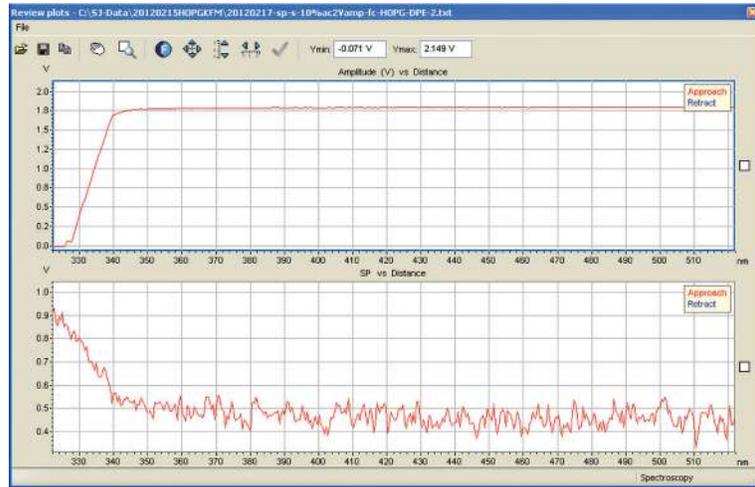


Figure 2. Mechanical oscillation amplitude and surface potential vs tip-sample distance plot.

KFM gives both higher accuracy of the measured potential value and better lateral resolution for surface features of nanometer scale.

Because the force gradient dies off quickly with increasing tip-sample distance, stable measurements are acquired only when the tip is very close to the surface, FM-KFM becomes unstable when the tip is more than 30 nm away from the surface, as demonstrated by Zerweck et al. in vacuum [8]. In ambient conditions, the distance could be larger due to water layers on the surface. With AM-KFM, stable measurements can be performed over a much larger z range because the electrostatic force extends longer distance than its gradient. Liscio et al. reported SP measurements with the tip was over 200 nm away from the surface [9].

Tip-Sample Distance

In general, the measured SP improves in accuracy as the tip-sample distance decreases, as shown in Figure 2. This has been confirmed by both numerical simulations and experimental measurements [8,10]. A simple look over Equation 3 and 4 can give the same conclusion. This is because both $(\partial C / \partial z)$ and $(\partial^2 C / \partial z^2)$ will increase in value as z decreases, thus lowering the error item in the equation. With FM-KFM, measurement can only be done when the tip is close to the surface (< 30 nm), and the measurement are usually more accurate with higher lateral resolution. With AM-KFM, however, the measured SP is always smaller than the real value even when the tip is really close to the surface.

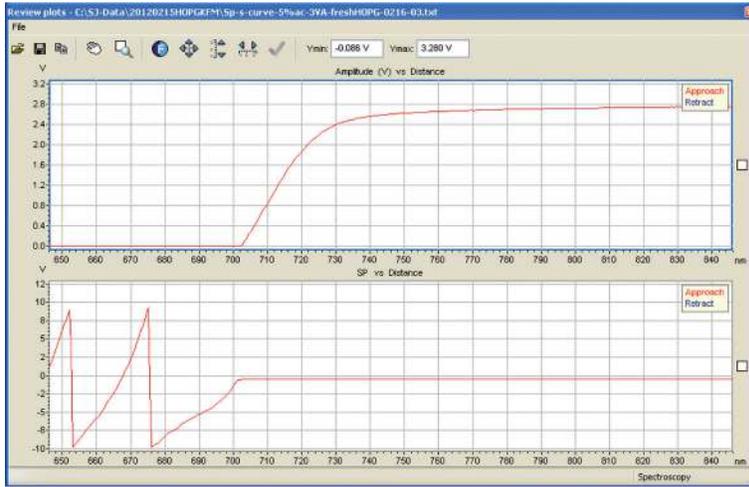


Figure 3. Mechanical oscillation amplitude and surface potential vs tip-sample distance plot, tip modification caused by crashing into the surface results in faulty measurements in surface potential.

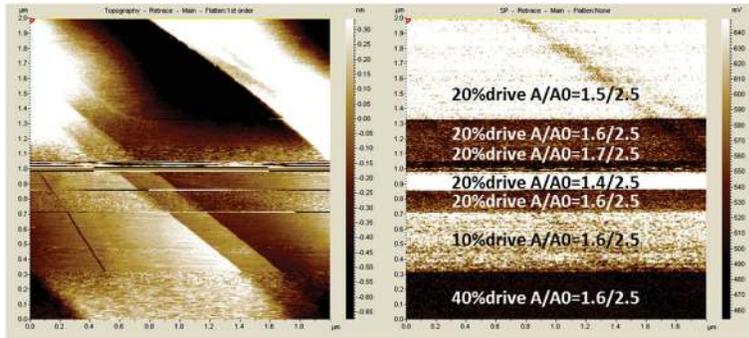


Figure 4. Effects of mechanical oscillation amplitude (top half with fixed EFM modulation at 20% drive and varying A/A_0) and ac modulation amplitude (bottom half with fixed A/A_0 and varying EFM modulation drive amplitude) on KFM imaging.

As shown in Figure 2, SP measured with AM-KFM reaches an asymptotic region as the tip becomes far from the surface, and the SP value measured in this region accounts only a certain percentage of the real value [9].

It is worth mentioning that even though the measurement become more accurate when the tip and sample get closer, one needs to take extra care not to crash the tip into the surface. This is because the accuracy of the measurement also depends on the consistency of the tip condition. Once the tip is modified, the SP value measured will be changed, so dramatically sometimes that recovery is almost impossible (See Figure 3).

AC Modulation Amplitude

Since the error in the KFM measurement is inversely dependent on the

V_{ac} as shown in Equations 3 and 4, therefore the larger the modulation amplitude the smaller the error. However, as V_{ac} increases, the tip oscillation amplitude induced by V_{ac} will also increase. When the tip oscillation can not be neglected, the F_{ω} will become more complex and time dependent. Consequently the fixed tip-sample distance approximation that leads to Equations 1 and 2 will no longer be valid. From the experiment point view, increase in V_{ac} will increase the F_{ω} , thus improve the signal-to-noise ratio of the SP measurement. However, a large V_{ac} tends to push the cantilever away from the surface resulting a lower SP and introduce more cross-talk to topography, see the lower part of Figure 4 where the setpoint and free amplitude of the mechanical oscillation (A/A_0) is held constant while the ac modulation is varied from 1 to 2, and to 4V peak-to-peak. Furthermore, large ac modulation can cause damage to sensitive samples. For example,

minimum bias is necessary for some semiconductor samples to prevent tip induced band-bending and possible breakdown of the structure.

Mechanical Oscillation Amplitude

In Single-Pass mode, the topography and SP are acquired simultaneously. The mechanical oscillation of the cantilever is usually at a much higher frequency than the KFM modulation, therefore the tip can be seen as kept at a 'static' distance above the surface by the mechanical oscillation. The effect of mechanical oscillation amplitude on KFM measurement is debatable. Zerweck et al. showed the accuracy by FM-KFM in vacuum will decrease with increasing mechanical oscillation amplitude, largely due to the average tip-sample distance become larger with higher amplitude [8]. In the top part of Figure 4, the ac modulation amplitude was kept to 2V while the A/A_0 was varied. The SP measured decreases with increasing A/A_0 . In practice, one should minimize the cross-talk by keeping the frequencies of the two excitation signal as far apart as possible. For example use a higher flexure mode for mechanical excitation, and a lower frequency for AM-KFM. For FM-KFM, use a stiffer cantilever for topography and a low frequency for KFM.

Relative Humidity

From Equation 1 and 2, the CPD and consequently the work function of the sample can be measured quantitatively with a calibrated tip of known work function. However, measured SP by KFM does not always agree with work function values obtained by other techniques, particularly those measurements performed in an ambient condition. This is because the SP measured depends strongly on the conditions of the surface and the cantilever. The effect of tip condition has already been mentioned earlier. Another major factor affecting the SP value is the relative humidity level in the imaging environment. Electric dipoles of the adsorbed water layer not only screens the surface of the sample,

but could also have a significant effect on the extremely hydrophilic Pt/Ir coated cantilever often used for KFM imaging. Therefore caution is necessary for quantitative interpretation of KFM measurements in ambient.

In summary, we have discussed a number of factors that are important for KFM measurements in this short note. A good understanding of those parameters will definitely help one to improve the use of KFM as a powerful technique for localized surface potential measurement, for the characterization of electronic properties of a wide range of materials including metal, polymer, organic film, semiconductor, and organic/inorganic composites. Figure 5 shows a KFM image that reveals the minute surface potential difference at the edge of a single graphite layer on HOPG.

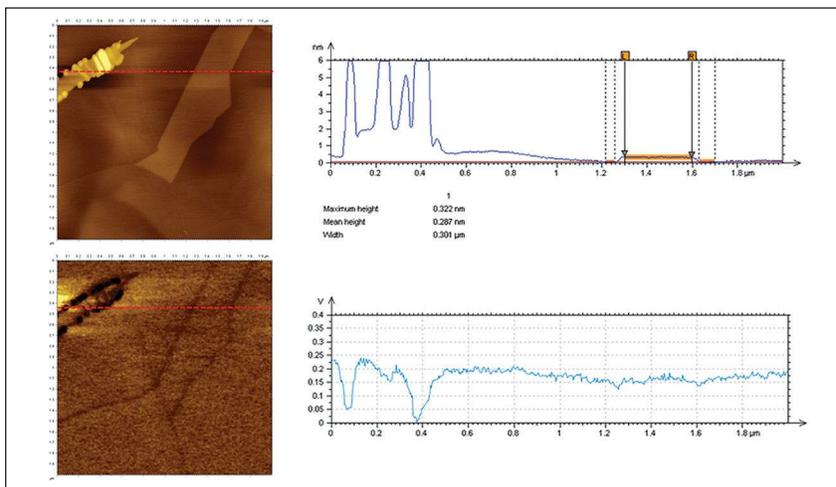


Figure 5. High resolution KFM imaging that revealed the topography of a single HOPG layer and the surface potential difference at the edge of the atomic step.

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