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Quantitative comparison of closed-loop and dual harmonic Kelvin probe force microscopy techniques

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Kelvin probe force microscopy (KPFM) is a widely used technique to map surface potentials at the nanometer scale. In traditional KPFM, a feedback loop regulates the DC bias applied between a sharp conductive probe and a sample to nullify the electrostatic force (closed-loop operation). In comparison, open-loop techniques such as dual harmonic KPFM (DH-KPFM) are simpler to implement, are less sensitive to artefacts, offer the unique ability to probe voltage sensitive materials, and operate in liquid environments. Here, we directly compare the two techniques in terms of their bandwidth and sensitivity to instrumentation artefacts. Furthermore, we introduce a new correction for traditional KPFM termed “setpoint correction,” which allows us to obtain agreement between open and closed-loop techniques within 1%. Quantitative validation of DH-KPFM may lead to a wider adoption of open-loop KPFM techniques by the scanning probe community. Published by AIP Publishing. https://doi.org/10.1063/1.5025432

I. INTRODUCTION

Atomic force microscope (AFM) based Kelvin probe force microscopy (KPFM) is an enabling technique which allows surface potential mapping at the nanometer scale. The contact potential difference (CPD) measured by KPFM can be used to fully quantify a sample’s local electronic properties (e.g., work function) under certain conditions (e.g., vacuum), provided the influence of the environment is minimized and the tip properties are known.1 More generally, AFM based KPFM in ambient environments may be influenced by sample preparation (e.g., surface oxidation or adsorbed dielectric layers2), by the environment (e.g., humidity3), and also by instrumentation (e.g., electronic offsets and electronic cross talk) as well as probe geometry.4,5 These effects combine to make traditional KPFM in ambient environments an accurate and useful technique for relative surface potential mapping, but it is often difficult to obtain the true electronic properties of the system.6,7

Traditional KPFM is a closed-loop KPFM (CL-KPFM) technique where a feedback loop is used to apply a DC bias to compensate the electrostatic force between a tip and a sample.8 Open-loop KPFM (OL-KPFM) techniques are increasingly being adopted as feedback-free methods,7,9,18–20,10–17 eliminating the need for the application of a DC bias8 and enabling the mapping of voltage-sensitive materials9,15,21,22 and surface potentials in liquid environments.10–12,16,17,23–25 These techniques are also of interest for their reduced sensitivity to electronic offsets and electronic cross talk instrumentation issues,4,7,32–35,13,22,26–31 which can affect both OL- and CL-KPFM operation to varying degrees.35 Dual harmonic KPFM (DH-KPFM) is one example of OL-KPFM which has been utilized to measure surface potential7,9–12,15,16,25,35,36 in ultra-high vacuum,7,9,36 air,7,15,35 and liquid10–12,16,25 environments.

Here we present a rigorous and quantitative comparison of traditional KPFM (lift mode, off-resonance) with DH-KPFM using a commercial AFM system. Previously we have demonstrated that traditional KPFM and DH-KPFM obtain the same potential difference between copper and graphene surfaces, but a global offset was observed between the two techniques.7 Here we demonstrate that by ruling out electronic cross talk and applying corrections for electronic offsets, the difference between the techniques can be reduced from 73 mV (34%) to 45 mV (17%). We also introduce a new type of correction (“setpoint offset”) for traditional KPFM which minimizes both the distance and bias dependence of the system, allowing for more accurate CPD values to be obtained. Once all necessary corrections have been quantified and applied, the CPD in air between a metal coated AFM probe and a highly oriented pyrolytic graphite (HOPG) sample is recorded and compared using traditional KPFM and DH-KPFM techniques and agreement within 2 mV (1%) is obtained. By demonstrating the quantitative agreement between traditional KPFM and DH-KPFM, the latter technique may become more widely adopted in the emerging field of liquid KPFM.37

II. BASIC PRINCIPLES OF KPFM

A. KPFM

In all KPFM-based techniques, a voltage is applied between a conductive AFM probe and a sample, \( V_{\text{probe}} = V_{dc} \).
The application of $V_{probe}$ results in an electrostatic force comprising static ($F_{dc}$) and dynamic ($F_{\omega}$ and $F_{2\omega}$) components as given by Eq. (1), where $C'_z$ and $V_{cpd}$ are the tip-sample capacitance gradient and CPD, respectively,

$$F_{dc} = \frac{-1}{2} C'_z \left[ (V_{dc} - V_{cpd})^2 + \frac{1}{2} V_{ac}^2 \right],$$  \hspace{1cm} (1a)$$

$$F_{\omega} = -C'_z (V_{dc} - V_{cpd}) V_{ac} \sin(\omega t),$$  \hspace{1cm} (1b)$$

$$F_{2\omega} = C'_z \frac{1}{4} V_{ac}^2 \cos(2\omega t).$$  \hspace{1cm} (1c)$$

A lock-in amplifier can be used to detect the cantilever’s oscillation in response to $F_{\omega}$ and $F_{2\omega}$ in terms of amplitude ($A_{\omega}$ and $A_{2\omega}$) and phase ($\theta_{\omega}$ and $\theta_{2\omega}$).

**B. Traditional KPFM**

According to Eq. 1(b), the first harmonic amplitude response, $A_{\omega}$, is linearly dependent on the applied DC bias. In traditional KPFM, $A_{\omega}$ is minimized by a feedback loop that controls the DC bias such that $V_{dc} = V_{cpd}$. A schematic of the experimental setup for traditional KPFM is shown in Fig. 1(a). From Eq. 1(b), the condition when $V_{dc} = V_{cpd}$ should be independent of $V_{ac}$ and $C'_z$. In practice however, $1/V_{ac}$ and $C'_z$ dependencies are often observed. In fact, the CPD values determined using traditional KPFM are influenced by a variety of factors: Instrumentation effects include electronic offsets and cross talk (between $V_{ac}$ and the photodetector output and/or between $V_{ac}$ and the piezoactuator), thermomechanical and electrical noise sources, the choice of feedback gains, and lock-in amplifier phase offset. The net effect of these factors is that the input signal to the feedback loop contains contributions, which are not associated with the electrostatic tip-sample forces. Under these conditions, the feedback loop will attempt to minimize a mixed signal leading to an error in the measured CPD value. This cumulative feedback effect can be minimized through careful calibration of electronic offsets in instrumentation, the use of shielded electronic cabling, and/or active cross talk compensation. The result of the feedback effect is that CPD values determined using traditional KPFM may be offset by up to $\sim 1$ V, depending on the instrumentation and experimental parameters used and often manifest as distance dependence, and a $1/V_{ac}$ dependence due to the influence of the capacitance gradient, $C'_z$.

**C. DH-KPFM**

DH-KPFM has emerged as a promising technique, both to minimize the influence of the feedback effect on measured CPD values and to enable the study of voltage sensitive materials and interactions at the solid-liquid interface. Initially implemented in ultra-high vacuum by Takeuchi et al., the method was extended to liquid by Kobayashi et al. and to ambient environments by Collins et al. A schematic of the experimental setup for DH-KPFM is shown in Fig. 1(b). In DH-KPFM, CPD is determined by measuring both $A_{\omega}$ and $A_{2\omega}$,

$$A_{\omega} = G_{\omega} |F_{\omega}| = G_{\omega} C'_z V_{cpd} V_{ac},$$  \hspace{1cm} (2a)$$

$$A_{2\omega} = G_{2\omega} |F_{2\omega}| = G_{2\omega} C'_z V_{ac}^2,$$  \hspace{1cm} (2b)$$

$$V_{cpd} = \frac{A_{\omega} \cos(\theta_{\omega})}{A_{2\omega} 4X_{gain}} V_{ac}.$$  \hspace{1cm} (2c)
where \( G_{\omega} \) and \( G_{2\omega} \) are the gains due to the cantilever transfer function at \( \omega \) and \( 2\omega \), respectively, and \( X_{\text{gain}} = G_{\omega} / G_{2\omega} \). Thus, \( V_{\text{setpoint}} \) can be determined using DH-KPFM when \( X_{\text{gain}}, A_{\omega}, A_{2\omega}, \) and \( \theta_{\omega} \) are known. \( X_{\text{gain}} \) can be calculated using the equation for a simple harmonic oscillator:\(^{11}\)

\[
G(\omega) = \frac{1}{k} \frac{1}{\sqrt{[1 - (\omega/\omega_0)^2] + [\omega/Q\omega_0]^2}}.
\] (3)

Electronic offsets and electronic cross talk, if present, can also affect CPD values determined by DH-KPFM.\(^{7,35}\) However, since there is no feedback loop present in DH-KPFM, there is no error associated with loop gains or trying to minimize a mixed signal (i.e., the feedback effect).\(^{5,35}\)

### III. EXPERIMENTAL DETAILS

#### A. Experimental setup

A MFP-3D AFM (Asylum Research, an Oxford Instruments Company, USA) and a HF2LI (Zurich Instruments, Switzerland) lock-in amplifier were used for all measurements. All measurements were performed using Pt/Ir-coated MikroMasch DPE18 AFM probes, having nominal resonant frequencies, \( \omega_0 = 75 \) kHz, and stiffness, \( k = 3.5 \) N/m, with \( V_{ac} = 2 \) V (12.5 kHz) applied to the probe (off-resonance). To remove any possible contamination,\(^{42,43,53}\) probes were consecutively rinsed in ethanol, isopropanol, and de-ionized water (milliQ, Gradient A10, resistance of 18.2 MΩ) and dried under a dry nitrogen flow before being exposed to UV ozone (UV ozone cleaner—ProCleaner™ Plus, BioForce Nanosciences, USA) for 30 min. The sample was a freshly cleaved HOPG surface. Electronics were calibrated using a semi-automated procedure written in IGOR Pro (Wavements, USA). Using a digital multimeter (Agilent Technologies, 34410A), we first calibrated one of the digital to analog converters (DAC) of the AFM controller as a reference and then calibrated all remaining signal pathways. To measure the bandwidth, we applied a chirp function to the sample and measured the resulting CPD, computing the cross correlation between drive and response. Standard uncertainty propagation rules were applied to all results unless otherwise stated. Uncertainties include the uncertainty of all electronic component calibrations as well as the noise associated with the signals measured. Uncertainties are reported as ± one standard deviation.

#### B. Automated data collection procedure

An automated data collection procedure was used to interleave the collection of traditional KPFM and DH-KPFM data as a function of tip-sample separation and bias (Fig. 2). For each combination of distance (8 points from 25 nm to 3.0 μm) and applied bias (13 points from ~1.5 V to 1.5 V with a step size of 250 mV), a contact mode force curve (10 nN setpoint force and 0.5 Hz rate) with no bias applied was used to reposition the probe. For each tip-sample distance, the CPD was determined using traditional KPFM and DH-KPFM consecutively by sampling for 5 s at a fixed tip-sample separation at a rate of 10 kHz using \( V_{ac} = 2 \) V (12.5 kHz) and fitting the respective bias vs. CPD curves at each distance.

![Flow chart of automated data collection procedure used for collection CPD data using traditional KPFM and DH-KPFM and frequency gain, all as function of bias and tip-sample separation.](image)

For DH-KPFM data sets, the frequency gain at each distance was also determined (see Sec. IV E).

### IV. RESULTS AND DISCUSSION

#### A. Electronic cross talk measurement

In order to check for the presence of electronic cross talk\(^4\) between the tip bias signal path and the photodiode signal path, an AC bias was applied to the tip bias line at an amplitude of 10 V (12.5 and 25 kHz) with the laser focused on the cantilever tip. The photodiode signal was then analyzed by using the lock-in amplifier at \(\omega\) and \(2\omega\) consecutively, and no signal was detected (estimated noise floor ~0.1 mV). This therefore eliminated the need to account for cross talk between these signals. Another possible source of electronic cross talk is between the tip bias signal path and the piezoactuation signal path, which could induce a mechanical oscillation of the cantilever at \(\omega\). Again, a signal of 10 V (12.5 and 25 kHz) was applied to the tip bias signal path, and the output of the photodiode with the laser focused on the cantilever chip was analyzed by using the lock-in amplifier at \(\omega\) and \(2\omega\) consecutively, and no signal was detected (estimated noise floor ~0.1 mV). This procedure was repeated after removing the circuit board and piezo-stack from the tip holder, and no difference was observed (estimated noise floor ~0.1 mV). These observations conclusively rule out the presence of any detectable cross talk in this commercial system, and as such additional shielding and/or compensation schemes were not required for our system.\(^{35}\)

#### B. Electronic offset calibration and corrections

As discussed above, any electronic offsets in instrumentation may lead to the measurement of incorrect CPD values.
To minimize these influences, it is necessary to calibrate all electronic pathways outlined in Fig. 1. Here we chose to correct for not only the DC offset value of each component but also any deviations that may occur as a function of the signal magnitude. To this end, both the linear slope and the offset of each component were determined by fitting the data collected in a semi-automated manner. This procedure constructs a look-up table of linear correction coefficients (Table I) and subsequently allows for correction of data offline during analysis. Note that electronic offsets vary between systems. While electronic offsets of a few tens of mV have been reported for other setups, in line with values reported here, electronic offsets for each AFM system will be unique and may also change with time. In order to correct for electronic offsets, each AFM system must be individually characterized.

C. Traditional KPFM optimization

Quantitative traditional KPFM requires feedback loop optimization as the choice of feedback loop parameters will ultimately determine accuracy, stability, and bandwidth of operation.14 The tuning procedure outlined by Jacobs et al.26 was implemented in this work, wherein a square wave (typically 300 mV with a frequency of 0.5 Hz) was applied to the HOPG surface. For gain optimization, the in-phase component signal, $X_\omega$, was used as the input to the proportional-integral (PI) feedback loop of the AFM controller.

D. Setpoint correction

The presence of an offset on the analog to digital converter (ADC offset) that provides the error signal ($X_\omega$) to the feedback loop also contributes to the feedback effect. In the case where all other artefacts are absent, and the amplitude measured at $\omega$ purely arises from electrostatic tip-sample interactions, this ADC offset results in $V_{dc} \neq V_{cpd}$. In our case, a real-time correction of the ADC offset in the error signal was not possible. Fortunately, as the shift in the error signal is constant, it is possible to satisfy the $V_{dc} = V_{cpd}$ condition by choosing an appropriate non-zero feedback setpoint. By ramping the setpoint value, the true minimization of $X_\omega$ can be observed (Fig. 3) when the setpoint is equal to the negative value of the offset of ADC1 measured for our setup (Table I). By using a setpoint of 23.27 mV (canceling the measured ADC1 offset), the electrostatic force of the tip-sample interaction is at the true minimum for the experiment and this allows for an accurate value of CPD to be determined. We will show later that this correction also removes the dependency of the CPD on the tip-sample distance and applied bias.

E. DH-KPFM

Quantitative DH-KPFM requires knowledge of the cantilever transfer function and thereby $X_{gain}$. Whilst $X_{gain}$ can be calculated using Eq. (3), this assumes that the transfer function of the cantilever is free from instrumentation artefacts and that $X_{gain}$ is independent of the tip-sample separation. Here we adopt an approach where we measured $X_{gain}$ directly at each tip-sample separation and applied bias by recording $A_\omega$ with excitation at $\omega$ and $2\omega$, consecutively.7,13,44 Other approaches based on half-harmonic excitation,15 resonance tracking,45 band excitation7,13,14,46 or G-mode19,20,47,48 may also be used to determine $X_{gain}$. Figure 4(a) shows the thermal spectra at a tip-sample separation of ~3 µm with excitation $A_\omega$ applied at 12.5 kHz. The measured $X_{gain} = 0.85 \pm 0.02$ does not show a significant tip-sample distance dependence [Fig. 4(b)] or surface bias dependence [Fig. 4(c)] and is consistently ~10% lower than the calculated $X_{gain} = 0.95 \pm 1 \times 10^{-6}$.

F. Bandwidth comparison

In order to compare the bandwidth of traditional KPFM and DH-KPFM, we evaluated the response of the system to a chirp excitation applied to the sample using a cross correlation calculation and measurement of the frequency response at –3 dB. The effective bandwidth of the DH-KPFM was determined to be 53 Hz in comparison with 5.6 Hz for traditional KPFM. The bandwidth for traditional KPFM is limited by the time constant of the lock-in amplifier (900 µs) and the feedback loop settings derived from the tuning procedure outlined by Jacobs et al.26 Further improvements in the bandwidth of traditional KPFM may be possible using modern all digital feedback controllers and/or alternative feedback loop tuning strategies. The bandwidth for DH-KPFM is also determined by the time constant of the lock-in amplifier ($\omega_1 = 900$ µs, $\omega_2 = 9.5$ ms). Note that whilst the use of

![FIG. 3. Measured $X_\omega$ as a function of setpoint. The true $X_\omega = 0$ condition is observed when the setpoint is 23.27 mV. Note that the calibrated ADC1 offset = $-23.27 \pm 7 \times 10^{-7}$ mV.](image-url)
TABLE II. Comparison of uncorrected, electronic offset corrected, and electronic offset + setpoint corrected traditional KPFM and DH-KPFM data.

<table>
<thead>
<tr>
<th>Technique</th>
<th>No corrections</th>
<th>Electronic offset corrections</th>
<th>Electronic offset + setpoint corrections</th>
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<tr>
<td></td>
<td>CPD (mV)</td>
<td>Vdc Sens (mV/μm)</td>
<td>Dist Sens (mV/μm)</td>
</tr>
<tr>
<td>Traditional KPFM</td>
<td>270 ± 4</td>
<td>2.6 ± 0.4</td>
<td>12 ± 2</td>
</tr>
<tr>
<td>DH-KPFM</td>
<td>198 ± 8</td>
<td>2 ± 1</td>
<td>1 ± 3</td>
</tr>
<tr>
<td>% difference</td>
<td>33.5</td>
<td>...</td>
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G. Comparison of quantitative traditional KPFM and DH-KPFM

We now examine the surface potential values obtained from traditional KPFM and DH-KPFM in air above a freshly cleaved HOPG surface as a function of applied bias and tip-sample separation. First, we compare the values obtained from an uncalibrated system where we observed a CPD value of 270 ± 4 mV for traditional KPFM compared to a value of 197 ± 8 mV for DH-KPFM (~34% or 73 mV difference). It is worth noticing the significant distance and bias dependence (“Dist” and “Vdc Sens,” respectively, in Table II) of the traditional KPFM data compared to the DH-KPFM data (Table II). Applying corrections for electronic offsets (Table I) in the system reduces the observed CPD for traditional KPFM and DH-KPFM to 225 ± 4 mV and 190 ± 8 mV, respectively (~17% or 45 mV difference), and minimizes the dc bias dependence of the traditional KPFM data, but the distance dependence remains significant. Furthermore, by applying the setpoint correction, the traditional KPFM CPD value reduces to 188 ± 4 mV, which corresponds to a deviation smaller than the error margin (~1% difference) of the corrected DH-KPFM CPD value. Under these conditions, the traditional KPFM values exhibit no significant bias or distance dependence. Therefore, in the absence of cross talk, with offset and setpoint correction applied, we have demonstrated that the CPD values measured with traditional KPFM and DH-KPFM agree within the margin of error. We would like to point out that the corrections applied to DH-KPFM changed the CPD values by only 10 mV, once again showing that this technique is less prone to instrumentation artefacts. This combined with the improved bandwidth of DH-KPFM provides more evidence for the utility of open-loop techniques for characterization of surface potential. We attribute the larger uncertainty of the DH-KPFM CPD to a combination of uncertainty in the measured X_gain and the noise in the A_ω and A_2ω signals. Figure 5 shows the effect of the corrections for offset and setpoint for traditional KPFM and offset corrections for DH-KPFM.

In order to demonstrate quantitative imaging of a heterogeneous sample, a silicon surface with a ~20 nm gold film covering approximately half of the scan area was measured for each experiment, independent of the technique applied, in order to select appropriate scan speeds. The larger bandwidth of DH-KPFM observed here may increase the speed at which accurate measurements can be acquired and aid in the study of charging dynamics in materials and electrical devices.57,49–51
FIG. 5. CPD measurements obtained using traditional KPFM and DH-KPFM as a function of tip-sample separation. (a) Comparison of traditional KPFM measurements: uncorrected (red), offset corrected (blue), and offset + setpoint corrected (black). (b) Comparison of DH-KPFM measurements: uncorrected (red) and corrected (blue). (c) Uncorrected traditional KPFM (red) and uncorrected DH-KPFM (blue). (d) Corrected traditional KPFM and corrected DH-KPFM. $V_{ac} = 2$ V (12.5 kHz).

FIG. 6. Imaging a silicon surface with a $\sim 20$ nm gold film. (a) Topography, (b) corrected traditional KPFM, and (c) corrected DH-KPFM. $V_{ac} = 4$ V (12.5 kHz).

using traditional KPFM and DH-KPFM. We found good agreement between the two techniques when scanning the same area (Fig. 6). The difference in measured CPD between

Figs. 6(b) and 6(c) was found to be $4.0 \pm 10.4$ mV (20% of images excluded from analysis due to drift).

DH-KPFM also allows the capacitance gradient to be measured as a function of tip-sample distance, as shown in Fig. 7. Here the capacitance gradient is determined by $C'_z = \frac{A_{260}}{V_{ac}^2}$. Gramse et al. have previously shown that such data can be fit with a model describing the geometry of the probe in order to extract the dielectric constant of the tip-sample interaction. Such measurements could also be made whilst using traditional KPFM if $A_{260}$ is also recorded.

V. CONCLUSION

We have examined the quantitative agreement between traditional KPFM (CL-KPFM) and DH-KPFM (OL-KPFM). We have shown that DH-KPFM allows for higher bandwidth of operation and that it is more robust to electronic offsets that
may be present in a given AFM system. By carefully measuring and applying corrections for offsets and applying a new “setpoint correction,” we were able to show that DH-KPFM and traditional KPFM agree within 1% for a calibrated system. Furthermore, the addition of the “setpoint correction” resulted in the removal of both bias and tip-sample separation dependence in CPD values for traditional KPFM operation. We also demonstrated quantitative agreement when imaging a heterogeneous sample as well as illustrating the potential for the measurement of dielectric constants.

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