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Calibration of the oscillation amplitude of electrically excited scanning probe microscopy sensors

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ABSTRACT
Atomic force microscopy (AFM) is an analytical surface characterization tool which can reveal a sample's topography with high spatial resolution while simultaneously probing tip-sample interactions. Local measurement of chemical properties with high-resolution has gained much popularity in recent years with advances in dynamic AFM methodologies. A calibration factor is required to convert the electrical readout to a mechanical oscillation amplitude in order to extract quantitative information about the surface. We propose a new calibration technique for the oscillation amplitude of electrically driven probes using the principle of energy balance. Our technique relies on the measurement of the energy input to maintain the oscillation amplitude constant. With the measurement of the energy input to the probe, a mechanical oscillation amplitude is calculated and a calibration factor to convert the electrical readout in volts to a mechanical oscillation amplitude in Ångströms is obtained. We demonstrate the application of the new technique with a quartz tuning fork including the qPlus configuration, while the same principle can be applied to other piezoelectric resonators such as length extension resonators or piezoelectric cantilevers. The calibration factor obtained by this technique is found to be in agreement with using the thermal noise spectrum method for capsulated and decapsulated tuning forks and tuning forks in the qPlus configuration.

INTRODUCTION
Dynamic scanning probe microscopy is a surface characterization technique where a sharp tip is attached to the end of an oscillating probe and acts as a sensing element to map the surface topography up to picometer resolution.1-4 With the advent of dynamic scanning probe techniques, quantitative measurement of local sample properties became popular.5-8 To extract quantitative information about the surface and to modulate the tip-sample separation in a controlled way, properties of the oscillating probe such as excitation frequency, excitation amplitude, frequency shift due to tip-sample interaction, phase difference between the excitation signal and the oscillation signal, quality factor, and oscillation amplitude have to be known.9-13 While the excitation frequency and the amplitude are directly controlled by the operator, the resonance frequency shift, phase difference, and quality factor are measured by well-established measurement electronics.14 Among these dynamic properties, the oscillation amplitude requires determination of a calibration factor between the electrical readout and the mechanical oscillation amplitude. The knowledge of the oscillation amplitude is important to quantitatively and even qualitatively interpret frequency shift measurements (“small amplitude” vs. “large amplitude” approximations).9-13 Accurate knowledge of the oscillation amplitude is the key to determine the tip-sample interaction laws from frequency shift data and, as recently demonstrated, needs to be known to determine if it is even in principle possible, i.e., a mathematically well-posed problem.15

Many techniques exist to determine the oscillation amplitude, including thermal excitation, interferometric techniques, electro-mechanical techniques, and frequency modulation-based techniques. These existing techniques
have limitations when applied to tuning forks and tuning fork-based sensors: Measuring thermal excitations of tuning forks at low temperatures is limited by the microscope’s mechanical and electrical noise detection. Specifically, the high Q factors (leading to mechanical noise sensitivity) and high spring constant (resulting in small amplitudes) impede measurement of the thermal noise spectrum of the tuning fork system at low-temperatures. Interferometric measurements can only be implemented with the integration of complex optical setups with microscopes that work with tuning fork-based sensors. Existing electro-mechanical techniques can only be applied to balanced tuning forks. Frequency modulation-based techniques rely on measuring the frequency shift induced by tip-sample interaction upon approach allowing the calibration of the oscillation amplitude due to the indirect measurement of current and frequency shift dependencies. In passing, we note that frequency shift-based methodologies can only be conducted with assembled sensors, and in particular oscillation amplitude determination based on tunneling currents requires a conductive sample.

In this manuscript, we propose a new calibration procedure that relies only on measuring the electrical energy input to maintain the oscillation amplitude of the probe constant. We demonstrate the technique with quartz tuning forks and quartz tuning forks in the qPlus configuration and compare our results with the thermal excitation technique at room temperature. The major advantage of our technique is that it directly delivers the calibration factor between the electrical readout and the mechanical oscillation, thereby eliminating the limitations of existing methodologies. As an outlook, we provide a pathway to track the non-negligible change in the spring constant when a tuning fork-based sensor is assembled and mounted. Although the spring constant of tuning forks can be assessed accurately (see supplementary material for details), the equivalent spring constant of the sensor assembly can be significantly different compared to tuning forks without tips leading to a large systematic error for quantitative force measurements if simply assumed constant.

BACKGROUND AND METHODS

As Fig. 1 summarizes, a sinusoidal voltage, \( V_{\text{drive}} \), is applied to a tuning fork and the resulting oscillating output current, \( I_{\text{out}} \), is measured. The applied sinusoidal voltage results in a mechanical oscillation of the tuning fork prongs with oscillation amplitude, \( A_{\text{osc}} \), due to the piezoelectric nature of quartz. The mechanical oscillation of the tuning fork’s prongs in turn induces an oscillating charge on the surface of the tuning fork, which is collected with electrodes. The time-varying charge is measured as an electric current which is converted by a transimpedance amplifier to voltage and typically demodulated with a lock-in amplifier. The lock-in outputs the phase between the excitation signal and the oscillation signal in degrees (\( \varphi \)) and oscillation amplitude, \( V_{\text{readout}} \), in volts.

In the absence of a tip-sample interaction, the dynamics of the probe can be expressed as a damped-harmonic oscillator. Equation (1) describes the total energy of a harmonic oscillator:

\[
E_{\text{mechanical}} = \frac{1}{2} k A_{\text{osc}}^2. \tag{1}
\]

In Eq. (1), \( A_{\text{osc}} \) is the mechanical oscillation amplitude and \( k \) is the equivalent spring constant of the probe. We calculated the spring constant of tuning fork-based sensors by using finite element methods (see supplementary material for details). We want to note that for probes with irregular shapes and/or material combinations, alternative techniques should be used to obtain \( k \) to avoid a systematic error of the calibration constant. The total mechanical energy of a balanced tuning fork is equal to \( k A_{\text{osc}}^2 \) as there are two oscillating prongs.

Due to the finite quality factor, \( Q \), of oscillating probes, the energy dissipated per oscillation cycle is represented by

\[
E_{\text{diss}} = \frac{2\pi}{Q} E_{\text{mechanical}}. \tag{2}
\]

The energy dissipated per second can be calculated by \( E_{\text{diss}} \times f_0 \), where \( f_0 \) is the resonance frequency of the oscillating probe. To keep the oscillation amplitude constant, this mechanical power loss is compensated by the electrical power, \( P_{\text{compensation}} \), supplied by the external circuit, such as \( E_{\text{diss}} \times f_0 = P_{\text{compensation}} \). Equation (3) shows the average electrical power input to compensate the dissipated energy,

\[
P_{\text{compensation}} = \frac{1}{2} V_{\text{drive}} I_{\text{m}} \cos \varphi. \tag{3}
\]

![FIG. 1. Schematic representation of the experimental setup. The piezoelectric probe is excited electrically, \( V_{\text{drive}} \), which results in a mechanical oscillation, \( A_{\text{osc}} \). The mechanical oscillation induces an oscillating current, \( I_{\text{out}} \), which is converted to voltage with the (transimpedance) amplifier. The output of the amplifier is demodulated with the lock-in. The phase difference between the excitation signal and the oscillation signal in degrees (\( \varphi \)) and oscillation amplitude in volts, \( V_{\text{readout}} \), are outputs of the lock-in amplifier.](image-url)
In Eq. (3), $V_{\text{drive}}$ is the amplitude of the sinusoidal excitation voltage, $I_m$ is the current induced by the mechanical oscillation, and $\varphi$ is the phase of $I_m$ with respect to $V_{\text{drive}}$. The phase, $\varphi$, is equal to zero at the resonance frequency, and Eq. (3) reduces to $\frac{1}{2} V_{\text{drive}} I_m$.

By equating the mechanical energy dissipation to electrical power input to maintain the oscillation amplitude constant, we can find the mechanical oscillation amplitude of a probe in terms of experimentally measurable quantities and the spring constant

$$A_{\text{osc}} = \sqrt{\frac{V_{\text{drive}} I_m Q}{2\pi f_0 k}}.$$  \hspace{1cm} (4)

It is important to note that $I_m$ needs to be extracted from the measured total current, $I_{\text{out}}$, which consists of $I_m$ that is due to the mechanical motion and the current passes through the stray capacitance in the resonator.\textsuperscript{26–29} This can be achieved by using the analytical formula by Lee et al.,\textsuperscript{29} which is based on an equivalent electrical circuit model of the piezoelectric resonator. In practice, the total current is measured by the lock-in amplifier as the output voltage, $V_{\text{readout}}$, which is related to $I_{\text{out}}$ as $I_{\text{out}} = \frac{V_{\text{readout}}}{G}$ with $G$ being the gain of the measurement setup (see supplementary material for details about $G$). By noting $I_m \propto V_{\text{drive}}$ and using $I_m = \frac{V_{\text{compensated}}}{G}$, the following linear relation can be derived (see supplementary material for the derivation):

$$A_{\text{osc}} = \alpha \times \frac{V_{\text{compensated}}}{G}.$$  \hspace{1cm} (5)

In Eq. (5), $\alpha$ is the calibration factor between the mechanical amplitude in Ångströms and the electrical signal in volts.

**RESULTS AND DISCUSSIONS**

The calibration of a decapsulated tuning fork with the principle of energy balance is demonstrated in Fig. 2. Several resonance curves are measured with different drive amplitudes. As Fig. 2(a) shows, the frequency response of the tuning fork deviates from the ideal Lorentzian shape with an anti-resonance peak due to the effect of stray capacitance. Figure 2(b) shows the corrected frequency response obtained from results in Fig. 2(a) using methods described in Ref. 29. Finally, the oscillation amplitude, $A_{\text{osc}}$, is calculated with Eq. (4). Figure 2(c) shows the linear relation between the calibration factor, $\alpha$, as presented in Eq. (5) of a tuning fork probe for different drive signals both with the direct readout, i.e., without correction of current through stray capacitance (red curve), and the corrected readout (blue curve).

To elaborate the calibration of the oscillation amplitude with the principle of energy balance, we applied the technique to three different capsulated and decapsulated tuning forks.
and compared our results with the thermal excitation technique (see supplementary material for details). As summarized in Table I, calibration with the direct readout is in agreement with less than 2.0% discrepancy compared to calibration using the corrected readout and thermal excitation for capsulated tuning forks. However, the calibration factor with the direct

| Type   | Capsulated, Q | Direct readout (Å/mV) | Corrected readout (Å/mV) | Thermal excitation (Å/mV) |
|--------|---------------|-----------------------|--------------------------|--------------------------|
| I, k = 1267 N/m | 61 200         | 1.38                  | 1.38                     | 1.37                     |
|        | 5100          | 1.42                  | 1.37                     | 1.37                     |
| II, k = 1939 N/m | 71 900         | 1.10                  | 1.08                     | 1.09                     |
|        | 5 800         | 1.16                  | 1.06                     | 1.06                     |
| III, k = 16 940 N/m | 101 000     | 0.29                  | 0.28                     | 0.28                     |
|        | 9 500         | 0.29                  | 0.28                     | 0.28                     |

FIG. 3 Calibration of an electrically excited tuning fork in the qPlus configuration with the principle of energy balance. (a) One of the prongs of the tuning fork is fixed, while the other prong oscillates freely for tuning forks in the qPlus configuration. (b) shows the direct electrical readout of the electrically excited tuning fork in the qPlus configuration for different drive amplitudes. (c) The effect of stray capacitance is corrected with the mathematical correction procedure. (d) The calibration constant of the oscillation amplitude is calculated both with the direct readout (red curve) and corrected readout (blue curve) by curve fitting to the experimental data. The confidence level of fits for the calibration factor, $\alpha$, is 1 pm/mV, which is consistent with former experimental work and results in 0.7% uncertainty due to the curve fitting. The horizontal axis of the plot in (d) is $V_{\text{readout}}$ for calibration with the direct readout and $V_{\text{compensated}}$ with the compensation of stray capacitance. For experiments presented in this figure, we used the type I tuning fork in the qPlus configuration, $Q = 1416$, and a resonance frequency of 32 592 Hz.
readout can deviate compared to decapsulated tuning forks. Moreover, this discrepancy upsurges when the quality factor decreases upon decapsulation for all tuning forks and reaches at least 9% for the type II tuning fork. We conclude that stray capacitance effects should be corrected to ensure accurate calibration.

Quartz tuning forks that have one free prong to which the tip is attached to the end while the fork’s other prong is fixed to a holder (“qPlus configuration”) have gained popularity in recent years for high-resolution imaging. Figure 3(a) schematically describes that when a tuning fork in the qPlus configuration is excited electrically, the electrical excitation voltage is applied to both prongs; however, only one of the prongs is free to oscillate (i.e., the effective impedance at resonance is half of the tuning fork configuration). Figure 3(b) shows the amplitude and phase of the tuning fork in the qPlus configuration with the direct electrical readout. We corrected the electrical readout to eliminate the effect of stray capacitance [Fig. 3(c)]. The calibration factor with the corrected readout, \(α_{\text{readout}} = 2.70 \text{ Å/mV} \) [Fig. 3(d), blue curve], is in agreement with the calibration of the same tuning fork in the qPlus configuration with thermal excitation, \(α_{\text{thermal}} = 2.72 \text{ Å/mV} \), the 1% difference well within the uncertainty of the thermal excitation technique. Similar to the previous observation, the calibration factor determined from a direct readout [Fig. 3(d), red curve] deviates by 15% with respect to the calibration with thermal excitation. Such a drastic difference emphasizes the importance of correcting the stray capacitance of tuning forks for accurate calibration of oscillation amplitude when using the principle of energy balance.

OUTLOOK AND SUMMARY

In the Results and Discussions Section, we have only compared tuning forks as fabricated, without any tip or tip-wires attached. Although the spring constant of the tuning forks can be quantified accurately with experimental and computational techniques (see supplementary material for details), the spring constant of the tip-tuning fork assembly is different than the tuning fork alone. Knowing the spring constant of the assembled tip-tuning fork system is particularly important for quantifying interaction potentials from atomic force microscopy (AFM) measurements and is normally not easily accessible. The spring constant of the sensor assembly is expected to be substantially different compared to the bare tuning fork as the mechanical constraints, i.e., boundary conditions, change due to the epoxy glue that is used to attach the tuning fork to the base, the epoxy that is used to attach the tip, the orientation of the tip, and the wire to collect tunneling current or apply a tip-bias in AFM. In the following, we will show how a measurable change in the calibration factor, \(α\), can be used to track the change in the spring constant as the sensor is assembled. The calibration factor, \(α\), scales as (see supplementary material for details)

\[
α \propto \left(\frac{1}{f_0 \times k \times \text{Number of Oscillating Prongs}}\right)
\]  

(6)

Since the change in \(f_0\) can easily be traced by a frequency sweep, the change in \(α\) can be used to quantify the change in the spring constant of the sensor, in particular in the qPlus configuration. As an example, the calibration factor, \(α\), of a qPlus sensor [2.70 Å/mV, Fig. 3(c)] has a 2% difference compared to the calibration of the same sensor in the tuning fork configuration (Table I, 1.37 Å/mV × 2 = 2.74 Å/mV) if the same effective spring constant is assumed and the number of oscillating prongs is taken into account [Eq. (6)]. The resonance frequency of the sensor in the qPlus configuration has only a 0.1% drop (Fig. 3) with respect to the decapsulated tuning fork (Fig. 2). According to Eq. (6), such a small change in the resonance frequency has a negligible effect on \(α\) and thus the proportionality presented in Eq. (6) reduces to \(α \propto \frac{1}{k}\). For this reason, the 2% decrease in the calibration factor implies the effective spring constant of the qPlus sensor as 1293 N/m. Similarly, the calibration factor of the type II tuning fork in the qPlus configuration is found as 1.97 Å/mV with the principle of energy balance, which reveals the effective spring constant as 2087 N/m. The reassessed spring constant of the type II tuning fork is 4% higher than the generally accepted value (2000 N/m) for similar qPlus sensors. Note, however, that the variation of the spring constant can be more dramatic due to the orientation of the tip and the wire to collect tunneling current or apply a tip-bias in AFM. With the reassessment of the spring constant, a systematic error when quantitatively reconstructing the tip-sample interaction laws can be avoided.

In summary, we demonstrate a new calibration technique for the oscillation amplitude of electrically driven piezoelectric probes using the principle of energy balance. We demonstrated the application of this new calibration technique with tuning forks including the qPlus configuration, while the same principle can be applied to other piezoelectric oscillators such as length extension resonators or piezoelectric cantilevers. Our experimental results show that the calibration with the principle of energy balance can be applied independently of the quality factor and the sensor configuration as long as the effect of stray capacitance is compensated. In addition to revealing the conversion factor between the electrical readout and the mechanical oscillation amplitude, our methodology provides a pathway to track the change in the effective spring constant of a sensor assembly in the qPlus configuration which is important for quantitative force spectroscopy experiments.

SUPPLEMENTARY MATERIAL

Supplementary material that describes the calculation of the spring constant with the finite element method technique, calibration of gain and phase shift of measurement electronics, thermal noise excitation, and derivation of the proportionality of the calibration factor is available online. Also, COMSOL Multiphysics code and MATLAB code are available online to calculate the spring constant of tuning forks.

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REFERENCES

1 G. Binnig, C. F. Quate, and C. Gerber, Phys. Rev. Lett. 56, 930 (1986).
2 T. R. Albrecht, P. Grutter, D. Horne, and D. Rugar, J. Appl. Phys. 69, 668 (1991).
3 R. Garcia, Amplitude Modulation Atomic Force Microscopy (Wiley-VCH, Singapore, 2010).
4 F. J. Giessibl, Rev. Mod. Phys. 75, 949 (2003).
5 F. J. Giessibl, Science 267, 68 (1995).
6 L. Gross, F. Mohn, N. Moll, P. Liljeroth, and G. Meyer, Science 325, 1110 (2009).
7 L. Gross, F. Mohn, P. Liljeroth, J. Repp, F. J. Giessibl, and G. Meyer, Science 324, 1428 (2009).
8 B. J. Albers, T. C. Schwendemann, M. Z. Baykara, N. Pilet, M. Liebmann, E. I. Altman, and U. D. Schwarz, Nat. Nanotechnol. 4, 307 (2009).
9 F. J. Giessibl, Phys. Rev. B 56, 16010 (1997).
10 H. Hölscher, W. Allers, U. D. Schwarz, A. Schwarz, and R. Wiesendanger, Phys. Rev. Lett. 83, 4780 (1999).
11 U. Dürig, Appl. Phys. Lett. 75, 433 (1999).
12 F. J. Giessibl, Appl. Phys. Lett. 78, 123 (2001).
13 J. E. Sader and S. P. Jarvis, Appl. Phys. Lett. 84, 1801 (2004).
14 Y. Martin, C. C. Williams, and H. K. Wickrundersinghe, J. Appl. Phys. 61, 4723 (1987).
15 J. E. Sader, B. D. Hughes, F. Huber, and F. J. Giessibl, e-print arXiv:1709.07571 (2017).
16 F. J. Giessibl, Appl. Phys. Lett. 76, 1470 (2000).
17 F. Welker, F. de Faria Elsner, and F. J. Giessibl, Appl. Phys. Lett. 99, 084102 (2011).
18 J. Rychen, T. Ihn, P. Studerus, A. Herrmann, K. Esslin, H. J. Hug, P. J. A. van Schendel, and H. J. Güntherodt, Rev. Sci. Instrum. 71, 1695 (2000).
19 G. Gucciardi, G. Bachelier, A. Mayah, and M. Allegrini, Rev. Sci. Instrum. 76, 036105 (2005).
20 J. Liu, A. Callegari, M. Stark, and M. Chergui, Ultramicroscopy 109, 81 (2008).
21 S. G. Hermann, H. Markus, and R. Hans-Peter, Nanotechnology 18, 255503 (2007).
22 A. Castellanos-Gomez, N. Agrait, and G. Rubio-Bollinger, Nanotechnology 20, 215502 (2009).
23 O. E. Dagdeviren and U. D. Schwarz, Meas. Sci. Technol. 28, 015102 (2017).
24 O. E. Dagdeviren and U. D. Schwarz, Beilstein J. Nanotechnol. 8, 657 (2017).
25 J. D. Hartog, Mechanical Vibrations (Dover Publications, 1985).
26 Y. Miyahara, M. Deschler, T. Fujii, S. Watanabe, and H. Bleuler, Appl. Surf. Sci. 188, 450 (2002).
27 Y. Qin and R. Reifenberger, Rev. Sci. Instrum. 78, 063704 (2007).
28 S. An, K. Lee, B. Kim, J. Kim, S. Kwon, Q. Kim, M. Lee, and W. Jhe, Curr. Appl. Phys. 13, 1899 (2013).
29 M. Lee, J. Jahng, K. Kim, and W. Jhe, Appl. Phys. Lett. 91, 023117 (2007).
30 F. J. Giessibl, Appl. Phys. Lett. 73, 3956 (1998).
31 N. A. Burnham, X. Chen, C. S. Hodges, G. A. Matei, E. J. Thoreson, C. J. Roberts, M. C. Davies, and S. J. B. Tendler, Nanotechnology 14, 1 (2003).
32 D. van Vörden, M. Lange, M. Schmuck, N. Schmidt, and R. Möller, Beilstein J. Nanotechnol. 3, 809 (2012).
33 J. Falter, M. Stiefermann, G. Langewisch, P. Schurig, H. Hölscher, H.uchs, and A. Schirmiseen, Beilstein J. Nanotechnol. 5, 507 (2014).
34 B. J. Albers, M. Liebmann, T. C. Schwendemann, M. Z. Baykara, M. Heyde, M. Salmeron, E. I. Altman, and U. D. Schwarz, Rev. Sci. Instrum. 79, 033704 (2008).
35 O. E. Dagdeviren, C. Zhou, E. I. Altman, and U. D. Schwarz, Phys. Rev. Appl. 9, 044040 (2018).