Calibration of oscillation amplitude in dynamic scanning force microscopy

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Abstract

A method to precisely calibrate the oscillation amplitude in dynamic scanning force microscopy is described. It is shown that the typical electronics used to process the dynamic motion of the cantilever can be adjusted to transfer the thermal noise of the cantilever motion from its resonance frequency to a much lower frequency within the typical bandwidth of the corresponding data acquisition electronics of a scanning force microscopy system. Based on this concept, two procedures for the calibration of the oscillation amplitude are proposed. One is based on a simple calculation of the root mean square deviation measured at the outputs of the electronics used to process the dynamic motion of the cantilever, and the second one is based on analysis of the corresponding spectrum and the calculation of the quality factor, the resonance frequency and the signal strength.

We show that the proposed scheme for amplitude calibration using thermal noise is experimentally and theoretically robust, with soft as well as with hard cantilevers. Moreover, it is directly related to well-defined quantities such as the force constant and thermal energy, in contrast to the calibration using amplitude versus distance curves, which requires non-trivial a priori assumptions regarding the amplitude versus distance relation.

(Some figures may appear in colour only in the online journal)

1. Introduction

Scanning force microscopy (SFM) is an extremely versatile tool for nanoscience which has a very high resolution, but usually only a modest precision. When SFM is used as a microscope, precise calibration of the piezo-movements is necessary, as well as careful control of piezo-hysteresis and nonlinearities. When used as a force measuring instrument a precise calibration of the displacement detector as well as the force constant of the cantilever is required. Dynamic SFM (DSFM) is by now probably the most extensive SFM mode, since it allows operation at very low forces and operates in the non-contact regime [1–4]. In DSFM the frequency (or phase) of the tip–sample system and the oscillation amplitude are the basic signals that are measured and should thus be calibrated for precise measurements. Precise measurement of frequency is easy, and is implemented directly in most instruments. Calibration of the oscillation amplitude is an issue that has received little attention (see, however, [5, 6]), and less than we think it deserves.

We think that calibration of the oscillation amplitude is important for two main reasons. First, the variation of oscillation amplitude is related to the dissipation in the tip–sample system. Thus, for quantitative dissipation measurements the precise oscillation amplitude has to be known. This is most easily recognized if a phase locked loop (PLL) is used to keep the system at resonance: then the oscillation amplitude at resonance is $a(\nu_0) = Q_{\text{eff}}a_0$, where $Q_{\text{eff}}$ is the effective quality factor, $\nu_0$ the resonance frequency and $a_0$ the (effective) excitation amplitude. If the PLL is not enabled, then the relation between dissipation and oscillation is less evident. In the common ‘tapping mode’ the dissipation is related to phase as well as amplitude [7]. The second reason

\footnote{Strictly, this is only correct in the harmonic approximation, otherwise nonlinearities will complicate the analysis, see for example [5].}
for calibration of the oscillation amplitude is its relation to the fundamental dynamics of the tip–sample system. In fact, for the oscillation amplitudes used typically in DSFM, the SFM system is not a linear system. Then the oscillation amplitude is a fundamental parameter, since it critically determines the nonlinearity of tip–sample interaction [5, 8, 9], the onset of bi-stability and chaos [10–12], as well as the precise interaction regime (attractive versus repulsive) used for imaging [13]. Finally, as shown recently, if correctly calibrated the oscillation amplitude can be used to obtain a reliable image of the ‘true topography’ that is independent of feedback parameters and scanning speed [14].

As a starting point for the discussion of the calibration of oscillation amplitude we recall that for static SFM measurements, the calibration of (static) deflection may be considered a similar problem. Different schemes for force calibration have been proposed [15–17] and in the present context we recall two of them. The first method is based on the slope of a force versus distance curve [18] acquired on a stiff sample, where it is assumed that in contact the measured cantilever deflection is equal to the piezo-displacement, therefore the slope $s_d$ of the deflection versus piezo-extension curve is $s_d = 1$. The second method for calibration of deflection is based on the equipartition theorem, which determines the mean thermal fluctuation of the cantilever [15–17, 19–22]:

$$c \frac{z^2}{2} (t)_{\text{rms}} = \frac{1}{2} kT \tag{1}$$

where $c$ is the force constant of the cantilever, $k$ is the Boltzmann constant, $T$ is the temperature of the cantilever and $z(t)$ is the displacement of the free end of the cantilever. We note that relation (1) has implicitly two unknowns: the force constant $c$ of the cantilever and a sensitivity $\beta$ (units: nm V$^{-1}$) to convert the measured signal $u(t)$ (volts) into physical length units (nanometers): $z(t) = \beta u(t)$. If the force constant $c$ of the cantilever is known, the thermal noise can be used to determine the sensitivity, $\beta$, and the deflection is thus calibrated.

With regard to the calibration of oscillation amplitude we think that, to the best of our knowledge, only the idea of ‘slope calibration’ has been transferred to DSFM. To implement the corresponding procedure, essentially, an amplitude versus piezo-extension curve is acquired and a slope of unity is assumed in the appropriate interaction region. Even though this procedure seems to be quite widely used, it is interesting that its validity has been discussed rather little. Moreover, as reported in [5] for the interaction of an oscillating tip with a repulsive harmonic potential, the slope of the amplitude versus piezo-extension curve can be calculated analytically (see relation (11) in [5]) and is not generally equal to unity. For the more general case of a repulsive harmonic potential and an attractive van der Waals potential, the slope cannot be calculated analytically, but simulations indicate that also in this case the assumption of a unity slope is not generally correct. Two other methods for the calibration of oscillation amplitude are discussed in [6]: one uses the tunneling signal and the other the frequency versus amplitude $\Delta \nu \propto \Lambda^{-3/2}$ (see, for example, [3]) well known in FM-DSFM. The first of these methods is applicable only in a UHV environment, and the second method is restricted to FM-DSFM and may suffer from systematic errors if not applied with care. In conclusion, we think that the issue of amplitude calibration in DSFM is still an open question. In the present work we will show that it is possible to use the equipartition theorem—relation (1)—to estimate the oscillation amplitude. Analyzing how thermal noise is processed by a typical DSFM dynamic unit (DSFM-DU) we prove that this method is theoretically correct, and we also show that it is experimentally robust.

2. Processing of thermal noise by a DSFM dynamic unit

2.1. Modeling

In the present work we discuss the response of the DSFM dynamic unit (DSFM-DU) in the frequency domain because we will analyze the response of the system to thermal noise $z_{th}(t)$. Unfortunately, thermal noise is non-trivial to describe using a ‘coherent’ superposition of harmonic signals, as it is implicitly assumed in the Fourier transform $z(t) = \int dv z(v) e^{i2\pi vt}$. The correct description of noise in the time domain should involve wavelet transforms [23, 24], which is however beyond of the scope of the present work. In the present work the DSFM-DU is used in a non-standard way: no oscillation is applied to excite the cantilever, and the reference oscillator frequency $\nu_r$ is not at the resonant frequency $\nu_0$ of the cantilever (see figure 1). Instead, the DSFM-DU is adjusted so that $\nu_r \approx \nu_0 - bw/2$, where $bw = 1/\tau$ (\(\tau\): time constant) is the bandwidth of the DSFM-DU. As shown schematically in the upper part of figure 1, the thermal peak of the cantilever then appears centered within the spectral range of the output of the DSFM-DU. Essentially, the reason for this frequency shift is that when the normal force signal is internally multiplied with the reference frequency $\nu_r$ of the DSFM-DU, frequency components at $\nu_0 - \nu_r$ and $\nu_0 + \nu_r$ appear. Usually, the DSFM-DU is operated with $\nu_0 = \nu_r$, then thermal noise is observed at very low frequencies $\nu \approx 0$, that is, at DC. At this low frequency the thermal noise is usually not clearly distinguished from the pink noise (1/\(\nu\)-noise) of typical electronic components. When $\nu_0 \neq \nu_r$, the thermal peak is shifted to the difference frequency $\nu_0 - \nu_r$ and is usually easily recognized.

In order to present a more precise description of how thermal noise is processed by a DSFM-DU, we recall that such electronics used for analyzing the dynamics of the cantilever may be implemented using a lock-in detection scheme [1, 2, 25, 26], as shown in figure 1: the input signal $u_n(t)$ is analyzed by multiplying it by a reference signal in quadrature—$a_r \cos(2\pi \nu_r t)$ and $a_r \sin(2\pi \nu_r t)$—and then filtered with an appropriate time constant $\tau$. The corresponding output signals of the DSFM-DU are the in-phase (‘phase’) and the out-of-phase (‘amplitude’) signals $u_x(t)$ and $u_y(t)$.

$2$ Note that in [26] the nomenclature $\langle q(t) \rangle_\tau$ and $\langle y(t) \rangle_\tau$ was used, that is, $u_x(t) \equiv \langle q(t) \rangle_\tau$ and $u_y(t) \equiv \langle y(t) \rangle_\tau$. 

Figure 1. Schematic description of a typical lock-in-type DSFM detection unit, of the processing of the signals in the frequency domain and of the electronics used to measure thermal noise in our experiments. As discussed in the main text, the signal to be analyzed by the DSFM detection unit is assumed to be centered around some frequency $\nu_0$. It enters the detection unit at the input ‘in’, is amplified by some factor $g$, which can be generally selected, and is usually high-pass filtered (for simplicity the corresponding components are not shown) before it is multiplied by two reference signals in quadrature at some frequency $\nu_{\text{ref}}$. After this multiplication, the signal is shifted to the frequencies $(\nu_0 - \nu_{\text{ref}})$ and $(\nu_0 + \nu_{\text{ref}})$. The resulting signals are then low-pass filtered, to remove the higher frequency component $(\nu_0 + \nu_{\text{ref}})$, and brought to the outputs $u_x(t)$ and $u_y(t)$. These signals can then be analyzed using different equipment: time domain data can be acquired for further processing using a digital oscilloscope, a spectrum analyzer, or directly with the acquisition electronics of the SFM system (termed DSP in the figure). Spectra can be acquired directly with the spectrum analyzer. Finally, noise measurements can be performed with a lock-in amplifier.

is used to excite the cantilever at resonance, then $\nu_r = \nu_0$, $u_x = 0$. In a recent work [26] we have analyzed in detail how such a DSFM-DU processes signals in the presence of thermal noise. In particular, it was shown that the DSFM-DU ‘frequency-shifts’ a signal $u_d(t) = \text{Re}[a(\nu)e^{2\pi i \nu t}]$ with

$$a(\nu) = x(\nu) + iy(\nu) = \frac{a_0}{1 - (\nu/\nu_0)^2 + i(\nu/\nu_0)/Q} = a_0 g(\nu)$$

(2)

at its input, to the sum and difference frequencies, where $g(\nu)$ is the complex ‘mechanical gain’ of the cantilever. The outputs ‘phase’ and ‘amplitude’ have frequency components at $\nu_{\Sigma} = \nu_0 + \nu_r$ and $\nu_{\Delta} = \nu_0 - \nu_r$. $u_x(t) = \text{Re}[M(t)]$ and $u_y(t) = \text{Im}[M(t)]$ with

$$M(t) = \frac{a(\nu)}{2} \left[ \frac{1}{1 + i2\pi \nu_{\Sigma} t} e^{-2\pi i(\nu_{\Sigma} + 1/2) t} + \frac{1}{1 + i2\pi \nu_{\Delta} t} e^{i2\pi i(\nu_{\Delta} + 1/2) t} \right]$$

(3)

where the matrix notation in [26] has been translated into the more compact complex notation. When the input signal is not at a well-defined frequency, but distributed around a central frequency $\nu_0$, $u_d(t) = \text{Re} \left[ \int d\nu u_d(\nu)e^{2\pi i \nu t} \right]$, then the outputs of the DSFM-DU will be:

$$u_x(t) = \frac{1}{2} \text{Re}[D(t)] \quad \text{and} \quad u_y(t) = \frac{1}{2} \text{Im}[D(t)]$$

(4)
with

\[ D(t) = \int dv_A u_d(v_{\text{ref}} + v_A) e^{2\pi i v_A t} \frac{1}{1 + 2\pi v_A \tau} \]

(5)

where we have assumed that the term with the sum frequency \( \nu_{\Sigma} \) (see relation (3)) can be neglected because the constant \( \tau \) of the DSFM-DU is much larger than the time \( 1/\nu_{\Sigma} \) (see also figure 1). The DSFM-DU therefore shifts, as stated above, the spectrum of the deflection signal \( u_d(t) \) centered at \( v_0 \) to the spectra of the in-phase and out-of-phase signals \( u_x(t) \) and \( u_y(t) \) centered at \( v_{\Delta} = v_0 - v_r \).

2.2. Measurement of thermal noise with a DSFM dynamic unit

In this section we will show that a DSFM-DU indeed detects and frequency shifts thermal noise. Figure 1 shows schematically the setup used to verify this statement. The data of interest—the deflection signal \( u_d \) or the DSFM signals \( u_x \) and/or \( u_y \)—can be analyzed with a digital oscilloscope, with a lock-in amplifier, with a spectrum analyzer or with the data acquisition module of the SFM system itself.

Figure 2 shows experimental data for the deflection signal \( u_d(t) \) and the output \( u_c(t) \) acquired with the DSFM-DU configured, as discussed above (no oscillation amplitude and \( v_{\text{ref}} \neq v_0 \)). The main graphs show spectra, and the insets time domain data. In this case, the spectra have been acquired using a spectrum analyzer [27] as well as the ‘noise’ function of a commercial lock-in amplifier [28]. The lines through the data points correspond to a ‘Lorentzian + offset’ fit function \( f(v) \) [19, 26],

\[ f(v) = \frac{\epsilon_{h0}^2 e^{g(v)}}{(1 - (v/v_0)^2)^2 + (v/(v_0Q))^2} + \epsilon_n^2 \]

\[ = \epsilon_{h0}^2 |g(v)|^2 + \epsilon_n^2 \]

(6)

where \( g(v) \) is the mechanical gain of the cantilever (see relation (2)). The corresponding parameters for the different fits are specified in table 1, and discussed in more detail below. The scattered points around the horizontal lines show the error of the experimental power spectrum to the corresponding fit, which show little tendency for both spectra, therefore we conclude that the chosen fit functions describe the experimental data well. The spectrum corresponding to the deflection signal \( u_d(t) \), and those corresponding to the ‘amplitude’ and ‘phase’ signals \( u_x(t) \) and \( u_y(t) \), lead to essentially the same results, in particular for the values of the resonance frequency \( v_0 \) and the quality factor \( Q \). Since the DSFM-DU internally amplifies the normal force signal, the signal strength of the deflection signal \( u_d(t) \) is smaller than that of the DSFM-DU outputs \( u_x(t) \) and \( u_y(t) \), leading to different thermal noise \( \epsilon_{h0} \) and electronic noise \( \epsilon_n \) for the deflection spectrum \( u_d(v) \) compared to \( u_x(v) \) and \( u_y(v) \). We therefore conclude that the outputs \( u_x(t) \) and \( u_y(t) \) of the DSFM-DU essentially reproduce the thermal noise spectrum of the deflection signal and that thermal noise can be ‘seen’ at its outputs.

3. Thermal noise calibration of the oscillation amplitude

Since the DSFM-DU processes internally amplified thermal noise, it is possible to apply the thermal calibration method based on the equipartition theorem to the calibration of the oscillation amplitude. As for the case of normal force calibration discussed in the introduction, the relation describing the root mean square deviation,

\[ \langle z^2(t) \rangle_{\text{ms}} = \frac{kT}{c} \]

(7)

has implicitly two unknowns: the force constant \( c \) of the cantilever and a sensitivity calibration \( \beta \) (units: nm V$^{-1}$) to convert the measured signal \( u(t) \) (volts) into physical length units (nanometers):

\[ z(t) = \beta u(t). \]

(8)

In the present work, we will assume that the force constant is known or has been determined independently (see, for
Table 1. Results obtained with the two calibration procedures applied to the measured deflection and amplitude noise acquired from a cantilever with a force constant $c = 1.6 \text{ N m}^{-1}$.

| Units | $u_{on}^{rms}$ | $u_{off}^{rms}$ | $u_{th}^{rms}$ | $\beta^{1/2}$ | $\beta_N$ | $\nu$ | $Q$ | $\epsilon_{th}$ | $\epsilon_n$ | $\beta^{1/2}_{10}$ | $\beta_N$ |
|-------|----------------|------------------|----------------|---------------|-----------|------|-----|----------------|--------------|----------------|-----------|
| DSP   | \langle u_d(t) \rangle | 5.4 | 0.7 | 5.3 | 9.7 | 39 | 67 438.0(±14 ppm) | 138.6(±0.6%) | 1.3(±0.5%) | 30(±1.9%) | 10.39(±0.8%) | 36.7 |
| LI    | $u_d(t)$ | — | — | — | — | — | 67 440(±40 ppm) | 137(±2.4%) | 0.35(±2.4%) | 3(±66%) | — | 38.7 |
| SA    | \langle u_d(t) \rangle | 1.8 | 0.39 | 1.7 | — | 30 | 67 441(±160 ppm) | 140(±6.3%) | 0.30(±5%) | 5(±12%) | — | 39 |
|       | $u_d(t)$ | 5.4 | 0.56 | 5.4 | 9.6 | 38 | 67 440(±5 ppm) | 137.8(±0.22%) | 1.33(±0.2%) | 20(±1.5%) | 10.12(±0.3%) | 35.8 |
example [16, 29], and will thus use (7) to calibrate the sensitivity $\beta$ of the DSFM-DU. We will present and discuss two procedures based on relation (7): a simple one which only requires estimation of the root mean square (RMS) noise of the output of the DSFM-DU, and a second one taking into account the full spectral response of the outputs $u_x(t)$ and $u_y(t)$.

### 3.1. Calibration based on root mean square estimation

The first calibration procedure is implemented experimentally by acquiring the outputs $u_x(t)$ and $u_y(t)$ for a certain period of time with the laser on and with the laser off. The noise power that is related to thermal noise is then, 

$$
\langle (u_{th}^2) \rangle = \langle u_{on}^2 \rangle_{rms} - \langle u_{off}^2 \rangle_{rms}
$$

with 

$$
\langle u_{on/offs}^2 \rangle_{rms} = \frac{1}{T} \int_0^T u_{on/off}^2(t) \mathrm{d}t.
$$

The reason to acquire data with the laser off is to estimate and subtract electronic noise that is not related to the thermal noise of the cantilever motion. In particular, for hard cantilevers, where thermal noise is quite small (see below), the calibration may be severely wrong if this electronic noise is not subtracted. Without subtraction, it is implicitly assumed that all measured noise (that is, also electronic noise) is thermal noise.

The voltage noise $\Delta u_{th}^x = \sqrt{\langle (u_{th}^x)^2 \rangle}$ of the output $u_x(t)$ is related to the ‘phase’ noise while the noise $\Delta u_{th}^y = \sqrt{\langle (u_{th}^y)^2 \rangle}$ is related to the oscillation amplitude. Relation (9) can, of course, also be applied to the fluctuations measured in the deflection signal $u_{th}(t)$, then $\Delta u_{th} = \sqrt{\langle (u_{th})^2 \rangle}$ is the deflection noise (in volts) of the cantilever motion. We recall that thermal noise is distributed equally between the in-phase and out-of-phase components. Then, if the amplification of the thermal noise is distributed equally between the in-phase and out-of-phase components of cantilever oscillation. For the outputs $u_x(t)$ and $u_y(t)$ as well as the deflection $u_{th}(t)$ are the same (the latter is usually not the case, see below), the thermal noise measured in the different channels is

$$
\Delta u_{th}^x = \Delta u_{th}^y = \Delta u_{th}^z / \sqrt{2}
$$

since the total deflection signal and the in-phase and out-of-phase are related by $u_{th}^x(t) = u_{th}^x(t) + u_{th}^y(t)$ and therefore $\beta^x = \beta^y$ (see, for example, [26]). The values $\Delta u_{th}^x$, $\Delta u_{th}^y$ and $\Delta u_{th}^z$ can be either computed directly from the time domain signals $u_{th}^x(t)$, $u_{th}^y(t)$ and $u_{th}^z(t)$, as defined by relation (10), or by adding (integrating) the noise spectrum in the frequency domain. Due to the different normalizations found for the Fourier transform and the power spectral density in the literature as well as in software packages, it is usually more secure to use the first option. Calibration factors are computed directly as (see relation (11) for the factor $\beta^x$).

$$
\beta^x = \Delta u_{th}^x / \sqrt{2} = \beta^y = \Delta z_{th} / \Delta u_{th}^z / \sqrt{2},
$$

with $\Delta z_{th} = \sqrt{kT/C}$ being the rms thermal noise movement (unit: nm, from relation (1)).

### 3.2. Calibration using the thermal noise spectrum

The second calibration procedure is based on estimating the area under the thermal noise spectrum using the values determined from the fit to a ‘Lorentzian function + electronic noise function’, $f(\nu)$ from relation (6). In this context we recall that $\int_0^\infty \mathrm{d}\nu \ |g(\nu)|^2 = Q \nu_0 \pi/2$, therefore the measured ‘thermal noise power’ as determined from the fit parameters is $\langle u_{th}^x \rangle^2 Q \nu_0 \pi/2$, where $\langle u_{th}^x \rangle^2$ is the strength of the thermal signal (units: V² Hz⁻¹). As discussed previously, in the frequency domain the DSFM-DU shifts the original deflection signal $u_{th}(\nu)$ from a signal centered around the resonance frequency $\nu_0$ to signals $u_x(\nu_0)$ and $u_y(\nu_0)$ centered at $\nu_0 - \nu_r \approx \nu_0 / 2$, where $\nu_r$ is the reference frequency of the DSFM-DU. In addition, the original signal $u_{th}(\nu)$ is amplified by the internal gains of the DSFM-DU. In principle, there are two possible ways of processing the data obtained from the DSFM-DU: the spectra $u_x(\nu_0)^2$ and $u_y(\nu_0)^2$ may be fitted either directly to the function $f(\nu)$ from relation (6)—resulting in a resonance peak near $\nu_0 / 2$ (see figure 2)—or the frequency shift induced by the DSFM-DU may be ‘undone’ by adding the reference frequency to the frequency of the power spectra $u_x(\nu + \nu_r)$ and $u_y(\nu + \nu_r)$.

- Option 1: $u_x(t)$ raw data → Fourier transform and power spectral density $\rightarrow |u_x(\nu)|^2 \rightarrow \text{fit} \rightarrow \{e_{th}, Q, \nu_0, \epsilon_n\}$.

- Option 2: $u_y(t)$ raw data → Fourier transform and power spectral density $\rightarrow |u_y(\nu)|^2 \rightarrow \text{frequency shift} \rightarrow |u_x(\nu + \nu_r)|^2 \rightarrow \text{fit} \rightarrow \{e_{th}, Q, \nu_0, \epsilon_n\}$.

The second option is the correct one, as relation (1) holds for the ‘true’ deflection signal with the thermal noise peak at the ‘correct’ resonance frequency $\nu_0$.

We therefore fit the frequency-shifted spectrum $|u_x(\nu + \nu_r)|^2$ to the ‘Lorentzian + offset’ function $f(\nu)$ from relation (6), in order to obtain the ‘strength’ $\epsilon_{th}$ of the Lorentz function, the quality factor $Q$, the resonance frequency $\nu_0$ and the noise $\epsilon_n$, which is not Lorentzian. For the experimental spectra of the output $u_x(t)$, relation (1) is rewritten as

$$
\frac{1}{2} kT = \frac{c}{2} \langle \epsilon_{th}(t) \rangle_{rms} = \frac{c}{2} \int \mathrm{d}\nu \ \epsilon_{th}^2 |g(\nu)|^2
$$

$$
= \frac{c}{2} \int \mathrm{d}\nu \ (e_{th} \beta^x)^2 |g(\nu)|^2
$$

$$
= \frac{c}{2} Q \nu_0 \pi \ (e_{th} \beta^x)^2 \Rightarrow \beta^d = \frac{1}{e_{th}^2} \sqrt{\frac{2kT}{\pi e Q \nu_0}}
$$

where $\beta^d$ (unit: nm V⁻¹) is the factor that converts the deflection signal $u_{th}(\nu)$ (in volts) to the physical amplitude $z(t)$ (in nm): $z(t) = \beta^d u_{th}(t)$. Similarly, $\beta^x$ and $\beta^y$ convert the signals $u_x(t)$ and $u_y(t)$ into the in-phase and out-of-phase components of cantilever oscillation. For the outputs $u_x(t)$ and $u_y(t)$ relation (1) is
the lower three scatter plots to data for gains light blue: signals with the laser off. The upper three scatter plots of the graph correspond to amplitude scatter plots (Graph (B), lower right graph: spectrum of the deflection versus time signal $u(t)$, measured with a digital oscilloscope. Inset: phase versus amplitude scatter plots $(u_x(t), u_y(t))$ of the data that was used to compute the spectra $u_i(v)$ shown left. Dark blue: signals with the laser on, light blue: signals with the laser off. The upper three scatter plots of the graph correspond to $(u_x(t), u_y(t))$ data for gains $g = 100, 30$ and 10; the lower three scatter plots to data for gains $g = 3, 2$ and 1.

\[
\frac{1}{2} k T = \frac{c}{2} \int \!dv \left( (e_{th}^x \beta_x)^2 + (e_{th}^y \beta_y)^2 \right) |g(v + v_0)|^2 \\
= \frac{c}{2} \frac{\pi}{Q} \left( e_{th}^x \beta_x \right)^2 \\
\Rightarrow \beta^x = \frac{1}{\sqrt{2 e_{th}^x \pi} \sqrt{\frac{2 k T}{Q} \frac{\pi}{Q}}} = \frac{1}{\sqrt{2 e_{th}^x \pi}} \sqrt{\frac{2 k T}{Q} \frac{\pi}{Q}} = \beta^x.
\]

The last relation in (14) is obtained because in our case the amplification of the two outputs $u_x(t)$ and $u_y(t)$ is the same; therefore, as discussed above, $\beta^x = \beta^y$.

4. Results

4.1. Soft cantilever

Here we will discuss the results obtained for a relatively soft cantilever (Olympus Silicon cantilever, length $= 240 \, \mu m$, nominal force constant $c = 2 \, N \, m^{-1}$) whose precise force constant was determined to be $c = 1.6 \, N \, m^{-1}$, calculated using Sader’s method [29] with the $Q$ factor and the resonance frequency $v_0$, obtained with the second method (see below and table 1).

First, we discuss the results obtained for RMS-calibration using the data shown in the insets of figure 2. The data acquired with the spectrum analyzer corresponds to time domain measurements. The data from the DSFM-DU is presented as a scatter plot $(u_x(t), u_y(t))$, as it would be visualized with an oscilloscope in $x - y$ mode. Data is shown for the laser on and off. The diameter of the scatter plot is proportional to the fluctuations in both directions. The data corresponding to the deflection signal $u_d(t)$ is shown ‘normally’ as a function of time, again data is shown for the laser on and off. The results obtained with this first method are summarized in the left column of table 1 (termed ‘first procedure’).

To check the second method, data was acquired using the different experimental options shown in the schematic representation of figure 1. Essentially, the idea is to acquire the spectra of the thermal noise, either directly using the noise function of the lock-in amplifier or with the spectrum analyzer; or by acquiring time domain data $u(t)$ using the spectrum analyzer, the digital oscilloscope or the acquisition electronics of the SFM system and calculating the power spectrum after data acquisition.

The parameters $Q, v_0$ and $e_{th}$ are obtained from the fits to the spectra shown in figure 2. The right columns of table 1 (termed ‘second procedure’, discussed in section 3) summarize the results obtained from the second calibration procedure when applied to the deflection data $u_d(t)$ and to the out-of-phase output $u_y(t)$ of the DSFM-DU. We will now comment on the different fields of this table. The lock-in amplifier is used to measure the noise density, and does not acquire time domain data, therefore the first procedure cannot be applied, and the corresponding fields are empty. The spectrum analyzer uses one to simultaneously measure the real-time data, as well as the spectrum of this data, therefore the first and second procedure can be applied. For this cantilever, with a relatively large thermal noise ($\Delta e_{th} = \sqrt{k T/c} \approx 50 \, pm$), the first and second calibration procedure give similar results, although the second procedure seems to have less error (see discussion below). In order to compare the results obtained from the deflection signal with that of the output(s) of the DSFM-DU a normalized calibration factor $\beta_N$ has been introduced that takes into account the internal gain of the DSFM-DU. This normalized calibration factor should be the same for the deflection data and the outputs of the DSFM-DU, which is indeed the case within the experimental error of the measurements.

4.2. Hard cantilever

Figure 3 shows the spectra of the deflection sensor and the outputs of the DSFM-DU for a hard cantilever (Olympus Silicon cantilever, length $= 160 \, \mu m$, nominal force constant $c = 50 \, N \, m^{-1}$) whose precise force constant was determined to be $c = 67 \, N \, m^{-1}$, as determined by Sader’s method [29].
Note that this cantilever has a very low thermal noise amplitude ($\Delta v_{th} = \sqrt{kT/c} \approx 8 \text{ pm}$), which is well below the detection limit of many SFM systems. Data is shown for different gains of the DSFM-DU, and the spectra have been acquired directly from the (calculated) power spectrum of the time domain signals acquired by the SFM-control unit. More precisely: the power spectrum is calculated from $u_x(t)$-images acquired with the tip far from the surface at maximum acquisition speed, without scanning and with no excitation applied to the driving piezo of the cantilever. The spectrum is calculated for each line and finally all spectra corresponding to the different lines $u_x(t)$ are averaged to obtain a (clean) mean spectrum.

Note that for hard cantilevers, the resonance frequency $f$ may be easily outside the acquisition bandwidth of the analog to digital converters of the SFM-acquisition electronics (usually 16 bits or more and thus rather slow) and therefore the corresponding thermal noise of cantilever motion cannot be measured in the digitalized normal force (deflection) signal $u_d(t)$. In contrast, the thermal noise peak is easily brought into the bandwidth of the analog to digital converters when the outputs $u_x(t)$ and $u_y(t)$ are used, since this noise is now at the much lower difference frequency. For this hard cantilever, the very small thermal noise signal is usually significantly smaller than the electronic noise or other non-thermal fluctuations. In our setup, this is indeed the case for the direct deflection signal as well as for the outputs of the DSFM-DU when its internal gain is smaller than 10. Then, it is essential to ‘normalize’ the thermal noise signal by subtracting the electronic noise (laser off) from the total noise (laser on), as discussed above (see relation (9)). Still, for low gains this procedure does not give satisfactory results; then only the second method is precise. Surprisingly the second procedure still works for gains as small as $g = 1$, as well as for the direct deflection signal. Note that for these low gains almost all noise is electronic noise. In fact, as can be observed in the scatter plots $(u_x(t), u_y(t))$ shown in the insets of figure 3, for low gains the signal measured is very similar with the laser on and off. The fit to the function $f(v) = v^2 + c_2^2$ is thus a very effective way of discriminating all non-thermal noise. For high gains the calibration factors rise by an amount that is not compatible with the error of our measurements for both procedures. This is not a problem of the calibration procedures, instead we think that this is due to low-pass filtering of our DSFM-DU that reduces the (nominal) signal strength and leads to a higher calibration value at high frequency (more nanometers of deflection are ‘needed’ per 1 V of signal).

5. Conclusion

We have presented two methods for calibration of the oscillation amplitude in DFSM, one based on simple calculation of the RMS value of the output signals $u_x(t)$ and/or $u_y(t)$ of the DSFM-DU, and the other one based on analysis of the corresponding spectrum $u_x(v)$ and/or $u_y(v)$ and calculation of the parameters $Q$, $v_0$, $e_h$ and $e_n$. From the results summarized in tables 1 and 2 we conclude that both methods give consistent results, even though the second method is considerably more precise and robust, in particular for the case of hard cantilevers, where thermal noise has a much smaller amplitude and the signal to noise ratio of thermal noise (which in this case is ‘good’ signal) versus other noise sources is much lower. The second method has several advantages: first, it explicitly ‘filters’ thermal noise from other noise sources, since it will only take into account a signal that has a Lorentzian spectrum; a signal not compatible with this shape is taken into account by the constant factor $e_n$ and the corresponding signal power is rejected for the calculation of the amplitude sensitivity. Also, fitting of a spectrum with many data points in order to obtain the four parameters $Q$, $v_0$, $e_h$ and $e_n$ results in effective data averaging, and thus additional improvement of the estimation of the thermal noise power. Finally, from a strictly theoretical point of view, the first method is not quite correct because it is only an approximation valid for high $Q$ factors. In fact, the first method only detects thermal noise in the small bandwidth $1/\tau \ll v_0$ around the resonance peak, but not all the noise below the Lorentz function, and in particular not the noise in the low frequency ‘tail’ (from DC to $v_0 - bv/2$, see [26] for a more detailed discussion). When the first method is used this noise is ‘filtered away’ by the DSFM-DU, and thus ‘lost’. Therefore the first method underestimates noise, and overestimates the amplitude sensitivity. Since the relation of thermal noise in the resonance peak to that in the low frequency ‘tail’ is $Q : 1$ (see [26], section 3) this error is negligible for experiments in air and vacuum, but is expected to be significant for the low $Q$ factors encountered in liquids. When the second method is used, this noise is not ignored, since the fit to the (frequency shifted) experimental spectrum describes the whole noise spectrum; therefore relation (14) ‘recovers’ all the thermal noise, even that which is filtered away by the DSFM-DU. Finally, we note that determination of the parameters $e_h$, $Q$ and $v_0$ using the outputs of the DSFM-DU involves a significant improvement of signal because of the internal gains of the DSFM-DU and the principle of lock-in detection, which implies an important reduction of bandwidth.

A scheme for calibration of oscillation amplitude has been presented that is based on the measurement of thermal noise after being processed by a typical DSFM-DU. This calibration scheme depends only on the knowledge of the force constant and the temperature of the cantilever, but not on a priori assumptions on specific tip-sample interaction, amplitude versus piezo-displacement or frequency versus amplitude relations. Moreover, the method is conceptually and experimentally simple: essentially only appropriate amplitude and/or phase signal data $(u_x(t)$ and $u_y(t))$ have to be acquired. In addition, it is experimentally robust and can be applied in UHV, air and liquid environment. Finally, even though the method has been presented for the calibration of oscillation amplitude, we note that it can also be applied to the calibration of the sensitivity of the deflection signal itself. Indeed, as discussed at the end of the section 4.1 (‘soft cantilever’), the normalized calibration factor $f_{by}$ is essentially the calibration factor for the deflection signal calculated from the known gains of the DSFM-DU. In particular, for hard cantilevers
Table 2. Results obtained with the two calibration procedures for a hard cantilever (force constant \( c = 67 \text{ N m}^{-1} \)).

| Units | 1st procedure | 2nd procedure | 3rd procedure | 4th procedure |
|-------|---------------|---------------|---------------|---------------|
| \( u_{\text{rms}}^{\text{on}} \) | mV | mV | mV | mV | mV | mV | mV | mV | mV | mV | mV | mV |
| \( u_{\text{rms}}^{\text{off}} \) | — | — | — | — | — | — | — | — | — | — | — | — |
| \( u_{\text{rms}}^{\text{th}} \) | — | — | — | — | — | — | — | — | — | — | — | — |
| \( \beta_{\text{G}} \) | — | — | — | — | — | — | — | — | — | — | — | — |
| \( \beta_{N} \) | — | — | — | — | — | — | — | — | — | — | — | — |
| \( v \) | Hz | Hz | Hz | Hz | Hz | Hz | Hz | Hz | Hz | Hz | Hz | Hz |
| \( Q \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) |
| \( \epsilon_{\text{th}} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) |
| \( \beta_{G} \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) | V Hz\(^{-1/2} \times (10^{-9}) \) |
| \( \beta_{N} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) | V Hz\(^{-1} \) |

For example:
- Oscillograph (Osci): 0.77 mV, 0.68 mV, 0.36 mV, 0.77 mV, 3160.66 (±7 ppm), 588 (±1.3%), 8.6 (±8%)
- DSP: 0.314 mV, 0.299 mV, 0.095 mV, 0.314 mV, 3160.60 (±120 ppm), 580 (±22%), 3.2 (±18%)
with very little thermal noise signal, this deflection calibration obtained by the DSFM-DU is much more precise than the calibration factor $\beta^d$ obtained directly from the deflection signal $u_d(t)$ (due to the internal gains of the DSFM-DU, the lock-in detection scheme and the data averaging). Moreover, it is a non-contact detection that does not require a force versus distance curve and thus avoids possible damage to the tip (recall that the cantilever is hard, thus high forces would be applied during a force versus distance curve). To conclude we think that the present calibration procedure is an important contribution which complements the methods for amplitude calibration used until now, and can be used also for calibration of the deflection/normal force signal in static SFM applications.

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