Dynamic Method for Calibration of Sensitivity in Scanning Force Microscopy

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Motivation

SFM has become a fundamental tool in many disciplines and fields. Among the different operation modes, the dynamic mode is without doubt the most important and versatile one. In this mode, the tip is oscillated at or near the resonance frequency of the supporting cantilever and the variation of resonance frequency or oscillation amplitude is used as feedback signal. For quantitative applications we believe that the calibration of the system is an issue that has not yet been resolved satisfactorily. In particular, most experiments are performed without a precise knowledge of oscillation amplitude, even though it has been shown [2] that due to the non-linearity of the SFM-system this amplitude can be a critical parameter for the behaviour of the total system.

Up to now, calibration of oscillation amplitude in a typical SFM-experiment can be performed in a variety of ways (see [3] for a recent overview), the most popular ones being based on the analysis of either the thermal noise spectrum of the cantilever [4] or a force versus distance curve [5]. In the first case, the force constant of the cantilever has to be known precisely. In the second case, contact between tip and sample has to be made, which is a serious disadvantage for fragile tips such as very sharp or functionalized ones. In this work a new technique is presented for the usual case of a piezo driven cantilever. This technique avoids the disadvantages just discussed, in addition it is very easy and fast. The essential idea underlying the technique is that the resonance amplitude of an oscillating free cantilever must somehow be related to the excitation amplitude of the driving piezo element. To find this relation, we solve the equation of motion for a piezo driven cantilever in a typical SFM-setup (see also [1]). The fundamental features of the model will be presented and discussed. First experimental results as well as simulations confirm the validity of the model.

References

[1] Hans-Jürgen Butt and Manfred Jaschke, *Calculation of thermal noise in atomic force microscopy*, Nanotechnology 6, pp. 1-7, 1995.

[2] A. S. San Paulo and R. García, *Unifying theory of tapping-mode atomic force microscopy*, Phys. Rev. B 66, 041406 (2002); and, in spanish, Alvaro San Paulo, *Dinámica de la Microscopía de Fuerzas con modulación de amplitud: descripción teórica e implicaciones experimentales*, PhD-Thesis CSIC-Dept. Física de la Materia Condensada (UAM), Madrid.

[3] N.A. Burnham, *et al.*, *Comparison of calibration methods for atomic–force microscopy cantilevers*, Institute of Physics Technology, Nanotechnology 14, pp. 1-6, 2003.

[4] J. L. Hutter and J. Bechhoefer, *Calibration of atomic-force microscope tips*, Rev. Sci. Instruments 64 (7), 1993.

[5] A.L Weisenhorn, P.K Hansma and T. R. Albrecht, *Forces in atomic force microscopy in air and water*, Applied Physics Letters, Vol. 54 No. 26, pp. 2651-2653, 1989.

Properties of the solutions

The family of "eigenfunctions" $\phi_i(\zeta)$ have important mathematical properties:

- They are eigenfunctions of the "self-adjoint" differential operator $\widehat{D}^{(4)} \equiv \partial^4/\partial\zeta^4$, $\widehat{D}^{(4)}\phi_i(\zeta) = \kappa_i^4\phi_i(\zeta)$.
- They are normalized with respect to the usual scalar product for functions, $\langle\phi_i, \phi_i\rangle = \int_0^1 |\phi_i(\zeta)|^2 d\zeta$.
- They are mutually orthonormal, $\langle\phi_i, \phi_j\rangle = \delta_{ij}$. In addition the extension is normalized, $\phi_i(1) = 2 \quad \forall i$, therefore $z_i(ky) = z_0\phi_i(\kappa\zeta)/2$, has an amplitude z_0 at the free end.
- Any function in the interval $[0, 1]$, can be decomposed as $f(\zeta) = \sum_n^\infty c_n\phi_n(\zeta)$ with $c_n = \int_0^1 f(\zeta)\phi_n(\zeta)d\zeta$. For a constant function $f(\zeta) = b_0$, we find using (2) and the boundary condition $\phi'''(1) = 0$,

$$c_n = \int_0^1 f(\zeta)\phi_n(\zeta)d\zeta = \frac{1}{\kappa^4}\int_0^1 b_0\frac{d^4\phi_n(\zeta)}{d\zeta^4}d\zeta = b_0\frac{\phi_n^{(3)}(0)}{\kappa^4} \tag{6}$$

Piezo driven cantilever — The important factor

Up to now we have treated only the homogeneus problem, i.e. no external forces acts on the cantilever. In present context is necessary to generalize the model to account for friction and external forces. The corresponding equation of motion is,

$$\rho S\omega^2 z(y) + EIz^{(4)}(y) + i\omega\gamma z(y) = f(y) \tag{7}$$

where $f(y)$ describes any loading force (per unit length) acting on each section of the cantilever. For an inertially driven cantilever, the driven force is an "external" inertial force, $f^{\text{ext}}(y) = \rho S\omega^2 z_p(y) = \rho S\omega^2 z_p^0\phi(\kappa\zeta)$. In analogy to the case of a driven harmonic oscillator we find for each mode

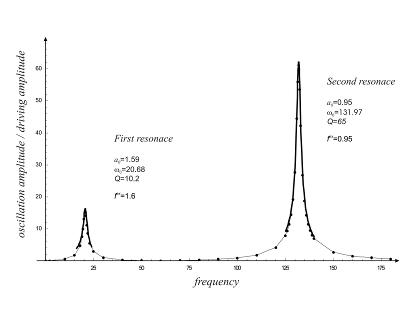
$$z_{lever}^n(\omega_n) = 2z_p^0 c_n\omega^2 / \sqrt{(\omega_n^2 - \omega^2)^2 + (\omega\omega_0/Q)^2} \tag{8}$$

and thus at resonance $z_{lever}^n(\omega_0) = 2z_p^0 Qc_n$, where Q is the quality factor. The model discussed above essentially predicts a (mode-dependent) constant factor $\mathscr{F}_n$ relating the oscillation amplitude of the clamped end of the cantilever to the oscillation amplitude of the free end. Using (6)

$$\mathscr{F}_n = \frac{z_{lever}^n(\omega_0)}{Qz_p^0} = 2\frac{\phi'''(0)}{\kappa^4} \tag{9}$$

Simulation results

To verify the validity of our model, a simple numerical simulation has been implemented. The equation of motion (7) has been discretised and solved as a function of driving frequency. A frequency interval containing the first two resonances was chosen. In total 3 different spectra were simulated each with a different friction coefficient. From the measured curves the corresponding Q-factor and the factor $\mathscr{F}_{\text{simu}}$=Amplitude $(\omega_0)/(Q$ Driving amplitude) were calculated.
The corresponding results are summarized in the table.



	$\mathscr{F}_n$	
γ	Mode 1	Mode 2
0.1	1.57	0.96
0.5	1.58	0.96
1.0	1.59	0.95

Experimental results

The experimental procedure discussed above was applied with three different rectangular cantilevers. Since optical beam deflection was used, the correction factor discussed above has to be taken into account. In our experiments, we found the following issues important:

- The resulting amplitude should not be too high. High oscillation amplitude when tip and sample are in contact result in a non-harmonic response of cantilever motion, possibly due to spurious frictional effects. To minimise these effects, a low-friction surface such as for example graphite should be used
- The experiments, that is the determination of the free cantilever motion and the corresponding calibration of the motion with tip and sample in contact should be performed at the resonance frequency. Note that for an "ideal" driving this is not necessary, since the usual "dither" piezos used for cantilever excitation have resonance frequencies well over 1 Mhz and therefore have a flat response with unitary gain at frequencies below 1 Mhz. Unfortunately once mounted in the whole SFM set-up, the response is not flat and spurious resonances appear, that have to be taken into account by the procedure just described.

Theory

The equation of motion of a SFM cantilever

A SFM cantilever can be considered a bar clamped on one side and free at the other. Its equation of motion is:

$$\rho S\ddot{z}(y,t) + EI\frac{\partial^4 z}{\partial y^4}(y,t) = 0 \tag{1}$$

with S the cross section, ρ, density, E, modulus of Young and I, moment of inertia. The first term represents the inertial force (per unit length) and the second term the elastic force per unit length. Separating variables, $z(y,t) = z(y)e^{i\omega t}$, we find,

$$-\rho S\omega^2 z(y) + EIz^{(4)}(y) = 0 \leftrightarrow z^{(4)}(y) = k^4 z(y) \qquad k = \left(\frac{\rho S\omega^2}{EI}\right)^{1/4} \tag{2}$$

We are thus looking for a function that essentially repeat itself after four derivatives. The general solution is $z(y) = z_0\left[\alpha_1\cos(ky) + \alpha_2\sin(ky) + \alpha_3\cosh(ky) + \alpha_4\sinh(ky)\right]$ where z_0 is a parameter that defines the strength of oscillation (with dimension of length), and the parameters α_i defines the "strength" of the four basic functions, and are determined by boundary conditions. In what follows, in addition to the physical solution $z(y)$, it will be useful to define a dimensionless "mathematical solution"

$$\phi(\kappa\zeta) = \alpha_1\cos(\kappa\zeta) + \alpha_2\sin(\kappa\zeta) + \alpha_3\cosh(\kappa\zeta) + \alpha_4\sinh(\kappa\zeta) \tag{3}$$

so that $z(ky) = z_0\phi(\kappa\zeta)$ where $k = \kappa/l$ and $y = \zeta l$. Now we impose the boundary conditions,

$$\phi(0) = 0; \quad \phi'(0) = 0; \quad \phi''(1) = 0; \quad \phi'''(1) = 0 \tag{4}$$

These boundary conditions lead to

$$\cos(\kappa\zeta) = \frac{-1}{\cosh(\kappa\zeta)} \tag{5}$$

which defines the solutions $\kappa_i = \{1.875,\ 4.609,\ 7.855, \ldots, (n+1/2)\,\pi\}$.

Verification of the model — Results

According to the theory just discussed, to experimentally verify the model it seems sufficient to:

(a) bring tip and sample into contact and drive the piezo element with a given driving voltage. For a stiff sample, cantilever motion equals piezo motion and the detected signal U^{pd} can be related to the tip motion via an appropriate calibration factor (see below),

(b) take a spectrum of the cantilever oscillation, that is, excite the driving piezo at different frequencies and record the response,

(c) measure the oscillation amplitude at resonance (at the same driving voltage as in (a)).

From the data obtained in (a)-(c) the experimental factor between the oscillation amplitude of the clamped end and the oscillation amplitude of the free end is:

$$\mathscr{F}^{\text{exp}} = \frac{U_{\text{resonance}}^{pd}/Q}{U_{\text{contact}}^{pd}} \tag{10}$$

Unfortunately, for the optical beam deflection technique, this simple procedure is not quite correct, since it does not take into account the fact that this method detects angles and not directly displacements. Therefore, a correction term has to be introduced. We first recall some basic points related to calibration issues in the optical beam deflection technique. The experimentally measured photodiode signal U^{pd} and the true deflection, measured either as displacement (unit: [m]) or angle are related by calibration factors:

$$z_{\text{stat}}(l) = e_{\text{stat}}^{\text{displacement}}U^{pd}; \qquad z'_{\text{stat}}(l) = e_{\text{stat}}^{\text{angle}}U^{pd} \tag{11}$$

In the static deflection mode displacement and angle are related by $z'(l) = 3z(l)/(2l)$, therefore we have

$$e_{\text{stat}}^{\text{angle}} = \frac{3}{2l}e_{\text{stat}}^{\text{displacement}} \tag{12}$$

Static and dynamic modes are different accordingly the calibration factors cannot be assumed the same, we define,

$$z_n(l) = e_n^{\text{disp}}U^{pd}; \quad z'_n(l) = e_n^{\text{angle}}U^{pd} \tag{13}$$

In analogy to (12) we find for the dynamic modes $e_n^{\text{angle}} = e_n^{\text{disp}}\phi'_n(kl)/l\phi_n(kl)$, and finally,

$$z_n(l) = e_n^{\text{disp}}U^{pd} = \frac{3}{2}\frac{\phi_n(kl)}{\phi'_n(kl)}e_{\text{stat}}^{\text{disp}}U^{pd}; \quad \mathscr{F}_n^{\text{beam-def}} = \frac{4\phi'''_n(0)\,\phi'_n(kl)}{3\kappa^4\phi_n(kl)} \tag{14}$$

Conclusion

- A model for a piezo driven SFM cantilever has been developed that allows to calculate the free oscillation amplitude as a function of the driving motion applied to the clamped end of the cantilever.
- Within this model, the free oscillation amplitude for each oscillation mode is determined by the quality factor of the oscillation and parameters related to the properties of each eigenmode.
- For the case of the beam deflection technique, correction factors have to be introduced to take into account that this mode detects variation on angles, and not directly variation of displacements.
- First numerical simulations of the behaviour of the cantilever at the first and second resonance frequency give a reasonably good agreement with the model (very good agreement for the first mode, less good agreement for the second mode, possibly the simulation routines have to be improved).
- Our experiments also give reasonable agreement with the model.

	$\mathscr{F}_n$	
Lever	Mode 1	Mode 2
soft (0.01 N/m)	1.6	0.6
hard 1 (0.75 N/m)	1.8	-
hard 2 (0.75 N/m)	1.9	-

We therefore propose as very simple and fast way of calibrating sensitivity in a SFM-setup

- Calibrate (once!) the response of the driving piezo at the resonance frequency of the cantilever (for example from a force vs. distance curve or from thermal noise)
- Then, for any new cantilever measure its oscillation spectrum, calculate its Q-factor and finally the sensitivity of the detection system follows as

$$\mathscr{F}_n^{\text{beam-def}} = \frac{2\phi'''_n(0)}{\kappa^4} = \{1.57,\ 0.87,\ 0.59, \ldots\} \tag{15}$$

or (for the optical beam deflection scheme)

$$\mathscr{F}_n^{\text{beam-def}} = \frac{4\phi'''_n(0)\,\phi'_n(kl)}{3\kappa^4\phi_n(kl)} = \{1.43,\ 2.77, \ldots,\ 8/3\} \tag{16}$$