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Note: Calibration of atomic force microscope cantilevers using only their resonant frequency and quality factor

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A simplified method for calibrating atomic force microscope cantilevers was recently proposed by Sader *et al.* [Rev. Sci. Instrum. **83**, 103705 (2012); Sec. III D] that relies solely on the resonant frequency and quality factor of the cantilever in fluid (typically air). This method eliminates the need to measure the hydrodynamic function of the cantilever, which can be time consuming given the wide range of cantilevers now available. Using laser Doppler vibrometry, we rigorously assess the accuracy of this method for a series of commercially available cantilevers and explore its performance under non-ideal conditions. This shows that the simplified method is highly accurate and can be easily implemented to perform fast, robust, and non-invasive spring constant calibration. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4901227]

The spring constant of an atomic force microscope (AFM) cantilever is required in many applications of the AFM.[1–4] Due to variability in the manufacturing process, *in situ* calibration of this parameter is critical for accurate and robust force measurements. Many different methods have been proposed to calibrate an AFM cantilever, including direct estimation from its dimensions, monitoring its static deformation due to an applied load or measurement of the cantilever's dynamic properties; see Refs. 4–8. While accurate nanomechanical characterization of AFM cantilevers has been extensively investigated and optimized, some methods require additional independent measurements that may include, for example, electron and/or high-resolution optical microscopy. These additional measurements can impose strong practical constraints that limit the broad application of these techniques. It is therefore desirable to have calibration methods that eliminate such requirements, and can be routinely implemented and used in the AFM with minimal effort.

The method of Sader *et al.*[9,10] utilizes the hydrodynamic flow generated by an oscillating cantilever to measure its spring constant, k,

$$k = \rho b^2 L \, \Lambda(\mathrm{Re}) \omega_R^2 Q. \tag{1}$$

This method requires the angular resonant frequency, $\omega_R = 2\pi f_R$, and quality factor, Q, of the cantilever in fluid (typically air), knowledge of its plan-view geometry and dimensions (b and L are the cantilever width and length) and its hydrodynamic function, $\Lambda(\mathrm{Re})$, where the Reynolds number Re depends on the resonant frequency and width of the cantilever, and the fluid density, ρ, and viscosity, μ. While this method facilitates rapid, accurate and non-invasive calibration of arbitrarily shaped cantilevers, it requires measurement/calculation of the hydrodynamic function for each cantilever geometry. This can hinder practical implementation, especially given the rapidly expanding range of commercially available cantilevers, e.g., see Fig. 1 of Ref. 10 and Table I.

To alleviate this requirement and facilitate its broad application, a simplification to Eq. (1) was proposed in Ref. 10 that is applicable to cantilevers of the same geometry, which also have identical widths and lengths. This simplified method relates the spring constant, resonant frequency, and quality factor of a reference cantilever, to the same parameters of the cantilever to be calibrated. The spring constant, k, of the uncalibrated cantilever can then be measured using

$$k = k_{\mathrm{ref}} \frac{Q}{Q_{\mathrm{ref}}} \left(\frac{f_R}{f_{R,\mathrm{ref}}} \right)^{2-\alpha}, \tag{2}$$

where f_R and Q are the resonant frequency and quality factor of the uncalibrated cantilever, respectively, $\alpha = 0.7$ (see Ref. 10, and below) and the subscript "ref" refers to the reference cantilever. The spring constant can be adjusted analytically for any difference in the imaging-tip positions between the reference and uncalibrated cantilevers; see discussion in

TABLE I. Reference cantilever devices of different plan-view geometry, denoted "type," measured using LDV in air. Uncertainty in the spring constants is based on a 95% confidence interval;[10] this dominates uncertainty in the resonant frequencies, f_R, and quality factors, Q, which are approximately $\pm 0.01\%$ and $\pm 1\%$, respectively. Nominal dimensions for all devices are listed. All devices are arrow-shaped, except TR800(L) which is V-shaped.[10] Types C and F are similar in shape, as are types A and E; they differ in their widths only.

Type	Reference devices	b (μm)	L (μm)	f_R (kHz)	Q	k_{LDV} (N/m)
A	AC240TS[10]	30	240	83.0	213	2.90 ± 0.13
B	TR800(L)[10]	30	200	22.9	57	0.194 ± 0.006
C	AC160TS-R3	40	160	306	477	29.0 ± 0.5
D	AC200TS-R3	40	200	136	281	6.94 ± 0.22
E	AC240TM-R3	40	240	64.5	147	1.58 ± 0.03
F	AC160TS[10]	50	160	370	646	57.3 ± 1.9

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Ref. 10. All that is required to use Eq. (2) is independent and accurate calibration of a single reference cantilever – laser Doppler vibrometry facilitates this measurement, see below. Thus if manufacturers were to calibrate a single sample cantilever and report this reference data, it would allow all cantilevers of the same geometry to be easily calibrated by the user.

Since the complete method in Eq. (1) is weakly dependent on variations in the plan-view dimensions of the cantilever,[10] Eq. (2) is also expected to be only weakly affected by such changes. Consequently, even though Eq. (2) is derived assuming the reference and uncalibrated cantilevers have identical plan-view dimensions (and geometry), variations in these dimensions are expected to exert a weak effect; a property that we assess experimentally here. This simplified method, Eq. (2), therefore provides a good compromise between the very high accuracy achievable using Eq. (1) and ease of implementation, by eliminating the need to explicitly measure the hydrodynamic function and the cantilever dimensions; the simplified method is also highly accurate, as we demonstrate below.

Recently, the performance of Eq. (2) was compared to other AFM calibration techniques, for which favorable agreement was observed.[11,12] However, these AFM techniques invariably produce results without strict error bounds – the accuracy of Eq. (2) is therefore currently unknown. In this study, we use laser Doppler vibrometry (LDV) to provide accurate benchmark results for the spring constants[10,13,14] and thus rigorously assess Eq. (2) for a range of different cantilevers. This shows that the simplified method in Eq. (2) exhibits high accuracy, at least equivalent to those of calibration methods currently used in the AFM, while dramatically simplifying implementation – all that is required is measurement of the cantilever resonant frequency and quality factor in air, which is already routinely performed.

Two sets of cantilevers are used to evaluate the accuracy of Eq. (2). The first are reported in Ref. 10, for which all required parameters of the reference cantilever in Eq. (2) are already available. The second set of cantilevers has similar geometries but different dimensions to those studied in Ref. 10, enabling a more detailed assessment of Eq. (2) and exploration of its robustness under variations in the plan-view dimensions. Six different cantilever geometries are studied here; see Table I. The cantilever sets, (i) AC160TS and AC240TS, and (ii) AC160TS-R3 and AC240TS-R3, differ only in their widths: cantilevers in (i) have widths of 50 and 30 μm, respectively, while (ii) both have a width of 40 μm.

The spring constant, resonant frequency, and quality factor of each reference cantilever are measured in air using a laser Doppler vibrometer [MSA-500 Micro System Analyzer, Polytec, Waldbronn, Germany], applying the protocol in Ref. 10; data are given in Table I. This protocol involves measuring the spring constant at a series of positions along the major axis of the cantilever, from which the spring constant at the imaging-tip position is determined. Use of this protocol ensures accurate measurement of the spring constant. The dynamic spring constant is reported here; a simple rescaling gives the static spring constant[10] – note that Eq. (2) is applicable to both spring constants. This LDV measurement protocol also provides the resonant frequency and quality factor of each cantilever; the measured uncertainty in the spring constant, typically ± 2 to $\pm 5\%$ based on a 95% confidence interval, dominates the overall uncertainty in Eq. (2).[10]

TABLE II. Spring constants obtained using the simplified method, Eq. (2), for cantilevers of the same type as in Table I. Uncertainty in the spring constants is based on a 95% confidence interval.

Type	Device	f_R (kHz)	Q	k (N/m) LDV	k (N/m) Eq. (2)
A	AC240TM[10]	65.9	162	1.65 ± 0.06	1.64 ± 0.07
	ASYMFM[10]	69.4	187	2.13 ± 0.06	2.02 ± 0.09
B	TR400(L)[10]	11.8	22	0.0293 ± 0.0027	0.0308 ± 0.0010
C	AC160TS-R3	300	468	26.8 ± 0.5	27.7 ± 0.5
	AC160TS-R3	304	478	29.0 ± 1.1	28.8 ± 0.5
D	AC200TS-R3	139	287	7.40 ± 0.36	7.21 ± 0.22
E	AC240TS-R3	65.7	133	1.36 ± 0.13	1.46 ± 0.03
	AC240TS-R3	59.2	123	1.26 ± 0.12	1.19 ± 0.02
	AC240TM-R3	65.2	149	1.58 ± 0.04	1.63 ± 0.03

The variable "type" listed in Table I classifies the different cantilever geometries; cantilevers of the same type have identical plan-view geometry, i.e., shape. The reference data in Table I is used with Eq. (2) to calibrate the spring constants of other, identically shaped cantilevers, solely through measurement of their resonant frequencies and quality factors. This is again performed in air using LDV, which also independently gives the spring constants for these other cantilevers with quantification of uncertainty. This allows a rigorous assessment of the accuracy of Eq. (2). Table II provides a comparison of the results.

Note that the resonant frequencies, quality factors, and spring constants of the cantilevers reported in Table II differ significantly from those of the reference cantilevers in Table I. Even so, the simplified method, Eq. (2), accurately determines the spring constants, with results agreeing to within measurement uncertainty. Type B is particularly noteworthy because the TR800(L) reference cantilever in Table I has a thickness double that of the TR400(L) device, and so the spring constants of these two cantilevers differ by an order-of-magnitude. Despite this disparity, the spring constant of the TR400(L) device is accurately captured using Eq. (2).

Importantly, the coefficient α in Eq. (2) is formally bound to the range $0.5 < \alpha < 1$, with the lower and upper values corresponding to the high and low-Reynolds number limits, respectively.[10] Since AFM cantilevers typically operate at intermediate Reynolds number,[15] a value for α midway in the range of $0.5 < \alpha < 1$ is expected; and indeed this is borne out in measurements.[10,11] Even so, cantilevers of the same type do not typically exhibit large variations in resonant frequency, in contrast to their spring constants – see Table II – the spring constant depends on the cantilever thickness cubed, whereas the resonant frequency varies linearly with thickness. Since the contribution from α in Eq. (2) only arises from resonant frequency differences, and given the above properties of α regarding the Reynolds numbers, measuring its true value and replacing $\alpha = 0.7$ with that value will in practice only exert a weak effect.

TABLE III. Spring constants using the simplified method, Eq. (2), but employing reference data from cantilevers in Ref. 10 that have different widths. Reference cantilevers for types A and F have widths of 50 and 30 μm, respectively, whereas AC160TS-R3 and AC240TS-R3 both have widths of 40 μm; see Table I.

		k (N/m)	
Type	Device	LDV	Eq. (2)
A	AC240TS-R3	1.36 ± 0.13	1.34 ± 0.01
	AC240TS-R3	1.26 ± 0.12	1.08 ± 0.01
	AC240TM-R3	1.58 ± 0.04	1.48 ± 0.02
F	AC160TS-R3	26.8 ± 0.5	31.6 ± 1.0
	AC160TS-R3	29.0 ± 1.1	32.8 ± 1.1

We now assess the robustness of the simplified method, Eq. (2), in situations where the plan-view dimensions of the reference and uncalibrated devices differ. As discussed above, the type A and F devices[10] and the type E and C devices have significantly different widths, but possess similar lengths and overall shapes; see Table I. These cantilevers thus provide an ideal platform upon which to examine the effect of dimensional variations.

The spring constants, resonant frequencies, and quality factors of the AC160TS and AC240TS devices reported in Table I are used as reference data for Eq. (2). The corresponding resonant frequencies and quality factors of the AC160TS-R3, AC240TS-R3, and AC240TM-R3 cantilevers, in Table II, are then used to determine their spring constants. Table III shows a comparison of this data with the true spring constants as measured using LDV. Despite the strong differences in plan-view dimensions and spring constants between the reference and uncalibrated cantilevers, the comparison in Table III shows that the simplified method accurately recovers the true spring constants. The errors in spring constant produced by Eq. (2) are larger than those reported in Table II, but given the large variations in plan-view dimensions here, the agreement is excellent. This verifies that the simplified method is relatively insensitive to variations in plan-view dimensions between the reference and uncalibrated cantilevers; it is unaffected by thickness variations, see Ref. 10 and above. Data in Table I and Eq. (2) can therefore be used immediately to accurately calibrate other cantilevers of the same type without requiring detailed knowledge of their dimensions.

In summary, we have experimentally assessed the accuracy of the simplified method, Eq. (2), for cantilever calibration.[10] This showed that the simplified method is capable of fast and accurate calibration of AFM cantilevers through measurement of their resonant frequencies and quality factors alone; cantilever dimensions are not used. The complete method in Eq. (1), which requires measurement/calculation of the hydrodynamic function, Λ (Re), clearly can provide greater accuracy; it is the foundation for Eq. (2) and eliminates approximation. Nonetheless, the simplified method is capable of accuracy at least equivalent to methods currently employed in the AFM[4–8] while dramatically simplifying implementation. This method is therefore expected to be of significant value to manufacturers and users, as it provides an easy, non-invasive, and routine approach for calibrating the spring constants of AFM cantilevers of arbitrary shape.

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