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Surface chemical analysis — Scanning-probe microscopy — Determination of cantilever normal spring constants

National foreword

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Analyse chimique des surfaces — Microscopie à sonde à balayage — Détermination de constantes normales en porte-à-faux de ressort

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 9, *Scanning probe microscopy*.

Introduction

Atomic force microscopy (AFM) is a mode of scanning probe microscopy (SPM) used to image surfaces by mechanically scanning a probe over the surface in which the deflection of a sharp tip sensing the surface forces mounted on a compliant cantilever is monitored. It can provide amongst other data, topographic, mechanical, chemical, and electro-magnetic information about a surface depending on the mode of operation and the property of the tip. Accurate force measurements are needed for a wide variety of applications, from measuring the unbinding force of protein and other molecules to determining the elastic modulus of materials, such as organics and polymers at surfaces. For such force measurements, the value of the AFM cantilever normal spring constant, k_z, is required. The manufacturers' nominal values of k_z have been found to be up to a factor of three in error, therefore practical methods to calibrate k_z are required.

This International Standard describes five of the simplest methods in three categories for the determination of normal spring constants for atomic force microscope cantilevers. The methods are in one of the three categories of dimensional, static experimental, and dynamic experimental methods. The method chosen depends on the purpose and convenience to the analyst. Many other methods may also be found in the literature.

Surface chemical analysis — Scanning-probe microscopy — Determination of cantilever normal spring constants

1 Scope

This International Standard describes five of the methods for the determination of normal spring constants for atomic force microscope cantilevers to an accuracy of 5 % to 10 %. Each method is in one of the three categories of dimensional, static experimental, and dynamic experimental methods. The method chosen depends on the purpose, convenience, and instrumentation available to the analyst. For accuracies better than 5 % to 10 %, more sophisticated methods not described here are required.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-2 and the following apply.

3.1
normal spring constant
spring constant
force constant
DEPRECATED: cantilever stiffness
k_z
<AFM> quotient of the applied normal force at the *probe tip* (3.2) by the deflection of the cantilever in that direction at the probe tip position

Note 1 to entry: See lateral spring constant, torsional spring constant.

Note 2 to entry: The normal spring constant is usually referred to as the spring constant. The full term is used when it is necessary to distinguish it from the lateral spring constant.

Note 3 to entry: The force is applied normal to the plane of the cantilever to compute or measure the normal force constant, k_z. In application, the cantilever in an AFM may be tilted at an angle, θ, to the plane of the sample surface and the plane normal to the direction of approach of the tip to the sample. This angle is important in applying the normal spring constant in AFM studies.

3.2
probe tip
tip
probe apex
structure at the extremity of a probe, the apex of which senses the surface

Note 1 to entry: See *cantilever apex* (3.3).

3.3
cantilever apex
end of the cantilever furthest from the cantilever support structure

Note 1 to entry: See *probe apex* (3.2), *tip apex* (3.2).

4 Symbols and abbreviated terms

In the list of abbreviated terms below, note that the final “M”, given as “Microscopy”, may be taken equally as “Microscope” depending on the context. The abbreviated terms are:

AFM	Atomic force microscopy
FEA	Finite element analysis
PSD	Power spectral density
SEM	Scanning electron microscopy
SPM	Scanning probe microscopy

The symbols for use in the formulae and as abbreviated terms in the text are:

A	amplitude of cantilever at a certain frequency
A_0	amplitude of a cantilever at its fundamental resonant frequency
A_white	mean amplitude of a cantilever associated with white noise
$B_\varPhi$	gradient determined from a straight line fit to values of L_x versus ${\varPhi_\text{x}}^{1/3}$
B_k	gradient determined from a straight line fit to values of L_x versus $\left(k_\text{z}^{L_\text{x}}\right)^{-1/3}$
C_1	correction factor for the thermal vibration method described in 8.2
C_2	correction factor for the thermal vibration method described in 8.2
d	distance between the probe tip and the cantilever apex
D	height of the probe tip
e	width of the V-shaped cantilever at a distance L_0 from the apex
E	Young’s modulus of the material of a cantilever
E_B	Young’s modulus of the base material of a cantilever
E_C	Young’s modulus of the coating material on a cantilever
f	frequency
f_0	fundamental resonant frequency of a cantilever
F	force of a nanoindenter
h	displacement of a nanoindenter
i	index of P_i, where $i = 1$ to 5
k_B	Boltzmann constant
k_z	normal spring constant
$k_\text{z}^{L_\text{x}}$	normal spring constant at the position L_x along a cantilever

k_z^R	normal spring constant of a reference cantilever
k_z^W	normal spring constant of a working cantilever
$k_{\mathrm{z}(tc=0)}$	normal spring constant of a cantilever with a coating thickness of 0
L	length of a rectangular cantilever or the effective length of a V-shaped cantilever
L_x	distance between the base of a cantilever and the effective position of a V-shaped cantilever
L_0	length of a V-shaped cantilever between the apex and the start of the arms
L_1	length of a V-shaped cantilever between the base and the start of the arms
P_i	label of one of the five positions on the reference cantilever axis
Q	quality factor of a cantilever
r	term defined by Formula (7)
t	thickness of a cantilever
t_B	thickness of the bulk material of a cantilever
t_C	thickness of a coating on a cantilever
T	absolute temperature of the cantilever measured in Kelvins
u_{A0}	standard uncertainty in A_0
u_B	standard uncertainty in B
u_{C1}	standard uncertainty in C_1
u_{C2}	standard uncertainty in C_2
u_d	standard uncertainty in the distance between the probe tip and the cantilever apex
u_E	standard uncertainty in the Young's modulus of a cantilever
u_F	standard uncertainty due to the calibration of force in the nanoindenter
u_{f0}	standard uncertainty in the resonant frequency
u_h	standard uncertainty due to the calibration of displacement in the nanoindenter
$u_{k\mathrm{z}}$	standard uncertainty in the normal spring constant
$u_{k\mathrm{zR}}$	standard uncertainty in the normal spring constant of the reference cantilever
u_L	standard uncertainty in the length of a cantilever
u_Q	standard uncertainty in the quality factor of a cantilever
u_t	standard uncertainty in the thickness of a cantilever
u_T	standard uncertainty in the absolute temperature
u_w	standard uncertainty in the width of a cantilever
u_{x1}	standard uncertainty in x_1

$u_{\alpha 1}$	standard uncertainty in α_1
u_ρ	standard uncertainty in the density of a cantilever
w	width of a cantilever
w_1	width of one side of a trapezium
w_2	width of one side of a trapezium
w_t	$w\cos\theta$
x_1	offset to account for the small uncertainty in the true position of the base of the cantilever compared to an arbitrary reference point
x_2	offset to account for the uncertainty in the true position of the probe tip compared to an arbitrary reference point
Z_1	term defined by Formula (4)
Z_2	term defined by Formula (5)
α	angle of the working cantilever with respect to the reference cantilever or surface
α_1	numeric constant used in Formula (11)
δ_R	average inverse gradient of the force-distance curve obtained with the working cantilever pressing on the reference cantilever or device
δ_W	average inverse gradient of the force-distance curve obtained with the working cantilever pressing on a stiff surface
θ	half angle between the arms of a V-shaped cantilever
Θ_2	term defined by Formula (6)
ν	Poisson's ratio of the cantilever material
ρ	density of a cantilever
ϕ_x	term defined by Formula (16)

5 General information

5.1 Background information

The spring constant, k_z, of an AFM cantilever is needed for quantitative force measurement. It is used to convert the deflection of the cantilever into a force. Applications that need this include the measurement of material properties at the nanoscale, such as elastic modulus, adhesive forces, and for studying the breaking of covalent bonds and protein unfolding. Depending on the application, k_z will be chosen to be in the range between 0,005 Nm^{-1} and 200 Nm^{-1}. There are two main shapes of cantilever: the rectangular "diving board" shape and the V-shaped. Both types vary slightly in basic shape and design between manufacturers and can be rectangular or trapezoidal in cross-section. Some cantilevers are also coated with a thin metallic layer. These factors all influence the value of k_z.

Many manufacturers provide data sheets for their cantilevers giving nominal values of k_z. Unfortunately, these values can be routinely in error by up to a factor of 3. One reason why similar cantilevers have very different values of k_z is that the spring constant is proportional to the thickness cubed and the thickness of AFM cantilevers is difficult to control accurately during manufacture. Since the cantilevers wear out, break, and need regular replacement, quick and accurate methods to determine k_z are required.

5.2 Methods for the determination of AFM normal spring constant

There are many methods to determine the normal spring constant and these are classified as the following.

a) The dimensional methods where k_z is determined from the cantilever material and the geometrical properties. In this method, any structural defects are not included.

b) The static experimental methods where k_z is determined by measurement of the static deflection of the cantilever under an applied force.

c) The dynamic experimental methods where k_z is determined by measurement of the dynamic properties of the cantilever.

In this International Standard, we describe procedures for a total of five methods with one or two methods in each category. Use one or more of the methods to determine k_z and its associated uncertainty, u_{kz}. Which method or methods are used depends on the time, equipment, and the accuracy that the user requires the spring constant to be measured to. Some advantages and disadvantages of the methods are given in Table 1.

Table 1 — Summary of the advantages and disadvantages of the methods in ISO 11775

Clause	Method	Advantages	Disadvantages
6	Dimensional measurement	Simple. Allows one to see why k_z varies from cantilever to cantilever.	Does not include defects. Slow and time consuming.
7	Static experimental measurement using a reference cantilever or a nanoindenter	Can be made traceable to SI.	May potentially damage the cantilever. Can be time consuming and in some cases, requires a nanoindenter.
8	Dynamic experimental measurement – thermal vibrational method	Fast if AFM instrument contains relevant software and hardware. Gives very good cantilever-to-cantilever comparability for cantilevers of a given design.	Uncertainty can be higher.

NOTE This International Standard does not include all the methods for calibrating k_z that are described in the literature.

6 Dimensional methods to determine k_z

6.1 General

The dimensional methods involve accurate measurements of a cantilever's geometry and knowledge of the material properties to determine k_z. The procedures described here use analytical formulae and are only applicable if the geometry is suitable. For other geometries, finite element analysis (FEA) is required and is not described here. Defects in the material, such as cracks or non-ideal geometry are not generally included.

6.2 k_z using formulae requiring 3D geometric information

6.2.1 Method

In order to determine k_z for a rectangular beam with a rectangular cross-section, as shown in Figure 1, measure the thickness t, the width w, and the distance $(L - d)$, which is the length of the cantilever, L, minus the distance from the free end of the cantilever to the probe tip, d. The measurement methods for these are given in 6.2.2. Also, obtain or measure, using an appropriate method, the value for the Young's modulus E of the cantilever.

Make at least seven independent measurements of those parameters that you are measuring by removing and replacing the cantilever. Evaluate the average values for these parameters and use them to calculate k_z using Formula (1) as detailed in 6.2.3.1, incorporating the averages of these independent measurements.

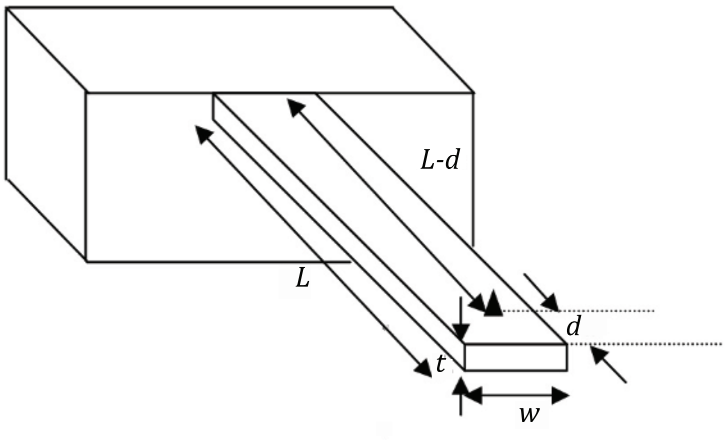
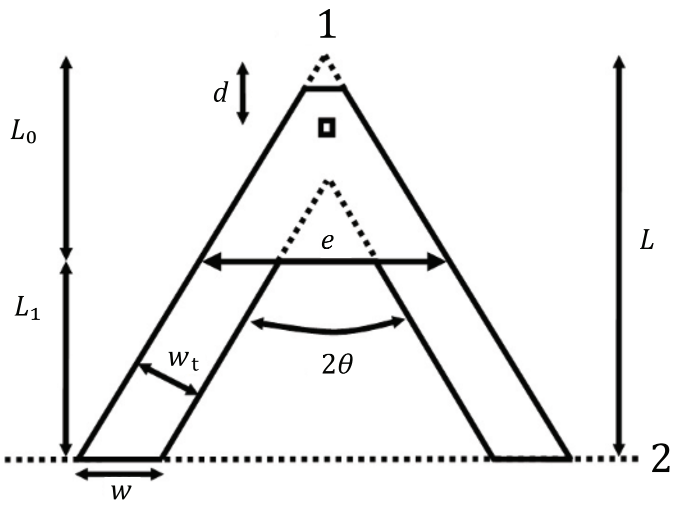


Figure 1 — Schematic of a rectangular shape cantilever with a probe tip a distance *d* from its free end

Similarly, if you are using a V-shaped cantilever, as shown in Figure 2, measure L_0, the length of a V-shape cantilever between the (virtual) apex and the start of the arms; L_1, the length of a V-shape cantilever between base and the start of the arms; d, the distance between the probe tip and the cantilever apex; e, the width of the V-shaped cantilever at the distance L_0 from the apex; and θ, the half angle between the arms. Also, obtain or measure, using an appropriate method, Young's modulus E and Poisson's ratio of the cantilever v. Make at least seven independent measurements of those parameters that you are measuring by removing and replacing the cantilever. Evaluate the average values for these parameters and use them to calculate k_z using Formulae (3) to (7).



Key

1 apex

2 base

Figure 2 — Schematic of a V-shaped cantilever

If the cross-section of the cantilever is trapezoidal and not rectangular, apply Formula (9) and follow the method given in 6.2.5.

If the cantilever has a significant coating, then account for this in the k_z calculation by following the method given in 6.2.6.

6.2.2 Measuring the required dimensions and material properties of the cantilever

6.2.2.1 Measuring the plan view dimensions of the cantilever

The plan view dimensions of the cantilever, including width and (L - d) for rectangular cantilevers or length and the offset of the probe tip from the cantilever apex for V-shaped cantilever, shall be measured using an appropriate method, for example, optical microscopy or SEM. The measurement instrument chosen shall be in calibration and operated in accordance with the manufacturer's documented instructions. Measure the width of the cantilever in at least three places along the length and determine an average width. More measurements will be required if the width of the cantilever is uneven in order to obtain a more accurate average width. A similar procedure applies in measuring L_0, d, and other dimensions for the V-shaped cantilever. In measuring d, the distance measured shall be from the apex or virtual apex of the V-shaped to the probe tip.

NOTE Using optical microscopy on typical commercial cantilevers, uncertainties in length and width are approximately 1 %. SEM can prove more accurate but is likely to be more time consuming and expensive.

6.2.2.2 Measuring the thickness of the cantilever

The thickness of the cantilever shall be measured using an appropriate method, for example, using SEM on the edge or side of the cantilever. The measurement instrument chosen shall be in calibration and operated in accordance with the manufacturer's documented instructions. Measure the thickness in a number of different locations along the cantilever's edge or side, and determine an average thickness.

NOTE 1 With careful, calibrated measurement, the uncertainty in thickness can be approximately 3 %.

NOTE 2 The number of measurements depends on the unevenness of the thickness. In addition, Formula (1) given in 6.2.3.1 assumes a rectangular cross-section with no taper along the length. Analytically, it can be seen that if there is an even taper in the thickness of 1 % change from end to end, then k_z is uncertain to approximately 1 %. Similarly, if it tapers in the middle and then returns to the original thickness, a 1 % change in thickness results in a change in k_z of approximately 1 %, as discussed in Reference [4].

6.2.2.3 Measuring the material properties of the cantilever

The Young's modulus, Poisson's ratio, and the density of the cantilever material, including coatings, if required, shall be determined from reference values if the cantilever is composed of known materials in a known crystal orientation. Otherwise, these values need to be measured by another suitable method. If no accurate values exist for these parameters and they cannot be measured, alternative methods to calibrate k_z shall be used as detailed in Clauses 7 and 8.

NOTE Cantilevers are typically made from silicon or silicon nitride. Silicon is highly anisotropic, so knowledge of the crystal orientation is critical. For the [110] direction, the Young's modulus is 168,9 GPa, with an uncertainty of approximately 1 %.[4] The modulus of silicon nitride cantilevers is less certain and can depend on the manufacturing technique. For example, the values of 146 GPa to 290 GPa have been reported for low pressure CVD growth and around 400 GPa for single crystal material.[5] The Poisson's ratio and density of silicon at room temperature are ~0,28 g cm^{-3} and 2,329 g cm^{-3}, respectively, but the Poisson's ratio depends on the crystal orientation. The Poisson's ratio and density of silicon nitride at room temperature are ~0,27 g cm^{-3} and ~3,3 g cm^{-3}, respectively, but both depend on the form and growth of the material.

6.2.3 Determining k_z for the rectangular cantilever

6.2.3.1 Determining k_z

For a rectangular beam with a rectangular cross-section, as shown in Figure 1, composed of a single material, once values for the Young's modulus, E, thickness, t, width, w, and $(L - d)$, which is the cantilever length, L, minus the tip distance, d, from the free end, have been determined; calculate the cantilever spring constant, k_z, using Formula (1).

$$k_z = \frac{Ewt^3}{4\left(L-d\right)^3} \tag{1}$$

Formula (1) involves the assumption that the bowing of the cantilever across the width, w, is negligible and is therefore applicable to practical cantilevers where $w << L$. The cantilever must be attached to a base and end effects here are usually small and ignored.

NOTE Formula (1) is reviewed in Reference [1].

6.2.3.2 Uncertainty

Determine the standard uncertainty in the spring constant, u_{kz}, by using

$$u_{kz} = k_z \left[\left(\frac{u_E}{E}\right)^2 + \left(\frac{u_w}{w}\right)^2 + \left(\frac{3u_t}{t}\right)^2 + \left(\frac{3u_L}{L-d}\right)^2 + \left(\frac{3u_d}{L-d}\right)^2\right]^{1/2} \tag{2}$$

where the uncertainties of the model are ignored. The main uncertainty arises from the measurement of t and to a lesser extent, d, L, and E.

NOTE Typical values often given are approximately u_E/E = 0,03, u_w/w = 0,01, u_t/t = 0,04, $u_L/(L\text{-}d)$ = 0,01, and $u_d/(L\text{-}d)$ = 0,01 so that u_{kz}/k_z = 0,12 and u_t is seen to be very important. u_E/E will be higher for silicon nitride cantilevers.

6.2.4 Determining k_z for the V-shaped cantilever

6.2.4.1 Determining k_z

For a V-shaped cantilever, shown in Figure 2, determine the dimensional and material properties of the cantilever and then calculate k_z using

$$k_z = \left[Z_1 + Z_2 + \Theta_2\left(\frac{w_t}{\sin\theta} - d\right)\right]^{-1} \tag{3}$$

where

$$Z_1 = \frac{12L_0}{Eet^3}\left\{\frac{\left(L_0-d\right)^2}{2} + d\left(L_0-d\right)\left[\ln\left(\frac{d}{L_0}\right) - 1\right] + L_0 d\ln\left(\frac{L_0}{d}\right)\right\} \tag{4}$$

$$Z_2 = \frac{L_1{}^2}{Ew_t t^3\cos^2\theta}\left[\frac{2L_1}{\cos\theta} + 3\left(w_t\cot\theta - d\cos\theta - r\sin\theta\right)\right] \tag{5}$$

$$\Theta_2 = \frac{3L_1\left(1+v\right)}{Ew_t t^3\cos\theta}\left(\frac{w_t}{\sin\theta} - d + r\cot\theta\right) \tag{6}$$

$$r = \frac{L_1 \tan\theta + \left(w_t - d\sin\theta\right)\left(1-\nu\right)\cos\theta}{2 - \left(1-\nu\right)\cos^2\theta} \tag{7}$$

Here, L_0 is the length of a V-shaped cantilever between the (virtual) apex and the start of the arms, L_1 is the length of a V-shaped cantilever between base and the start of the arms, d is the distance between the probe tip and the cantilever apex, e is the width of the V-shaped cantilever at the distance L_0 from the apex, w_t is $w\cos\theta$, where θ is the half angle between the arms, and ν is the Poisson's ratio of the cantilever material.

NOTE Formula (3) is reviewed in Reference [1], but note that there is a small change in the symbols that are defined by Formulae (4), (5), and (6) in order to simplify Formula (3).

6.2.4.2 Uncertainty

The uncertainty calculation for k_z for a V-shaped cantilever based on Formula (3) is complex. However, to a first approximation for the uncertainty calculation, this cantilever may be considered to be an unskewed rectangular beam of width $2w$, length L, and tip position d.

This simplified model can be used to calculate to a good approximation the uncertainty in k_z of a V-shaped cantilever. Hence, determine the uncertainty in k_z using

$$u_{kz} = k_z \left[\left(\frac{u_E}{E}\right)^2 + \left(\frac{u_{2w}}{2w}\right)^2 + \left(\frac{3u_t}{t}\right)^2 + \left(\frac{3u_L}{L-d}\right)^2 + \left(\frac{3u_d}{L-d}\right)^2 \right]^{1/2} \tag{8}$$

where the main contribution to the uncertainty is typically from t and L. Formula (8) differs from Formula (2) in the second term in the square brackets related to the uncertainty in w, which is generally small. The contribution arising from the uncertainty in θ is also generally small and is ignored here.

NOTE 1 Typical values often given are approximately $u_E/E = 0{,}03$, $u_{2w}/2w = 0{,}01$, $u_t/t = 0{,}04$, $u_L/(L\text{-}d) = 0{,}01$, and $u_d/(L\text{-}d) = 0{,}01$ so that $u_{kz}/k_z = 0{,}12$ and u_t is seen to be very important. u_E/E will be higher for silicon nitride cantilevers.

NOTE 2 These formulae are described in Reference [1].

NOTE 3 More complex methods to calculate the uncertainty in k_z may be appropriate for those requiring higher accuracy than those given by this International Standard.

6.2.5 k_z for the trapezoidal cross-sections

Some cantilevers, rather than having a rectangular cross-section, have a trapezoidal cross-section of the upper and lower widths, w_1 and w_2. Measure these widths using the methods given in 6.2.2.1. Determine, to a first approximation, the width of the cantilever for use in 6.2.3 and 6.2.4 using Formula (9).

$$w = \frac{w_1 + w_2}{2} \tag{9}$$

Determine k_z for a diving board cantilever with a trapezoidal cross-section, using Formula (9) to calculate w and use Formula (1) to determine k_z. Follow a similar procedure for a V-shaped cantilever with a trapezoidal cross-section, but calculate k_z using the formulae detailed in 6.2.4.

NOTE Formula (9) leads to a small overestimation in k_z, which is less than 2 % if w_1 is in the range $0{,}6 < w_2 < 1{,}6$ and less than 1 % if w_1 is in the smaller range $0{,}7 < w_2 < 1{,}4$ [2].

6.2.6 k_z to account for coatings

The changes in k_z arising from a coating on the cantilever, which is often added to improve the reflectivity from the laser beam used for monitoring the cantilever deflections, may be described by a simple equation. Coating thicknesses, which are small compared to the thickness of the cantilever, k_z, can be calculated as a linear combination using the relationship

$$k_z = k_{z(t_C=0)}\left[1+3\left(\frac{t_C}{t_B}\right)\left(\frac{E_C}{E_B}\right)+3\left(\frac{t_C}{t_B}\right)^2\left(\frac{E_C}{E_B}\right)\right] \tag{10}$$

where t_C and t_B are the thicknesses, and E_C and E_B are the Young's moduli of the coating and the bulk material of the cantilever, respectively. Here, $k_{z(tc=0)}$is simply the calculated spring constant ignoring the coating. Therefore, to determine k_z, measure or obtain, using appropriate methods, t_C, t_B, E_C, and E_B then determine k_z using Formula (10). The uncertainty arising from the coating is small compared with the uncertainty of the uncoated cantilever and its contribution can therefore often be ignored.

NOTE Formula (10) is derived in Reference [1]. This shows that, for example, a V-shaped cantilever adding a 40 nm gold coating increases the spring k_z by approximately 5 %.

6.3 k_z using plan view dimensions and resonant frequency for rectangular tipless cantilevers

6.3.1 Determining k_z

The determination of k_z by the dimensional methods described in 6.2 and 6.3 requires accurate information about the cantilever dimensions and, in particular, the thickness. The spring constant is proportional to the cube of the thickness. It is also the most difficult dimension to control during manufacturing and so it is subject to the greatest variability in its value leading to large uncertainties in k_z. If the measurement of the cantilever thickness is difficult or an SEM is unavailable, the method detailed in this section can be used to determine k_z.

Measure the plan dimensions (w and L) of a rectangular cantilever using the methods described in 6.2.2.1. Determine the resonant frequency f_0 of the cantilever by mechanically vibrating the cantilever as a function of frequency. Obtain or measure the Young's modulus E and density ρ of the cantilever material following the method described in 6.2.2.3.

The resonant frequency f_0 of a rectangular tipless cantilever in vacuum of Young's modulus E, density ρ, thickness t, width w, and length L, is calculated to be

$$f_0 = \frac{t}{4\pi}\left(\frac{\alpha_1}{L}\right)^2\sqrt{\frac{E}{3\rho}} \tag{11}$$

where α_1 = 1,875 1. Solving for t and substituting into Formula (1) yields

$$k_z = \frac{48\pi^3\sqrt{3}}{\alpha_1^6}\frac{wL^3}{\left(1-d/L\right)^3}\frac{\rho^{3/2}}{\sqrt{E}}f_0^3 \tag{12}$$

where d is the distance of the tip from the free end of the cantilever.

Determine the cantilever spring constant using Formula (12). This formula is based on the resonant frequency in vacuum. If it is not possible to measure this, then measure the resonant frequency in air and apply a small correction factor. This correction factor increases the measured frequency value by around 1 %, depending on the values of E, L, w, ρ, and t.

The value of α_1 is for a rectangular cross-section cantilever beam with no tip that is clamped at one end. For different shapes and types of cantilever, the value of α_1 should be determined by an appropriate method.

NOTE 1 Formulae (11) and (12) assume a cantilever comprised of a single material with a constant cross-section and does not account for defects. The method is reviewed in Reference [1].

NOTE 2 The increase in resonant frequency between air and vacuum has been measured to be approximately 0,4 %[3] to 0,6 %, although others found it to be 2 % to 4 %, as reviewed in Reference [1]. Unless the resonant frequency is measured in vacuum, a modest uncertainty will be introduced.

NOTE 3 The resonant frequency is determined here via a driven method rather than the thermal method as detailed in 8.2 in order to obtain a better signal quality. An interlaboratory study showed that the scatter in measured f_0 using this method was 0,7 %.

NOTE 4 For cantilevers with a tip, further considerations are required. The tip adds mass to the cantilever, which decreases the measured resonant frequency to an extent proportional to f_0^{-2}. The volume of the tip can be estimated using microscopy, and the mass is then the product of this volume, and the tip density and a suitable correction factor may be determined. Further details and a method to determine different values for α from the ratio of first and second harmonic frequencies can be found in Reference [2].

6.3.2 Uncertainty

Determine the standard uncertainty in the spring constant of a tipless cantilever using

$$u_{kz} = k_z \left[\left(\frac{6u_{\alpha 1}}{\alpha_1} \right)^2 + \left(\frac{u_E}{2E} \right)^2 + \left(\frac{3u_\rho}{2\rho} \right)^2 + \left(\frac{3u_{f0}}{f_0} \right)^2 + \left(\frac{u_w}{w} \right)^2 + \left(\frac{3u_L}{L} \right)^2 + \left(\frac{3u_d}{L-d} \right)^2 \right]^{1/2} \qquad (13)$$

in the usual case where d/L is small.

NOTE Typical values often given are approximately $6u_{\alpha 1}/\alpha_1$ = 0,03, $u_E/2E$ = 0,015, $3u_\rho/2\rho$ = 0,005 to 0,02 (depending on the cantilever material with the latter number for non-silicon cantilevers), $3u_{f0}/f_0$ = 0,03 (including the uncertainty due to f_0 being measured in air), u_{2w}/w = 0,01, $3u_L/L$ = 0,03 and $3u_d/(L-d)$ = 0,03 so that u_{kz}/k_z = 0,06 and u_α is seen to be important.

7 Static experimental methods to determine k_z

7.1 General

Static experimental methods involve the application of a constant force or a set of constant forces applied to the cantilever and the subsequent measurement of the deflection. These methods generally, but not exclusively, use a pre-calibrated reference beam or device to push on the working cantilever or vice versa.

There are a number of different static experimental methods that are described using

a) one or more calibrated reference cantilevers, and

b) a calibrated nanoindenter.

7.2 Static experimental method with a reference cantilever

7.2.1 Set-up

Obtain or calibrate a reference cantilever of known spring constant, k_z^R. A tipless rectangular cantilever with constant cross-section along its length is preferred. The spring constant of the reference cantilever should approximately match that of the working cantilever, k_z^W. The piezoelectric *z*-scanner of the AFM shall be in dimensional calibration and the AFM shall be operated in accordance with the manufacturer's documented instructions. This should be operated in closed loop mode or an alternative method used to deal with the effects of the piezoelectric scanner nonlinearity, hysteresis, creep, and drift. An AFM equipped with top view optics is recommended for the highest positional accuracy of the reference cantilever on the working cantilever.

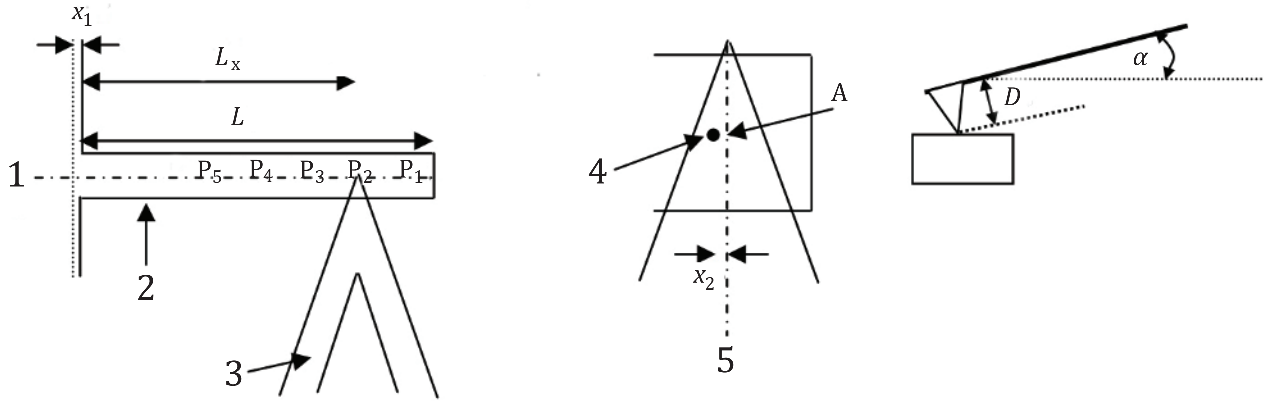
NOTE 1 One method to calibrate a reference cantilever is to use the nanoindenter on cantilever measurement method as detailed in 7.3. Another method is to acquire or calibrate a cantilever that has been traceably calibrated for instance via mass artefacts or quantum-based standards using electromechanical force balances.

NOTE 2 If a tipless cantilever with marked probing positions along its length is available, it may aid the ease or accuracy of undertaking this method.

7.2.2 Determining k_z

The deflection constant for the working cantilever shall be measured using the method outlined in 7.4.1. This shall be done at the start and end of the experiment.

The reference cantilever shall be mounted securely in the sample position and aligned to be perpendicular to the long axis of the working cantilever, which shall be mounted in the cantilever holder, as shown in Figure 3. The perpendicular alignment makes it easy to move the working cantilever along the reference cantilever and aids the location of the tip position. Land the working cantilever tip as near the axis of the reference cantilever as possible and near the free end. Conduct five force-distance curves, recorded as cantilever deflection in volts versus piezoelectric z-scanner extension in nanometres. The cantilever deflection should be kept within the elastic limit of the cantilever. The cantilever should bend less than 5 % of its total length and be kept within the linear region of the photodiode detector. For most cantilevers, a movement of less than 200 nm is suggested. Calculate the average gradient of each resultant plot of deflection voltage signal versus z-scanner displacement. This should be measured over the maximum range over which the curve linearity is less than a desired target uncertainty. Measure the distance between a suitable reference point on the test cantilever and the reference cantilever base. Ideally, this should be the tip position of the test cantilever, but if this cannot be seen, then use a reference point on the symmetry axis of the working cantilever, as shown by point A in Figure 3 b). Repeat this procedure for at least three to five evenly spaced positions, L_x, as far apart as possible along the outermost 70 % of the length of the reference cantilever, as shown by the P_i's in Figure 3 a).



a) Plan view

b) Higher magnification of a)

c) Side-view

Key

1 ref. cantilever axis
2 ref. cantilever
3 working cantilever
4 tip under working cantilever
5 long axis of working cantilever

NOTE In this particular example for the working cantilever position L_x denotes the distance between P_2 and the base of the cantilever

Figure 3 — Schematic of the static experimental method using a reference cantilever

At each position, calculate the spring constant of the working cantilever using

$$k_z^W = k_z^R \left(\frac{L + x_1}{L_x + x_1 + x_2} \right)^3 \left(\frac{\delta_R}{\delta_W} - 1 \right) \cos^2\alpha \left(1 - \frac{3D}{2L} \tan\alpha \right) \tag{14}$$

where L is the length of the calibrated cantilever, D is the height of the probe tip, L_x is the distance of the working cantilever along the reference cantilever from its base, and α is the angle of the working cantilever with respect to the reference cantilever, which is typically around 11°.

In Formula (14), δ_R is the average inverse gradient of the force-displacement curve obtained with the working cantilever pressing on the reference cantilever at L_x and δ_W is the average inverse gradient of the force-displacement curve obtained with the working cantilever pressing on a stiff surface. These should be the average of the advance and retraction curves.

The spring constant of the test cantilever at each position is given by a cubic relationship relating the position, L_x, along the cantilever to the length of the reference cantilever, L. This is exact for a rectangular reference cantilever and a good approximation for V-shapes. However, the exact location of the working cantilever's tip is difficult to measure and so a suitable reference point has been used on the back of the working cantilever at an unknown offset, x_2, from the true tip position, as shown in Figure 3 b). Measure this offset using an optical microscope or an SEM. This offset (x_2) is included, along with a second offset (x_1) to account for small uncertainties in the position of the base of the reference cantilever.

Rearranging Formula (14) gives

$$L_x = \left(\frac{k_z^R}{k_z^W} \phi_x \right)^{1/3} \left(L + x_1 \right) - x_1 - x_2 \tag{15}$$

where

$$\phi_x = \left(\frac{\delta_R}{\delta_W} - 1 \right) \cos^2\alpha \left(1 - \frac{3D}{2L} \tan\alpha \right) \tag{16}$$

Plot L_x against $\phi_x^{1/3}$ and determine the gradient, B_Φ, and the y-intercept of the resultant straight line. From Formula (15), it can be seen that the y-intercept of the line is ($x_1 + x_2$). Therefore, from the y-intercept and measuring x_2, determine the small correction factor, x_1.

Rearranging Formula (15) and substituting B_Φ into this formula gives Formula (17).

$$k_z^W = k_z^R \left(\frac{L + x_1}{B_\phi} \right)^3 \tag{17}$$

Measure, using an optical or electron microscope, the cantilever's length, L and tip height, D. Then from Formula (17), determine the spring constant of the working cantilever, k_z^W.

Figure 4 shows the results for a V-shaped working cantilever on a rectangular reference cantilever.

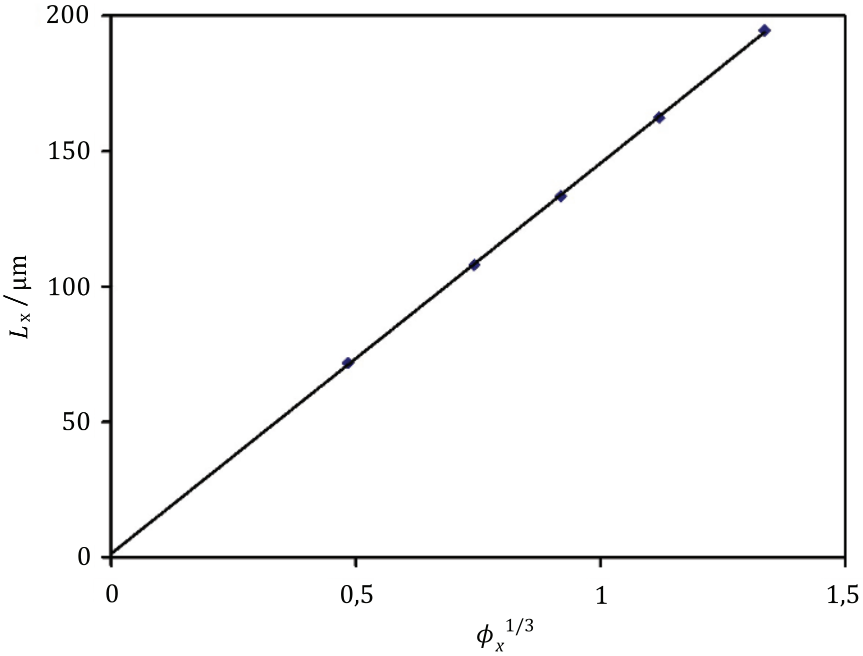


Figure 4 — Graph of L_x against $\phi_x^{1/3}$ in order to determine from Formula (15) the spring constant of the working cantilever where the dots are experimental data and the line is a straight line linear regression fit

7.2.3 Uncertainty

The standard uncertainty in the spring constant is given by

$$\left(\frac{u_{kz}}{k_z}\right)^2 = \left(\frac{3\sqrt{{u_L}^2 + {u_{x1}}^2}}{L + x_1}\right)^2 + \left(\frac{3u_{B\phi}}{B_\phi}\right)^2 + \left(\frac{u_{kzR}}{k_z^R}\right)^2 \tag{18}$$

where the standard uncertainty in the gradient, $u_{B\Phi}$ contains contributions from both abscissa and ordinate values and are dominated by random terms.

NOTE 1 In calculating $u_{B\Phi}$, the standard uncertainty from fitting the straight line is the dominant contribution provided that the measured force-distance curves are truly linear and pass through the origin. Typical values often given are approximately less than 0,01 for the first two terms in Formula (18) and u_{kzR}/k_{zR} = 0,1 so that u_{kz}/k_z = 0,11 and u_{kzR} is seen to be very important.

NOTE 2 The effect of the tilt angle of the cantilever is described in Reference [6]. In some cases, the tip height, *D*, is very much smaller than the length of the cantilever and hence can be ignored. The effect of sliding friction between the cantilevers may cause hysteresis between advance and retraction (also sometimes referred to as loading and unloading) curves. This effect is described in Reference [7] and can be compensated for by using the mean gradient of the advance and retraction curves when calculating δ_R.

NOTE 3 The random uncertainty in *B*, u_B, which takes into account uncertainties in both the abscissa and ordinate values, can be determined, for example, using the freely downloadable software XGenline v8.1.[1] A useful reference on error analysis is in Reference [8].

NOTE 4 This method is described in Reference [4].

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1) XGenline is the trade name of a free software developed by National Physical Laboratory. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7.3 Static experimental method using a nanoindenter

7.3.1 General

In this method, use a nanoindenter that is capable of landing on an AFM cantilever and is calibrated in both force and displacement. Force calibration can be achieved by attaching SI-traceable calibrated masses to the nanoindenter or through direct realization of force through quantum-based standards *in situ*. Displacement can either be calibrated by scanning over traceably calibrated step height standards or displacement can be SI-traceably realized directly by *in situ* laser interferometry.

7.3.2 Determining k_z for a tipped or tipless cantilever

Use a sharp nanoindenter probe with a tip radius less than 5 µm, for example, an etched tungsten tip. A nanoindenter equipped with side view 45° optics is required for highest positional accuracy of the nanoindenter on the working cantilever.

The working cantilever shall be mounted securely in the sample position and aligned to be perpendicular to the viewing optics.

Land the nanoindenter probe tip as near the axis of the working cantilever as possible and as close to the tip position as possible. Conduct 10 force-distance curves using the nanoindenter, recorded as force in nN versus nanoindenter deflection in nanometres with a movement of less than 200 nm. Calculate the average inverse gradient of the force-distance curve; this is the inverse spring constant of the working cantilever at this point. Measure L_x, the distance between a suitable reference point on the nanoindenter probe tip and the working cantilever base. Repeat the procedure for at least three to five positions, L_x, along the outermost 70 % of the length of the cantilever. The spring constant at the end of the cantilever, k_z, is given by

$$k_z = k_z^{L_x} \left(\frac{L_x + x_1 + x_2}{L + x_1} \right)^3 \tag{19}$$

where $k_z^{L_x}$ is the spring constant of the cantilever at the position L_x along the length. Formula (19) contains two length correction factors, x_1 and x_2. The first factor, x_1, accounts for any difference between the observed and effective base end of the cantilever and x_2 accounts for an unknown offset of the nanoindenter tip position compared to its true position and the second, as shown in Figure (5).

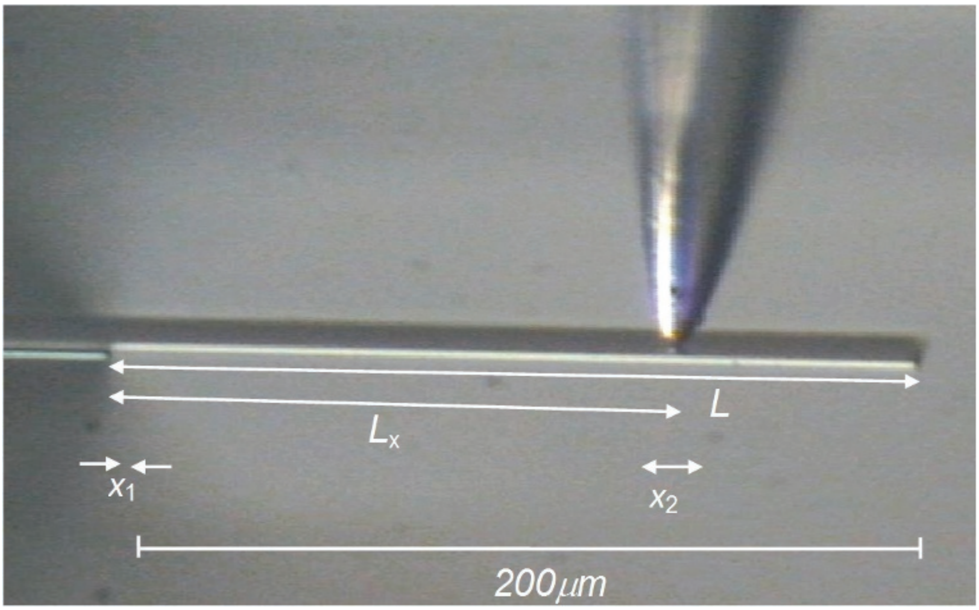

Figure 5 — Optical image of the static experimental method using a nanoindenter showing an etched tungsten tip pressing on a rectangular tipless working cantilever

In order to determine the offsets ($x_1 + x_2$) and k_z, Formula (19) can be rearranged to give Formula (20).

$$L_{\mathrm{x}} = \left(k_{\mathrm{z}}^{L\mathrm{x}}\right)^{-1/3} \left(k_{\mathrm{z}}\right)^{1/3} \left(L + x_1\right) - x_1 - x_2 \tag{20}$$

Plot L_x against $\left(k_{\mathrm{z}}^{L\mathrm{x}}\right)^{-1/3}$ and fit a straight line through the data. Determine the intercept, which gives the small correction factor, ($x_1 + x_2$) and the gradient, B_k, which is a function of the spring constant of the working cantilever. The offset x_2 is typically small enough to be ignored, otherwise, determine or estimate it using a suitable method. Calculate the spring constant using Formula (21).

$$k_{\mathrm{z}} = \left(\frac{B_k}{L + x_1}\right)^3 \tag{21}$$

Formula (21) gives the spring constant at the cantilever apex. For a cantilever with a tip, when the spring constant at the probe tip is required, Formula (21) becomes

$$k_{\mathrm{z}} = \left(\frac{B_k}{L + x_1}\right)^3 \left(\frac{L}{L - d}\right)^3 \tag{22}$$

where d is the distance between the cantilever apex and the probe tip.

7.3.3 Uncertainty

The uncertainty in the spring constant can be estimated using

$$\left(\frac{u_{k\mathrm{z}}}{k_{\mathrm{z}}}\right)^2 = \left(\frac{3\sqrt{u_L{}^2 + u_{x1}{}^2}}{L + x_1}\right)^2 + \left(\frac{3u_{Bk}}{B_k}\right)^2 + \left(\frac{u_F}{F}\right)^2 + \left(\frac{u_h}{h}\right)^2 \tag{23}$$

where u_F is the uncertainty due to the calibration of force F in the nanoindenter, u_h is the uncertainty due to the calibration of displacement h, and u_B is the uncertainty in the gradient. When k_z at the probe tip is calculated using Formula (22), then the last two terms from Formula (2) should be added to account for u_d.

Figure 6 shows example results for the nanoindenter on cantilever method.

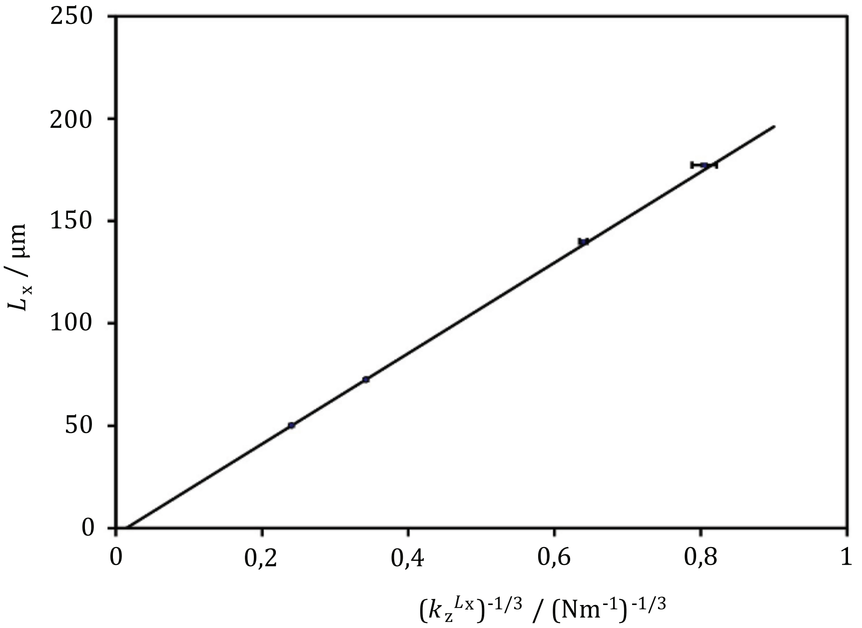

Figure 6 — Nanoindenter on cantilever results for the rectangular cantilever, plotted in terms of L_x versus $\left(k_z^{L_x}\right)^{-1/3}$ where the gradient of the line is related to the spring constant of the cantilever by Formula (21)

NOTE 1 In calculating u_{Bk}, the standard uncertainty from fitting the straight line is the dominant contribution provided that the measured force-distance curves are truly linear and pass through the origin. Typical values often given are approximately 0,005 for the first term in Formula (23), $3u_{Bk}/B_k = 0{,}05$, $u_F/F = 0{,}05$, and $u_h/h = 0{,}035$ so that $u_{kz}/k_z = 0{,}08$ and u_F and u_h are seen to be very important.

NOTE 2 This method is described in Reference [4].

NOTE 3 In this method x_2 is usually very small and can be ignored.

Depending on the instrumentation used, this method may not be suitable for the calibration of the most compliant cantilevers and the indenter may have to be landed near the base end only. For the stiffest cantilevers, typically with $k_z > 100$ N/m, the machine compliance of the nanoindenter may contribute non-negligibly to the measurement of k_z. This should be measured using an appropriate technique and taken into account, for example, through the addition of terms to Formula (22).

NOTE 4 An alternative method to determine k_z for a tipped cantilever using a nanoindenter is to use a flat punch indenter tip or sphere with a large radius of curvature (>500 µm) and place the cantilever in the sample position with the AFM probe tip facing the nanoindenter. The nanoindenter is landed on the AFM tip and force-distance curves undertaken by the nanoindenter at the exact location of the AFM cantilever tip to be calibrated. The AFM probe tip can be damaged. Uncertainty is determined using Formula (22) where the first term is zero and B becomes the uncertainty in the nanoindenter force-displacement curve. The method using an electrostatic force balance rather than a nanoindenter is described in Reference [10].

7.4 Measurement methods

7.4.1 Static deflection calibration

This calibration is used to convert the deflection of the cantilever from a voltage to a distance, generally recorded in nanometres. In most commercial AFMs, the deflection of the cantilever is typically monitored by the optical-lever technique, in which a laser beam is reflected off the cantilever into a position-sensitive photodiode detector. In systems where an interferometer is used, this does not apply.

The calibration is determined from average gradient of at least five force-distance curves, recorded as cantilever deflection in volts versus piezoelectric z-scanner extension in nanometres, on a stiff sample whose effective spring constant greatly exceeds that of the cantilever in use, for example, silicon. For low spring constant cantilevers (e.g. below 0,05 N/m), stiction may be an issue on a silicon surface, so highly ordered pyrolytic graphite can be used. The cantilever deflection should be kept within the elastic limit of the cantilever and within the linear region of the photodiode detector.

The optical-lever technique measures the inclination of the cantilever at the position of the laser illumination and does not directly measure cantilever deflection. Thus, the calibration factor obtained is dependent on the position of the laser spot relative to the tip. Hence, this calibration needs to be undertaken every time the cantilever is changed or the laser spot moved on the cantilever.

In general, the laser spot should be positioned close to the probe tip position near the apex of the cantilever and then the laser position should be refined to achieve a maximum reflected intensity value, in the first instance, by maximizing the intensity by moving the laser along the short axis of the cantilever. The optimum position can be determined by moving the laser spot to different positions along the cantilever and measuring the deflection calibration or sensitivity until the most sensitive position is found close to the probe position.

Another consideration is that the relationship between the cantilever gradient and the deflection depends on the geometry of the loading force. Thus, the calibration factor only applies to measurements of deflection produced under equivalent loading situations.

NOTE This is for the static deflection calibration. If a dynamic deflection calibration is required then an appropriate correction factor can be used. See 8.2.1.2 for the case using the thermal vibration method.

8 Dynamic experimental methods to determine k_z

8.1 General

Dynamic experimental methods generally involve finding the cantilever's resonant frequency combined with other measurements. These other measurements, for example, could involve adding masses to the cantilever to measure the change in the resonant frequency. In this International Standard, the method of thermal vibrations is detailed.

8.2 Dynamic experimental method using thermal vibrations using AFM

8.2.1 Determining k_z

8.2.1.1 Mount the working cantilever in the AFM head and a stiff sample, for example, a clean silicon wafer, in the sample position. Bring the tip into contact with the substrate and measure the deflection calibration constant using the method outlined in 7.4.1. Retract the cantilever far from the surface to avoid the influence of gradients in long-ranged forces or squeeze film damping of air between the cantilever and sample surface. Collect deflection versus time data at a bandwidth sufficient to completely sample the resonance frequency of the cantilever, i.e. at a rate comfortably higher than $2f_0$. Convert the data into a power spectral density of units of m^2/Hz. Follow the manufacturer's instructions if undertaking the measurements using software and hardware built into commercial AFM instruments.

Fit the fundamental resonance peak in the PSD to a simple harmonic oscillator function.

$$A = A_{\text{white}} + \frac{A_0 f_0^4}{Q^2}\left[\left(f^2 - f_0^2\right)^2 + \frac{f^2 f_0^2}{Q^2}\right]^{-1} \tag{24}$$

In Formula (24), A_{white} is the mean noise background, f_0 is the frequency of the fundamental resonant peak, and the amplitude A_0 and quality factor Q are related to the amplitude and width of the fundamental resonant peak, respectively. Figure 7 shows a typical PSD for a rectangular cantilever where the solid line is a fit to the curve described by Formula (24). Repeat the measurement seven times, removing and replacing the cantilever, and determine the average values for A_0, f_0, and Q.

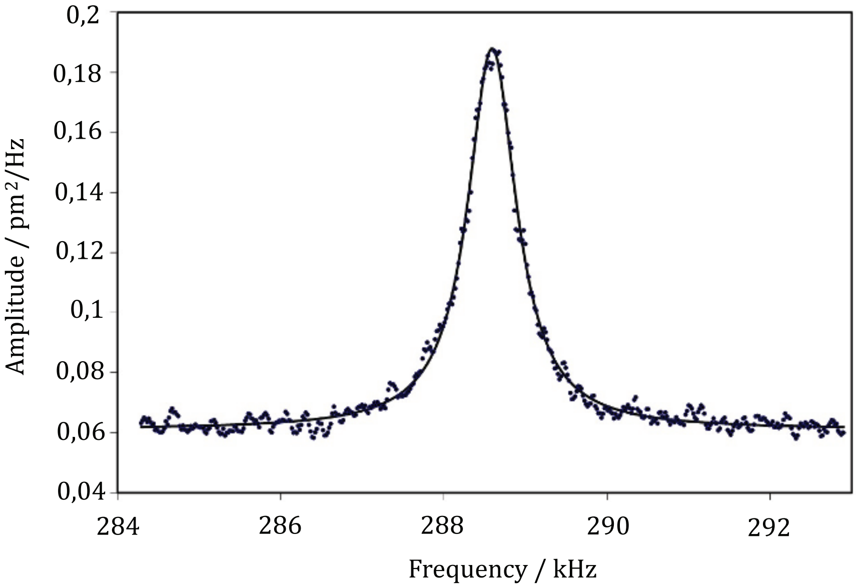

Figure 7 — Example power spectral density function for the thermal vibrations of a cantilever with a fit to the resonant peak using Formula (24) indicated by the solid line

Calculate the spring constant using

$$k_z = \frac{2C_1 k_B T Q}{C_2^2 \pi A_0 f_0} \tag{25}$$

where k_B is the Boltzmann constant, T is the absolute temperature of the cantilever, and C_1 and C_2 are correction factors. T should be measured as close to the cantilever as possible.

NOTE 1 Calibrating the deflection constant using the method outlined in 7.4.1 may result in damaging or breaking the tip, especially for stiff cantilevers. Therefore, the calibration could be undertaken after the experiment of interest has taken place.

NOTE 2 Some manufacturers' software plot and analyse the thermal data using m/√Hz and this will give a different Q from that above and is not appropriate for use with this International Standard.

8.2.1.2 There are two correction factors in Formula (25) and their determination needs careful consideration. C_1 is taken as 0,970 7 and arises from a consideration of the normal vibrational modes of the cantilever. C_2 arises from the fact that typical AFM instrumentation measures cantilever displacement via the optical-lever method, for which the deflection signal is strictly related to the inclination of the cantilever rather than its true displacement. As the thermal vibrations are measured over all frequencies with an unloaded cantilever whose end is free to fluctuate, the relationship between the deflection signal and the true cantilever displacement differs from that calculated by the quasi-static calibration determined in 7.4.1. Thus, a correction factor C_2 dependent on cantilever geometry must be applied. For rectangular cantilevers, this correction factor is given by

$$C_2 = \frac{\chi}{\cos\alpha}\left(\frac{1-\dfrac{2D}{L}\tan\alpha}{1-\dfrac{3D}{2L}\tan\alpha}\right) \tag{26}$$

where χ is the conversion factor between the static deflection sensitivity constant and the dynamic sensitivity constant which is typically 1,05.

NOTE 1 In some typically older AFM instruments, the results for stiffer cantilevers can be erroneously low. The results using this method or any other method can be checked using a traceably calibrated reference cantilever. This also provides a method to obtain a traceable calibration.

NOTE 2 Conversion factors arise from the damping effect of the ambient air and range from 1,029 to 1,101 for selected cantilevers of widely differing plan views.[11] When the laser spot size becomes comparable to the cantilever length then the size of the laser spot and its position can have effects on the value of χ.

8.2.2 Uncertainty

The major contributors to the uncertainty in this method are the uncertainty in the deflection calibration constant, the uncertainties in the fit parameters for the resonant peak, and the uncertainties in the correction factors. The uncertainty in k_z can be calculated to a first approximation using Formula (27).

$$u_{kz} = k_z\left[\left(\frac{u_{C1}}{C_1}\right)^2+\left(\frac{2u_{C2}}{C_2}\right)^2+\left(\frac{u_{f0}}{f_0}\right)^2+\left(\frac{u_{A0}}{A_0}\right)^2+\left(\frac{u_Q}{Q}\right)^2+\left(\frac{u_T}{T}\right)^2\right]^{1/2} \tag{27}$$

NOTE 1 Typical values often given are approximately $u_{C1}/C_1 = 0{,}02$, $2u_{C2}/C_2 = 0{,}09$, $u_{f0}/f_0 = 0{,}001$, $u_{A0}/A_0 = 0{,}02$, $u_Q/Q = 0{,}025$ and $u_T/T = 0{,}018$ so that $u_{kz}/k_z = 0{,}1$ and u_{C2} is seen to be very important.

NOTE 2 This method is described in References [12] to [14].

NOTE 3 u_{C2} includes the uncertainty contributions from χ and α.

Annex A
(informative)

Inter-laboratory and intra-laboratory comparison of AFM cantilevers

A.1 Overview and aims

An inter-laboratory and intra-laboratory comparison of the determination of the spring constants of AFM cantilevers was undertaken. This was done partly to aid development of this International Standard and to ensure comparability between methods. The comparison was to calibrate the spring constant of a total of 64 cantilevers of two different spring constants using four methods. These methods included the cantilever on reference cantilever, thermal methods, and two methods using dimensional measurements. One of each set of cantilevers was sent to 30 participating laboratories. Participants calibrated the cantilevers using the methods described here as well as a range of other methods. The two cantilevers were both rectangular in shape with trapezoidal cross-sections. One was a compliant cantilever (denoted C), which the participants were told was between 0,1 N/m and 0,9 N/m spring constant and the other a stiffer cantilever (denoted S) with a spring constant between 15 N/m and 60 N/m.

A.2 Intra-laboratory results

The results for the intra-laboratory results are summarized in Figure A.1.

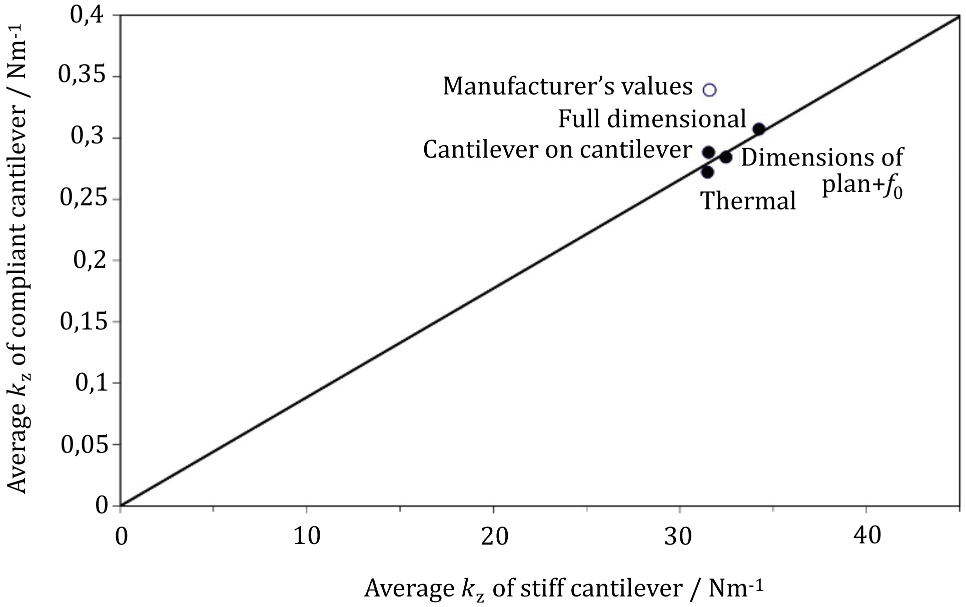

Figure A.1 — Average spring constant values for the stiff cantilevers plotted against those from the compliant cantilever using 4 methods in an intra-laboratory comparison plus the values from the manufacturers

The standard deviations of individual measurements for the 31 C Cantilevers and 34 S Cantilevers and the standard deviations of the means are given in Table A.1. The standard deviations shown illustrate the scatter of the experimental measurements and do not include the uncertainty arising from the use of constants such as E used in calculating k_z. In Table A.1, we see that the standard deviations of the means are only 0,5 %, to 2,7 %, while the means for the different methods used in this standard are distributed over a range of ±5 %. Furthermore, the scatters of individual measures range from 1,2 % to 12,8 % and so at least seven separate measurements are required in the methods of the standards.

Table A.1 — Standard deviations, and in parentheses, relative standard deviations of results for the 31 C Cantilevers and 34 S Cantilevers in the intra-laboratory study

	C cantilever		S cantilever	
	SD[a] of the mean Nm^{-1}	SD[a] of the distribution Nm^{-1}	SD[a] of the mean Nm^{-1}	SD[a] of the distribution Nm^{-1}
Cantilever on cantilever	0,004 1 (1,4 %)	0,022 5 (7,8 %)	0,57 (1,8 %)	3,30 (10,5 %)
Full dimensional	0,007 (2,7 %)	0,039 (12,8 %)	—	—
Dimensions of plan + f_0	0,001 4 (0,5 %)	0,007 9 (2,8 %)	0,07 (0,2 %)	0,37 (1,2 %)
Thermal	0,001 9 (0,7 %)	0,011 (4,0 %)	0,36 (1,1 %)	2,04 (6,5 %)

NOTE Dimensions were measured by the manufacturer and were all the same for the S Cantilever.

[a] Standard deviation.

A.3 Inter-laboratory results

The results for the inter-laboratory results are shown in Figure A.2. The error bars are standard deviations. For the nanoindenter on cantilever method, no participants reported results for the C Cantilever and only one participant reported a result for the S Cantilever.

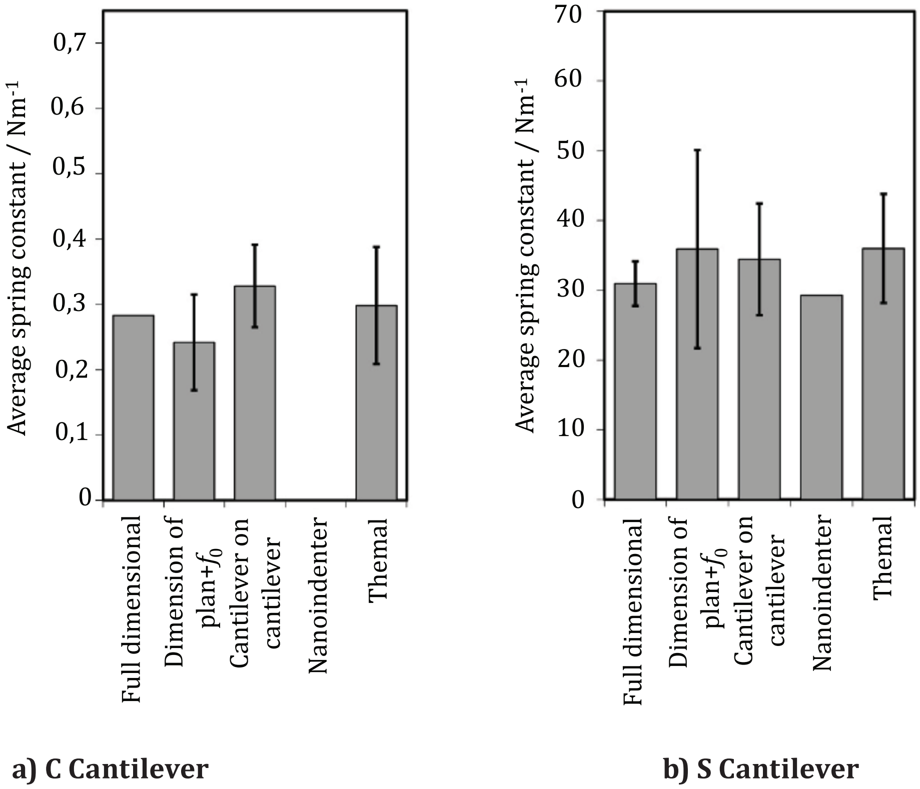

Figure A.2 — Interlaboratory results

A.4 Conclusions

The results for the spring constants are highly consistent. The manufacturers' values of k_z, provided by them using nominal values for the dimensional method, overestimated k_z by typically 18 % for the C Cantilevers.

The intra-laboratory results for the thermal method, the dimensional method using plan view dimensions and resonant frequency, and the cantilever on reference cantilever all agreed. The standard deviation of the mean for each method was within the size of the markers shown in Figure A.1. The average k_z for all these methods for the C Cantilever was 0,288 N/m with a standard deviation of 0,015 and for the S Cantilever it was 32,44 N/m with a standard deviation of 1,28.

The inter-laboratory results were more scattered but involved a range of experience of k_z calibration laboratories ranging from beginners to experts. A number of procedural problems were discovered to have given poor results and which led to the revised procedures described here. Participants greatly favoured the thermal method for its ease of use. However, results here were scattered as a result of fitting to different functions and use of different equipment.

NOTE A summary of the inter-laboratory and intra-laboratory results are provided in this Annex. Further details are given in Reference [15].

Bibliography

[1] Clifford C.A., & Seah M.P. The determination of atomic force microscope cantilever spring constants via dimensional methods for nanomechanical analysis. Nanotechnology. 2005 Sept., **16** (9) pp. 1666–1680

[2] Lübbe J., Doering L., Reichling M. Precise determination of force microscopy cantilever stiffness from dimensions and eigenfrequencies. Meas. Sci. Technol. 2012 Apr, **23** p. 045401

[3] Lübbe J. TEMMEN.M., SCHNIEDER, H., REICHLING, M., Measurement and modelling of non-contact atomic force microscope cantilever properties from ultra-high vacuum to normal pressure conditions. Meas. Sci. Technol. 2011 Mar, **22** p. 055501

[4] Clifford C.A., & Seah M.P. Improved methods and uncertainty analysis in the calibration of the spring constant of an atomic force microscope cantilever using static experimental methods. Meas. Sci. Technol. 2009, **20** p. 125501

[5] Khan A., Philip J., Hess P. Young's modulus of silicon nitride used in scanning force microscope cantilevers. J. Appl. Phys. 2004, **95** pp. 1667–1672

[6] Hutter J.L. Comment on tilt of atomic force microscope cantilever: effect on spring constant and adhesion measurements. Langmuir. 2005 March, **21** (6) pp. 2630–2632

[7] Pratt J.R., Shaw G.A., Kumanchik L., Burnham N.A. Quantitative of sample stiffness and sliding friction from force curves in atomic force microscopy. J. Appl. Phys. 2010, **107** p. 044305

[8] Software can be freely downloaded from the UK National Physical Laboratory, XGENLINE v8-1, Smith I M, (further information is available at http://www.npl.co.uk/mathematics-scientific-computing/mathematics-and-modelling-for-metrology/xgenline-8.1)

[9] Taylor J.R. An introduction to error analysis: The study of uncertainties in physical measurements. University Science Books, 1997

[10] Kim M.-S., Pratt J.R., Brand U., Jones C.W. Report on the first international comparison of small force facilities: a pilot study at the micronewton level. Metrologia. 2012, **49** (1) pp. 70–81

[11] Sader J.E., Sanelli J.A., Adamson B.D., Monty J.P., Wei X., Crawford S.A. Spring constant calibration of atomic force microscope cantilevers of arbitrary shape, *Rev. Sci. Instrum.*, 2012, Vol. 83, pp. 103705-1 to 103705-16

[12] Hutter J.L., & Bechhoefer J. Calibration of atomic-force microscope tips. Rev. Sci. Instrum. 1993, **64** pp. 1868–1873

[13] Butt H.-J., & Jaschke M. Calculation of thermal noise in atomic force microscopy. Nanotechnology. 1995, **6** pp. 1–7

[14] Stark R.W., Drobek T., Heckl W.M. Thermomechanical noise of a free v-shaped cantilever for atomic-force microscopy. Ultramicroscopy. 2001, **86** pp. 207–215

[15] Clifford C.A., & Seah M.P. International Interlaboratory Study of AFM Spring Constant Calibration[2]

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[2] To be published.

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