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

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

Reference number
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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_{Φ} gradient determined from a straight line fit to values of L_x versus $\Phi_x{}^{1/3}$

B_k gradient determined from a straight line fit to values of L_x versus $\left(k_z^{L_x}\right)^{-1/3}$

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

$k_z^{L_x}$ 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

$k_{z(tc=0)}$ 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_{kz} standard uncertainty in the normal spring constant

u_{kzR} 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.

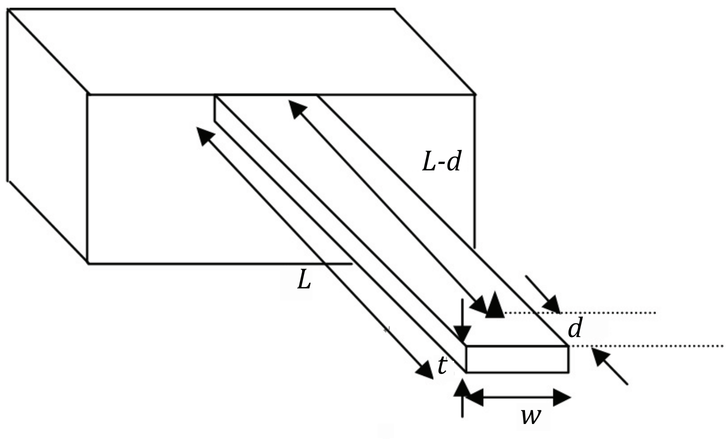
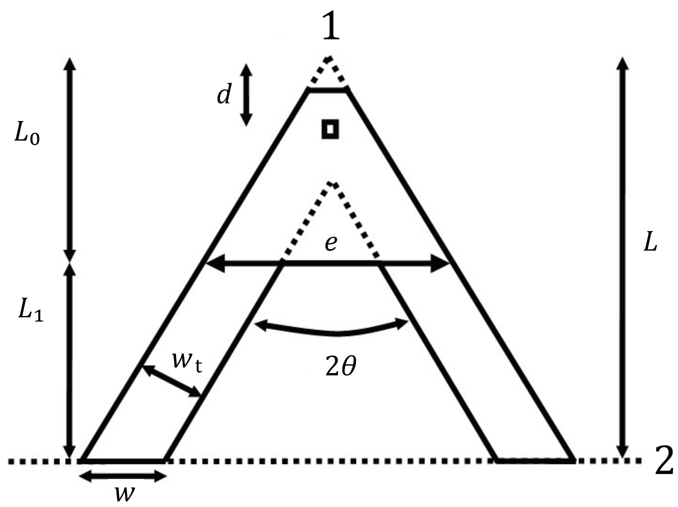


Figure 1 — Schematic of a rectangular shape cantilever with a probe tip a distance *d* from its free end

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

