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Surface chemical analysis — Scanning probe microscopy — Procedure for the determination of elastic moduli for compliant materials using atomic force microscope and the two-point JKR method

Analyse chimique des surfaces — Microscopie à sonde locale — Lignes directrices pour la détermination des modules d'élasticité des matériaux souples en utilisant un microscope à force atomique

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Foreword

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 9, *Scanning probe microscopy*.

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Introduction

Atomic force microscope (AFM) is a member of the scanning probe microscope (SPM) family and is used to image surfaces by mechanically scanning a probe over the surface. In AFM, a surface force is monitored as the deflection of a compliant cantilever, which has a probe tip at its free end in order to interact with surfaces. AFM can provide amongst other data: topographic, mechanical and chemical information about a surface depending on the mode of operation and the property of the probe tip. Accurate force measurements and sample deformation measurements are needed for a wide variety of applications, especially to determine the elastic moduli of compliant materials such as organics and polymers at surfaces. For quantitative force measurements, it is necessary to select an adequate contact mechanic model used to calculate the elastic modulus, and also use the appropriate calculation procedure.

This document describes a procedure for the determination of the elastic moduli for compliant materials using AFM. Force-distance curves are obtained on the surfaces of compliant materials and are used for the calculation of elastic modulus based on Johnson-Kendall-Roberts (JKR) two-point method.

Surface chemical analysis — Scanning probe microscopy — Procedure for the determination of elastic moduli for compliant materials using atomic force microscope and the two-point JKR method

1 Scope

This document describes a procedure for the determination of elastic modulus for compliant materials using atomic force microscope (AFM). Force-distance curves on the surface of compliant materials are measured and the analysis uses a two-point method based on Johnson-Kendall-Roberts (JKR) theory. This document is applicable to compliant materials with elastic moduli ranging from 100 kPa to 1 GPa. The spatial resolution is dependent on the contact radius between the AFM probe and the surface and is typically approximately10-20 nm.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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*

ISO 11775, *Surface chemical analysis — Scanning-probe microscopy — Determination of cantilever normal spring constants*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
force-distance curve
force-displacement curve
<AFM> pairs of force and distance values resulting from a mode of operation in which the probe is set at a fixed (x, y) position and the probe tip is moved towards or away from the surface as the force is measured

Note 1 to entry: The force is usually monitored using the cantilever deflection.

[SOURCE: ISO 18115-2:2013, 5.56]

3.2
normal spring constant
spring constant
force constant
k_z
<AFM> quotient of the applied normal force at the probe tip by the deflection of the cantilever in that direction at the probe tip position

Note 1 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 2 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 AFM can 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.

[SOURCE: ISO 18115-2:2013, 5.92, modified — Note 1 to entry has been deleted and the following notes renumbered.]

3.3
pull-in force
pull-on force
force exerted by the surface on the probe tip at snap-in

[SOURCE: ISO 18115-2:2013, 5.123]

3.4
pull-off force
force required to pull the probe free from the surface

Note 1 to entry: This force is generally measured from the force-distance curve as the value between the force minimum and the zero of force as the probe moves away from the surface.

[SOURCE: ISO 18115-2:2013, 5.124]

3.5
tip radius
<excluding scattering NSOM/SNOM> radius describing the surface curvature in a region at the apex of a stylus or probe tip

Note 1 to entry: It might be necessary to describe the tip by radii in different azimuths.

Note 2 to entry: In practice, tips can only approximate a sphere for a very small region at the tip.

[SOURCE: ISO 18115-2:2013, 5.161]

3.6
tip-sample contact radius
maximum radius of the contact area between the tip and the sample at the maximum indentation depth

[SOURCE: ISO 18115-2:2013, 5.163]

3.7
work of adhesion
energy required when two condensed phases, forming an interface of unit area, are separated reversibly to form unit areas of the free surfaces of those two phases

Note 1 to entry: This term is sometimes also known as the work of separation or the Dupré work of adhesion.

[SOURCE: ISO 18115-2:2013, 5.175]

3.8
Hertzian model
model of tip and surface contact between elastic solids that ignores any surface forces and adhesion hysteresis

Note 1 to entry: This approach, derived by Hertz and described in Reference [1] describes the contact between elastic solids. It ignores any surface forces and adhesion hysteresis and applies at high loads where there are no surface forces present.

[SOURCE: ISO 18115-2:2013, 4.4]

3.9
DMT model
Derjaguin-Müller-Toporov model
model of tip and surface contact in which adhesion forces are taken into account but the tip-sample geometry is constrained to be Hertzian[2]

Note 1 to entry: This approach applies to rigid systems with low adhesion and small radii of curvature. The adhesion forces are taken into account but the tip-sample geometry is constrained to be Hertzian, i.e. Hertzian mechanics with an offset to account for surface forces.

[SOURCE: ISO 18115-2:2013, 4.3]

3.10
JKR(S) model
Johnson-Kendall-Roberts (-Sperling) model
model of tip and surface contact in which adhesion forces outside the contact area are ignored and elastic stresses at the edge of the contact area are infinite[3]

Note 1 to entry: In this work, adhesion forces outside the contact area are ignored and elastic stresses at the edge of the contact area are infinite. At contact, short-range attractive forces suddenly operate, and the tip-sample geometry is not constrained to remain Hertzian. Adhesion hysteresis is described and loading and unloading are abrupt processes. This approach applies to highly adhesive systems with low stiffness and high radii of curvature.

[SOURCE: ISO 18115-2:2013, 4.5]

3.11
Maugis model
Maugis-Dugdale model
model of tip and surface contact between a sphere and a flat surface incorporating the elastic modulus and work of adhesion[4]

Note 1 to entry: This analysis is a complex mathematical description of the contact mechanics between a sphere and a flat surface which applies in all material possibilities through a parameter that is a function of reduced elastic modulus, reduced curvature radius, work of adhesion and the tip-sample interatomic equilibrium distance. At the limits, when this parameter tends to infinity or zero, the Maugis mechanics tend to the JKR(S) or DMT mechanics, respectively.

[SOURCE: ISO 18115-2:2013, 4.6]

3.12
COS model
Carpick-Ogletree-Salmeron model
model of tip and surface contact between a sphere and a flat surface giving a simple general equation that approximates Maugis' solution to within 1 % accuracy[5]

Note 1 to entry: The general equation is amenable to conventional curve-fitting routines and provides a rapid method of determining the approximate value of the parameter described by Maugis.

[SOURCE: ISO 18115-2:2013, 4.2]

4 Symbols (and abbreviated terms)

The abbreviated terms are:

AFM	Atomic force microscopy
a	Tip-sample contact radius
D	Cantilever deflection
E	Elastic modulus of sample
k_z	Normal spring constant
K	Elastic coefficient of sample
F	Normal load
F_1	Normal load of Hertzian model
F_a	Molecular attraction
$F_{\text{pull-off}}$	Pull-off force
R	Tip radius
u_D	Standard uncertainty in the cantilever deflection
u_E	Standard uncertainty in the elastic modulus
u_{kz}	Standard uncertainty in the normal spring constant
u_R	Standard uncertainty in the tip radius
u_δ	Standard uncertainty in the indentation depth of sample
w	Work of adhesion
Z	vertical scanner displacement
z_0	Atomic equilibrium separation
δ	Indentation depth of sample
μ	Tabor parameter
ν	Poisson's ratio of sample

5 Review of contact mechanics

5.1 Introduction

Contact mechanics plays a key role in understanding the phenomena occurring around the contact between a probe tip and a surface. It can give answers about, for example, how much contact radius and indentation depth are induced by a given load and how much stress is applied around a probe apex. In particular, contact mechanics is necessary to calculate the mechanical properties from the experimental force–distance curves.

This clause introduces several contact mechanics theories with the aim of applying them to force spectroscopy AFM measurements. The following discussion assumes the contact between a spherical probe tip with radius of curvature (tip radius), R and a flat surface with elastic modulus, E and Poisson's ratio, ν. The so-called elastic coefficient, K is defined in Formula (1):

$$K = \frac{4}{3} \frac{E}{1 - \nu^2} \tag{1}$$

5.2 Hertzian model

The Hertzian theory about the contact between two elastic bodies relates the contact radius a to the normal load F_1 by Formula (2):

$$F_1 = \frac{Ka^3}{R} \tag{2}$$

It also relates the indentation depth, δ to the contact radius a by Formula (3):

$$\delta = \frac{a^2}{R} = \left(\frac{F_1{}^2}{RK^2} \right)^{1/3} \tag{3}$$

This Formula is used for a spherical probe with a tip radius of R.

NOTE This theory was developed by Hertz in 1882[6].

5.3 Derjaguin-Muller-Toporov (DMT) Model

The DMT model assumed, in addition to Hertzian repulsion, the existence of molecular attraction forces, which would not be able to change the contact profile appreciably. The net normal load F is given by $F = F_1 + F_a$, where F_1 is Hertzian repulsion given by Formula (2) and F_a is the molecular attraction (<0). Thus the profile predicted by the DMT model is the same as Hertzian profile, but smaller net normal load is required. Owing to the assumption that the indentation depth has the identical form with Formula (3), F and δ can be directly related by Formula 4:

$$F = KR^{1/2} \delta^{3/2} + F_a \tag{4}$$

The attractive interaction F_a depends on the profile near the contact perimeter and is typically represented as a function of the contact radius a. At the point contact ($a \rightarrow 0$), elastic displacement on the surface profile vanishes in the case of DMT model, hence F_a can be approximated to Bradley's value for the pull-off force, $F_{pull\text{-}off}$, as shown in Formula 5:

$$F_a \approx F_{pull-off} = -2\pi wR \tag{5}$$

where w is the work of adhesion.

NOTE This adhesive contact theory was developed by Derjaguin *et al.* in 1975[2]. By 1960s, several experimental results had been reported that are contradictory to the Hertz theory, especially at low loads. These observations strongly suggested the intervention of attractive surface forces in the elastic contact of two bodies.

