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Surface Chemical Analysis — Atomic force microscopy — Procedure for in situ characterization of AFM probe shank profile used for nanostructure measurement

Analyse chimique des surfaces — Microscopie à balayage de sonde — Procédure pour la caractérisation in situ des sondes AFM utilisées pour mesurer la nanostructure

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Foreword

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

Introduction

Atomic force microscopes (AFMs) are of increasing importance for imaging surfaces at the nanoscale. The imaging mechanism involves a dilation of the surface form by the AFM probe shape. In practice, the radii of probe tips are in the range of 1 nm to 200 nm, which is the same order of magnitude as that of many important surface features. AFM images may, therefore, be strongly affected by the shape and size of the AFM probe used for imaging. In addition, the mechanism used to control the distance between the AFM probe and the sample surface can create artefacts in AFM images, because the effective probe shape characteristic depends on the control parameters. The probe radius and its half-cone angle are often used for the specification of AFM probes. However, practical probes are often not described so simply. Therefore, a quantitative expression for probe shank shape is required. This International Standard describes two methods for the detailed determination of probe shank shape: a projection of the probe profile (PPP) and the effective probe shape characteristic (EPSC), both of which are projected onto a defined plane and which, in turn, include the effect of the probe controlling mechanism. The PPP provides a continuous profile, whereas the EPSC provides a few discrete characteristic points. PPP, used in conjunction with a probe shape characteristic (PSC) measurement, gives the quality of the probe for general applications, whereas EPSC indicates the usefulness of the probe for depth measurements in narrow trenches and similar profiles. The true surface shape can be recovered and estimated from the measured surface with an accurate model of the true probe shape. This International Standard provides methods for the quantitative determination of aspects of AFM probe shank shape, to ensure that the probe is adequate to measure surfaces with narrow trenches and similar profiles and to ensure reproducible AFM imaging.

Surface Chemical Analysis — Atomic force microscopy — Procedure for in situ characterization of AFM probe shank profile used for nanostructure measurement

1 Scope

This International Standard specifies two methods for characterizing the shape of an AFM probe tip, specifically the shank and approximate tip profiles. These methods project the profile of an AFM probe tip onto a given plane, and the characteristics of the probe shank are also projected onto that plane under defined operating conditions. The latter indicates the usefulness of a given probe for depth measurements in narrow trenches and similar profiles. This International Standard is applicable to the probes with radii greater than $5u_0$, where u_0 is the uncertainty of the width of the ridge structure in the reference sample used to characterize the probe.

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 18115-2:2010, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

ISO/TS 80004-4:2011, *Nanotechnologies — Vocabulary — Part 4: Nanostructured materials*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-2, ISO/TS 80004-4 and the following apply.

NOTE Some of the terms and definitions are reprinted here for convenience.

3.1
aspect ratio of the probe
ratio of the probe profile length at a certain position to the probe profile width at that position

3.2
deflection sensitivity
sensitivity factor converting the output of an AFM optical displacement detection system for a cantilever in the contact mode to the displacement of the tip

3.3
error signal
feedback control system signal whose amplitude and sign are used to correct the position and/or alignment between the controlling and the controlled elements

3.4
effective probe shape characteristic
EPSC
relationship between the probe profile width and probe profile length for a given probe, including the effects of the true probe shape, artefacts due to the feedback controlling mechanism in the AFM modes employed, and other imaging mechanisms of AFM projected onto a defined plane

Note 1 to entry: The defined plane is usually the *x*-*z* plane.

3.5
narrow-ridge structure
isolated plateau with thin width having wide gaps on either side

3.6
peak force mode
AFM intermittent contact mode using frequencies well below resonance in which the maximum force is used for measurement or for imaging

3.7
probe apex
structure at the extremity of a probe, the apex of which senses the surface[4]

3.8
probe profile width
projected width of a probe at a defined probe profile length, which may be for a defined azimuth or projection plane

Note 1 to entry: The defined projection plane is usually the *x*-*z* plane.

3.9
probe profile length
length, measured from the probe apex along the instrument's *z* (vertical)-axis, to a defined point on the probe axis

3.10
probe shape characteristic
PSC
relationship between the probe profile width and the probe profile length for a given probe projected onto a defined plane

Note 1 to entry: The defined projection plane is usually the *x*-*z* plane.

3.11
projected probe profile
PPP
measured profile of the probe projected onto a defined plane

Note 1 to entry: The defined projection plane is usually the *x*-*z* plane.

Note 2 to entry: Figure 1 a) shows schematically the relationship between the probe profile width, *w*, and length, *l*, and b) the definition of the aspect ratio, *a*.

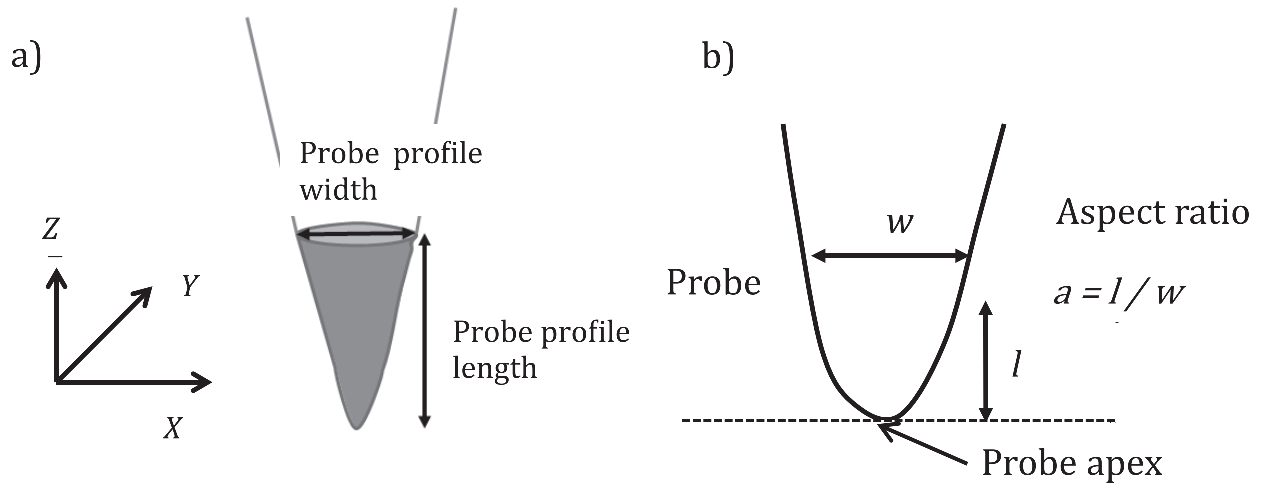


Figure 1 — Probe profile width (*w*), defined here for projection on the x-z plane, and probe profile length (*l*)

4 Symbols and abbreviated terms

In the list of abbreviated terms below, note that the “M” in the abbreviation “AFM,” defined here as an abbreviation for “Microscopy,” also is used as an abbreviation for “Microscope” depending on the context. The following are the abbreviated terms.

AFM	atomic force microscopy
AM	amplitude modulation
EPSC	effective probe shape characteristic
CRM	certified reference material
PID	proportional integral derivative (controller)
PSC	probe shape characteristic
PPP	projected probe profile
TEM	transmission electron microscopy

The following are the symbols for use in the formulae and as abbreviations in the text.

A_0	free oscillation amplitude of the cantilever before approaching the probe to the sample
A_{sp}	oscillation amplitude of the cantilever for AFM imaging
a	aspect ratio of the probe
D_0	distance between the side wall of an isolated ridge structure and the adjacent wall
D_j	line distance between the two side walls of the j_{th} trench structure
e_m	maximum error signal, in nanometres, measured during the recording of the probe shape data

f	numbers of the frames
H_0	average depth of the trenches on either side of the ridge structure
H_j	depth of the j_{th} trench structure
j	index number for the j_{th} measurement of trench
l	length of the probe profile
l_j	maximum measured depth for the j_{th} trench
L_0	width of the ridge structure
m	index number for m_{th} measurement of the probe profile length
n	index number for n_{th} measurement of the probe profile length
p_m	probe profile length at m_{th} measurement
p_n	probe profile length at n_{th} measurement
q	difference of probe profile length between PSC and EPSC data
r	corner radius of the reference sample
r_{r}	corner radius of the ridge structure provided by the CRM supplier
r_{t}	maximum corner radius of the trench structure
r_j	corner radius of the side wall of the j_{th} trench structure provided by the CRM supplier
s	maximum slope of the PSC curve
s_E	maximum slope estimated from the EPSC data
u	combined standard uncertainty of the measurement of the probe profile length
u_0	standard uncertainty of the width of the ridge structure
u_{s}	standard uncertainty of the random component obtained by the probe profile length measurement
u_{t}	standard uncertainty of the gap width of the multiple-trench structure
w	projected profile width of the AFM probe in the x-z plane
w'	apparent width of the ridge structure
w_j	measured width of the AFM probe at j_{th} measurement
ΔL	error in l caused by the presence of a non-zero value of r_{r}

5 Procedure for probe characterization

5.1 Methods for the determination of AFM probe shapes

There are two methods to determine AFM probe shank profiles:

a) narrow-ridge method to determine the probe projected profile (PPP) and the probe shape characteristic (PSC);

b) multiple-trench method to determine the effective probe shape characteristic (EPSC) for depth measurement.

Either one or both of the above methods shall be used to determine aspects of the probe shank profile. Suitable applications for each method are given in Table 1. The approximate profile of an AFM probe tip, i.e. the profile obtained by removing that of the tip apex, is determined by the narrow-ridge method using a reference sample. The resulting profile is given as the PPP onto a given plane. The PSC is an expression of the relationship between the probe profile width and length obtained from PPP. The EPSC is the PSC determined at a few points using a multiple-trench structure. The narrow-ridge method is used mainly for the evaluation of AFM measurements for convex nano-structures, i.e. protrusions, whereas the multiple-trench method, under the two-point contact condition, is mainly used for depth measurements in narrow trenches and similar profiles. The two methods generate results that differ to an extent that depends on the measurement conditions, such as humidity and the parameters used to control the probe during AFM imaging. The appropriate probe characteristic shall be used for the relevant analysis.

NOTE Examples of PSC and EPSC are shown in Annex A.

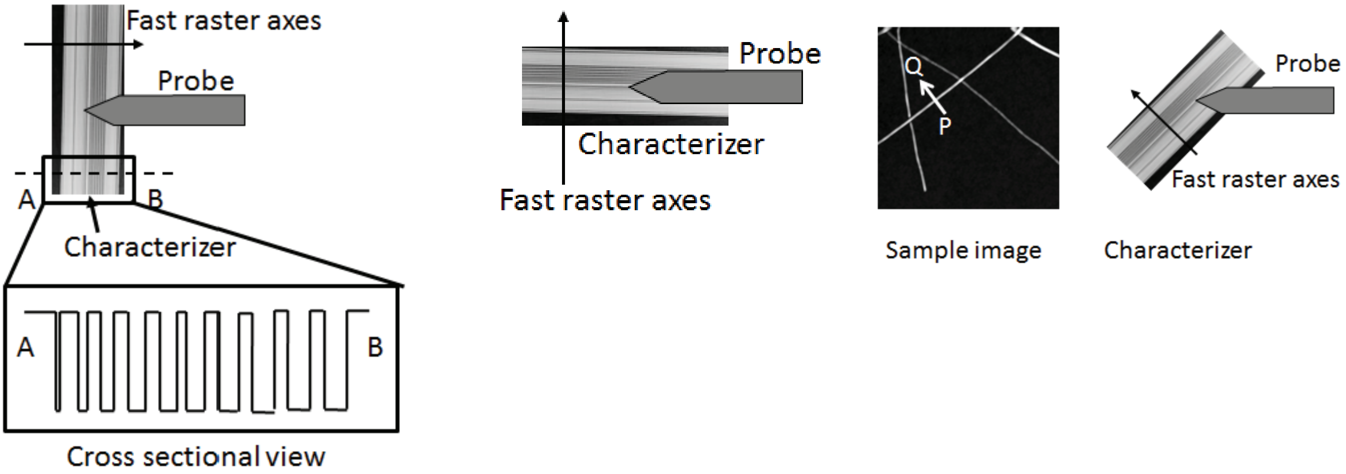
Table 1 — Summary of the methods

Subclauses	Method	Suitable application	Properties to be determined
5.2 to 5.4.1, 5.4.2, 5.5	Narrow-ridge method	Conventional AFM measurements and analysis of protrusions in AFM images	i) Projected probe profile (PPP), ii) Probe shape characteristic (PSC)
5.2 5.4.1, 5.4.3, 5.5	Multiple-trench method	Depth measurements, such as those of contact holes and trenches	Effective probe shape characteristic (EPSC)

5.2 Reference sample setting

A reference sample with either a ridge structure, suitable trench structures, or both types of structures is required and is described in Annex B. An example of a reference structure is shown in Annex C. It is recommended to select a reference sample with a small corner radius and various sets of trench structures. If the corner radius is large, the uncertainty region at the tip apex increases. The reference sample shall be set on the sample holder so that the relevant structures are placed either perpendicular or parallel to the fast raster axis of the AFM scanner, as shown in Figure 2 a) and Figure 2 b). Specifically, if the probe shape is to be projected onto the plane through the longitudinal direction of the cantilever, the lines of the pattern in the reference sample and the longitudinal direction of the cantilever shall be aligned perpendicular to each other, as shown in Figure 2 a) and Figure 2 b) is the complementary case for characterizing the probe shape projected onto the plane normal to the longitudinal direction of the cantilever. If instead the probe shape is required for a different specific orientation [for example, P-Q direction in Figure 2 c)], align the direction of the pattern lines in the reference sample to be perpendicular to that specified orientation, as shown in Figure 2 c). The reference structures of the sample should be rigidly fixed onto the specimen holder, and the orientation of the structures of the reference sample with respect to the fast raster axis of the scanner shall be set within an error of 1° of perpendicular orientation. When the ambient humidity changes, the effective probe shape often changes due to the altered amount of water adsorbed on the probe surface[3]; thus, humidity shall be recorded.

The reference specimen and the cantilever shall be electrically grounded to avoid electrostatic damage and the accumulation of contaminants collected by electrostatic charge.



a) Perpendicular to the fast raster axis

b) Perpendicular to the fast raster axis

c) Perpendicular to a specific direction (P-Q direction) in an AFM image

Figure 2 — Alignment of the AFM probe to the lines of a pattern in the reference sample

5.3 Requirements of AFM and AFM imaging

The *x*-, *y*-, and *z*-axes of the AFM shall be calibrated for their orthogonality and for the measurement of length scales to the accuracy required by the user.[2] The accuracy level should be considered carefully since this may limit the accuracy with which the probe shape may be defined. The topographic image and error-signal image shall both be measured and displayed synchronously for monitoring and optimization of the feedback parameters. The scan length along both *x* and *y*-axes should be set to cover the necessary structures of the reference material. If a closed-loop scanning system is used, the position noise/lateral noise ratio should be considered to determine the desired resolution. The pixel size should be chosen so that it is consistent with the desired resolution of <1 nm.

EXAMPLE For a raster width of 1 000 nm, 2 000 pixels per line scan should be used.

At least 10 raster lines shall be measured. The scan rate and other feedback parameters for imaging shall be optimized to achieve an amplitude error-signal of <1 nm. Other control parameters should be set to values similar to those that will be used for the actual measurements of the sample of interest, if possible. Other experimental conditions shall be adjusted by following the instrument manual or other documented and validated procedures. Such conditions include the alignment of the laser and detectors, initialization of other hardware, and adjustment of the excitation frequency (for dynamic mode operation). After setting up the AFM, check the thermal drift in the image. If the thermal drift remains, see ISO 11039.[1] The imaging mode of the AFM, such as contact mode, intermittent contact mode (AM-mode), and others, shall be set to obtain the topographic and error signal images. A common procedure for setting up the AFM is to adjust the setpoint such that the forward and backward profiles of the probe match. However, a difference between the PSC and EPSC may remain. In AM mode, by increasing the free oscillation amplitude, decreasing the operating amplitude (low setpoint), or both, the difference between PSC and EPSC may be minimized. However, tip-wear increases when high amplitudes and high setpoints are used. For reducing tip wear, the phase contrast should be maintained at low levels, without abrupt phase jumps and apparent topographic jumps. If the difference between PSC and EPSC is reduced, the free oscillation amplitude and setpoints should be adjusted to realize the maximum apparent depth of the trenches. Another method to reduce the difference between PSC and EPSC is to use an operating mode that is controlled by cantilever deflection, such as contact mode or