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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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ISO 13095 was prepared by Technical Committee ISO/TC 201, *Surface Chemical Analysis*, Subcommittee SC 9, *Scanning Probe Microscopy*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

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 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 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 probe's profile 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 shape is required. This document describes two methods for the detailed determination of probe 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. To recover and estimate the true surface shape, the measured surface must be eroded mathematically by an accurate model of the true probe shape. This International Standard provides methods for the quantitative determination of aspects of AFM probe shape, to ensure that the probe is adequate to measure surfaces with high-aspect-ratio structures 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

Thi 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 reference(s)

The following referenced documents are indispensable for the application 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.

ISO18115-2:2010 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 below from ISO 18115-2:2010 Surface chemical analysis – Vocabulary – Part 2: Terms used in scanning-probe microscopy apply and are reprinted here for convenience [1].

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 in nanometers

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 – the defined plane is usually the *x*-*z* plane; see Annex A.

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 senses the surface [ISO 18115-2, 6.120]

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 – 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 – 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 – the defined projection plane is usually the *x-z* plane.

Figure 1 shows schematically the relationship between the probe profile width *w* and length *l* (a) and the definition of the aspect ratio *a* (b).

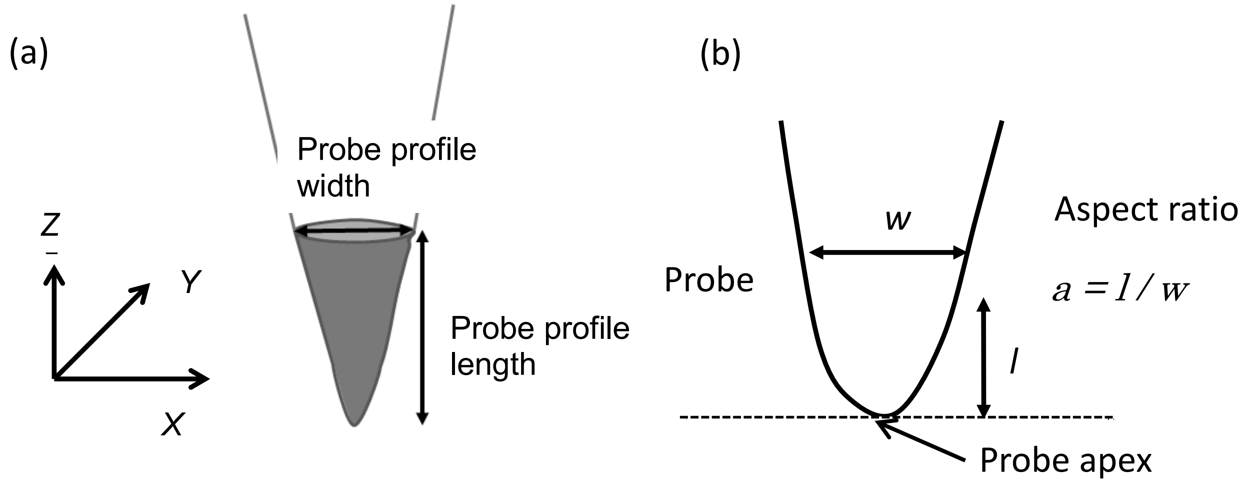

Figure 1 — The probe profile width (*w*), defined here for projection on the x-z plane, and probe profile length (*l*)

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 abbreviated terms are:

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 symbols for use in the equations and as abbreviations in the text are:

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 nanometers, 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 D.