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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*.

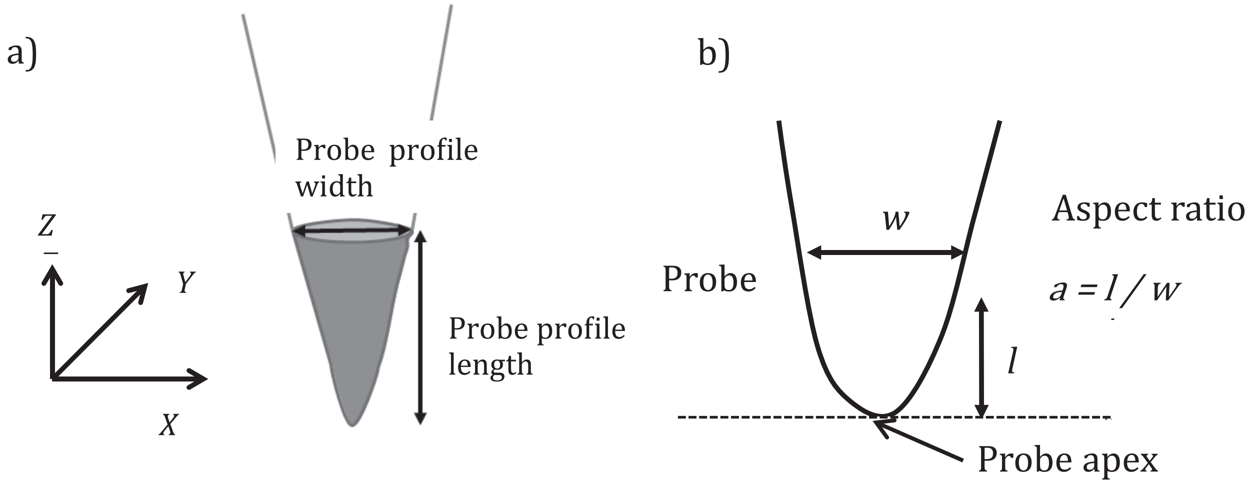


Figure 1 — Probe profile width (*w*), defined here for projection on the x-z plane, and probe profile length (*l*)

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.

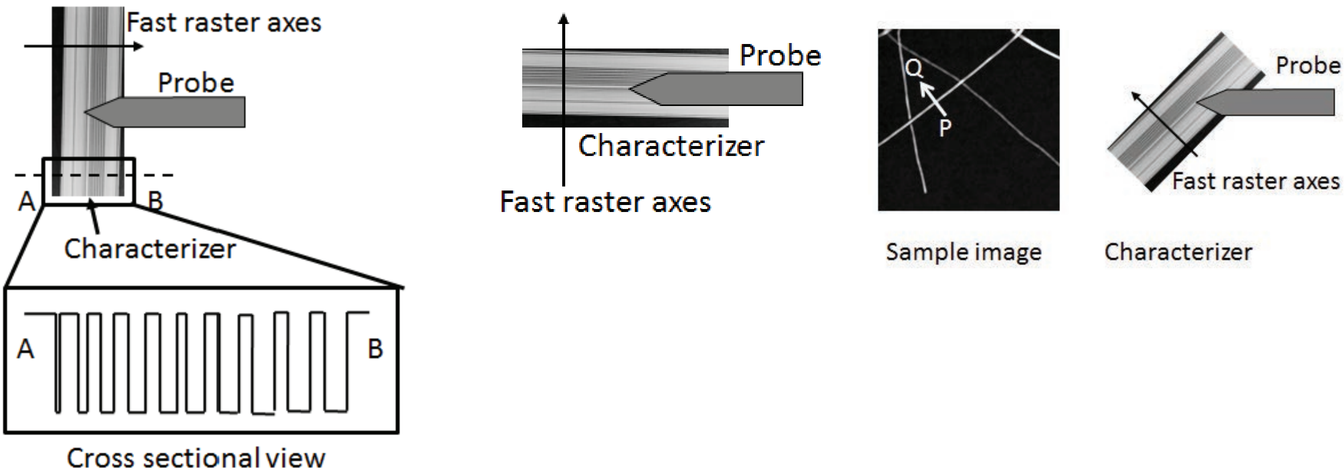
Table 1 — Summary of the methods

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 peak force mode. If static contact mode is used, then it is recommended to use hopping scanning mode (a scanning mode in which, at each pixel, the surface is approached with the probe to a setpoint, then the height of the probe is recorded at this point and the probe is retracted[5]). To preserve the shape of the probe, careful changes of the controlling parameters are required to reduce the difference of the probe profile width between PSC and EPSC.

NOTE An example of an EPSC measurement, with associated reproducibility, is provided in Annex D.

5.4 Measurement of probe shank profile

5.4.1 General Remarks

A narrow-ridge structure shall be measured for PPP analysis, and a multiple-trench structure shall be measured for EPSC analysis under two-point contact conditions. The choice of experimental parameters such as the oscillation amplitude of the cantilever and the setpoints may affect the EPSC measurement results. These parameters shall be optimized by following the instrumental manual or other documented and validated procedures. The recommended absolute value of the maximum error signal e_m is smaller than 1 nm (i.e. $| e_m | < 1$ nm). The measured surface plane of the profile of the reference sample shall be as level as possible. The plane correction for probe profile analysis is described in Annex E.

5.4.2 Method using narrow-ridge structure for obtaining PPP and PSC

The reference sample, which includes a narrow-ridge structure with known line width, uncertainty of line width, and edge radius, shall be obtained. Details of the reference sample are shown in Annex B. The procedure for determining the PPP and the PSC are given below.

a) Measure the line profile of the narrow-ridge structure along the fast raster axis of the AFM instrument. The scan length along this axis shall be sufficiently wide such that the depth measured on either side of the ridge reaches to 90 % of the total depth, H_0.

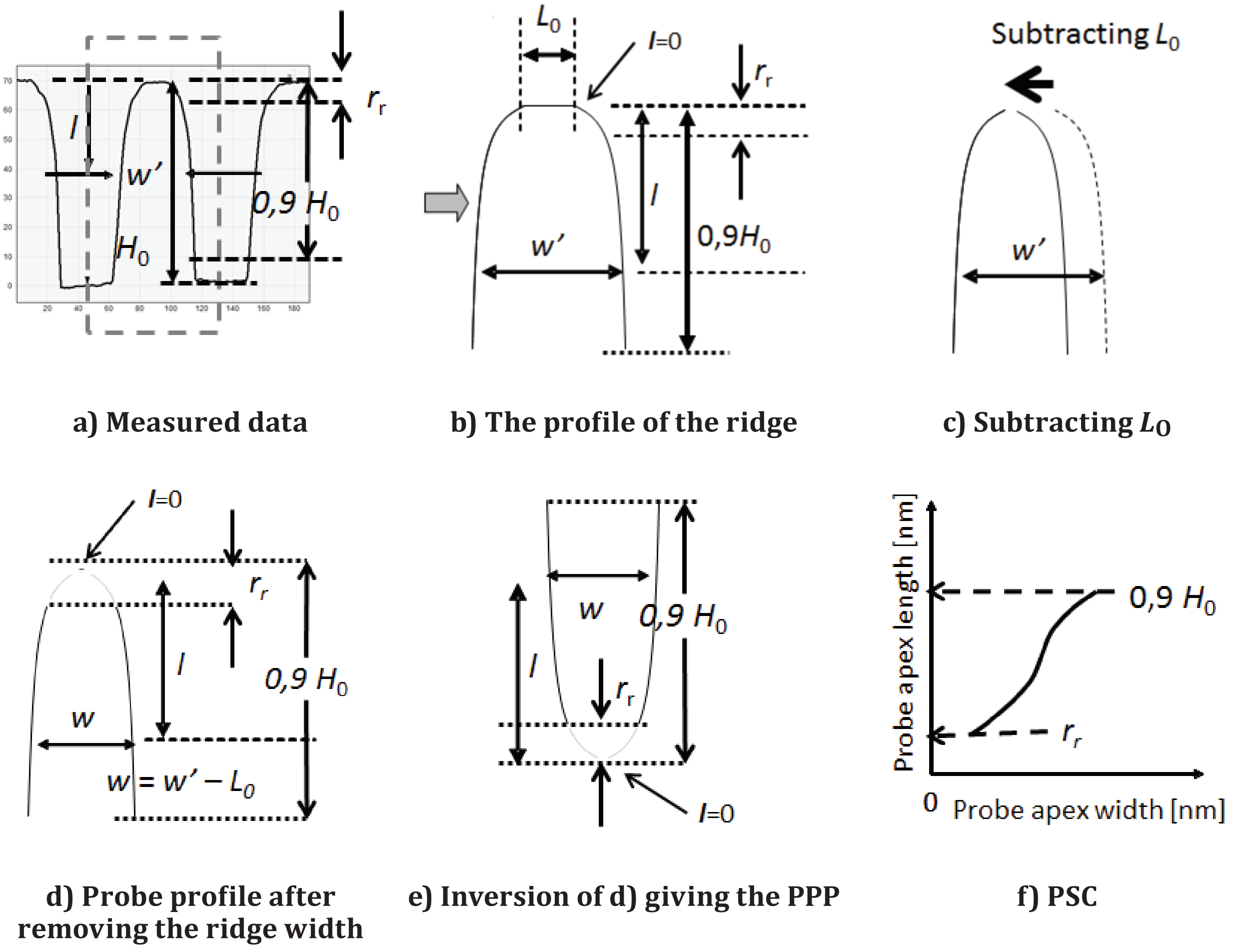
   NOTE If the probe reaches the bottom of the adjacent trenches, spurious wings appear in the probe shape; hence plotted data are limited to depths up to 0,9 H_0.

b) Plot the measured line profile taking the *x*-axis as the fast scanning axis and the *z*-axis as the measured height. The scale units shall be in nanometres for both axes. The top of the profile, averaging over the plateau which includes the width of the ridge structure, shall be set on the *z*-axis at 0 nm ($l = 0$ nm), and the maximum scale depth on either side shall be over 0,9 H_0, as shown in Figure 3 a).

c) Draw two horizontal lines on the plot obtained in step b, as shown in Figure 3 b). One line is at the *z*-axis depth of r_r, where r_r is the radius of the ridge corner and is determined separately, and the other line is at the depth of 0,9 H_0.

d) Re-plot the line profile obtained in step b by subtracting the value of the width of the ridge structure (L_0) from the *x*-axis values for the data for the right hand side of the ridge structure in the height region between r_r and 0,9 H_0, as shown in Figure 3 c) and Figure 3 d). Invert this plot as shown in Figure 3 e), this provides the PPP.

e) Plot the relationship between the width w (*x*-axis) and length l (*z*-axis) of the profile obtained in step d. Use “probe profile width” as the *x*-axis label, and “probe profile length” as the *z*-axis label. The minimum and maximum values of the *z*-axis shall be r_r and 0,9 H_0, respectively, as shown in Figure 3 f). This provides the PSC.



a) Measured data **b) The profile of the ridge** **c) Subtracting L_0**

d) Probe profile after removing the ridge width **e) Inversion of d) giving the PPP** **f) PSC**

Figure 3 — Determination of PPP and PSC using a narrow-ridge structure

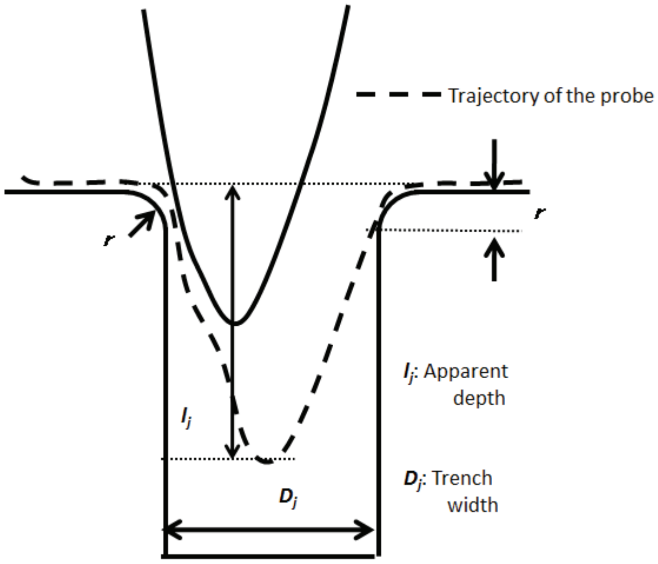
5.4.3 Method using multiple-trench structure for obtaining EPSC

A reference sample that includes a multiple-trench structure with different known gap distances and associated uncertainties shall be obtained. Details of a representative reference sample are shown in Annex B. The procedure for determining the EPSC is given below.

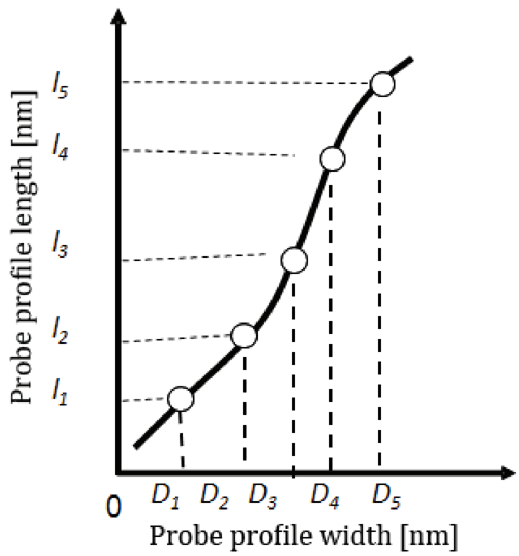
a) Measure the line profile of the multiple-trench structure along the fast raster axis of the AFM instrument. The scan length along this axis shall be sufficiently wide so that more than three trench structures are measured. Confirm that flat regions appear at both sides in each trench profile.

b) Draw a straight line connecting the flat region appearing on either side of the j_{th} trench, and measure the maximum depth l_j from that line, as shown in Figure 4 a).

c) Measure the maximum (apparent) depth l_j for each trench structure. Plot l_j as a function of the gap width D_j, which is included in the data sheet for the reference sample. Label the x-axis as “probe profile width” and the *z*-axis as “probe profile length.” The units of the *x* and *z*-axes shall be in nanometres, as shown in Figure 4 b) for an example with five trenches.



a) Schematic drawing of the probe and the j_{th} trench with the trajectory of the probe ape



b) EPSC measured from the multiple-trench structure

Figure 4

5.5 Uncertainty of the measurement of the probe shank profile

Divergence from the true shape of the probe can arise due to deviations of the reference sample from its ideal shape, measurement errors of the instrument, and variation of the contact area due to the positional relationship between the probe and sample surface. EPSC is affected by contact area, but PSC is not, because point contact is realized on the narrow-ridge structure. Since sharp AFM tips will wear with use, and in order to reduce the amount of work needed to obtain PSC (or PPP) and EPSC, a simple uncertainty estimate is described below.

5.5.1 Uncertainty of the PPP or PSC measurement

The uncertainty of the PPP or PSC measurement of the probe shank profile arises mainly from the standard uncertainty of the width, u_0, of the ridge structure, the standard deviation in the error of the probe control signal, e_m, in nanometres, the maximum slope, s, in the PSC curve, and corner radius, r_r, of a ridge structure. Calculate the standard uncertainty of the measurement, u_s, as follows:

a) From the image of the selected area containing the ridge structure, select f line scans evenly distributed through the image where $f > 7$;

b) Calculate the probe profile length (p_1, p_2, ...) for selected f line scans. Obtain PSC [see Figure 3 f)] for each selected line scan as the first step, and then calculate p at a width of $H_0/2$;

c) Calculate the standard uncertainty, u_s, of the probe profile length measurement.

$$u_s = \frac{\sqrt{\sum_{m=1}^{f}\left(p_m - \frac{1}{f}\sum_{n=1}^{f} p_n\right)^2}}{\sqrt{(f-1)\sqrt{f}}} \quad (1)$$

Then the combined standard uncertainty, u, of the PPP and PSC measurement is given by the following Formula (2), with putting $e_m = 0$ if e_m is not available from the instrument.

$$u(\mathrm{PSC}) = \sqrt{{r_r}^2 + (su_0)^2 + {e_{m(\mathrm{PSC})}}^2 + {u_{s(\mathrm{PSC})}}^2} \quad (2)$$

5.5.2 Uncertainty of the EPSC measurement

The EPSC is to be used only to define the limitations for depth measurement. For the EPSC, the combined uncertainty, u, for the measurement of probe profile length is given by a formula similar to that of 5.5.1, by replacing r_r with r_t, and s with s_E, and u_0 with u_t. Then the combined standard uncertainty, u, of the EPSC measurement is given by Formula (3).

$$u(\mathrm{EPSC}) = \sqrt{{r_t}^2 + (s_E u_t)^2 + {e_{m(EPSC)}}^2 + {u_{s(EPSC)}}^2} \quad (3)$$

with putting $e_m = 0$ if e_m is not available. Here, u_s in the formula is obtained by applying the similar steps described in 5.5.1 for the measurement of multiple trench structures instead of the ridge structure.

When both the PSC and EPSC data are obtained, it is allowed to estimate the combined standard uncertainty, u, of the EPSC measurement by adding q to the combined uncertainty of the 5.5.1. That is, with putting $e_m = 0$ if e_m is not available,

$$u(\mathrm{EPSC}) = \sqrt{{r_r}^2 + (su_0)^2 + {e_{m(\mathrm{PSC})}}^2 + {u_{s(\mathrm{PSC})}}^2 + q^2} \quad (4)$$

where q is the difference of probe profile length between the PSC and EPSC data. In case the use of r_t (maximum corner radius of the trench structures) leads to over estimation of uncertainty, Formula (4) would give better estimation of the combined uncertainty.

6 Reporting of probe characteristics

The following characteristics of the probe and specifications of the reference sample shall be reported:

a) PPP of the AFM tip given by step 4 in 5.4.2, as shown in Figure 3 e);

b) PSC given by step 5 in 5.4.2;

c) EPSC given by step 3 in 5.4.3;

d) uncertainty of the PPP or PSC or EPSC measurement obtained as described in 5.5;

e) specifications of reference sample used for analysis. That is, the width and its uncertainty of a ridge structure, the edge radius of a ridge structure, and the average depth of the trenches on either side of a ridge structure for PPP or PSC measurement. Sets of the gap distance and the trench width of multiple trench structures, and the uncertainty associated with each gap distance for EPSC measurement.

The following experimental parameters and conditions shall also be reported, model of the cantilever, PID feedback parameters, setpoints, line scanning speed (with units nm/s) or line scanning rate (with unit Hz), pixels and lines in the image, temperature, humidity.

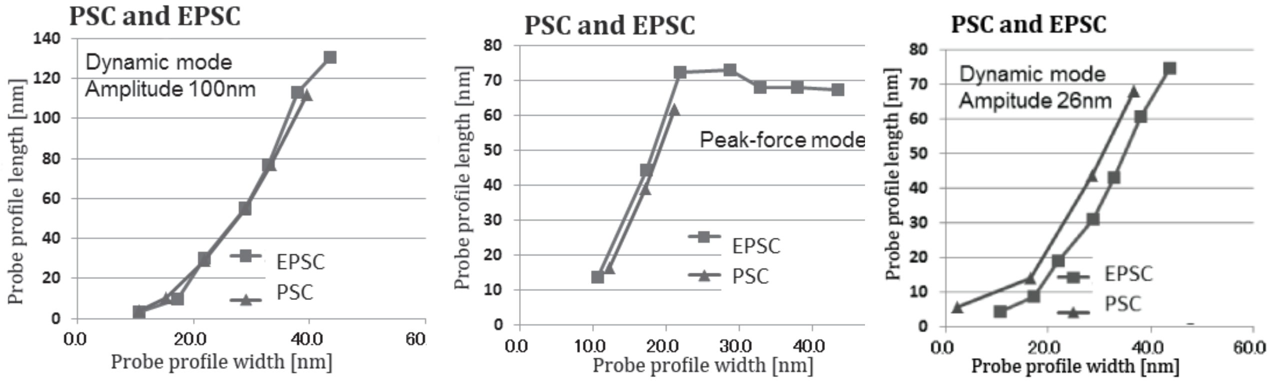
An example of a report is shown in Annex F.

Annex A
(informative)

Dependence of AFM images on measurement mode and settings

A.1 Results of the inter-laboratory test

Figure A.1 a), Figure A.1 b), and Figure A.1 c) are typical results of an inter-laboratory comparison of probe profile measurement. Figures A.1 a) and Figure A.1 c) show the PSC and EPSC in intermittent contact mode. If the cantilever oscillation (free oscillation amplitude) is large enough (typically more than 60 nm peak to peak), the PSC and EPSC are in good agreement. The PSC and EPSC are also in good agreement using the Bruker “peak-force” mode, as shown in Figure A.1 b). These results show that either a large oscillation amplitude or a low amplitude with a low setpoint (see A.2) are required for consistency of the PSC and EPSC. The operating amplitude for the intermittent contact mode used to acquire Figure A1 c) is 26 nm peak-to-peak. By using a low amplitude with a low setpoint, degradation of the probe is minimized, but small differences between the PSC and EPSC remain, as shown in Figure A.1 c).



a) Country A: intermittent contact mode **b) Country B: “peak-force” mode** **c) Country C: intermittent contact mode**

Figure A.1 — Results from inter-laboratory comparison

NOTE 1 The amplitude given in the figures is the peak-to-peak oscillation amplitude observed before the probe approaches the sample surface

NOTE 2 The trench depths of the reference samples used in these inter-laboratory tests ranged from 70 nm to 140 nm.

A.2 Example AFM image for a trench structure: dependence on the amplitude setpoints

Figure A.2 shows the change observed in the measured depth of trenches as a function of the setpoint for the operating amplitude for AFM imaging in intermittent contact mode. The measured depth of the trench image changes as a function of the operating amplitude of cantilever oscillation, as shown in Figure A.2. The EPSC measured from multiple trenches depends on this oscillation amplitude, whereas the PSC obtained from the sharp-ridge structure is not as sensitive to oscillation amplitude, as shown in Figure A.3 b). The process is reversible, and a lower setpoint (reduced A_{sp} value for a given A_0) is required for the best determination of the trenches. Thus, the effective probe shape, measured from multiple trenches, changes as a function of the setpoint, as shown in Figure A.3 a) and Figure A.3 b). Figure A.2 was obtained using a free peak-to-peak amplitude of oscillation (A_0) of 42 nm, with operating peak-to-peak amplitudes of oscillation (A_{sp}) of 17 nm, 27 nm, 32 nm, and 37 nm at a scanning rate of 0,05 Hz (100 nm/s).

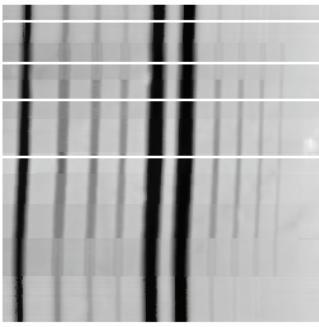
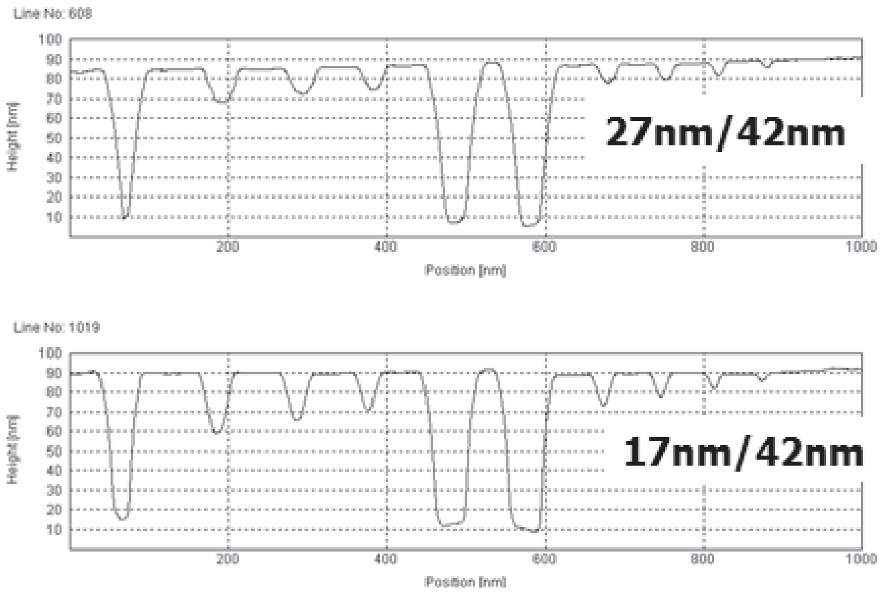
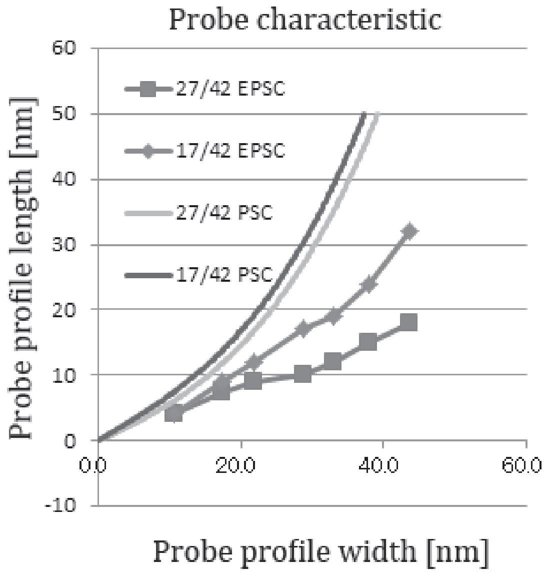


Figure A.2 — Trenches observed in an AFM image as a function of the setpoint (operating amplitude) in intermittent contact mode



a) Line profile from AFM images



b) PSC measured from the ridge structure and EPSC measured from multiple trenches, each for two A_{sp} values with A0 = 42 nm

Figure A.3

Annex B
(normative)

Reference sample preparation

B.1 Requirements for the reference sample

The reference sample for probe characterization shall contain one or both of two kinds of structures: (i) an isolated narrow-ridge structure and (ii) a set of trenches of different widths. The side walls of both structures shall be fabricated vertically relative to the top surface so that the walls and top surface meet at right angles within 1°. Figure B.1 shows a typical cross-section for a reference sample that contains the two essential structures, the dimensions of which are given in Table B.1. An example of a fabricated structure is shown in Annex C. The ridge width and trench widths shall be specified in the region between $2r_r$ or $2r_j$ from the top and halfway down the ridge and trench structures. It is best to use a measurement close to the top surface, yet further away than $2r_r$ or $2r$, as shown in Figure B.2 b). Parameters such as the width of the ridge structure, L_0, uncertainty of the width, u_0, and height of the ridge structure, H_0, shall be given in the data sheet of the reference sample for PPP analysis using the ridge structure. If the EPSC for depth measurement is required,[6] the trench widths (D_1, D_2, D_3, ...), edge radius, r_r, of the ridge structure in Figure B.2 and Table B.1, trench depths, and corner radius of the trench structure, r_j, shall all be given with their uncertainties.

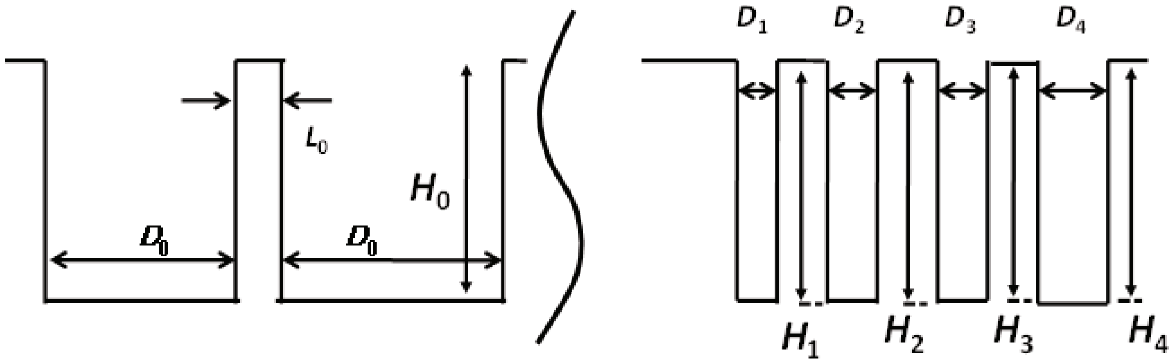


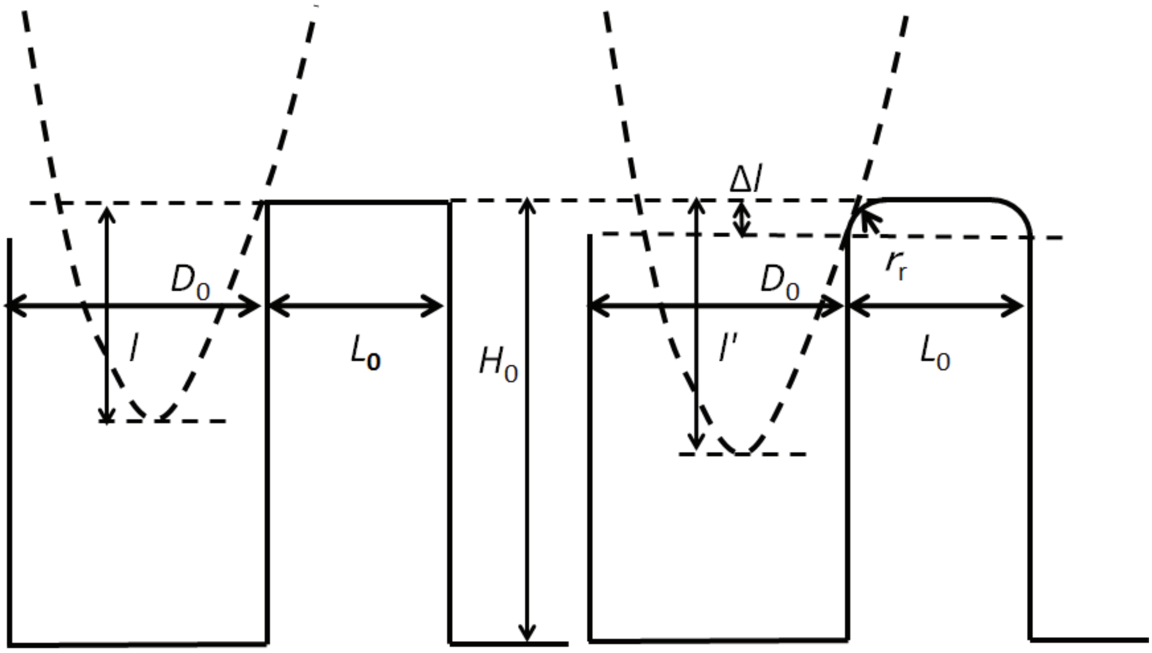
Figure B.1 — Typical design of a reference sample with multiple trenches and a narrow-ridge structure

Table B.1 — Symbols for design parameters of the reference samples shown in Figure B.1

	Ridge structure	Trench 1	Trench 2	Trench 3	Trench 4
Ridge width	L_0				
Trench (gap) width	D_0	D_1	D_2	D_3	D_4
Ridge height/trench depth	H_0	H_1	H_2	H_3	H_4

B.2 Narrow-ridge structure

The narrow-ridge structure is recommended to have a width L_0 narrower than 50 nm and an edge radius r_r smaller than 10 nm [see Figure B.2 a)]. The distance from the wall of the ridge structure and the other side wall D_0 shall be greater than either 50 nm or 120 % of the maximum probe profile width to be determined. The corner radius, r_r of the ridge structure affects the resolution of the probe shank profile, and leads to an uncertain zone at the probe apex. Because of this corner radius effect, the probe profile length is biased to be longer than the true value by Δ*l* ($0 < \Delta l < r_r$), as shown in Figure B.2 b).



a) Ideal narrow-ridge structure

b) Practical narrow-ridge structure with edge radii

Figure B.2

B.3 Multiple-trench structure

The multiple-trench structure shall contain several trenches with different trench widths. The minimum trench width shall be in the range from 3 nm to 50 nm, and the maximum trench width shall be in the range from 20 nm to 200 nm. The edge sharpness of each trench directly influences the accuracy of the probe characterization. It is reasonable to use different sharpness values for the edge of each trench part, depending on the gap dimension of the trench part. A typical value requested for the edge radius is smaller than either 8 nm or 10 % of the gap width in each trench part. Each pair of *l* and *w* values for the EPSC can be determined from one of the trenches, provided that the probe apex does not reach the base of the trench. A valid measurement is shown in Figure B.3. The minimum trench depth shall be 20 nm, or the maximum probe profile length to be determined

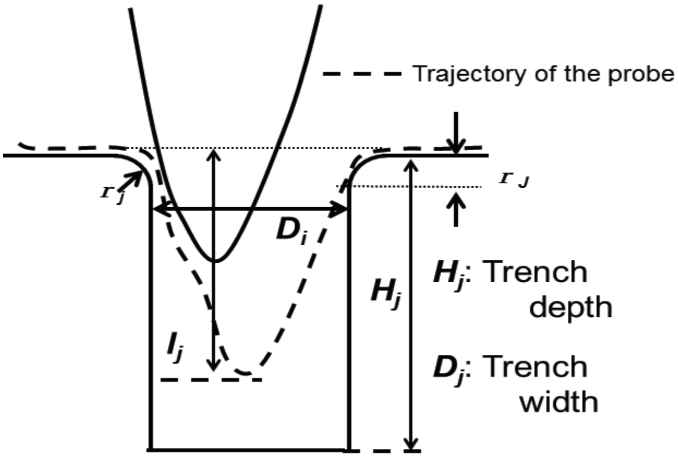


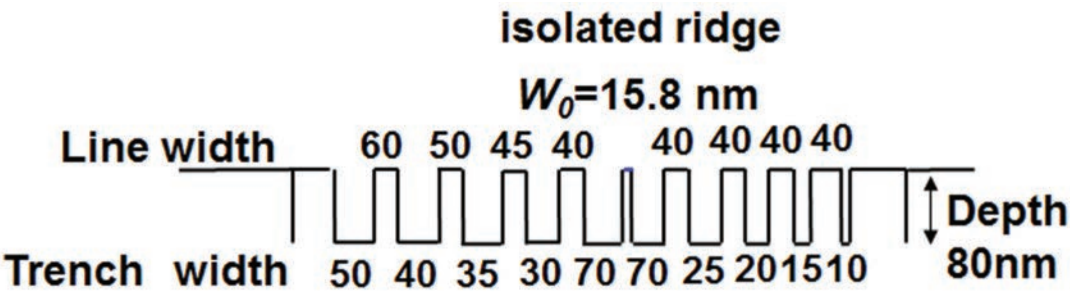
Figure B.3 — Typical trench structure

Annex C
(informative)

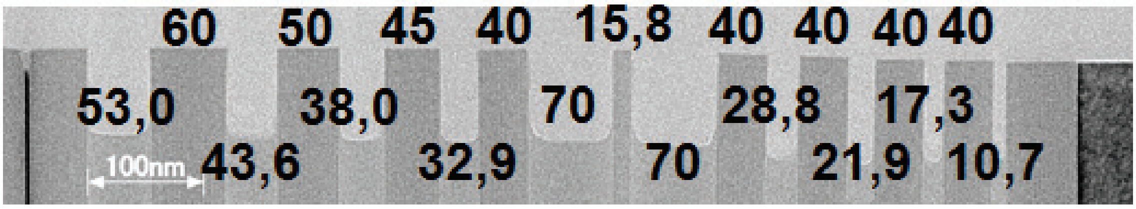
Example of a reference structure

C.1 General

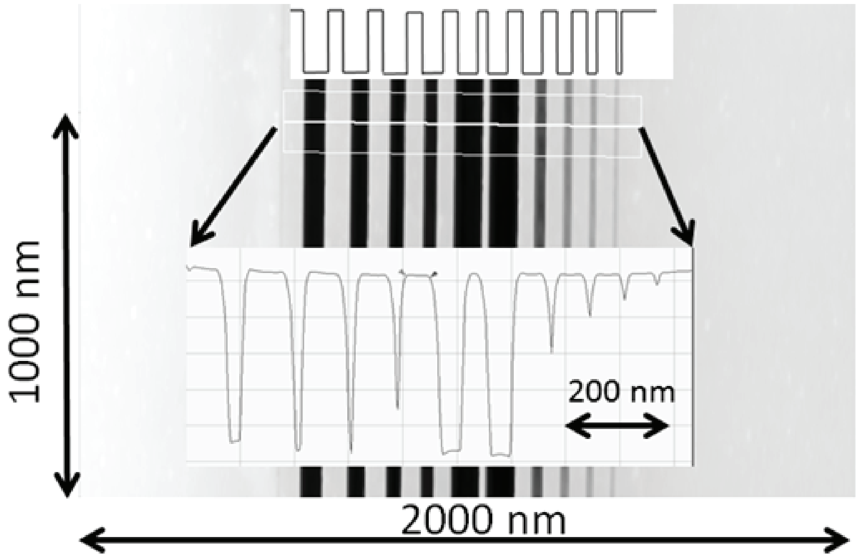
Details of the structure of a fabricated reference sample are shown in Figure C.1.[7][8] This includes trench structures with eight different widths and a (15,8 ± 1,0) nm wide ridge. The angles of the side walls from the top surface are all 90° ± 1°. The edge radius at the upper corners is less than 2 nm as shown in Figure C.1 b). An example of an AFM image and line profile obtained from this reference sample are shown in Figure C.1 c).



a) Designed structure of a reference sample



b) Cross-sectional TEM image of the reference sample



c) AFM image and line profile of the reference sample

Figure C.1

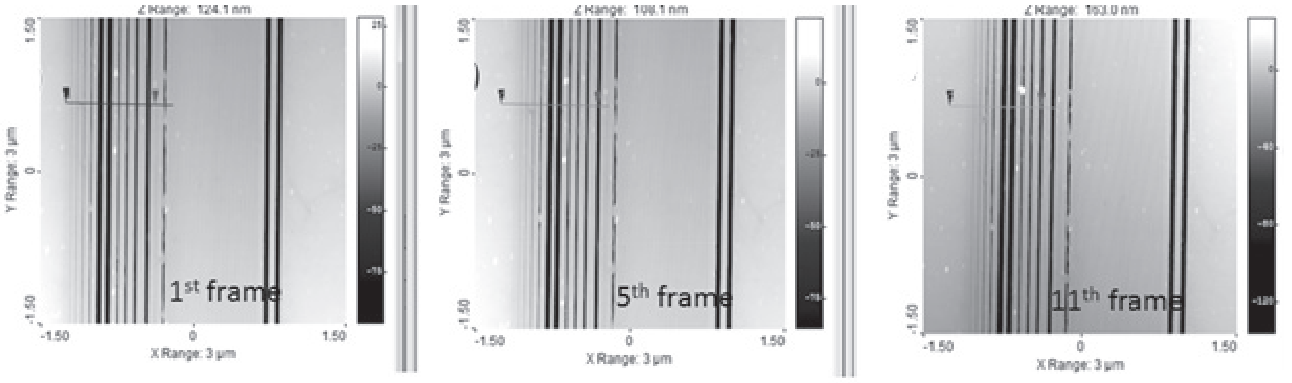
Annex D
(informative)

Results of EPSC measurement repeatability test

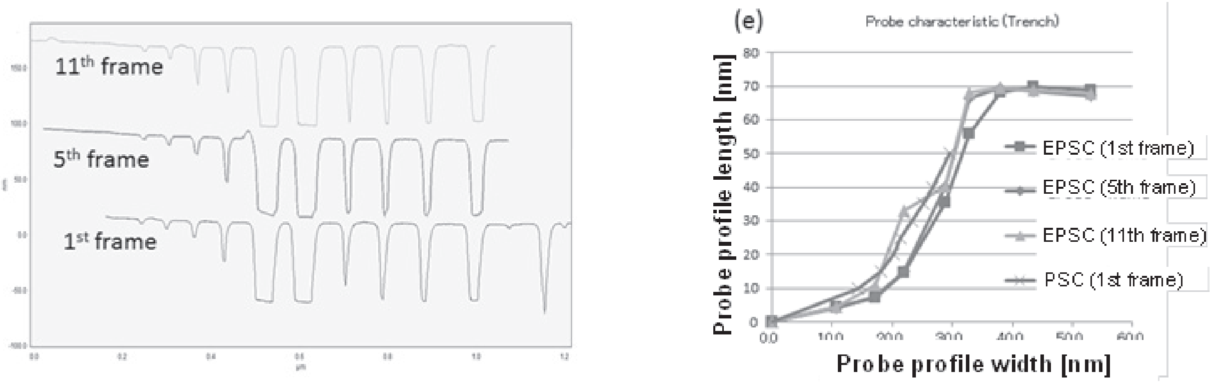
D.1 Repeatability of EPSC measurements

AFM images illustrating the repeatability of the EPSC measurement using a multiple-trench structure are shown in Figure D.1. The reference sample used in this experiment is the same as that shown in Figure C.1 a). The field of view of the AFM image is 3 µm × 3 µm, and raster lines were at 512 lines/frame. These images were obtained in peak-force mode using a Dimension ICON (Bruker)[1)] AFM with a ScanAsyst-Air[1)] cantilever with a setpoint around 1 nN. For every image, 2 048 pixels and 512 raster lines were obtained, and the scan rate was 0,3 Hz. Automatic controlling mode of feedback gain was used in the experiment. Three AFM images from the experiment are shown in Figure D.1 a), Figure D.1 b), and Figure D.1 c). The EPSC was evaluated using the line profiles shown in Figure D.1 d), and the resulting plot is shown in Figure D.1e). No significant degradation in the data was observed after 5 632 raster lines. The EPSC also became sharper after the 11th scan, possibly because the setpoint had risen due to thermal drift. Enlarged portions of the AFM images in Figure D.1 a), Figure D.1 b), and Figure D.1 c) are shown in Figure D.2 for clarity.

1) Dimension ICON (Bruker)and ScanAsyst-Air are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.



a) AFM image for the 1st scan b) AFM image for the 5th scan c) AFM image for the 11th scan



d) Line traces for the images in a), b), and c) as indicated

e) EPSC for the traces in d) for the 8 trenches

NOTE 3 × 3 µm, 2 048 pixels × 512 lines (Total scan lines: 5 632 lines)

Figure D.1

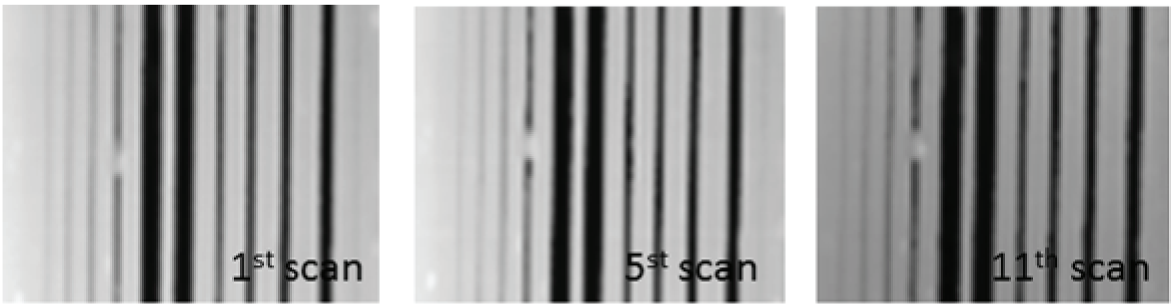


Figure D.2 — Magnified images of Figure D1 a), Figure D.1 b), and Figure D.1c)

Annex E
(informative)

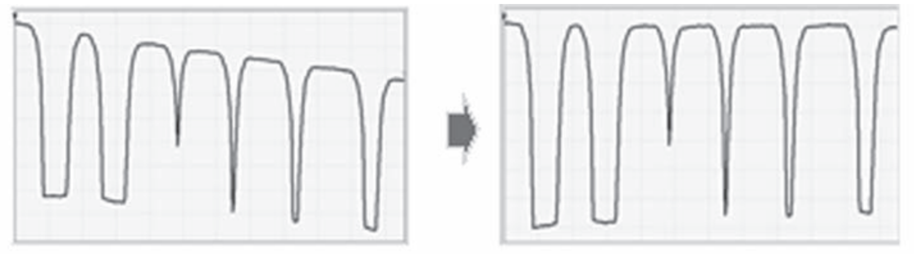
Plane correction for probe shank profile analysis

When the line profile of the AFM image is tilted, the surface level shall be corrected so that the surface plane is as level as possible, as shown in Figure E.1 a). If the surface plane is unlevel due to the vertical bow of the scanner or to non-flat features in the reference sample, local correction of the plane for each structure is recommended for data analysis, as shown in Figure E.1 b). The plane correction procedure is as follows.

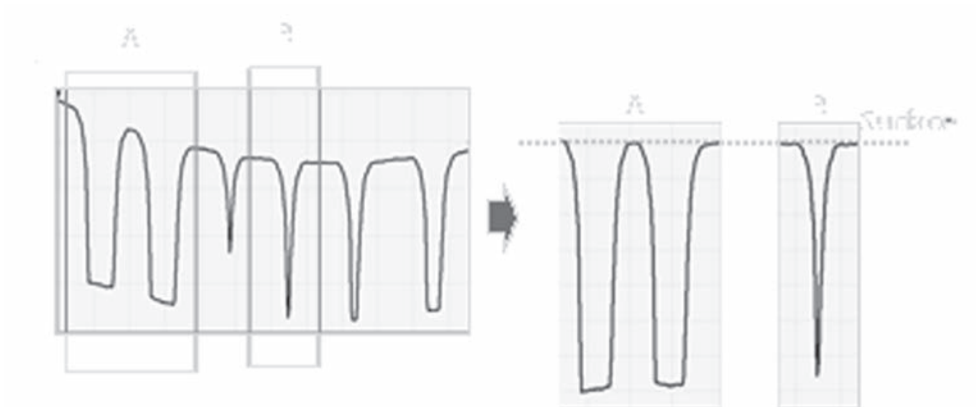
a) Display the profile of the reference sample.

b) The top level of the profile shall be horizontally level (i.e. not sloping). Correction to achieve a level surface can be adjusted relative to individual features, such as a single trench, or to the whole structure.

NOTE If the inclined angle of the top of the surface level is small, such as 0,2°, then the surface is nearly flat locally. However, for a 1 000 nm-long structure with a 0,2° tilt angle, the difference in height between the two ends of the structure's profile would be 3,5 nm.



a) Plane correction for all structures



b) Local plane correction

Figure E.1 — Plane correction for the analysis

Annex F
(informative)

Example of a report

Table F.1 — Report of AFM probe shapes

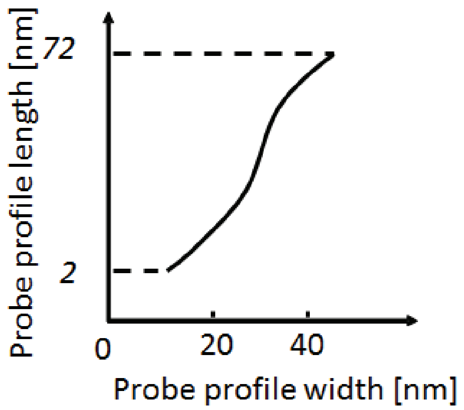
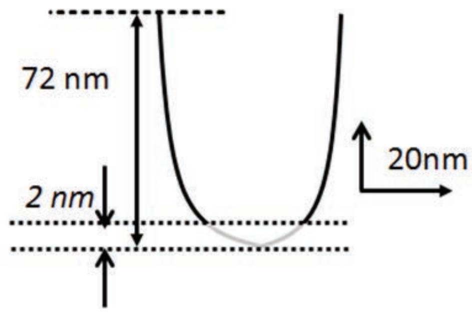
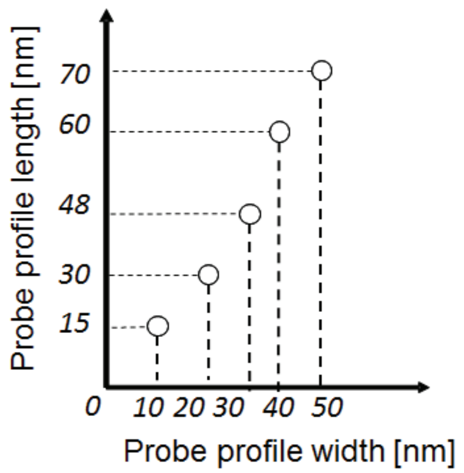
<table>
<tr><td>Probe shape characteristic (PSC) measured by narrow-ridge method (5.4.2)

</td><td>Projected probe profile (PPP) measured by narrow-ridge structure method (5.4.2)

</td></tr>
<tr><td>Effective probe shape characteristic (EPSC) measured by multiple-trench method (5.4.3)

</td><td>Specifications of the reference sample
Ridge width: L_0 = 15,8 nm, u_0 = 0,5 nm, D_0 = 100 nm, H_0 = 80 nm, r_r = 2 nm
Trench width 1, 2, 3, 4, 5: 10 nm, 20 nm, 30 nm, 40 nm, 50 nm
Trench depth 1–5: 80 nm
u_t = 1,5 nm, r_t = 2,0 nm, r_j = 2,0 nm (j = 1–5)</td></tr>
<tr><td>Measurement mode of an AFM instrument</td><td>Intermittent contact mode, amplitude control, hopping mode</td></tr>
<tr><td>Cantilever used in the measurement (cantilever, resonance frequency, quality factor, and deflection sensitivity)</td><td>Cantilever type A-1
(321 KHz, 450, 90 nm/V)</td></tr>
<tr><td>Free oscillation amplitude</td><td>100 mV (28 nm p-p)</td></tr>
<tr><td>Setpoint</td><td>300 mV p-p (27 nm p-p)</td></tr>
<tr><td>PID controlling parameters for the probe</td><td>Proportional gain = 8, Integral gain = 3,5</td></tr>
<tr><td>Line scanning rate</td><td>0,15 Hz</td></tr>
<tr><td>Numbers of pixels and lines</td><td>Fast raster: 2 048 pixels, Raster lines: 32 lines</td></tr>
<tr><td>Temperature, humidity</td><td>25°C, 35 %</td></tr>
</table>

Table F.1 *(continued)*

AFM model	AFM system 1
Origins of errors (corner radius of the ridge structure r_r in the reference material, line width uncertainty of the narrow-ridge structure u_0, and maximum error signal e_m)	r_r = 2,0 nm, u_0 = 1,0 nm, e_m = 1,0 nm
Combined standard uncertainty, u, of the narrow-ridge method	2,4 nm

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