INTERNATIONAL STANDARD

ISO 11952

Second edition
2019-05

Surface chemical analysis — Scanning-probe microscopy — Determination of geometric quantities using SPM: Calibration of measuring systems

Analyse chimique des surfaces — Microscopie à sonde à balayage — Détermination des quantités géométriques en utilisant des microscopes à sonde à balayage: Étalonnage des systèmes de mesure

Reference number
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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

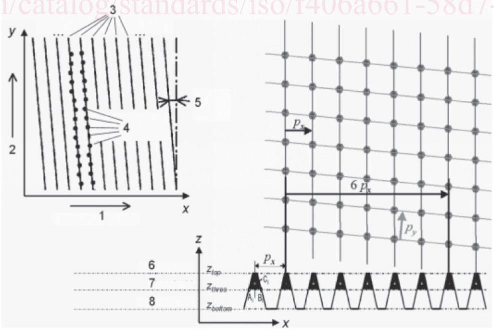
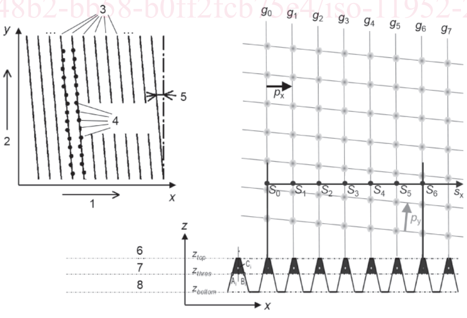
Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

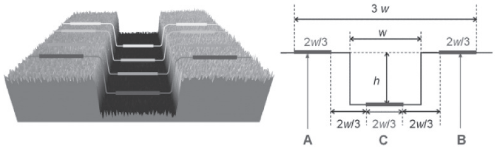
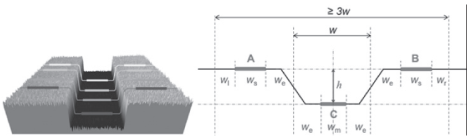
For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 11952:2014), of which it constitutes a minor revision. The changes to the previous edition are as follows:

		Previous edition	Revised edition
Figure 1		“interferometery”	“interferometry”
Figure 1	Note	“The calibration of a user’s SPM by means of traceably calibrated measurement standards is the object of this International Standard (done by the user).”	“The calibration of a user’s SPM by means of traceable calibrated measurement standards is the object of this document (done by the user).”
Clause 4	N_i	“N_i”	“N_{ij}”
Clause 4	*r*	“tip”	“tip radius”
Clause 4	α_m	“thermal expansion coefficient”	“thermal expansion coefficient of the specimen”
Clause 4	*ytx*	“positional deviation Δx measured along a y-coordinate line”	“straightness deviation Δx measured along a y-coordinate line”
Clause 4	*yty*	“straightness deviation Δy measured along a y-coordinate line”	“positional deviation Δy measured along a y-coordinate line”
Clause 4	*ztz*	“straightness deviation Δz measured along a z-coordinate line”	“positional deviation Δz measured along a z-coordinate line”
6.3.1	Title	“Kinds of external influence”	“Sources of external influences”
6.3.1	First sentence	“As SPMs are most sensitive to interference from the environment, the following quantities are to be accounted for”:	“As SPMs are very sensitive to interference from the environment, the influences of the following quantities need to be determined”:

		Previous edition	Revised edition
6.3.1	Fourth bullet	“mechanical vibrations (e.g. structural vibrations, foot fall sounds/human traffic, pumps)”;	“mechanical vibrations (e.g. structural vibrations, human traffic, pumps)”;
6.4	a) and b)	a) For measurement b) For installation or familiarization of the staff	a) and b) switched
7.3.4	Second bullet	“Adjust the z-position of the scanner in such a way that the z-scanner operates symmetrically around the central position in the z-deflection range (see also Figure 18).”	“Adjust the z-position of the scanner in such a way that the z-scanner operates symmetrically around the central position in the z-deflection range as illustrated in Figure 17 around its medium (central) deflection, i.e. 50 % of its range”
7.3.7	Fourth bullet	“adjust the z-position of the scanner in such a way that the z-scanner operates, e.g. by 20% (see also Figure 20) above or below the central position in the z-deflection range (see also Figure 18 and 20).”	“adjust the z-position of the scanner in such a way that the z-scanner operates above or below the central position in the z-deflection range, i.e. symmetrically around 10 %, 30 %, 70 % and 90 % (as illustrated in Figure 17), in addition to the basic z calibration performed around 50 % deflection”
Figure 9	Title	“Flow diagram of calibration of the lateral axes[35]“	“Calibration of the lateral axes: materials, steps and methods[35]”
7.4.4	Seventh paragraph	“needs to” “great”	“should” “large”
7.4.4	Eighth paragraph	“(relatively feeble)”	“(low)”
7.4.5	2)	“(see also Figure 18)”	“as illustrated in Figure 15 and shown for the medium (central) deflection case in Figure 17.”
7.4.7	Figure 10		
7.4.7	4) and Note 1	“Appurtenant”	“relevant”
7.4.8	Second bullet	“In good gratings, the mean values of the pitches of all the straight lines are a good approximation and should be used for further evaluation. If this is not the case, the parallelism of the straight lines is to be forced by fitting as above.”	“In good gratings, the fit lines g_0 to g_n are nearly parallel so that the mean value of the gradients of all these straight lines is a good approximation and should be used for further evaluation. If this is not the case, the parallelism of the straight lines is to be forced by fitting as above.”
7.4.8	Eighth bullet	“(example in Figure 15)”	“(the example in Figure 12 shows a polynomial fit of the third degree)”

		Previous edition	Revised edition
7.4.8	Note 2	“In the case of clear deviations of the specimen temperature (e.g. in deep-temperature applications) from the reference temperature 20 °C in particular, for which the calibration of the measurement standard is valid, the thermal expansion is to be accounted for.”	“The certified pitch values of a transfer standard are valid for a certain reference temperature, typically 20 °C. In case of significant deviations of the sample temperature from the reference temperature (e.g. in low-temperature chambers or if the sample is heated in the particular setup), the material-dependent thermal expansion is to be taken into account.”
7.5.7.2.1	Second paragraph	“For the one straight line — besides the parallelism requirement — the determination uses only an area C in the middle of the indention or elevation whose width can be selected by the user; it is usual to select one (according to ISO 5436 1) to two-thirds (Figure 18) of the total width w of the indention/elevation.”	“For the one straight line — besides the parallelism requirement — the determination uses only the section C in the middle of the indention or elevation whose width w_m can be selected by the user; it is usual to select one (according to ISO 5436-1) to two-thirds (like in the example shown on the left of Figure 18) of the total width w (defined as full-width at half maximum) of the indention/elevation.”
7.5.7.2.1	Third paragraph	“Taking account of the parallelism requirement, the second straight is selected through two areas A and B which lie symmetrically about the indention/elevation and usually show the same width as C. The distance of A”	“Taking account of the parallelism requirement, the second straight is selected through two sections A and B which lie symmetrically about the indention/elevation. The lengths w_s in sections A and B are identical, but might be different from w_m. The sections A and B should not start/end with the beginning/end of the profile (scanline), as irregularities in height measurement are to be expected especially at the beginning/end of scanlines. A spacing w_l to the left of section A and w_r to the right of section B should be allowed for. As a general rule, the total length of the measured profile should be at least 3*w*. The distance we of A”
7.5.7.2.1	Fourth paragraph	“As to the mathematics, the determination of the step height, *h*, is reduced to the calculation of only one regression line by appropriately shifting the points area by area by +*h*/2 and −*h*/2, respectively.”	“As to the mathematics, the determination of the step height, *h*, is reduced to the calculation of only one regression line by least squares approximation with h being the fit variable. This reduction to only one regression line is achieved by introducing a vertical shift of the data points in the sections A, B and C according to the following rules”:
7.5.7	Figure 18 title		

		Previous edition	**Revised edition**
		“Step height determination according to ISO 5436-1 (left: step height measurement standard 6 nm)”	“Step height determination according to ISO 5436-1 (left: example of a step height measurement standard 6 nm, right: profile section C of length w_m and profile sections A and B each of length w_s taken into account for step height analysis, spacings of lengths w_e from the edge and of lengths w_l to the left of section A and w_r to the right of section B)”
Table C.1	Sum	“+1,49”	“+1,50”
Annex E	Fifth paragraph	“The bars in Figure E.1 give the uncertainty”	“The bars in Figure E.1 give the standard uncertainty”
Bibliography	[16]	DZIOMBA T., KOENDERS L., WILKENING G., FLEMMING M., DUPARRÉ A. Entwicklung einer Kalibrierrichtlinie für Rastersondenmikroskope; tm — Technisches Messen 72 (2005) 5, S. 295–307; siehe auch http://www.tm-messen.de/	KLAPETEK P., Quantitative data processing in scanning probe microscopy. Elsevier, Amsterdam, The Netherlands, ISBN: 978–0–12–813347–7, 2018
Bibliography	[26]	Koenders, L.; Dziomba, T.; Thomsen-Schmidt, P.; Senoner M.: Normale für die dimensionelle und analytische Nanometrologie. PTB-Mitteilungen 114 (2004) 1, S. 16–24	Yacoot A., Koenders L. Recent developments in dimensional metrology using AFMs. *Meas. Sci. Technol.* 2011, 22, p. 12201
Bibliography	[39]	ZHAO X. PTB-report F-20. Wirtschaftsverlag NW, Bremerhaven, Germany, 1995	Zhao X. “Scanning Probe Microscope with high resolution capacitive transducers”, PTB-report F-32, Wirtschaftsverlag NW — Verlag für neue Wissenschaft GmbH, Bremerhaven, www-nw-verlag.de ISBN 3-89701-207–3, 1998, 158 pages

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

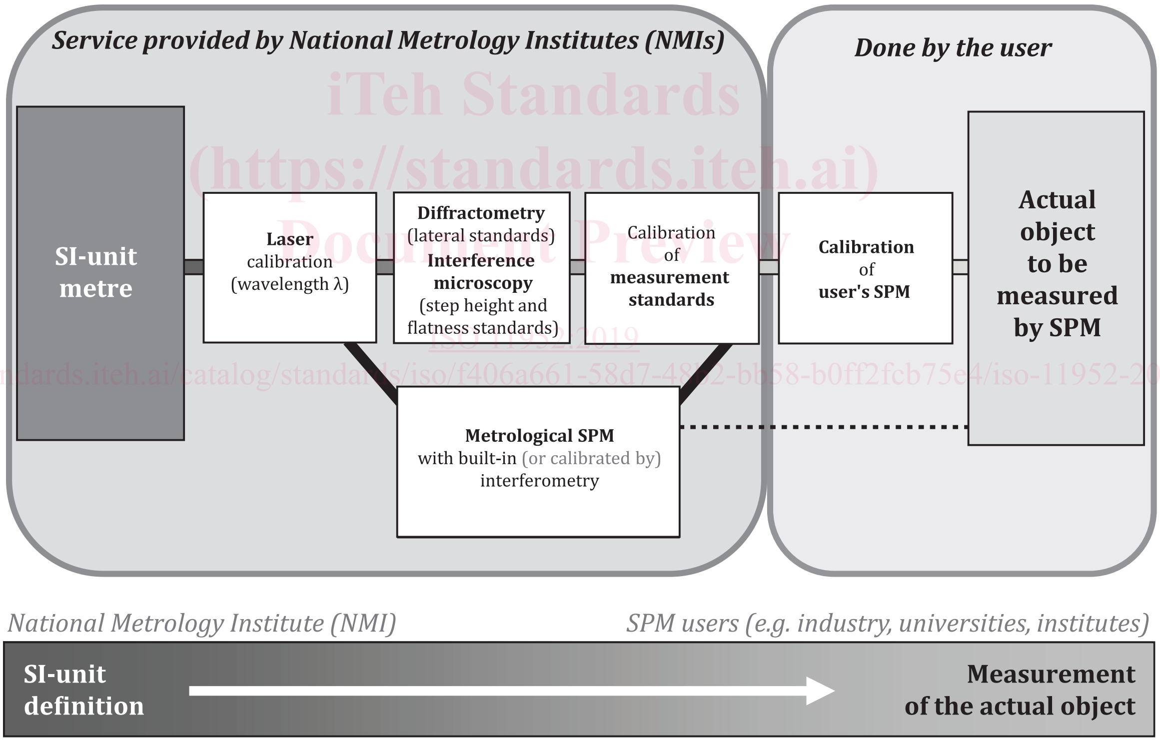
Introduction

The progress of miniaturization in semiconductor structuring, together with the rapid advance of many diverse applications of nanotechnology in industrial processes, calls for reliable and comparable quantitative dimensional measurements in the micro- and submicrometre range[9]. Currently, a measurement resolution, in or below the nanometre region, is frequently required. Conventional optical or stylus measurement methods or coordinate measuring systems are not able to offer this level of resolution.

For this reason, scanning-probe microscopes (SPMs) are increasingly employed as quantitative measuring instruments. Their use is no longer confined only to research and development, but has been extended to include industrial production and inspection.

For this category of measuring instrument, standardized calibration procedures need to be developed, as have already been established, for example, for contact stylus instruments (see ISO 12179). For efficient and reliable calibration of SPMs to be carried out, the properties of the measurement standards used need to be documented and accounted for in the calibration (see Figure 1). At the same time, the procedure for the calibration should be clearly defined.

Only if this prerequisite is satisfied will it be possible to perform traceable measurements of geometrical quantities.



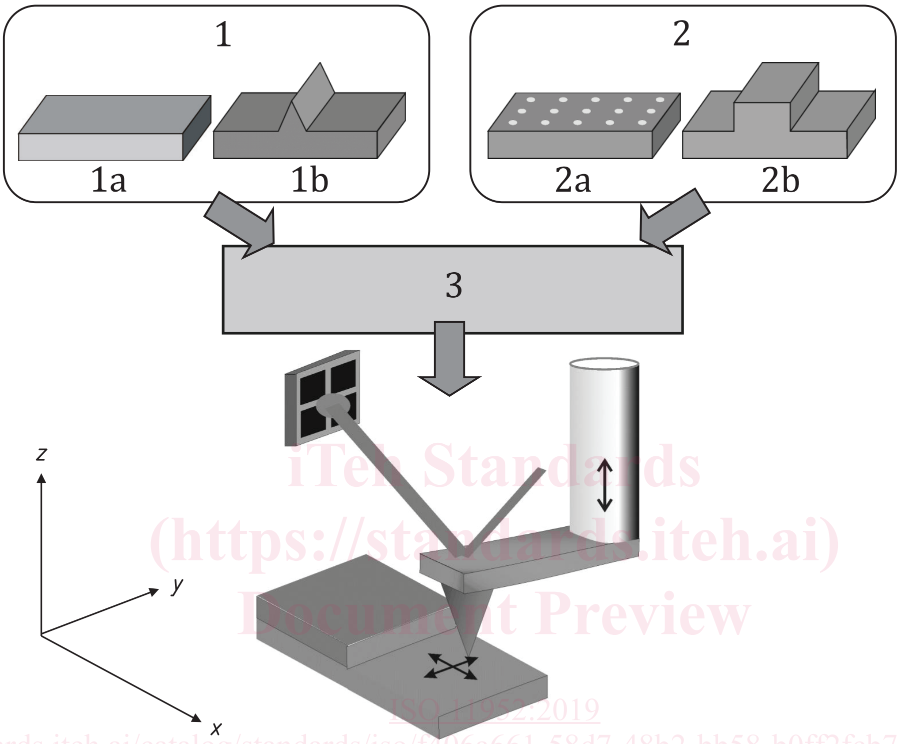
NOTE The calibration of a user's SPM by means of traceable calibrated measurement standards is the object of this document (done by the user).

Figure 1 — Traceability chain for SPMs

An SPM is a serially operating measuring device which uses a probe with a tip of adequate fineness to trace the surface of the object to be measured by exploitation of a local physical interaction (such as the quantum-mechanical tunnel effect, interatomic or intermolecular forces, or evanescent modes of the electromagnetic field). The probe and the object to be measured are being displaced in relation to one another in a plane (hereinafter referred to as the *x*-*y*-plane) according to a defined pattern[10], while the signal of the interaction is recorded and can be used to control the distance between probe and object. In this document, signals are considered which are used for the determination of the topography (hereinafter called the "*z*-signal").

This document covers the verification of the device characteristics necessary for the measurement of geometrical measurands and the calibration of the axes of motion (*x*, *y*, *z*)[11], i.e. the traceability to the unit of length via measurement on traceable lateral, step height and 3D measurement standards (see Figure 2).

While this document aims at axis calibrations at the highest level and is thereby intended primarily for high-stability SPMs, a lower level of calibration might be required for general industry use.



Key

1	measurement standards for verification purposes
1a	flatness
1b	probe shape
2	measurement standards for calibration purposes
2a	1D and 2D lateral
2b	step height
3	calibration of the measurement standards by reference instruments (certified calibration, measurement value including uncertainty)

Figure 2 — Verification and calibration of SPMs with test specimens and measurement standards

This document is mainly based on the guideline VDI/VDE 2656, Part 1, drafted by a guideline committee of the VDI (Verein Deutscher Ingenieure/Association of German Engineers) from 2004 to 2008, with the final whiteprint of that guideline being released in June 2008.

Surface chemical analysis — Scanning-probe microscopy — Determination of geometric quantities using SPM: Calibration of measuring systems

1 Scope

This document specifies methods for characterizing and calibrating the scan axes of scanning-probe microscopes (SPMs) for measuring geometric quantities at the highest level. It is applicable to those providing further calibrations and is not intended for general industry use, where a lower level of calibration might be required.

This document has the following objectives:

- to increase the comparability of measurements of geometrical quantities made using SPMs by traceability to the unit of length;
- to define the minimum requirements for the calibration process and the conditions of acceptance;
- to ascertain the instrument's ability to be calibrated (assignment of a “calibrate-ability” category to the instrument);
- to define the scope of the calibration (conditions of measurement and environments, ranges of measurement, temporal stability, transferability);
- to provide a model, in accordance with ISO/IEC Guide 98-3, to calculate the uncertainty for simple geometrical quantities in measurements using an SPM;
- to define the requirements for reporting results.

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 11039, *Surface chemical analysis — Scanning-probe microscopy — Measurement of drift rate*

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

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

IEC/TS 62622, *Artificial gratings used in nanotechnology — Description and measurement of dimensional quality parameters*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-2 and IEC/TS 62622 and the following 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
scanner bow
additional deflection in the *z*-direction when the scanner is displaced in the *x*-*y*-direction

Note 1 to entry: Scanner bow is also known as out-of-plane motion (see also *xtz*, *ytz* in Clause 4).

3.2
look-up table
table in which a set of correction factors for the scanner are filed for different modes of operation (e.g. scan ranges, scan speeds, deflections)

3.3
step height
height of an elevation (bar) or depth of a groove (see ISO 5436-1); on atomic surfaces, the distance between neighbouring crystalline planes

3.4
levelling
correction of the inclination between the ideal *x*-*y*-specimen plane and the *x*-*y*-scanning plane

4 Symbols

x, y, z	position value related to the respective axis
C_x, C_y, C_z	calibration factors for the *x*-, *y*-, and *z*-axes
h	step height
w	width of a structure of the specimen
N_{ij}	*i*th pitch value in a profile used for the determination of the pitch/period (number of pitch values *i* over all lines $j = 1,\ldots, Nj$)
p_x	pitch or period in the *x*-direction
p_y	pitch or period in the *y*-direction
a_x	vector in the *x*-direction of a grating (not to be confused with p_x)
a_y	vector in the *y*-direction of a grating (not to be confused with p_y)
γ_{xy}	non-orthogonality of 2D gratings
P-V	peak-to-valley value
r	tip radius
Rq (Sq)	root mean square deviation of the assessed roughness profile (Rq) or of the assessed area (Sq)
T	temperature
α_m	thermal expansion coefficient of the specimen
T_L	temperature of the air
T_m	temperature of the specimen during measurement
j_x	angle of rotation about the *x*-axis

j_y	angle of rotation about the y-axis
j_z	angle of rotation about the z-axis
θ	levelling angle
x_L	value of the measurement standard for shift in the x-direction
x_m	shift in the x-direction measured with the x-displacement transducer
xtx	positional deviation Δx measured along an x-coordinate line
xty	straightness deviation Δy measured along an x-coordinate line
xtz	straightness deviation Δz measured along an x-coordinate line
xrx	rotational deviation j_x measured along an x-coordinate line
xry	rotational deviation j_y measured along an x-coordinate line
xrz	rotational deviation j_z measured along an x-coordinate line
xwy	measured rectangularity deviation in the coordinate plane x-y
xwz	measured rectangularity deviation in the coordinate plane x-z
y_L	value of the measurement standard for displacement in the y-direction
y_m	displacement measured with the y-displacement transducer in the y-direction
ytx	straightness deviation Δx measured along a y-coordinate line
yty	positional deviation Δy measured along a y-coordinate line
ytz	straightness deviation Δz measured along a y-coordinate line
yrx	rotational deviation j_x measured along a y-coordinate line
yry	rotational deviation j_y measured along a y-coordinate line
yrz	rotational deviation j_z measured along a y-coordinate line
ywz	rectangularity deviation measured in the coordinate plane y-z
z_L	value of the measurement standard for displacement in the z-direction
z_m	displacement in the z-direction measured with z-displacement transducer
ztx	straightness deviation Δx measured along a z-coordinate line
zty	straightness deviation Δy measured along a z-coordinate line
ztz	positional deviation Δz measured along a z-coordinate line
zrx	rotational deviation j_x measured along a z-coordinate line
zry	rotational deviation j_y measured along a z-coordinate line
zrz	rotational deviation j_z measured along a z-coordinate line
$\cos(\varphi_\mathrm{i})$	rotational correction, for example in pitch measurement

$\cos(\theta_\mathrm{i})$	tilt-related correction, for example in pitch measurement
λ_s	short-wavelength filter (see ISO 4287 for details)
λ_c	long-wavelength filter (see ISO 4287 for details)
Λ	correlation length
ϕ_{xy}	angle between the x- and y-direction, counterclockwise
ϕ_{xz}	angle between the x- and z-direction, counterclockwise
ϕ_{yz}	angle between the y- and z-direction, counterclockwise
Rqx	noise in the x-direction
Rqy	noise in the y-direction
Rqz (Sqz)	noise in the z-direction in a measured profile (or within a measured area)
v	scan speed (i.e. distance travelled by the probe tip per unit time, not to be confused with the scan rate, i.e. the number of scanlines recorded per unit time)

5 Characteristics of SPMs

5.1 Components of an SPM