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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

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Contents

Page

Foreword

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

Introduction

The progress of miniaturization in semiconductor structuring, just as the fast advance of the extremely versatile nanotechnology applications, in a great number of industrial processes calls for reliable and comparable quantitative dimensional measurements in the micro- and submicrometre range [1]. By now resolutions in or even below the nanometre region are already frequently required, i.e. resolutions conventional optical or stylus measurement methods or coordinate measuring systems are not able to offer.

This is why scanning probe microscopes (SPM) are increasingly employed as quantitative measuring instruments, their use being no longer confined only to research and development but increasingly extended also to industrial production and inspection.

For this category of measuring instruments, too, standardized calibration procedures therefore need to be developed as have, for example, been established already long ago for contact stylus instruments (ISO 12179). In many cases, specifically developed measurement standards are used here. For calibrations of SPMs to be carried out not only reliably but also efficiently, the properties of the standards used should have been documented and be accounted for in the calibration and, at the same time, the procedure for the calibration should be clearly defined.

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

This standard is mainly based on the guideline VDI/VDE 2656 – Part 1 drafted by a guideline committee of VDI (Verein Deutscher Ingenieure, Association of German Engineers) in the years 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 standard is restricted to scanning probe microscopes and their ***characterization*** and ***dimensional calibration***. A scanning probe microscope 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, 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 [2], while the signal of the interaction is recorded and can be used to control the distance between probe and object. In this standard signals are considered which are used for the determination of the topography (hereinafter called "*z*-signal").

This ISO standard 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*), i.e. the traceability to the unit of length via measurement on traceable lateral, step height and 3D standards (figure 1). Such ***characterization*** and ***calibration*** of the SPM (figure 2) is a prerequisite for its use for dimensional measurements e. g. in industry [3].

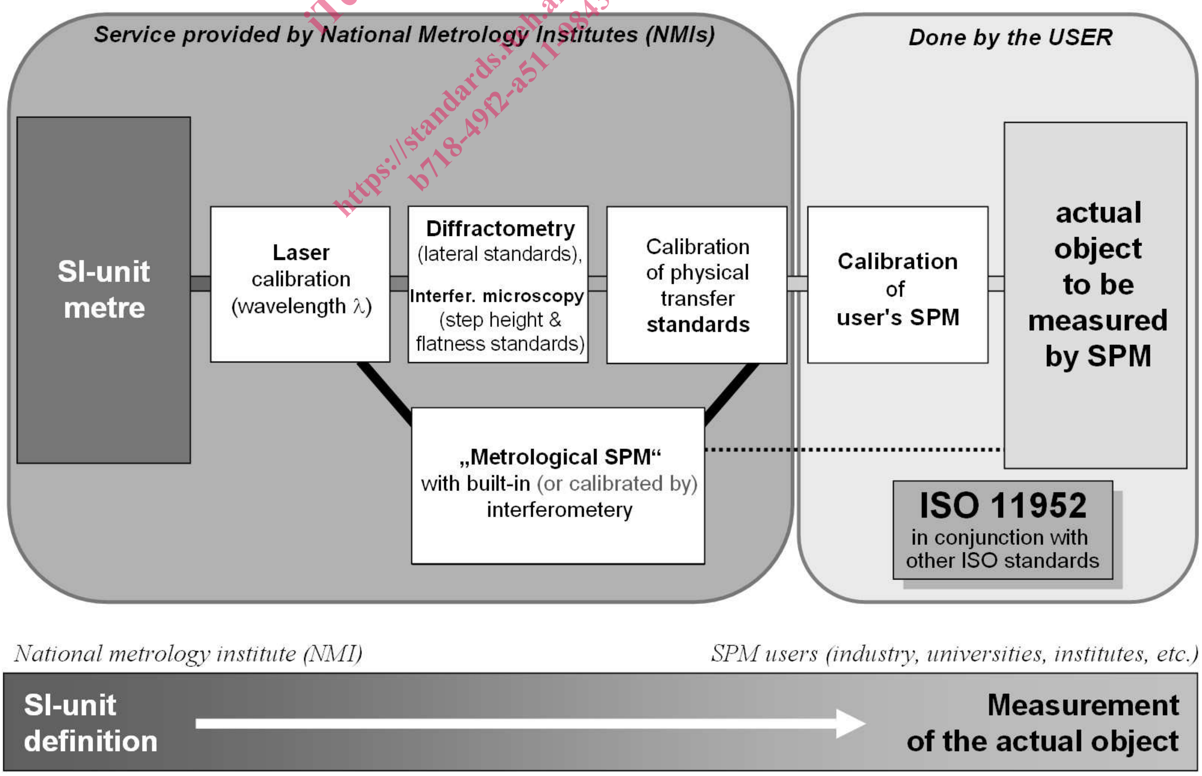

Figure 1: Traceability chain for scanning probe microscopes:
The calibration of user's SPM by means of traceably calibrated standards is the object of this standard ("*done by the user*")

With the implementation of this standard the following objectives are pursued:

- increase in the comparability of measurements of geometrical quantities using scanning probe microscopes by traceability to the unit of length
- definition of minimum requirements for the calibration process and the conditions of acceptance
- ascertainment of the calibratability (assignment to calibratability categories)
- fixing of the scope of a calibration (conditions of measurement and environments, ranges of measurement, temporal stability, transferability)
- provision of a model according to GUM to calculate the uncertainty for simple geometrical quantities in measurements using a scanning probe microscope
- definition of the requirements for a result report

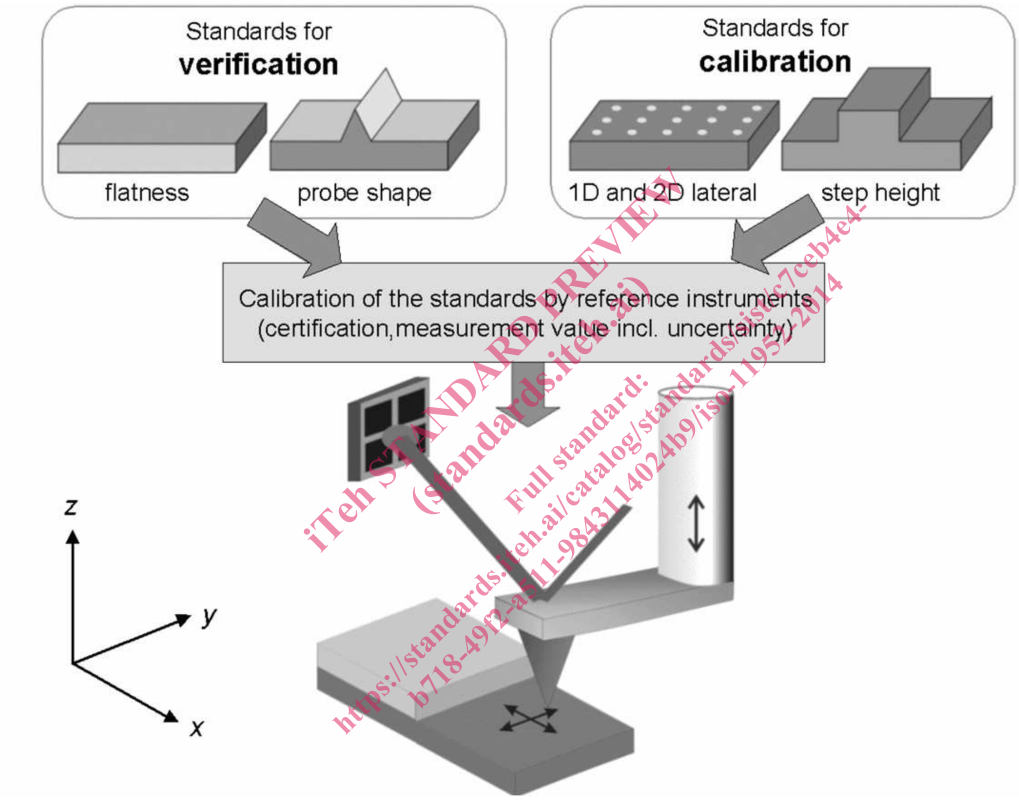

Figure 2: Verification and calibration of scanning probe microscopes with samples and standards

2 Normative references, terms and definitions

2.1 Normative references

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.

- ISO 18115-2 Surface chemical analysis – Vocabulary – Part 2 Terms used in scanning probe microscopy (including amendments)
- ISO Guide 30: 1992, Terms and definitions used in connection with reference materials
- ISO Guide 34: 1996, Quality system guidelines for the production of reference materials

- ISO 11039 Surface chemical analysis — Scanning probe microscopy — Definition and measurement methods of drift rates of SPM

The following documents are not mandatory for the basic dimensional calibration of SPM, but may prove indispensable depending on the intended applications of the SPM:

- ISO 11775 Surface chemical analysis — Scanning probe microscopy — Determination of cantilever normal spring constants
- ISO 11939 Surface chemical analysis — Scanning probe microscopy — Standards on the measurement of angle between an AFM tip and surface and its certified reference material
- ISO 13095 Surface chemical analysis — Scanning probe microscopy — procedure for in-situ characterization of AFM probes used for nanostructure measurements
- ISO 13096 Surface chemical analysis — Scanning probe microscopy — Guide to describe AFM probe properties
- ISO/IEC TS 13126 Nanotechnologies — Artificial gratings used in nanotechnology — description and measurement of dimensional quality parameters

Furthermore, in the field of surface measurement, a corpus of standards is already available for contact stylus instruments. So definitions and terms according to these standards are used here especially for:

- definitions of device components: ISO 3274
- calibration standards: ISO 5436-1
- definition of surface (profile) characteristics: ISO 4287
- conditions of measurement and evaluation: ISO 4288
- definitions of properties of general-purpose microscopes: ISO 12853
- calibration of contact stylus instruments: ISO 12179

2.2 Terms used

In addition to acronyms and terms defined in ISO 18115 and the other standards mentioned above, the following terms are used:

scanning sample	During scanning, the sample is displaced in the *x*-*y*-direction and, if need be, tracked in the *z*-direction. The probe acts as a zero detector.
scanning probe	During scanning, the probe is displaced in the *x*-*y*-direction and tracked in the *z*-direction of the surface.
scanner bow or out-of-plane motion	When displaced in the *x*-*y*-direction, the scanner is accidentally deflected in the *z*-direction (see also *xtz*, *ytz*).
look-up table	table(s) in which correction factors for the scanner for different modes of operation (ranges, speeds, deflections...) are filed
open loop	position values for the *x*- and *y*-axis are not used for position control
closed loop	For scanning, the position values measured by a displacement transducer are used to track the *x*- and *y*-axis via a control to scheduled position values.
position	centre of gravity of a structure or of a feature in coordinates of the device
step height	height of an elevation or depth of a groove (ISO 5436-1), in atomic surfaces the distance between neighbouring crystalline planes
levelling	correction of the inclination between ideal *x*-*y*-sample plane and *x*-*y*-scanning plane
distance	distance of two positions on the surface; it is defined as the distance of the centres of gravity of the respective structures unless otherwise stated
pitch	mean distance of similar structural features of the surface (period)

3 Symbols

x, y, z	position value related to the respective axis
C_x, C_y, C_z	calibration factor for the x-, y- and z-axis
h	step height
w	width of a structure of the sample
N_j	number of pitch values i over all lines $j = 1,..., Nj$
(o, x_R, y_R, z_R)	rectangular coordinate system with the coordinate origin o and the coordinate axes x, y and z (abscissa, ordinate and applicate axes)
p_x	pitch or period in the x-direction
p_y	pitch or period in the y-direction
a_x	vector in x-direction of a grating (not to be confounded with p_x)
a_y	vector in y-direction of a grating (not to be confounded with p_y)
γ_{xy}	non-orthogonality of 2D gratings
P-V	peak-to-valley value
r	radius
Rq (Sq)	root mean square deviation of the assessed roughness profile (Rq, so-called rms value) or of the assessed area (Sq)
T	temperature
α_m	thermal expansion coefficient
ϑ	temperature of air
T_m	temperature of sample in the measurement
φ_x	angle of rotation about x-axis
φ_y	angle of rotation about y-axis
φ_z	angle of rotation about z-axis
Θ	levelling angle
x_L	value of the 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 φ_x measured along an x-coordinate line
xry	rotational deviation φ_y measured along an x-coordinate line
xrz	rotational deviation φ_z measured along an x-coordinate line
xwy	measured rectangularity deviation in the coordinate plane xy
xwz	measured rectangularity deviation in the coordinate plane xz
y_L	value of the standard for displacement in the y_L-direction
y_m	displacement measured with the y-displacement transducer in the y-direction
ytx	positional deviation Δx measured along a y-coordinate line
yty	straightness deviation Δy measured along a y-coordinate line
ytz	straightness deviation Δz measured along a y-coordinate line
yrx	rotational deviation φ_x measured along a y-coordinate line

yry	rotational deviation φ_y measured along a *y*-coordinate line
yrz	rotational deviation φ_z measured along a *y*-coordinate line
ywz	measured rectangularity deviation in the coordinate plane *yz*
z_L	value of the standard for displacement in the z_L-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	straightness deviation Δz measured along a *z*-coordinate line
zrx	rotational deviation φ_x measured along a *z*-coordinate line
zry	rotational deviation φ_y measured along a *z*-coordinate line
zrz	rotational deviation φ_z measured along a *z*-coordinate line
$\cos(\varphi_i)$	rotational correction e. g. in pitch measurement
$\cos(\theta_i)$	tilt-related correction e. g. 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 *x*- and *y*-direction, counterclockwise
ϕ_{xz}	angle between *x*- and *z*-direction, counterclockwise
ϕ_{yz}	angle between *y*- and *z*-direction, counterclockwise
Rqx	noise in *x*-direction
Rqy	noise in *y*-direction
Rqz (Sqz)	noise in *z*-direction in a measured profile (within a measured area, resp.)
v	scan speed (i.e. distance travelled by the tip per unit of time, not to be confounded with the scan rate, i.e. the number of scanlines recorded per unit of time)

4 Characteristics of scanning probe microscopes

4.1 Components of a scanning probe microscope

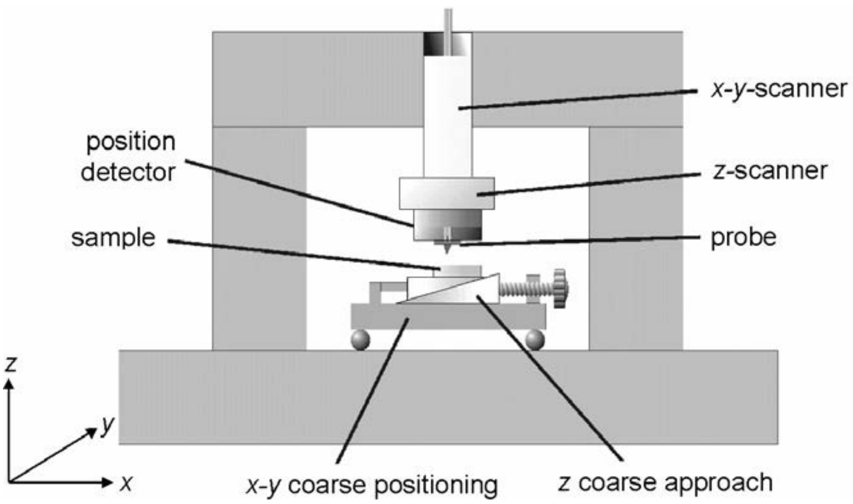

Figure 3: Schematic sketch of a scanning probe microscope system

Several components shown in Figure 3 are defined in ISO 18115. Here they fulfil the following functions:

Probe: equipped with a tip at its apex, it probes the sample surface exploiting a local physical interaction whose changes can be detected, e.g. as cantilever bending in the case of an atomic force microscope

Position detector: Transformation of the probe's interaction response (e. g. bending or oscillation of the cantilever) into an electrical signal.

z-scanner: Adjusting element for tracking the sample-probe distance during *x-y*-scanning to a constant value of the physical interaction used for distance control (e.g. of the action of force on the probe in the case of an atomic force microscope), approximately to ensure a constant distance between sample and probe.

x-y-scanner: Adjusting element for lateral displacement of the probe (alternatively: of the sample) in the *x-y*-plane (plane parallel to the seating face of the sample), which is used, among other things, to record a location-dependent interaction signal that contains information about the local property of the sample (here above all the topography: local height).

Sample holder: where appropriate, with coarse positioning and coarse approach mechanics.

Casing/mounting: Construction and materials between mountings for scanner and sample forming the measurement circle.

4.2 Metrological categories of scanning probe microscopes

SPMs can generally be subdivided into the three following categories depending on their metrological equipment:

A) Reference devices with integrated laser interferometers allowing direct traceability via the wavelength of the laser used to the SI unit of length [1]

B) SPMs with position measurement using displacement transducers, e.g. capacitive/inductive sensors, strain gauges, encoders calibrated by temporarily connecting laser interferometers to the device or by measuring on high-quality standards. Two types are to be distinguished here:

  B1) active position control: tracking to scheduled position by means of a closed loop (so-called closed-loop configuration)

  B2) with position measurement but without closed loop for position control (so-called open-loop configuration)

C) SPMs in which the position is determined from the electrical voltage applied to the adjusting elements and, if need be, corrected using the look-up table; calibration against standards

This definition of metrological categories implies that it is not possible for certain devices to be assigned as a whole to a single category but that with respect to their scan axes they must be considered separately.

4.3 Block model of a scanning probe microscope

From the schematic SPM model in Figure 3, the abstract block model in Figure 4 is obtained. The characteristics of its essential *components* are given below and need to be investigated individually in the course of verification and calibration.

*For **category C**:*

- casing/mounting (mechanical, acoustic, electromagnetic and thermal characteristics)
- sample holder, where appropriate with coarse positioning and coarse approach mechanics (accoustic

[1] Instruments of category A are often referred to as "metrological SPM", although the definition of a "metrological SPM" in ISO 18115-2 Amd1 does not necessarily imply laser interferometric position control

mechanical and thermal characteristics)

- *z*-scanner
- *x*-*y*-scanner
- detector loop, e.g. by the beam deflection method: beam on rear side of cantilever in the case of AFM, detection of the reflex with a position-sensitive photodiode, position-dependent signal serves as input for the loop to track scanner or probe in the *z*-direction
- probe

***Additionally** for **category B**:*

- *x*-, *y*- and/or *z*-displacement transducer, e.g. encoder, capacitive or inductive displacement transducer, strain gauge: category B2
- where appropriate, active (closed-loop) position control: category B1

Additionally** for **category A:

Traceability by integrated laser interferometers, i.e. systems as category B, equipped with

- integrated laser interferometers for position measurement/control
- where appropriate, additionally provided with angle sensors

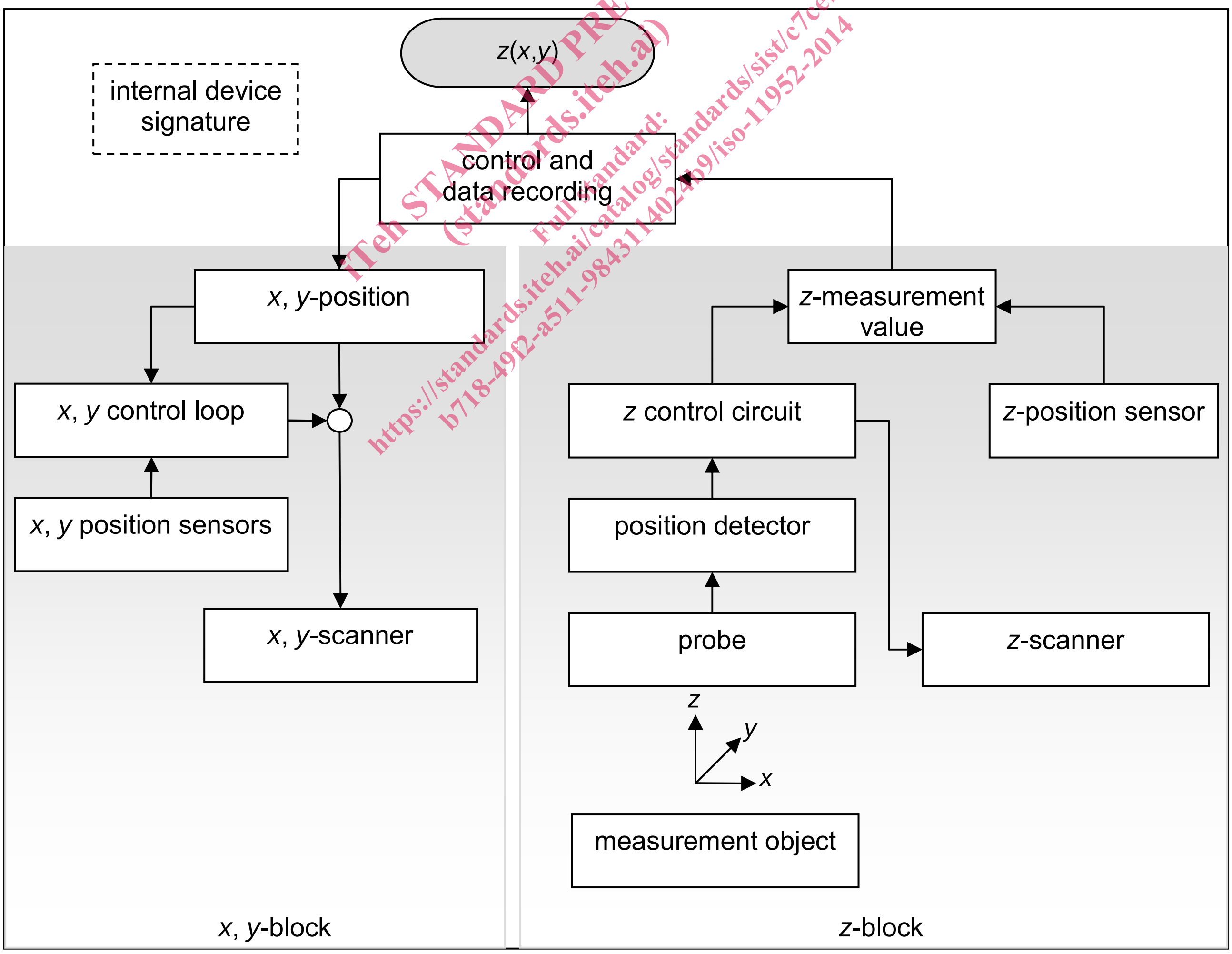

Figure 4: Block model of a scanning probe microscope