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NEW DEVELOPMENTS OF MEASUREMENT STANDARDS AND PROCEDURES FOR MICRO AND NANOMETROLOGY AT THE PTB

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Abstract:

The contribution provides an overview on new developments of standards and measurement methods for 3D-micro- and nanometrology with a focus on recent work at the PTB. These developments target at a reduction of measurement uncertainty of instruments used for dimensional characterization of micro- and nanostructures. The developments in 3D-micrometrology will focus on standards for calibration of micro-CMM, for dimensional micro-CT instrumentation and for microcontour measurement. For different applications in nanometrology calibration standards for unidirectional measurands like 1D-, 2D- and 3D-position as well as step height and for bi-directional measurands like linewidth or diameter will be described. In addition the potential to make use of crystalline lattice structures to provide traceability for dimensional measurements at the nanoscale will be addressed.

Keywords: 3D-micrometrology, nanometrology, metrological traceability, standards, measurement uncertainty

1. INTRODUCTION

Measurement technology plays a decisive role for development and optimization of manufacturing processes as well as quality assurance of manufactured components and products. Dimensional measurands are of particular importance because the function of components often is critically dependent on their dimensional properties. This also holds for manufacturing processes in micro- and nanotechnology.

Dimensional measurands are to be referred to the unit of length, the metre. Metrological traceability of the measurement results requires a chain of calibrations based on suitable standards, which eventually are linked to the SI unit of length via national standards [1]. This calibration chain can be shortened by the use of special measurement instruments with integrated interferometric measurement axes.

For the determination of the measurement uncertainty of measurement results according to internationally accepted guidelines [2] the uncertainty contributions of the calibrated standards used for the measurement have to be appropriately taken into account. This requirement is also valid in micro- and nanotechnology. In this contribution we concentrate on aspects of development and calibration of suitable standards for application in micro- and nanotechnology. The standards and their associated calibration procedures were developed by the PTB, often in close cooperation with other partners, targeting at a wide applicability of the calibration standards.

2. STANDARDS FOR MICROCOORDINATE METROLOGY

2.1 Background

The task of micro-coordinate metrology is the characterization of sub-mm structures. Special microprobes were developed for tactile probing. Examples are the piezo-resistive microprobe [3] and the tactile-optical fibre probe [4]. These microprobes use probing elements smaller than 0.3 mm and are applied in special micro-coordinate measuring machines (e.g. Werth VideoCheck, Zeiss F25) with highly precise and high resolution positioning axes.

In addition to tactile microprobes also non-contacting sensors are used for measurement of microstructures. The range of optical sensors covers optical point sensors (e.g. Foucault sensor, chromatic sensor) and areal measuring sensors (e.g. confocal microscopes, white light interferometers, focus variation sensors) [5]. In recent years also dimensional computed tomography (CT) was developed as coordinate measurement systems for microcomponents [6]. This technology allows a complete characterization of components including inner structures with a high density of measurement points.

To assure the comparability of specifications, the testing of micro-coordinate measuring machines (CMM) should be aligned with established procedures for macro-CMM based on the parameters probing deviation and length measurement deviation [7]. For this, suitable standards are needed.

Small reference spheres (Ø 0.5 mm and below) for testing of the probing characteristics are available, however their comparably large form deviations as well as the diffuse surface scattering properties required for testing of optical sensors still are challenging aspects.

Even bigger challenges are due to the requirements on material measures of length required for the testing of length measuring deviations. Examples are shown in the following chapter. Finally, special challenges arise for the determination of the measurement uncertainty for measurement results on microcomponents, this in particular holds for non-contacting probes. Reasons for this are the comparably large influence of workpiece characteristics (e.g. the surface properties) and the increased number of influence parameters in comparison to tactile probing. To cope with this challenge, so-called workpiece-like standards have been developed and one example is discussed below.

2.2 *Examples of standards*

2.2.1 Hemisphere plate [8]

On this standard, nine hemispheres (radius approx. 2.5 mm) of silicon nitride are fixed on a Zerodur plate (edge length of 90 mm). The distances of the centres of the hemispheres are the material measures of length. Bores close to the hemispheres allow tactile reversal measurements. In addition optical measurements in transmission illumination with subsequent image processing can be realized. The tactile calibration of the hemisphere distances is realized at the PTB with an expanded uncertainty of $U_{\mathrm{cal}} =$ 0.2 µm.

Fig. 1: Micro hemisphere plate.

2.2.2 Sphere calotte cube

The sphere calotte cube (edge length 10 mm, made from titanium) developed by the PTB [9] shows a raster of spherical calottes with 400 µm radius on 3 side faces. This material standard thus combines 3 ball plates in mutually orthogonal positions. Titanium was chosen to have a good X-ray transmission in CT measurements, which can be even enhanced using an optional hollowing of the cube. The calottes are produced by electric discharge machining (EDM). Because of the diffuse reflection characteristics, also optical measurements in regions with stronger slopes are possible. The distances of the centers of the calottes are the lengths realizations embodied in the cube standard. Single calotte positions are slightly mismatched to the regular raster pattern to realize 5 different parallel length measures, which are necessary for testing of length measurement deviations according to existing standards and guidelines.

Fig. 2: Sphere calotte cube.

The calibration of the sphere calotte cube is performed with a conventional CMM, which results in expanded calibration uncertainties for the calotte distances of $U_{\mathrm{cal}} = 1$ µm.

2.2.3 Micro-Tetrahedron

The micro-tetrahedron is a new class of standards developed at the PTB by using micro-manipulation techniques. In the standard configuration ruby spheres with diameters from 0.2 mm to 3 mm are glued together to form micro-tetrahedrons, which then embody 6 length values in different directions in space.

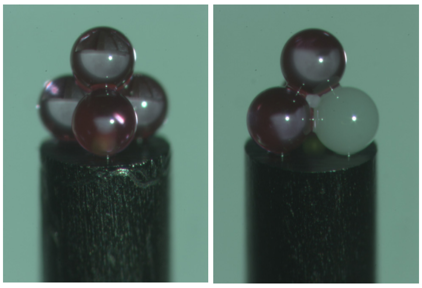

Fig. 3: Manufactured micro-tetrahedrons with sphere diameters of 1 mm.

For the investigation of the material influence in CT measurements, micro-tetrahedrons with different sphere materials were also realized. For this, one ruby sphere was replaced by a sphere with similar X-ray absorption (Si_3N_4) or of stronger X-ray absorption, like e.g. steel or ZrO_2. It should be noted that microspheres were also proposed by some authors as standards to determine the structural resolution of CT measurements, however this issue is still evolving.

The calibration of the tetrahedron is realized for sphere diameters of 0.5 mm and bigger with tactile coordinate metrology. The expanded measurement uncertainties obtained by calibration with a conventional CMM for the distances of the sphere centers with diameters of 1 mm – 3 mm are $U_{\mathrm{cal}} = 1$ µm. For sphere diameters of 0.5 mm the calibration was performed with a micro-CMM (F25) with an expanded measurement uncertainty of $U_{\mathrm{cal}} = 0.3$ µm. Tetrahedrons with sphere diameters smaller than 0.5 mm currently cannot yet be calibrated reliably. However, first measurements have already been performed with synchrotron-CT [10].

2.2.4 Micro-contour standard

As a workpiece-like standard for tactile and optical sensors for small features the micro contour standard [11] was developed at the PTB, see fig. 4. The standard realizes cylinder segments, sloped areas and steps in different dimensions. The manufacturing was done by wire erosion resulting in (cooperative) surface characteristics, suitable for optical sensing.

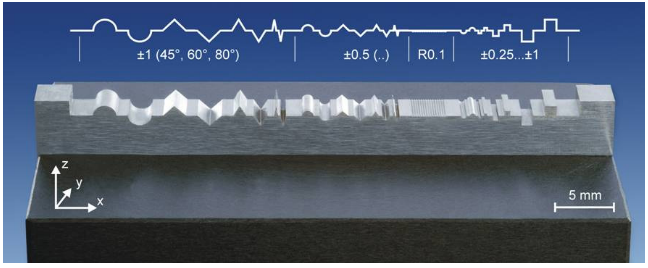

Fig. 4: Micro contour standard.

The calibration is realized by a patented procedure on an optical CMM in transmission mode. Here, thin sheets with similar contour are measured which have been manufactured in the same process as the contour standards. The resulting expanded measurement uncertainties are U_{cal} = 0.5 µm - 1.5 µm for the coordinates of the cylinder segments.

2.2.5 *Micro-pyramid standard*

Starting with the experiences gained from the application of pyramidal standards with total dimensions on the micrometer scale [12] (see chapter 3.2.4), a new 3D standard was developed which allows the characterization of micro/nano-CMM with measurement volumes of a few centimeters [13]. The measurements on these standards do not only allow a calibration of the axes scales but in addition a determination of the guiding deviations and the crosstalk of the coordinate axes.

The layout of the standard is shown in fig. 5. The single segments with an inverse pyramid each have dimensions of 6.5 mm x 6.5 mm x 1.5 mm. A pyramid is characterized by four plateau areas on which micromarkers are patterned at defined positions. The standard is made from silicon by micro manufacturing methods (photolithography and subsequent etching). Each marker center embodies a defined 3D coordinate on the standard and thus allows the instrument to be calibrated in the measurement volume. If the 3D-coordinates of the markers are known with sufficiently small measurement uncertainty, e.g. by prior calibration in a metrological 3D measuring instrument, the position data can be regarded as reference data of an ‘ideal’ measuring instrument. A correct application of the standard then allows to determine and correct the deviations of the device under test including not only the scale factors but also the crosstalk between the axes. The determination of the obtainable calibration uncertainty for micro-CMM using this type of standards currently is under investigation.

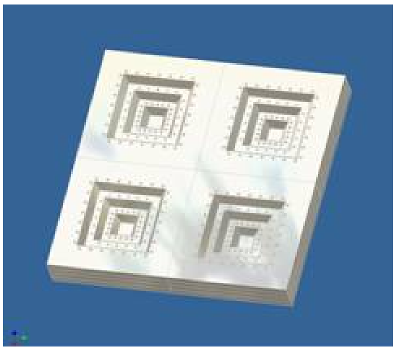

Fig. 5: Micro pyramid standard made of silicon.

3. STANDARDS FOR NANOMETROLOGY

3.1 *Background*

The term nanotechnology defines the investigation, use and manufacturing of structures, molecular materials and systems with a dimension of typically 1 nm up to 100 nm. Because of these small dimensions of the structures, the requirements on the instrumentation used for characterization of nanostructures are quite different from those used in micrometrology.

For applications in nanometrology based on tactile sensors only cantilever probe tips as used in scanning probe microscopes are used, which under certain conditions allow to achieve atomic resolution in surface probing. In addition non-contacting measurement methods are used. Here microscopic measurement methods like e.g. scanning electron microscopy (SEM) or transmission electron microscopy (TEM) are used, but also optical microscopy with short wavelength (DUV) and non-imaging optical methods, like e.g. scatterometry in a wavelength range from visible down to EUV.

All measurement methods used in nanometrology are dependent on a proper signal modeling to support the analysis of the instrument’s signal contrast. Without a proper signal modeling which takes into account the interaction of the probe with the measurement structure the metrological traceability of measurement results with the demanded measurement uncertainties in the nm-rage could not be obtained [14].

3.2 *Examples of standards*

In recent years several standards for dimensional nanometrology were developed at the PTB, often in close cooperation with partners from companies, research institutes or other national metrology institutes (NMI) [15]. The main focus was on characterization and calibration of scanning probe microscopes (SPM) [16] and other high resolution measuring instruments.

In addition to standards for calibration of the scan axes (1D and 2D grating standards for lateral scan axes, step height standards for the vertical axis, newly developed 3D-standards for all 3 axes) also flatness standards and standards for characterization of the tip shape were developed (linewidth standards and tip characterizers with sharp edges).

Because of the different measurement volumes of SPMs, their maximum permitted sample sizes and positioning capabilities, the suitable calibration standards should be carefully chosen for each application. Most of the available types of standards could be calibrated by PTB and also in other NMIs. Some of the standards have proven their stability and applicability in international comparison measurements, e.g. in the so-called NANO comparisons organized by the BIPM CCL[1]: NANO2 step height standards (2001-2003), NANO4 1D-gratings (1998-2000) and NANO5 2D-gratings (2005-2008). In addition, several EURAMET- and bilateral comparisons were run successfully.

To achieve a harmonized approach to characterization and calibration of measurement instruments, guidelines and standards were developed and finalized in recent years, which also contained some advice concerning the choice of suitable calibration standards.

Optical measuring instruments are dealt with in the guideline VDI/VDE 2655: Sheet 1.1 for interference microscopes and Sheet 1.2 for confocal microscopes. For SPM the guideline VDI/VDE 2656 should be used. Future sheets of the series of VDI/VDE-guidelines will also cover roughness and form measurements.

3.2.1 *Step height standards*

For calibration of the vertical scan axis of measuring instruments several step height standards are available, based on protrusions or grooves with rectangular or trapezoidal profile. Because most instruments show systematic nonlinearities along their vertical axis, measurements at different steps are necessary, which cover the full measurement range. Commercially available sets of calibrated step height standards cover the range from 6…8 nm up to several µm.

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[1] BIPM CCL - KCDB: Bureau International des Poids et Mesures (Paris), Consultative Committee on Length - Key comparison data base

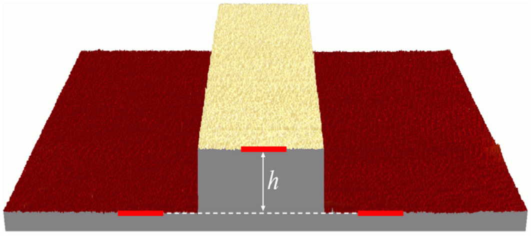

Fig. 6: 3D view of a scanning force microscope image of a 40 nm step height standard (image size: 24 µm x 20 µm).

Most step height standards are made from silicon by etching techniques. Alternative materials are quartz glass and metals (rarely used). Attention has to be paid to avoid effects due to material contrast. Therefore, sometimes step height standards are homogeneously coated with an optional additional metal layer.

The evaluation of the measurement data is done linewise or area-based in close agreement to ISO 5436-1. In SPM applications also histogram-based evaluations are used in addition. The expanded measurement uncertainties of calibrations by means of PTB's metrological SPM is approx. 1 nm for step heights of 7 nm and approx. 10 nm for step heights of 10 µm.

3.2.2 1D- and 2D-grating standards

Periodical gratings show advantages over other types of lateral standards because they allow to average over many grating periods and they in principle also allow to characterize non-linearities in the x-y-plane if the positions of the individual grating structures are known with sufficiently small uncertainty [17]. 2D-gratings offer the advantage to characterize both lateral scanning axes and their squareness error in just one measurement.

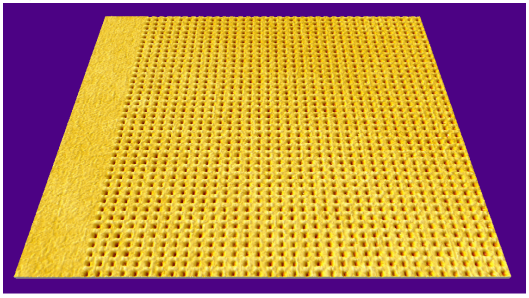

Fig. 7: 3D view of a scanning force microscope image of a Nanosensors 2D100 pitch standard (image size: 4 µm x 4 µm, pitch: 100 nm).

Commercially available lateral standards show period lengths of a few µm down to 100 nm (2D) or 25 nm (1D), with prototypes of only 35 nm (2D) respectively a few nm (1D) being tested at PTB. Gratings with period lengths above 500 nm usually are produced by optical (interference) lithography, while finer gratings are mainly produced by e-beam lithography (EBL).

Very fine gratings with pitches well below 100 nm have been realized by molecular beam epitaxial growth techniques as alternating series of thin films from different materials (so-called hetero-structure standards). Here the layer system is cut after the epitaxial growth process to make the hetero-structure accessible to measurement probes [18]. In a bilateral comparison with the NMIJ (Tsukuba, Japan) it could be proven that also these fine gratings with a nominal grating pitch of only 25 nm could be reliably calibrated [19].

The evaluation of grating measurements can be made in the spatial or the frequency domain, often also combining both approaches, as e.g. in the commercially available analysis software SPIP (Image Metrology A/S) [20]. At PTB a combined FFT/FT-Method was developed as well as a refined centre of gravity method for the determination of single grating elements [21].

Using the metrological large range SPM of the PTB, the achievable expanded measurement uncertainties for the mean pitch of the gratings, averaging over several hundred periods, is a few ten pm only. Alternatively gratings with pitches down to 140 nm can be calibrated by diffractometry.

3.2.3 Linewidth standards

In the lithographic production process of integrated circuits, the positions, widths and overlay of the structures successively projected onto the wafer surfaces and afterwards processed have to be reliably measured and controlled. The smallest lateral structure dimensions (critical dimension, CD) of currently produced ICs are about 22 nm, developments for further reduction of CD are under way.

The templates which are used in lithography are the masks, which carry the patterns to be projected on to the wafer in a scale of 4:1. For compensation of diffraction effects the masks also carry functionally relevant assist features with dimensions of about 30 nm, which also have to be qualified within the quality control processes of the mask manufacturer.

In cooperation with partners from industry the PTB has developed a linewidth photomask standard and tested its applicability in a comparison measurement [22]. This photomask standard adds on the already available silicon linewidth standards [23]. On the refined photomask standard line and box features with smallest structure sizes of nominal 30 to 40 nm are available in fine variations of CD.

The linewidth mask standards are calibrated with UV- and DUV-optical transmission microscopy and SEM, supported by additional SPM characterizations of the edge transition. The expanded measurement uncertainties obtained by SEM for the top linewidth are $U_{\mathrm{cal}} =$ 15 nm.

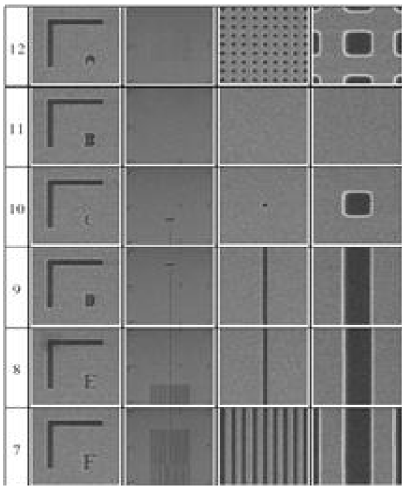

Fig. 8: SEM images of structures on a linewidth standard for mask metrology.

It should be noted that recently PTB also made progress in calibrating rectangular Si lines by CD-SPM, traced back to TEM measurements on single crystal silicon.

3.2.4 3D-pyramidal standard

As an alternative or as add-on to grating and step height standards, also 3D standards are available since several years [12]. They are based on 3D-structures representing multistep pyramids. The terraces of the pyramids and the substrate are patterned with so-called nanomarkers, whose centres serve as 3D-reference points (like the landmarks known from geodesy).

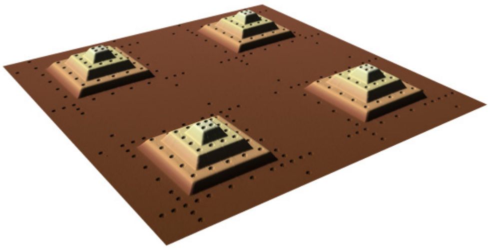

Fig. 9: 3D view of a scanning force microscope image of a 3D standard (image size: 84 µm x 84 µm, base line of pyramids approx. 20 µm, height approx. 3 µm).

The size of the 3D-pyramids is chosen to fit the 3D-measurement volume of most standard SPMs. The pyramids consist of platinum, being patterned onto the substrate with the focussed ion beam (FIB) method. In a subsequent step the ring-shaped nanomarkers are written by FIB as well.

For an efficient data analysis a special software package was developed by the manufacturer of the micro-pyramids (m2c, Potsdam). The single nanomarkers here are automatically recognized in the 3D data set. In a second step the measured 3D-coordinates are compared with a reference coordinate data set (obtained from the metrological large range-SPM at PTB).

By use of this standard in combination with a suitable measurement strategy and software not only the calibration factors for the 3 scan axes but also the crosstalk between them could be determined. In addition to their use in SPM calibration, these 3D standards are also used in stereogrammetric SEM and confocal laser scanning microscopes.

The 3D standards described in section 2.2.5 with dimensions in the mm-range represent an extension of this concept of 3D standards and target to close the gap between nano- and macroscale standards.

3.2.5 Crystal lattice as standards

The limitations of the different processes applied to manufacture the standards described in the text before also set limitations of the achievable calibration uncertainties. In particular for standards representing smallest nominal values, like e.g. step height standards below 10 nm, linewidth standards or lateral standards with pitches below 25 nm, the critical dimensions are already close to crystalline lattice dimensions, triggering concepts to try to make use of the regularity of crystalline lattices for metrology.

Such atomic structures could be prepared under certain conditions and imaged with SPM, in particular with the scanning tunneling microscope (STM) [24]. From the metrological point of view, however, a principle disadvantage of the atomic lattice structures at the surface of single crystals is that the corresponding measurands (step height, nearest neighbor distance) are based on calibration values of the bulk lattice, which might be different from the conditions at the surface. This is known from investigations of crystalline surfaces under UHV conditions, which showed that modified atomic configurations occur at the surface, which change not only the structure but also the distance of nearest neighbour atoms. This shows the necessity to establish a direct route of traceability for suitable crystalline surface structures as has been done for the lattice distance of bulk silicon atoms within the framework of the Avogadro project [25]. An example for the application of the bulk silicon lattice constant for calibration of silicon single crystal linewidth standards is described in [26], making use of transmission electron microscopy to image columns of atoms in thin silicon lamella.

For atomic step heights at silicon surfaces first investigations exist which confirm the bulk values of 0.31 nm but with comparably larger measurement uncertainties [27]. In the near future smaller uncertainties are expected, due to improved instrumentation like e.g. high precision interferometry. A similar situation exists for the lateral crystalline structures. On graphite surfaces the distance between second neighbouring surface atoms could be traceably determined by STM imaging and interferometry to be 0.246 nm with an expanded uncertainty $U = 7.2$ pm [28]. Figure 10 shows the potential of atomic surface terraces as step height standards.

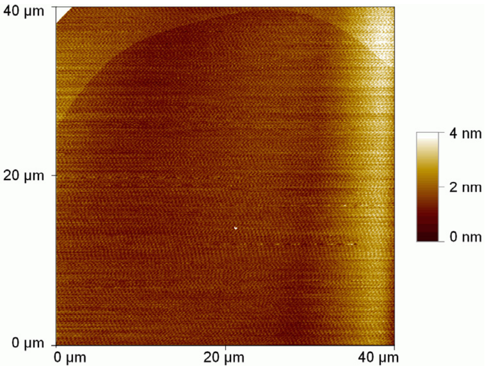

Fig. 10: Scanning probe microscopy image of a large, atomically smooth Si(111) surface measured under normal lab conditions in air. In the upper part a mono-atomic step with a height of 0.31 nm can be seen. The deviations visible in the image are related to small nanometric deviations of the scanning instrument.

4. SUMMARY AND OUTLOOK

An up-to-date overview of developments of standards and measurement procedures for application in micro- and nanometrology was presented from a PTB perspective. The developed standards as well as calibration procedures and the achieved calibration uncertainties were shortly described.

Future developments of standards will concentrate on challenging requirements like e.g. even smaller 3D microstandards of different materials for CT metrology (standard for structural resolution), further standards which make use of crystalline lattices and 3D nanostructures which can be produced precisely including evaluation of self-organization techniques for bottom-up manufacturing.

For the development of standards and procedures in micro- and nanometrology it is recommended to follow the established procedures and guidelines from macrometrology wherever possible.

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