Surface Topography: Metrology and Properties

PAPER

Bottom-up approach for traceable calibration of tip geometry of stylus profilometer

Gaoliang Dai, Xiukun Hu and Johannes Degenhardt
Physikalisch-Technische Bundesanstalt, 38116, Braunschweig, Germany
E-mail: gaoliang.dai@ptb.de
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Abstract
A novel approach for accurate and traceable calibration of stylus tip geometry is introduced in the paper. The approach consists of several steps. Firstly, the geometry of an AFM tip is calibrated to a kind of line width standard whose geometry is traceably calibrated to the lattice constant of crystal silicon. Then, the stylus tip to be calibrated is measured by the calibrated AFM tip in an AFM, thus its tip geometry can be accurately determined after the contribution of the AFM tip geometry being corrected from the measured AFM image. After being calibrated, the stylus tip can be applied in measurements of vast microstructures and surfaces, where the measurement results can be in turn corrected using the characterized stylus tip geometry. In such a way, the stylus tip geometry and its measurement results can be finally traceable to the lattice constant of silicon, using this bottom-up approach. Detailed experimental examples are illustrated. For a stylus type RFTHB-50 studied in this paper, its tip radius is measured as 1.727 μm with a standard deviation of 0.007 μm. It is significantly smaller than its nominal value of 2 μm, indicating the need of the calibration. The application of calibrated stylus tip in measurements of microspheres is demonstrated. Compared to conventional tip characterization methods based on tip characterizers the proposed method has advantages of (i) no risk of damaging sharp edges of tip characterizers, (ii) capable of directly characterizing the 3D geometry of stylus tip, (iii) high accuracy.

1. Introduction
Tactile profilometry is one of the most applied techniques for surface metrology in research and industry. In a stylus profilometer, usually a diamond stylus tip with a typical radius of a few μm is applied in scanning surfaces. The stylus probe is usually fixed to an arm structure, where a feedback system is often used to apply a specific amount of torque on the arm, thus to introduce a desired probing force, typically in a range of μN-mN. The surface’s profile can be represented by the z-position of the stylus together with the lateral displacements of the scanner. Based on the measured profile, dimensional properties of the surface such as roughness, form, contour and/or step height (layer thickness) etc can be characterized.

The history of tactile profilometry can be dated back to the time when surface measurement was started in the early 1930s, by Schmaltz in Germany quickly followed by Abbott and Firestone in the USA [1]. By 1938 Abbott et al [2] had set up a company to manufacture and sell the profilometer. Since then, various stylus profilmeters have been developed and applied [3–6], strengthened by modern electronic and computing technologies.

Stylus profilometry is a kind of matured technique today. Its characteristics, calibration, material measurements, filtering technique and data evaluation have been well standardized in a series of international standards. For instance, there is ISO 3274 about the nominal characteristics of stylus profilmeters [7], ISO 5436 describing the material measures [8], ISO 12179 explaining the calibrations of a stylus profilometer [9] and ISO 16610-21 about the Gaussian profile filter [10]. As three-dimensional (areal) surface parameters are more reliable than 2D profile parameters [11], more and more areal parameters are applied today. For this purpose, the standard ISO 25178–601 [12] has been developed which defines the metrological characteristics of contact (stylus) areal surface texture.
measuring instruments, as well as the standard ISO 25178–701 [13–15] which describes the calibration and measurement standards for contact (stylus) instruments. In addition, a new standard ISO 21920 [16–18] is being prepared for an update and extension of previous documentary standards.

Calibration of stylus profilometer is a fundamental issue. It usually involves the calibrations of

(i) scaling factor and linearity of dimensional axes, typically using e.g. a set of lateral gratings (type C in ISO 5436–1) and depth setting standards (type A in ISO 5436–1) [8, 18],

(ii) coordinate system using e.g. a plane glass, a reference sphere and/or a prism (type E in ISO 5436–1) [8],

(iii) probing force using e.g. a kind of reference spring [19, 20],

(iv) overall performance using e.g. a kind of roughness standard (type D in ISO 5436–1) [8],

(v) stylus tip geometry and/or monitoring of the tip status using e.g. a sharp protruding edge such as a razor blade (type B3 in ISO 5436–1) [8].

Thanks to the developments of metrological atomic force microscopy (AFM) [21, 22], metrological profilometry [4] and reference springs [19, 20], great progresses have been achieved for accurate calibrations of the scaling factors, coordinate system, and probing force.

The impact of stylus tip geometry on measurement results becomes an increasingly important issue. It is particularly due to the paradigm shifts that have occurred in the discipline of surface metrology, as reviewed by Jiang et al. [23, 24]: (i) profile to areal characterization; (ii) stochastic to structured surfaces, and (iii) simple geometries to complex free-form geometries. In tactile measurements, the measurement results can be interpreted as the dilated results of surface structures by the tip geometry [25]. In form or contour metrology, the stylus tip geometry needs to be characterized with a (much) higher accuracy than the needed accuracy of form/contour measurements. In roughness metrology, the contribution of the stylus tip geometry can be interpreted as a limit on its measurement bandwidth. A critical wavelength, $\lambda_c$, defined as the smallest wavelength of a surface that a stylus probe can realistically reproduce can be represented as equation (1) [26]. Although a measurement uncertainty of tip radius by $\pm 0.5 \mu m$ is mostly sufficient in industrial roughness measurement practice, (much) higher metrology accuracy is demanded at national metrology institutes (NMIs), accreditation laboratories and sophisticated industrial applications.

$$\lambda_c = 2\pi \sqrt{AR}$$

where $A$ is the amplitude of the surface profile and $R$ is the tip radius.

The effects of stylus tip geometry on roughness metrology have been well studied [27–30]. Their results indicate the importance of the characterization of the tip geometry to better interpret the roughness values, and to compare the measurements of different surface measuring tools in context of bandwidth characteristics.

Applying a tip characterizer with sharp edge features (e.g. a razor blade) is a typical method for characterizing the stylus tip geometry. In industrial applications, silicon gratings with a trapezoidal or rectangular profile, which can be conveniently manufactured using the state-of-the-art wet-etching processes, are usually applied [31]. However, this method involves some problems in practice. The first issue concerns the (possible) damage of the sharp edge features of the tip characterizer during measurements. It is due to the fact that the sharper the edge feature is, the easier it gets damaged particularly under the large probing force of the stylus ($\mu N$ to mN). The second issue concerns the measurement efficiency. As the tip characterizer is usually etched as a kind of 1D grating, only one 2D profile of the tip can be determined in one sample orientation. To obtain the 3D geometry of the tip, the characterizer has to be rotated for measurements with different orientations. The third problem relates to the data processing. To illustrate this important issue, a schematic diagram is shown in figure 1. In this figure, a tip characterizer with a trapezoidal shape (shown as “S”) is measured by a tip geometry (shown as “T”), resulting in a measured profile $S_m$. Assuming the tip characterizer has an infinitely sharp edge, the segments “AB” and “CD” (figure 1(b)) of $S_m$ can be regarded as two segments of the tip shape, where the points B and C denote the top corner positions of the trapezoidal shape. By combining the segments “AB” and “CD” as shown in figure 1(c), the stylus tip geometry can be reconstructed theoretically. This method will, however, encounter a big problem in practice: it is very difficult to accurately determine the position “B” and “C” from the measured profile $S_m$, particularly in presence of measurement noise and the deviation of the real structure/tip shape from its ideal shape. Consequently, as illustrated in figure 1(b), if the corner positions were wrongly evaluated as (B’, C’) or (B”, C”), the tip geometry would be overestimated as T’ or underestimated as T”, leading to significant measurement errors. Furthermore, in practice the tip characterizer cannot have an infinitely sharp edge, consequently, its real edge radius will introduce a measurement bias as well.

Recently, Brand et al. [32] introduced a silicon standard which consists of 26 rectangular grooves with varying width from 0.3 $\mu m$ to 3 $\mu m$ in one row, in a similar design concept as [33]. When a stylus tip measures these grooves, the measurable depth of the grooves varies depending on the tip geometry and the

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groove width, thus offering a solution for characterizing the tip geometry. Although the principle of this method is straightforward, it has similar problems in its practice application as mentioned above. For instance, the (possible) damage of sharp edges will significantly impact the measurable depth of the grooves, leading to measurement error. The standard needs to be rotated in different orientations for determining 3D tip geometry. Although the determination of edge positions "B" and "C" as shown in figure 1 is no more needed, it requires accurate calibration of the groove width before the tip characterizer can be applied.

To solve the challenging problems above, this paper introduces a novel bottom-up approach for accurate, traceable, and 3D characterization of stylus tip geometry. The paper is organized as follows: section 2 introduces the concept of the proposed calibration approach. Section 3 gives a detailed measurement example illustrating the realization of the proposed concept. Section 4 introduces an application example of the calibrated stylus in measurements of microspheres.

2. Concept

2.1. Bottom-up traceability approach for nanometrology

The bottom-up traceability approach has been recently developed by several NMIs [34–36] for nanometrology. The key concept of the approach is to apply the crystal lattice constant as an internal ruler for traceable nanometrology. To illustrate its concept, a simplified schematic diagram is shown in figure 2(a), where the width of a feature can be calculated as the number (N) of crystal lattice planes within the feature’s cross section multiplied with the silicon crystal constant \(a_{111}\). This constant \(a_{111}\) has been traceably measured as 313.560 11(7) pm for silicon through a combination of x-ray and optical interferometry [37]. This approach was recently recommended as a secondary realization of the metre for nanometrology by the Consultative Committee for Length (CCL) of the Bureau International des Poids et Mesures (BIPM) [38]. The state-of-the-art high resolution transmission electron microscopy (HR-TEM) is capable of resolving the spatial periodicity of a crystal lattice, offering a technically feasible solution for the realization of the approach.

The realization of the bottom-up traceability approach has been systematically studied and published elsewhere [34–36, 39]. Figure 2(b) illustrates the geometry of a line feature including the parameters of line width, corner rounding and sidewall angle, measured by using the bottom-up approach. Its measurement accuracy reaches sub-nm [34–36], thus offering a solid metrological basis for true 3D nanometrology.

For the sake of a completed introduction, one important issue of applying the bottom-up approach needs to be addressed: the dissemination of the TEM

![Figure 1. Schematic diagram showing the basic principle of the characterization of stylus tip geometry using a tip characterizer with sharp edges.](image-url)
results of silicon samples for calibration applications. This issue is related to the fact that the sample needs to be destructively prepared into thin “lamella” so that it can be measured by a TEM. Consequently, the feature is no longer available for further calibration applications. To solve this problem, a strategy based on the data fusion concept is applied as explained in figure 2(c) [39]. The strategy applies two groups of specimens, one as the reference structures and the other as the TEM target structures. The measurement procedures consist of three steps. First, the differences of the feature geometries of the two specimens are measured by e.g. a critical dimension AFM (CD-AFM). Second, the TEM target structures are destructively prepared and measured by a TEM, and thus their true dimensions are determined using the bottom-up approach mentioned above. Third, the data of the two steps are fused to determine the geometry of reference structures. The benefit of this approach is that the reference structures are kept undestroyed for further calibrations. The challenge issue is that the overall measurement uncertainty is a combination of measurement uncertainties of all steps. Consequently, each step needs to be performed as accurate as possible to achieve the best possible accuracy. With actual research progress achieved at NMIs, the expanded measurement uncertainty of above methodology may reach about 0.7 ∼ 1.6 nm, as confirmed in a bilateral comparison between the NIST and PTB [40]. It in turn allows the traceable calibration of AFM tip geometry with nm accuracy [41].

2.2. Extension of the Bottom-up traceability approach to micrometrology

The bottom-up traceability approach mentioned above can be further extended for the characterization of microstructures such as stylus tips as illustrated in figure 3. The approach is realized in several steps. Firstly, an AFM tip geometry is calibrated to a nano standard, referred to as a critical dimension (CD) standard type IVP5100-PTB [39], which was jointly developed by the PTB and the company Team Nanotec. The geometry of the line structure of the IVP5100-PTB sample was traceably calibrated to the lattice constant of crystal silicon as mentioned above. In such a way, the geometry of the AFM tip, S_{AFM Tip}, can be characterized accurately and traceably with a measurement uncertainty of about 1 nm [41].

With S_{AFM Tip} calibrated, the AFM tip can then be applied to directly measure the geometry of a stylus tip in an AFM. In this measurement, the stylus tip is mechanically clamped and acts as a measurement specimen. As the measured AFM image is the dilated result of the stylus tip geometry by the AFM tip geometry, it has to be corrected by the known S_{AFM Tip} to obtain the real stylus tip geometry, S_{Stylus Tip}.

After the stylus tip has been calibrated, it can be applied for traceable and accurate calibrations of vast micro- and nanostructures and surfaces. The contribution of the stylus tip geometry can be in turn corrected from the measurement results using the characterized S_{Stylus Tip}. With the traceability chain mentioned above, it is understandable that these measurements are thus ultimately traceable to the lattice constant of crystal silicon. This approach is referred to as a bottom-up approach, as the lattice constant of crystal silicon is typically (much) smaller than the size of micro- and nanostructures being measured, and smaller than the optical wavelength which is typically applied for the realization of the traceability chain of length metrology.

To understand the advantage of the proposed calibration concept, we do a thought experiment. Let’s consider two measurement scenarios: (i) measurement of a stylus tip (S_{Stylus Tip}) using an AFM tip (S_{AFM Tip}) by an AFM; (ii) measurement of an AFM tip using a stylus tip by a stylus profilometer, where the AFM tip acts as a tip characterizer. It can be easily understood that the measurement results of both scenarios, S_m, are the same from the morphological point of view, i.e.

\[
S_m = S_{Stylus Tip} \oplus S_{AFM Tip}
\]

where, \( \oplus \) denotes the morphological operator of dilation.

Figure 2 Concept of the new bottom-up traceability approach for dimensional metrology of nanostructures.
From equation (2) we can derive $S_{\text{stylus Tip}}$ from the measured $S_m$ and the known $S_{\text{AFM Tip}}$, thus the two methods seem to be equivalent. However, there are significant differences of the two approaches in practice. In stylus measurements a tiny structure like a sharp AFM tip will be easily damaged due to the large probing force of the stylus probe (in range of $\mu$N to mN). In contrast, the AFM can easily perform measurements with high resolution, (almost) non-destructively and in 3D, owing to its low tip-sample interaction force (nN or even below) and advanced measurement modes (e.g. intermittent-contact or noncontact). It can thus avoid the problems of classical tip characterizers as discussed in the introduction. Furthermore, as the AFM tip geometry is traceably calibrated and its contribution to the measurement result is corrected, it is capable of offering (much) higher measurement accuracy.

In practice, the prosed concept can also be applied in a simplified way. If the demanded calibration accuracy of the stylus geometry is low, the calibration step of the AFM tip characterization can be skipped. In such a case, the AFM tip can be regarded as infinitely sharp. Since the radius is typically a few nm for a new AFM tip, and 10…20 nm for a slightly worn tip, its measurement bias can be well estimated as a part of measurement uncertainty in low-demanding measurements.

3. Experimental realization

In this section we will introduce the performed experiments and obtained results in the realization of the concept introduced in section 2.

3.1. Characterization of the AFM tip

Characterization of the AFM tip is illustrated in figure 4. The measurement is performed by an AFM instrument developed by authors at the PTB, referred to as a "low-noise 3D-AFM". This AFM has been developed recently and has not been published yet, therefore a brief introduction is given here. The AFM works in the so-called sample-scanning principle, where a low-noise 6-axes piezo stage (type P-915K514 of the Physik Instrument GmbH) is applied for scanning samples in x-, y- and z-axes in measurements. The stage P-915K514 has a motion range of $12 \ \mu m \times 12 \ \mu m \times 10 \ \mu m$ ($x, y, z$) and applies capacitive sensors for measuring its motions in six degree-of-
freedoms (DOFs). The noise of the scanner is low, measured as a standard deviation of 0.12 nm, 0.11 nm and 0.12 nm for the motion of x-, y-, and z-axes, respectively, with a sampling rate of 2 kHz in closed loop. A special design feature of this low-noise 3D-AFM is that it combines three different measurement functions, i.e. CD-AFM, tilting AFM and normal AFM, in one device, thus offering great capabilities for 3D nanometrology. In addition, a special probing and measurement strategy referred to as the vector approaching probing (VAP) method developed at PTB \[42\] has also been realized in this AFM.

In this study, normal intermittent-contact AFM measurement mode is applied, thus the measurement can also be (conveniently) realized in commercial AFMs. An AFM probe type PPP-NCLR (Nanosensors\textsuperscript{TM}) is applied. The measurement is performed on a selected line feature of our internal CD standard type IVPS100-PTB over a measurement area of 0.3 \(\mu\)m \(\times\) 1 \(\mu\)m with 301 \(\times\) 8 pixels, i.e. a pixel distance of 1 nm/pixel along the fast scan axis. The VAP measurement mode is applied with a probing speed of 500 nm s\(^{-1}\) for preventing tip wear and tip breakage \[43\]. The measurement of one image takes about 7 min.

For clarity, a measured profile is depicted in red in figure 4(a) shown as raw data after 1st order levelling, which is typically applied to remove the contribution of sample tilting due to its mounting deviation. The pixels in the profile are not evenly distributed, which is due to the fact that the lateral pixel distance is kept constant (i.e. 1 nm pixel\(^{-1}\)) consequently the vertical distance of pixels strongly depends on the slope of the measured profile. In the figure, the calibrated feature geometry (width of 105.9 nm with a corner rounding of 6.0 nm) of the IVPS100-PTB determined by the bottom-up approach is also depicted.

With the measured AFM images and the reference feature geometry of the IVPS100-PTB, the AFM tip geometry can be evaluated by a software package developed by authors [41]. An evaluated 2D tip profile along the y-axis is illustrated in figure 4(b). Totally 14 evaluated tip profiles are plotted in red, where the variation span of profiles is about 1.7 nm, showing very good measurement repeatability. Their averaged profile is calculated as the final result as shown in black. The tip region can be fit by a circle using the least-squares method, where the tip radius is estimated as 18.5 nm.

Since the IVPS100-PTB uses line features for tip characterization, it can only determine one 2D profile of the tip in one sample orientation. To determine 3D geometry of the AFM tip, the IVPS100-PTB is rotated and measured in six different orientations. After performing the same measurement and data evaluation process as mentioned above, 2D profiles along
different orientation can be obtained. In figure 4(c) the 2D profiles of the tip along the x- and y- axis are compared. The two profiles have different shape, because the AFM tip manufactured by wet-etching process does not have an ideal conical shape. The 2D profiles measured at different orientations can be fitted and interpolated to obtain a 3D view of the tip geometry, as shown figure 4(d).

Table 1 lists the tip radius evaluated at the AFM tip apex along different directions. It can be seen that the radius may differ from 16.0 nm to 21.8 nm. As the AFM tip does not have an ideal sphere shape, the characterized 3D tip geometry shown in figure 4(d) rather than a fitted sphere is applied for correcting its contribution in the following measurements.

| $\phi$ (°) | 91 | 62 | 51 | 45 | 21 | 1 |
|-----------|----|----|----|----|----|---|
| r (nm)    | 18.5 | 15.8 | 16.1 | 16.0 | 18.6 | 21.8 |

3.2. Characterization of stylus tip geometry

The characterized AFM tip described above is then applied to calibrate a commercial stylus probe type RFTHB-50 (Mahr GmbH). The stylus has a nominal radius of 2 $\mu$m and a cone angle of 90°. The measurements are performed in the metrological large range atomic force microscope (Met. LR-AFM) developed by the authors [44]. The Met. LR-AFM is based on a high precision nano positioning stage, referred to as a nano positioning and measuring machine (NMM) [45] developed at TU-Ilmenau and manufactured by the SIOS Meßtechnik GmbH. This Met. LR-AFM, although still named as an “AFM”, may combine different probing sensors to the NMM. Consequently, it works as a metrological AFM when an AFM sensor head is used, and as a metrological stylus profilometer when a stylus probing head is applied. In this measurement, the stylus probe is applied as a sample specimen. To prevent the motion of the stylus tip during measurement, it is mechanically clamped. When the stylus tip is mounted, it points upwards (as illustrated in figure 5), so that it can be conveniently measured by the AFM tip as a “normal” microstructure. The position of the stylus tip apex was first determined before AFM area scans are taken. It can be easily done under the assistance of an optical microscope embedded in the AFM, and some trial profile measurements within a few minutes. In this study, we have measured the stylus tip over three different area sizes with different pixel densities: (i) $2.995 \times 2.995 \text{ } \mu\text{m}^2$ with $600 \times 600$ pixels; (ii) $3.995 \times 3.995 \text{ } \mu\text{m}^2$ with $800 \times 800$ pixels; and (iii) $5.99 \times 5.99 \text{ } \mu\text{m}^2$ with $600 \times 600$ pixels. Each measurement is repeated twice. The scan speed is again set low, i.e. $1 \mu\text{m} \text{ s}^{-1}$, to protect tip wear and tip breakage. One image takes about 1.0 ~ 1.5 h. The measurement time can be shortened if the pixel density is reduced.

To evaluate the geometry of the stylus tip, the measured AFM image is corrected by the characterized tip geometry using the same set of software package developed in [41]. The obtained geometry of the stylus tip is shown in figure 5(a). To illustrate the measurement quality, the same 2D profiles obtained in 6 AFM images (3 areas $\times$ 2 measurement repeats pro area) are depicted in red in figure 5(b). The averaged profile of 6 profiles is calculated as the result of the stylus tip geometry and depicted in black. The deviation of 6 individual profiles from the averaged profile is shown in figure 5(c). The deviation is well within $\pm$ 5 nm, indicating very good measurement repeatability.

To estimate the radius of the stylus tip, the data points at the cap region (with a depth up to 500 nm as marked in the figure 5(b)) of the evaluated tip geometry is fitted to a sphere. The obtained radius is 1.727 $\mu$m. It is significantly smaller than the nominal value of 2 $\mu$m, indicating the need of calibration of the stylus tip. The measurement standard deviation of six repeat measurements reaches 0.007 $\mu$m, indicating high measurement precision of the proposed method. It is to be mentioned that stylus tip geometry may significantly deviates from an ideal sphere shape, as shown in an insert figure of figure 5(b) as a zoom-in view of its 2D profile. Thus, to well correct the contribution of the stylus tip geometry in its application measurements, its measured 3D shape of the stylus tip geometry rather than its fitted sphere is applied.

4. Application

After the tip geometry of the stylus has been calibrated as mentioned above, it can be applied in accurate calibrations of vast microstructures and surfaces. Here we demonstrate measurements of microspheres as an example.

The NMM combined with the calibrated stylus probe is applied as a measurement tool for this experiment. Before the measurement, the mechanical clamp which was previously introduced to the stylus probe is removed. During the measurement, the stylus probe works as a zero detector, i.e. the sample is moved up and down during scanning so that the output of the stylus probe is kept constant.

Three sapphire spheres denoted as D300, D500 and D1000 with nominal diameters of 300 $\mu$m, 500 $\mu$m and 1000 $\mu$m, respectively, are measured. A radial scanning strategy is applied in the measurement. Using this scan strategy, profiles are taken in a radial direction through the centre of the pattern. Such a measurement strategy was first designed for measuring rotation symmetrical structures, such as Rockwell indenters [46]. The radial scan length is 200 $\mu$m, 360 $\mu$m and 720 $\mu$m for the measurements of D300, D500 and D1000, respectively. Thus, the measurement
profile covers a surface slope range of over ±45°. The scanning speed is set as 20 μm s⁻¹. Totally 30 profiles are measured in one image with an angular resolution of 6°. One image takes 22 to 47 min.

A photo of the stylus tip when it is measuring the sphere D500 is shown in figure 6(a), as acquired from the CCD camera of the optical vision microscope monitoring the measurement. A measured image of the D500 is illustrated in figure 6(b), shown as the raw data before tip correction. The correction of the contribution of stylus tip geometry is shown in figure 6(c), where the original measured profile is shown in black and corrected profile in red. The shape of the stylus in the figure is for schematic illustration purpose only.

To illustrate the measurement performance, the same 2D profile of five repeated measurements of the sphere D500 are plotted in figures 7(a), (c), (e), respectively. After calculating the averaged profile of the five repeated profiles, the residual deviation between the measured and averaged profiles are plotted in figures 7(b), (d) and (f), respectively. Interestingly, it can be seen that the measurement repeatability changes with respect to the surface slope, θ, of the measurement point. When the tip is probing at the top of the sphere (i.e. θ = 0°), the measurement has the best measurement repeatability of about ±5 nm (p-v). However, the repeatability is worsened to about ±20 nm (p-v) when the surface slope is increased to θ = ±45°. This behavior may be attributed to the measurement principle of the stylus probe, which is a vertical probing sensor only. When it probes an inclined surface, the lateral component of the probing force may lead to measurement instability.

The tip corrected 3D data sets of the microspheres are then fitted to an ideal sphere shape using the least-squares method to calculate their radii. The results are listed in table 2. For three spheres measured, the evaluated mean radius is 150.8596 μm, 248.9966 μm and 499.9548 μm, respectively. They are quite close to their nominal values. The standard deviation of five measurements reaches 0.5 nm, 0.5 nm and 0.6 nm,
indicating excellent measurement repeatability of the metrology tool. However, it should be mentioned that the measurement repeatability stated above does not stand for the measurement uncertainty. To rigorously confirm its measurement accuracy, thorough measurement uncertainty analysis needs to be carried out.

Figure 6. Measured result of the sphere D500 having a nominal diameter of 500 μm, shown as (a) a photo of the stylus tip and the sphere; (b) measured raw data in 3D; (c) a cross-sectional profile before and after the geometry of stylus tip is corrected.

Figure 7. Repeatability of measured 2D-profiles along the y direction of three sphere samples: D300 (a), D500 (c) and D1000 (e). The measurements are repeated 5 times for each sample. The right panel shows the residuals of all 5 profiles subtracted by the averaged profile for each sample, D300 (b), D500 (d) and D1000 (f), respectively.
and a comparison with other methods/tools for the verification of estimated measurement uncertainty is needed in the future.

Although a measurement example of microspheres is demonstrated in this section, the profilometry are widely applied in vast research and industries for measurements of form, contour and surface roughness. After the stylus tip geometry has been accurately characterized, its contribution can either be corrected to enhance the measurement accuracy or be applied to estimate measurement uncertainty. However, one important issue that should be stressed is, that the morphological operator “dilation” and “erosion”, which mathematically underpins the function of tip geometry in measurements, is not a pair of reversible functions. Consequently, the correction of tip contribution using “erosion” cannot guarantee that its contribution is fully corrected. Let’s take a simple example: if a kind of dense surface structures cannot be probed in stylus measurements due to the limit of the probe size, the information of the dense structures is lost in the raw data of measured profile. Such lost information cannot be recovered even after the correction of the tip geometry. Another important issue to be addressed concerns the aspect of error propagation of the tip geometry to measurement applications. Generally, an uncertainty of the stylus tip radius/geometry results in an uncertainty of the measured topography. In a study performed by Krüger-Sehm et al.\cite{47}, for instance, the error propagation of the uncertainty of stylus tip radius, $\sigma(r_i)$, on the uncertainty of topography points after $\lambda s$-filtering is analyzed as:

$$\sigma(r_i) = \sqrt{\frac{1}{3} \frac{20 \text{ nm}}{\mu m} \cdot \sigma(r_i)}$$

Table 2. Radius calculation result.

| Repeat | D300   | D500   | D1000  |
|--------|--------|--------|--------|
| 1      | 150.8600 | 248.9967 | 499.9540 |
| 2      | 150.8601 | 248.9971 | 499.9544 |
| 3      | 150.8595 | 248.9969 | 499.9550 |
| 4      | 150.8593 | 248.9961 | 499.9552 |
| 5      | 150.8590 | 248.9960 | 499.9554 |
| Mean   | 150.8596 | 248.9966 | 499.9548 |
| Std. dev. | 0.0005 | 0.0005 | 0.0006 |  

5. Conclusion

Stylus profilometry is a kind of widely applied technique for the measurement of form, contour and roughness of surfaces. The geometry of a stylus tip is an important influence factor of measurements, as the measured profile is the dilated result of surface features by the tip geometry. Thus, the tip geometry sets a bandwidth limit of the profilometer in roughness measurements and biases the measurement results in form and contour metrology. To correct its contribution, the geometry of the stylus tip needs to be characterized accurately.

This paper introduces a novel method for accurate and traceable calibration of stylus tip geometry. The method is based on several steps. Firstly, the geometry of an AFM tip is calibrated to a kind of line width standard, e.g. the CD standard type of IVPS100-PTB whose geometry is traceable calibrated to the lattice constant of crystal silicon. The calibrated AFM tip is then applied to scan the geometry of the stylus tip, where the stylus probe is clamped and acts as a sample specimen. The geometry of the stylus tip can be accurately determined after correcting the contribution of the AFM tip geometry from the obtained AFM image. After being calibrated, the stylus tip can then be applied in measurements of vast microstructures and surfaces, where the measurement results can be corrected again using the calibrated stylus tip geometry. Thus, with the unbroken measurement chain described above, the stylus tip geometry and its further measurement results are finally traceable to the lattice constant of crystal silicon.

Detailed experimental examples are introduced in the paper illustrating the calibration of the AFM tip geometry and of the stylus tip geometry. For instance, for the given stylus type RFTHB-50 in this study, the stylus tip radius is measured as 1.72 $\mu m$. It is significantly smaller than its nominal value of 2 $\mu m$, indicating the need of calibration. The application of calibrated stylus tip is demonstrated in measurements of microspheres. For three spheres with nominal radii of 150 $\mu m$, 250 $\mu m$ and 500 $\mu m$, their radii are measured as 150.8596 $\mu m$, 248.9966 $\mu m$ and 499.9548 $\mu m$, respectively. The standard deviation of five repeat measurements reaches 0.5 nm, 0.5 nm and 0.6 nm for three microspheres, respectively, indicating excellent measurement repeatability of the metrology tool.

Compared to the conventional characterization method using e.g. a kind of a tip characterizer, the proposed novel method has several advantages:

(i) It avoids the damage risk of sharp edges of tip characterizers;

(ii) It is capable of directly characterizing 3D geometry of stylus tip;
(iii) It avoids the ambiguity in determining profile segments in reconstructing tip geometry (figure 1);

(iv) It can be achieved with high accuracy and is traceable to a nature constant—the lattice constant of crystal silicon.

Finally, we suggest that a proper tip characterization method should be selected based on the metrology demands in practice. For applications where measurement requirements are low, for instance, the measurement uncertainty of tip radius in the level of hundreds of nm or even higher, the conventional characterization method using e.g. a razor blade could be a simple and convenient solution; For applications where the measurement uncertainty is at the level of tens of nm, the proposed method can be applied with the calibration step of the AFM tip characterization being skipped. In such a case, the AFM tip can be regarded as infinitely sharp. Since the radius is typically a few nm for a new AFM tip, and 10…20 nm for a slightly worn tip, its measurement bias can be well estimated as a part of measurement uncertainty. As the AFM tip characterization needs significant measurement efforts, this simplification can greatly reduce the application efforts of the proposed method. Finally, for applications where the best measurement accuracy is aimed, the full calibration strategy proposed in this paper can be applied.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

ORCID iDs

Gaoliang Dai https://orcid.org/0000-0002-1611-0074
Johannes Degenhardt https://orcid.org/0000-0001-5508-5556

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