Calibration of stylus profilometers using standards calibrated by metrological SFMs

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Abstract. A method for calibrating stylus profilometers on the basis of metrological scanning force microscopes (SFMs) is proposed. Two kinds of such calibrations are presented. First, step height standards set (feature height from 7 nm up to 1000 nm), calibrated by metrological SFMs with expanded measurement uncertainties (k=2) of only 1 to 1.5 nm, are applied to calibrate the z-axis scaling factor of profilometers. Second, roughness standards (ISO 5436-1 type D1), calibrated by a metrological large range SFM, are used to achieve dynamic calibrations of profilometers. The proposed method significantly improves the performance of the calibrated profilometers. For instance, the expanded measurement uncertainty (k=2) of a PTB profilometer is reduced from 11.52 nm to 6.26 nm on Pt (total height of profile), and 7.57 nm to 3.25 nm on D (depth of the groove) calibrations of a groove sample (wide groove with flat bottom, nominal Pt = 2.7µm).

1. Introduction

Stylus profilometers are widely used for measurements of roughness and surface topography in research and industry. For achieving traceable measurements, these profilometers are usually calibrated in the xy plane and along the z-axis using transfer standards. Prior to the usage, these transfer standards have to be calibrated, e.g. by metrological profilometers or optical interference microscopes. Presently, the expanded uncertainties $U$ of the calibration standards are mostly at a level of several nanometres (ca. 3 - 5 nm) [1]. Since good stylus profilometers are very stable and reproducible, the propagated errors from the calibration standards have been the dominant error sources of profilometers.

To overcome this lack, a method for calibrating stylus profilometers using standards calibrated by metrological scanning force microscopes (SFMs) is presented. As the metrological SFMs have expanded measurement uncertainties of only 1 to 1.5 nm for step height measurements up to 2 µm which are significantly smaller than that of other kinds of instruments, this method largely reduces the uncertainties of the stylus profilometers. Besides, the high reliabilities of these metrological SFMs have been confirmed in several international comparisons, e.g. NANO 2 [1].

In this study, two kinds of calibrations are presented. First, a set of step height standards with nominal feature heights from 7 nm to 1000 nm has been calibrated by the metrological SFMs. The standard set is then applied to calibrate the scaling factor of a profilometer along the z-axis. Second, a roughness standard (ISO 5436-1 type D1), calibrated using a large range SFM over twelve different
positions with profile length of 5.6 mm, has been employed to perform dynamic calibrations of a profilometer.

2. Metrological SFMs
SFMs are a new class of universal two and half dimensional coordinate measurement instruments. They sense sample surfaces using very tiny SFM tips which may have a radius of 10 nm or even below, and with low forces (a few nanoNewtons). The SFM tip is in contact, sometimes in intermittent-contact or noncontact, to the surface during measurements. The image is formed by monitoring the deflection of the SFM cantilever as the sample is scanned beneath the tip. Metrological SFMs are specially designed SFMs equipped with laser interferometers for position control and measurement, and are thus capable of performing quantitative traceable dimensional measurements.

Three metrological SFMs referred as Veritekt B, Veritekt C and a large range SFM (LR-SFM), have been set up at PTB. The LR-SFM has a capable measurement volume of 25 mm × 25 mm × 5 mm, whereas Veritekt B and C have a capable measurement volume of 70 µm × 15 µm × 15 µm. The operation principles and instrumentations of these instruments have been published elsewhere [2, 3].

3. Calibration of scaling factor using nano step height standards
Standards with nominal step heights from 7 nm to 1000 nm have been applied for the calibration of z-axis scaling factor of a profilometer “Nanostep” [4]. The standards are manufactured with a SiO₂ layer on a silicon substrate using optical lithography, and their surfaces are coated with a chromium layer. In this study, both the Veritekt C and the metrological LR-SFM are applied to calibrated the standards set.

In order to achieve a better calibration performance, the same defined reference area of each standard is measured by both the metrological SFMs and the to-be-calibrated profilometer. Such reference area can be easily located during measurements with the help of a specially designed “island” feature on the layout of the standard. In such a way, the calibration error caused by the non-uniformity of the standards in step-height is expected to be less than 0.1 nm. Measurement data of both methods are evaluated using the same method defined in ISO 5436 line by line. As an example, typical measurement results taken on a standard with nominal step height of 1000 nm are shown in figure 1.

The calibration is carried out in several steps. Firstly, each standard is calibrated using the metrological SFMs. The obtained step height values are given in the row marked as SH₅F₅ in table 1. Secondly, the standard set is transferred to and then measured by the profilometer “Nanostep” with its z-axis scaling factor m set as the default value (m=1). The result is shown in the row marked as SH₅N₅. Thirdly, the values of SH₅F₅ is plotted versus the values of SH₅N₅. The plotted curve is fit to a linear function SH₅F₅ = m*SH₅N₅, and m=1.00688 is obtained. Finally, the value m representing the scaling
factor of the z-axis of the profilometer can be set to the profilometer. In order to verify the calibration procedure, a set of new step height results \( SH_{NSK} \), standing for the correct step height measured by the profilometer after its calibrated scaling factor \( m \) being applied, is calculated. The residual calibration error \( \Delta SH \), calculated as \( SH_{SFM} - SH_{NSK} \), is less than 1 nm for all standards, as listed in Table 1. This fact not only implies the good linearity of both instruments, but also indicates the good performance of the calibration method.

Table 1. Calibration results of the stylus profilometer “Nanostep” using step height standard sets. The expanded uncertainties of the SFM results given in the table are calculated with \( k=2 \).

| Sample | SH8 (nm) | SH20 (nm) | SH70 (nm) | SH300 (nm) | SH800 (nm) | SH1000 (nm) |
|--------|----------|-----------|-----------|------------|------------|------------|
| \( SH_{SFM} \) | 6.9±1.0   | 21.4±1.0  | 69.2±1.1  | 289.9±1.2  | 780.7±1.4  | 1014.3±1.4 |
| \( SH_{NS1} (m = 1) \) | 6.7       | 20.4      | 68.2      | 287.6      | 774.6      | 1008.1     |
| \( SH_{NSK} (m = 1.00688) \) | 6.75      | 20.54     | 68.67     | 289.58     | 779.93     | 1015.04    |
| \( \Delta SH \) | 0.15      | 0.86      | 0.53      | 0.32       | 0.77       | -0.74      |

Using the proposed calibration method, the propagated errors from the calibration standards can be significantly reduced, and consequently the measurement accuracy of stylus profilometers can be improved. For instance, the uncertainty budgets of the calibrated profilometer “Nanostep” are compared in Table 2 before and after the proposed calibration method has been undertaken, indicating a reduction of the measurement uncertainty of about 50%.

Table 2. Comparison of measurement uncertainty budgets of the profilometer “Nanostep” on Pt (total height of profile) and D (depth of the groove) calibrations of a groove sample without filtering before (shown as \( \bar{U}_i^2 \)) and after (shown as \( \bar{U}'_i^2 \)) the proposed method has been undertaken. In the calculation, the nominal Pt value is 2.7 \( \mu \)m. The uncertainty (\( k = 2 \)) of the calibration standard is 7 nm and 1.5 nm in two cases. The roughness of the reference plane Pt is 8 nm.

| Error source | Degree of freedom \( v_i \) | Pt calibration | D calibration |
|--------------|----------------------------|----------------|--------------|
|              | \( \bar{U}_i^2 \), (nm²)   | \( \bar{U}_i^2 \), (nm²)   | \( \bar{U}_i^2 \), (nm²)   | \( \bar{U}_i^2 \), (nm²)   |
| Uncertainty of the standard | 50              | 1.23E+1 5.63E-1 | 1.23E+1 5.63E-1 |
| Difference in the calibration position | 50              | 3.33E-3 3.33E-3 | 3.33E-3 3.33E-3 |
| Measurement reproducibility | 5               | 8.00E-1 8.00E-1 | 2.00E-1 2.00E-1 |
| Topography of the sample | 5               | 4.50E-1 4.50E-1 | 4.50E-1 4.50E-1 |
| Non-uniformity of the sample | 50              | 3.33E-3 3.33E-3 | 3.33E-3 3.33E-3 |
| Error of guidance system | 50              | 8.33E-2 8.33E-2 | 8.33E-2 8.33E-2 |
| Instrument noise | 5               | 3.33E-1 3.33E-1 | 4.44E-3 4.44E-3 |
| Leveling of the profile | 200             | -           | 1.33E0 1.33E0 |
| Uncertainty of a profile point, \( u^2(zg) \) | -               | 1.39E+1 2.24E0 | -           | -           |
| Uncertainty, \( u^2 \) | -               | 3.32E+1 9.81E0 | 1.44E+1 2.63E0 |
| Expanded uncertainty \( U (k = 2) \) | -               | 11.52 nm 6.26 nm | 7.57 nm 3.25 nm |

4. Dynamic calibration of stylus profilometer using roughness standard

In order to achieve dynamic calibrations of stylus profilometers, roughness standards of ISO 5436-1 type D1 and D2 are usually applied. In this study, a type D1 roughness standard calibrated by the metrological LR-SFM is applied to calibrate a metrological stylus profilometer. It is worth being mentioned that the used metrological profilometer is the routine device for the calibration of roughness standards at PTB.

In [5] we have already shown that our metrological LR-SFM is capable of quantitative and accurate measurements of micro roughness, however, the standard was measured only at one position. In this paper, we carried out the investigation more carefully by performing measurements at 12 different
positions with profile length of 5.6 mm and using a pixel distance of 100 nm. Each profile has a
different start position along the x-axis as shown in figure 2. The averaged roughness values at 12
different positions are applied as the final calibration result. The reason of using such calibration
strategy is to eliminate the influence of the measurement positions on the calibration results. As an
example, profiles taken on the position 1 (start position: 0 mm) of the roughness standard both by the
LR-SFM and the stylus profilometers are depicted in figure 3. Both profiles have very similar
roughness structures.

Table 3. Averaged roughness values measured by the metrological SFM and the stylus profilometer.
The expanded uncertainties of the stylus results given are calculated with $k=2$.  

| Parameters | Rq, nm | Ra, nm | Rz, nm | Rp, nm | Rv, nm |
|------------|--------|--------|--------|--------|--------|
| LR-SFM     | 222.27 | 176.72 | 1198.82| 439.92 | 758.91 |
| Stylus profilometer | 221.01±13.0 | 175.35±5.3 | 1211.93±60.0 | 440.16±13.0 | 771.76±22.8 |
| Difference | 1.26   | 1.37   | -13.11 | -0.24  | -12.85 |

Both data sets are evaluated using the PTB reference software [6] under the same filtering
conditions ($\lambda_c = 800 \, \mu\text{m}$, $\lambda_s = 2.5 \, \mu\text{m}$). The final roughness parameters listed in table 3 show an
excellent agreement between the stylus and SFM results, indicating the calibration factor of the
metrological SPM was set correctly. The residual deviation are due to factors like the tip shape
difference [5], the deviation of the measurement positions, etc.

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