TURNING SCANNING PROBE MICROSCOPY INTO A MORE QUANTITATIVE METHOD

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Scanning Probe Microscopy (SPM) is used today not only in research and development but increasingly also in many fields of industrial fabrication and inspection. High-technologies such as semiconductor fabrication and nanotechnology attach great importance to the quantitative information these instruments provide. Thus, SPM is widely regarded as one the key measurement methods for future technologies. Consequently, National Metrology Institutes (NMI) face the challenge to provide measurement technology, transfer standards and written documentary standards or guidelines on SPM characterization & calibration methods in order to enable the SPM user to calibrate his instrument traceable to the SI unit metre. Furthermore, when it comes to the further reduction of measurement uncertainty, intensive research is required to understand the interaction between probe and sample on the nanometre and sub-nanometre level. This report gives an overview of several SPM-related research activities at PTB.

1. Introduction

In the course of the evolution of many high-technologies such as microelectronics, micromechanics and also biotechnology, the size of technical structures is being decreased continuously. In many technical applications, the feature size has already reached the lower sub-micron scale and often needs to be measured with an uncertainty in the nanometer range. Scanning Probe Microscopes (SPMs) are therefore increasingly used today as quantitative measurement instruments.

SPMs are serially operating measuring devices which use a probe of adequate fineness to trace the surface of the object to be measured exploiting a local physical interaction between probe and sample (such as the quantum-mechanical tunnel effect, interatomic or intermolecular forces, evanescent modes of the electromagnetic field). Depending on the kind of interaction exploited, SPM allows to obtain information on different physical properties of the sample surface. The most common method is Scanning Force Microscopy (SFM), relying on probe-sample forces, to trace the *topography* of the object. Scanning Tunnelling Microscopy (STM) allows to study the density of electronic states; it is therefore restricted to conductive samples and probes and thus of limited relevance to the broader industrial application. For the storage media industry, Magnetic Force Microscopy (MFM) constitutes an indispensable high-resolution method to probe the surface magnetisation. While there is increasing cooperation with the groups working on MFM, this report will focus on the purely dimensional topographic aspects of SPM, as these are most important to ultra-precision industries [1].

2. Traceability for SPM

In order to ensure worldwide comparability of SPM measurement results, traceability to the SI unit metre needs to be established for this measurement method in a similar way as already routinely practised for methods operating on a larger scale, e. g. coordinate measuring instruments or profilometers. Such a traceability chain as realized by PTB (Fig. 1) requires the following elements:

- very stable high-accuracy SPM instrumentation (usually at NMIs) with direct traceability to the SI unit by built-in laserinterferometers that monitor the translation of the SPM stage during measurement (so-called "**Metrological SPM**"); at PTB, the lasers used for interferometry are calibrated to an I_2-stabilized laser whose frequency was compared to the Caesium clocks. In this way, the metre definition is directly applied [2].
- Certified calibration of **physical transfer standards** either by metrological SPMs or - as far as applicable - by other directly traceable instrumentation such as diffractometry (lateral standards) or interference microscopy (step height and flatness standards).
- Application of the certified standards for the **calibration of the SPM by the user** "outside" in industry, in other reasearch institutes, in measurement service companies, etc. A prerequisite for the correct execution of the calibration process are written documentary standards or **guidelines** on SPM characterization & calibration methods.

In the following, a set of current PTB research activities serves to illustrate these elements.

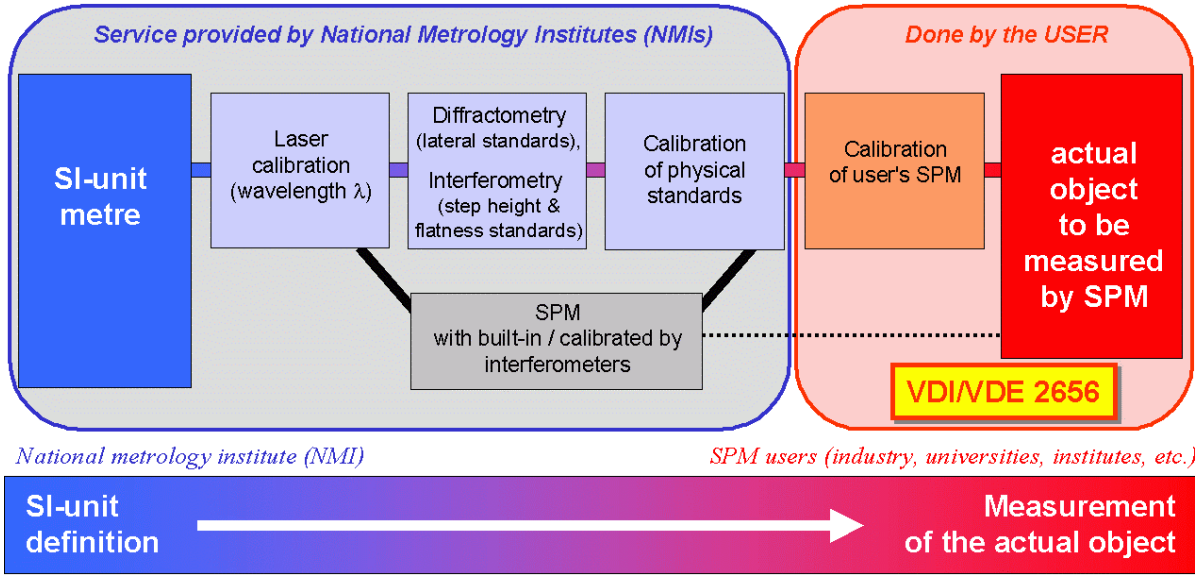

Fig. 1 Traceability chain for SPM

3. Metrological SPMs - Metrological Large-Range SPM at PTB

Several NMIs have developed special Metrological SPMs in the past few years. The typical scan range is some 10 micrometres in both lateral directions and a few micrometres in the vertical direction. This is sufficient for the calibration of most SPM-relevant standards.
The Metrological Large-Range SPM (Met.LR-SPM) realized at PTB [3] on the basis of the NanoMeasuringMachine (SIOS GmbH, Ilmenau, Germany) allows an interferometrically controlled translation as large as 25 mm x 25 mm x 5 mm; in conjunction with the flexible programming of the scan process and data acquisition with PTB-made software, larger gratings and extended height steps e. g. of several 100 µm in width can be calibrated mostly with SPM-typical high resolution. Consequently, standards also for methods other than SPM can be certified with so-far unsurpassed accuracy. The large scan range pays out especially at larger gratings typically used as lateral standards: Due to the averaging over many periods and the interferometrically determined positions, uncertainties as low as some 10 picometres are reached for high-quality standards [4][5]. In addition to this, the uniformity of the grating can be investigated easily; this is particularly important if jumps occur in the grating (Fig. 2) [6].
Several European and international key comparisons ("Euromet707" and "Nano2" on step height standards; "Nano1" and "Nano4" on lateral standards) served to compare the metrological or other traceable high-accuracy SPMs at NMIs to each other, and partly also to other measurement instruments: It could be shown that the SPM results agree well to those of e. g. high-resolution profilometry or interference microscopy, with SPM uncertainties typically being smaller.

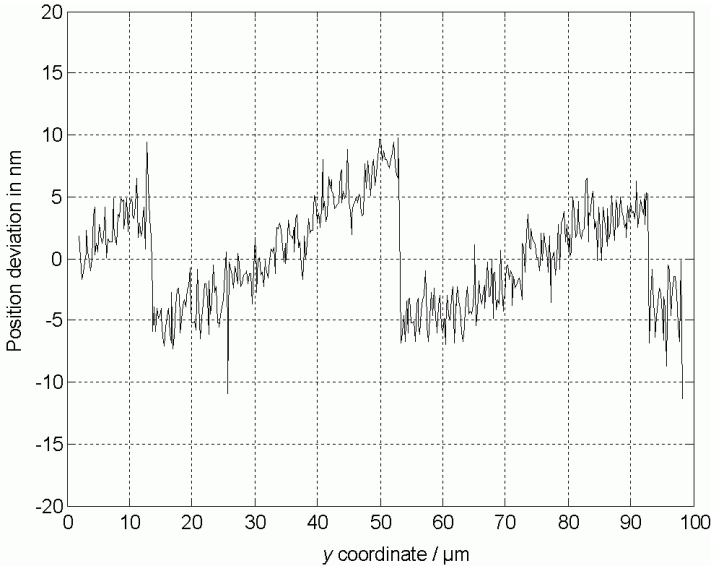

Fig. 2

Example of a uniformity investigation (by Met.LR-SPM at PTB) of a pit grating used as lateral SPM standard: deviation of the individual pit position from the ideal (perfect) grating. The plot reveals jumps in the gratings at *y* positions 14 µm, 53 µm, and 93 µm, i. e. the distance between these pairs of adjacent rows of pits is ~10 nm / ~13 nm / ~8 nm smaller than usual on this grating.

4. Transfer standards for SPM - novel 3D standards and nano-geometry standards

A number of transfer standards suited for SPMs has already been developed and are commercially available. Most of them are either lateral standards based on a homogeneous 1D or 2D grating (Fig. 2), step height standards with one or several steps of a discrete height, or flatness standards. With these types of standards, the calibration of the three axes and of the out-of-plane motion can be accomplished. However, SPM calibration faces several other challenges as well that cannot - or only with an extraordinary effort - be addressed with these standards alone.

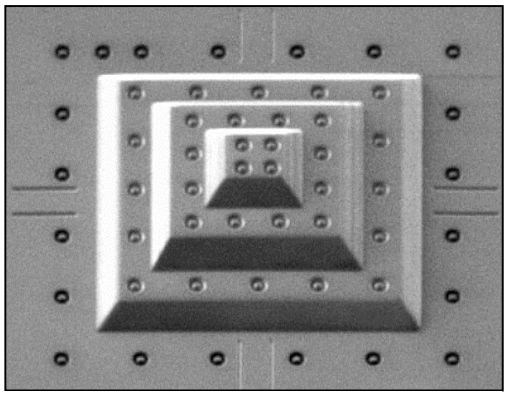

Fig. 3 Scanning Electron Micrograph (approx. 30 µm x 30 µm) of a 3-step pyramid (height ~3.6 µm) decorated with nanomarkers for landmark-based 3D calibration of SPM and other high-resolution instruments

One of these challenges is coupling between the three axes *x*, *y* and *z*. While 2D lateral standards allow to determine the coupling between *x* & *y*, the cross-talk of the *z*-axis on *x* any *y* can hardly be determined with these established standards. A novel 3D standard has therefore been developed by BAM and PTB which allows a complete 3D calibration with just one type of standard, i. e. the coupling of all three axes can be determined easily and automatically [7]. The 3 D standards consist of simple geometric bodies (e. g. pyramids with different height steps, Fig. 3) on the substrate. Special marks are written on the bodies and the remaining substrate surface, so-called nanomarkers. Contrary to the established lateral & step height standards, these nanomarkers neither need to be written in a uniform pitch pattern nor on discrete height levels, as the individual three spatial coordinates of each nanomarker are used (so-called "landmark"-based calibration). Besides SFM, these 3D standards are well suited for stereogrammetric Scanning Electron Microscopy (SEM) and Confocal Laser Scanning Microscopy (CLSM) and thus allow correlative analyis of different instruments.

Another challenge is the determination of nanoroughness and nanogeometry. While roughness measurements e. g. by profilometry are standardized already for a long time, appropriate nanostandards for SPM are currently under development at PTB. In a joint project with IPM-RAS, nanogeometry roughness standards based on Ge/Si nanoislands are under investigation. Preliminary extensive studies have shown that these islands are very homogeneous in size and that the roughness across the sample surface is isotropic in all directions, i. e. there is no distinguishable angle dependence. The height of the islands can be reproducibly varied from a few to some ten nanometres by control of the growth parameters.

These Ge/Si nanoislands are also investigated as tip characterizers at PTB. Due to their homogeneity, they help to determine the tip shape. A great advantage compared e. g. to Au

nanospheres adsorbed on a substrate is that these Ge/Si islands are firmly connected with the crystallographic lattice of the substrate. This allows to fabricate the negative of their surface features by hot embossing into plastics, as they stand the high pressure and temperature exerted on the master. In this way, even multiple negative copies of the same master can be created, and positive master and negative copy can be used as complimentary reference samples for tip shape characterization as well as complimentary roughness determination (Fig. 4). Current investigations focus on the similarity of master and copy.

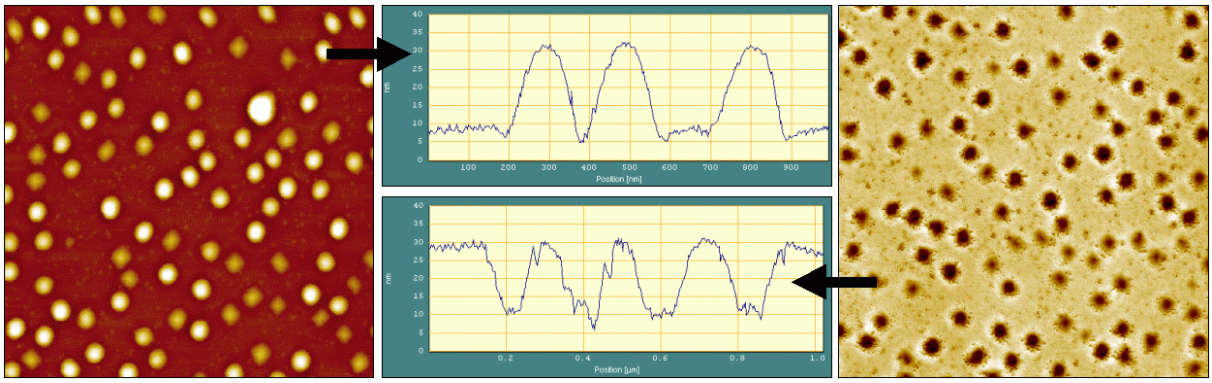

Fig. 4 Ge/Si islands on Si substrate fabricated by IPM-RAS. *Left image:* SFM image of the original, *right image:* SFM image of the replica (negative copy) in plastics. Size of both SFM images: 3 µm x 3 µm, with the same height colour coding. *Centre:* Two cross-sectional profiles of length ~1 µm through 3 islands (top) and 4 indents left by islands (bottom), height scale of both diagrams is 40 nm. Hot embossing courtesy of M. Rahlves, IMR Universität Hannover

5. Development of guidelines and international standards of SPM calibration

While detailed knowledge of the standards' properties is a prerequisite for their practical application, the calibration procedure itself also deserves careful consideration [8]. Up to now, there is no internationally accepted guideline on how to perform SPM calibrations.
In an attempt to fill this gap, an SPM calibration guideline has been drafted by a committee of VDI (Verein Deutscher Ingenieure, the Association of German Engineers) chaired by L. Koenders and T. Dziomba of PTB. This guideline VDI/VDE 2656 [9] has been released in preliminary version in November 2006 and is currently under review for the final version.
As SPMs are highly sensitive high-resolution instruments, they demand a verification & calibration strategy quite different from those already established for instruments operating at larger scales. In VDI/VDE 2656, much space is therefore given to an illustration of instrument characterization, followed by instructions for the out-of-plane calibration of the scanner's *x*-*y* movement, the calibration of the lateral axes and of the vertical axis. The use of 3D standards is explained as additional or alternative calibration method.
The draft of VDI/VDE 2656 serves as basis for considerations on dimensional SPM standardization within ISO TC 201 SC 9.

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