Part I Instrumentation – Overview I ____

1

1

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Abstract

1

An overview of PTB's activities in the field of dimensional nanometrology using scanning probe microscopes (SPMs) is presented. The chapter is divided into two parts: the development of (1) high-resolution probing systems and (2) complete SPM metrology systems. The subject of SPM-probing system design comprises, among other things, the concept of the "sensor objective" to combine conventional microscopy with scanning probe techniques. In the field of complete metrological SPM systems, the measuring properties of one of the existing SPM metrology systems have been significantly improved by including laser interferometers directly into the position control loop and by a clear reduction of the nonlinearity of the interference signals. In addition, the application spectrum of metrological SPM has been considerably extended by the establishment of an SPM system with a measuring volume of 25 mm \times 25 mm \times 5 mm.

1.1 Introduction

In many fields of material sciences, biology, and medicine, conventional scanning probe microscopes (SPMs) serve to visualize small structures with dimensions down to atoms and molecules as well as to characterize object-specific properties (magnetism, friction, thermal conductivity, and the like). For a large part of the investigations, the image information obtained with the SPM is completely sufficient for the qualitative investigation of the sample. Because of their high spatial resolution, use of these microscopes is also of great interest for metrological applications. This is why the PTB has begun using SPMs in dimensional metrology as one of the first national metrology institutes [1, 2].

A fundamental requirement for precise length measurements is, however, the addition of a length measuring system to the microscope scanning system. For this purpose, the piezo actuators that serve for positioning and scanning of sam-

ple or measuring head, are in many cases position-controlled via additional sensors (strain gauges, capacitive or inductive sensors) by which disadvantages of piezo elements, such as hysteresis or creeping, are compensated [3–10]. The use of these additional sensor systems does not release the user from performing regular calibrations. In the majority of cases, the SPM is calibrated with the aid of special standards with microstructures of defined geometry. Detailed information on such calibration standards can be found in another article of this edition [11]. The disadvantage of discrete calibration via standards is, however, that linearization of the positioning measuring systems is based on a few reference points only which are given by the material measure. This leads to higher calibration effort when objects of different height or structure periods are to be measured.

This is why users with high demands on the uncertainty of SPM measurements have in the past few years proceeded to equip the individual axes of the positioning system with laser interferometers. This allows the positioning values to be continuously traced back to the wavelength of the laser light and thus to the SI unit "meter". The fundamental idea is to treat the SPM like a miniaturized three-coordinate measuring machine and to correct its metrological properties with the device's control software.

As in the case of coordinate measuring machines, the SPM measuring systems can be divided into probing system and positioning unit. The structure of the present chapter reflects this aspect. The first part describes the PTB activities in the development of high-resolution probing systems based on scanning probe microscope techniques. The second part deals with precise positioning units and with the complete SPM measuring and calibration devices that are available at PTB.

1.2

High-Resolution Probing Systems

PTB's development of probing systems based on SPMs is aimed at constructing and optimizing these measuring heads for use in dimensional nanometrology. Needless to say that the sensor systems described cannot only be used for metrological applications, but are of general interest for scanning probe microscopy and coordinate measuring techniques.

The scanning force microscopes (SFMs) are those of the family of SPMs that are of special importance for dimensional metrology. This is mainly due to the fact that their use is not limited to conductive surfaces as it is, for example, the case for scanning tunnel microscopes. The design principle of an SFM is shown in Figure 1.1. In this case, the deflection detection system of the cantilever moving relative to the surface is based on an optical beam deflection principle, thus keeping the cantilever with the integrated measuring tip in a constant distance to the surface. The sample is then investigated line by line, and the profiles are subsequently composed in a computer to form an image.

1.2 High-Resolution Probing Systems 5



In addition to the properties important from the viewpoint of metrology such as stability, sensitivity, and noise behavior, different other aspects have been incorporated into PTB's device development:

- Combination of the SPM measuring heads with optical microscopes: here, the optical function extends from visualization to quantitative dimensional or analytical methods.
- The use of different detection principles: the movement and position of the measuring tip is measured by an external optical procedure or via an intrinsic electrical measuring principle.
- The use of different measuring tip materials: in recent developments, special diamond tips are used in addition to silicon and silicon nitride tips.

1.2.1

Sensor Objective with Beam Deflection Detection

As the name already suggests, the concept of the so-called sensor objective directly takes up the combination of microscope objective and sensor, the sensor in this case working as a scanning probe microscope. The special feature of this sensor head development is that existing optical standard microscopes are used as a basis: because of the compact geometry and the special design, the sensor objective (composed of SPM module and imaging optics) can be directly screwed into the turret of an optical microscope [12]. This allows two microscopy worlds to be ideally combined.

In measuring operation, the advantages of the combined system become obvious. Firstly, the well-proven conventional light microscopy is used for fast and extensive surface investigation. The spectrum of tasks extends from the orientation on the measurement object to quantitative optical measurements (see Section 1.2.4). Then local measurement is performed with the slower serial scanning probe procedure in the measurement area defined for calibration or, generally, at those points of the sample which require a high resolution.

Figure 1.2 shows a version of the sensor objective screwed into a standard microscope. During measurement the sample is scanned with a piezo scanning stage whose position is controlled with capacitive sensors. In this objective design, the optical part of the measuring head consists of a combination of mirror optics. These allow the cantilever with the integrated measuring tip to be viewed simultaneously with the sample surface. This clearly improves user-friendliness as far as the adjustment and the selection of the measurement area are concerned. When optionally operated as optical near-field microscope, the same optics can be used for coupling and/or for collecting optical radiation into or from the near-field probe [12]. This allows very local optical investigations and spectroscopic surface characterizations to be performed even on submicroscopic structures [13].

In topography mode (SFM mode), even single atomic terraces of a GaAs substrate wafer (step height: approx. 0.28 nm) have been resolved with this measuring setup despite the relatively large measuring circle (sample, microscope body, granite stand, positioning stages – cf. Figure 1.2) [13]. These measurements were performed in a dynamic SFM mode using conventional silicon cantilever probes. Traditional beam deflection technique was applied to detect the bending of the cantilever. All optoelectronic elements of the beam deflection system have been arranged outside the measuring head, since a spatial integration was not intended when this version of the measuring head was constructed. This arrangement can be optimized, in particular, with respect to its mechanical stability. The further objective of the PTB development went, however, beyond the integration of the beam deflection system into the measuring head. This is why measuring heads based on probes with monolithically integrated deflection detection have been developed (see Sections 1.2.2 and 1.2.3).



Fig. 1.2 Conventional standard microscope with screwed-in sensor objective – the version shown here allows the device to be operated as optical near-field microscope in addition to scanning force and optical microscopy. The enlarged image section in addition shows a diagrammatic representation of the beam path inside the objective.

1.2.2 Sensor Objective with Piezolever Module

One possibility of integrating the deflection detection system into the sensor probe (i. e., as near as possible to the measuring tip) consists in utilizing the piezoresistive effect of the cantilever material (here silicon) [14]. Comparable to the strain gauge principle, the movement of the cantilever can thus be directly converted into a measurable electrical signal. This means that an adjustment of a light beam on the cantilever is not necessary. This improves user-friendliness of the system and avoids possible errors as a result of inexact adjustment. Stepheight measurements have, for example, shown that scattered light or reflections from the surface can lead to disturbing interference patterns or that the roughness of the rear side of the cantilever affects the measurement when optical methods are used for deflection detection. These error sources are avoided by monolithically integrated deflection sensors.

For realization of the piezoresistive cantilevers (briefly referred to as "piezolevers"), the piezoresistive elements were arranged in the form of a complete Wheatstone bridge and incorporated into the silicon cantilevers by ion implantation. This work was performed in cooperation with NanoWorld Services GmbH, Forschungszentrum Jülich and Surface Imaging Systems (SIS) GmbH [15]. As a special option, one of the Wheatstone resistors is realized as an electrically controllable resistor that allows the measuring bridge to be nulled.

During the design of our very compact SFM measuring head, which is based on these piezolevers, special attention was directed toward the requirement for detachable contacts of the cantilever chips [16]. In the piezolever SFMs so far realized, the cantilever chips were glued on small ceramic boards and the contacts were bonded. To avoid these complex additional process steps, the cantilever chips should be directly clamped and, at the same time, electrically contacted. To achieve this spring contacts were used that are made of gold-plated platinum beryllium (see Figure 1.3(b)). These "fingers" are arranged on a steel spring that is pressed-on or flapped-back with the aid of a very small cam to allow the probes to be exchanged. The complete holder must be exactly preadjusted and work free from mechanical play in order to contact the electrodes on the rear side of the chip reproducibly with the fingers, to exert enough force on the chip and to achieve good contacting. As can be seen in Figure 1.3(b), the contacts are only 50 μ m apart from each other. The latter emphasizes the desired mechanical precision of the contacting mechanism.

The dimensions of the whole SFM module that comprises both a piezo element for the dynamic excitation of the cantilever and the electrical connections for the sensor signals were reduced to 4 mm \times 3.5 mm \times 35 mm only (see Figure 1.3(c)). This compact design allows the combination with different measuring heads and measuring microscope objectives. Topographic measurement results obtained with this piezolever module are described and shown in Section 1.2.4 (Figure 1.6) together with interference-optical measurements.

8 1 Metrological Scanning Probe Microscopes – Instruments for Dimensional Nanometrology



Fig. 1.3 Piezolever module combined with a standard microscope objective. Part (b) shows the finger contacts for fastening and electrical contacting.

A great advantage of the mirror optics used in the sensor objective version described above (Section 1.2.1) was the fact that the dimensions and the optical parameters could be calculated by optical computational programs and manufactured with diamond turning machines. This finally allowed the whole sensor objective to be designed and constructed at our own options and the space required for the SFM module and the positioning mechanics to be taken into account. As described, the compact piezolever module does not require so much space. This is why these aspects are no longer important and the combination with a commercial microscope objective as shown in Figure 1.3(a) furnishes a solution that is more universal. This combination – microscope objective and SPM module – has been realized for all measuring head versions so far developed (cf. also Figure 1.4(a)).

1.2.3

Sensor Objective with Tuning Fork Module

Another possibility of integrating the deflection detection system into the measuring probe consists in using a cantilever arm made of quartz [17]. In operation, this quartz is – just like the tuning fork in a quartz clock – excited to swing after an electrical voltage has been applied. The measurement of the distance between the probe and the surface and thus imaging the surface is performed by recording the



Fig. 1.4 "Tuning fork" module with positioning mechanics and adapter ring for the microscope objective as well as a micrograph of the tuning fork lever arm with the diamond tip (b).

current flowing through the quartz. This signal is proportional to the lever arm vibration and reacts very sensitive to changes of the damping when the distance between the tip and the surface varies.

Diamond tips designed at PTB are fastened on these tuning forks to allow high lateral resolution of the measurement (tip radii < 100 nm) [18]. Figure 1.4(b) shows a quartz probe with tip. The selection of diamond as tip material is based on both the mechanical properties (stability and resistance to abrasion) and the optical properties that are important for the future use of the probes in optical near-field microscopy.

To test the efficiency of the tuning fork measuring head, topographic measurements were performed on structures with dimensions in the nanometer range. The samples used here are made of self-organized InAs quantum dots on a GaAs substrate. These quantum dots have pyramidal geometries (width approx. 20-30 nm, height approx. 4-6 nm). The mechanical stability of the whole microscope is sufficiently high to image such nanostructures. Investigations of the noise resulted in values of less than 0.6 nm (root mean square value) on a profile 2 μm in length.

Because of their extremely slim construction and their adjustment-free deflection detection, the tuning fork sensors can be tilted relative to the surface without any problem. This also allows measurements to be performed on object areas difficult to access such as structure edges or inclined areas. These properties allow these as well as the piezolever sensors to be used as sensitive probes in a coordinate measuring machine. Relevant developments have already been initiated at PTB.

1.2.4

Sensor Head for Combined Scanning Probe and Interference Microscopy

Up to now, imaging optics in SFMs only served as visualization tools to determine the area of interest for the measurement and to aid during probe alignment. In the sensor head realization described in this chapter, the functionality has been

considerably progressed. The combination of SFM and interference microscope allows an optical measuring technique to be integrated into the measuring head that can be traced back to the SI unit "meter" [19, 20].

This measuring system is based on the developments of the compact SFM measuring heads so far described and has been conceived so that it can be operated in different interference microscopes. For the PTB measuring setup, a commercial interference microscope (MicroMap, Nikon) was selected as the basic instrument. Because of the identical mechanical connecting plate, the newly developed sensor only replaces the exchangeable interference objective (see Figure 1.5). The basic instrument makes use of both the evaluation software and the displacement mechanics for phase-shifting interferometry or white light interferometry.

For the realization of the sensor head, two possibilities came into consideration: (1) modification of a commercial interference objective by adding an SFM module with the aid of an adapter (cf. Sections 1.2.2 and 1.2.3) or (2) new internal development of the whole interferential sensor head with additional SFM module. A solution according to (1) can directly be achieved by adapting the adapter ring mentioned in Section 1.2.2 and shown in Figure 1.3(a). In view of the planned improvement of the optical properties of the objective, which will be explained



Fig. 1.5 View of the combined SFM and interference microscope composed of sensor head and commercial basic instrument.

in the following, preference has, however, been given to the internal development of the measuring head.

Core piece of the newly developed sensor head is a Michelson interferometer in which the illumination is not performed via the internal, filtered microscope white light lamp, but via external laser sources coupled to optical fibers. This way, an essential heat source is removed from the measuring setup and the mechanical stability is improved. Even more important is the fact that due to the small illumination aperture of the optical fiber aperture correction becomes negligible in the interference-microscopic evaluation. This clearly reduces the measurement uncertainty.

At present, a HeNe laser ($\lambda = 632.80$ nm) or a frequency-doubled Nd-YAG laser ($\lambda = 532.26$ nm) can optionally be used as external laser sources in the measuring setup. If desired, this allows operation in the multiwavelength interferometry mode by which, compared to operation with only one wavelength, the range of unambiguous measurements of the interference microscope is extended.

For combination with a scanning probe microscope, the compact SFM module with piezolevers already described in Section 1.2.2 was mounted on the sensor head below the beam splitting cube. The cantilever can be seen in the image section of the optical microscope (both in the "live image" and in the interference-microscopic image; see Figure 1.6: on the left above) so that measurement area selection is very user-friendly. The interference-optical measurement (e.g., in phase-shifting mode) is performed simultaneously over the whole image section; in the current configuration, the optical measuring range amounts to approx. 900 μ m × 900 μ m. It can, however, also be varied by using different optical systems. In the case of a higher optical magnification it has, however, to be taken into account that the depth of focus is reduced and the advantage of an optical survey image is no longer valid. In a second step, the object area to be investigated with a high lateral resolution is moved below the SFM measuring tip with the aid



Fig. 1.6 Topography image of an 80-nm step-height standard (H80) (a) image recorded in the interference-optical mode (range 900 μ m \times 900 μ m) – the SFM cantilever can be seen in the circle marked at the upper-left corner; (b) Section measured with the integrated SFM module (range 40 μ m \times 20 μ m).

of a manually operated linear *x*, *y*-stage. After the surface has been successfully approached to the tip, the measurement object is moved line by line with a position-controlled piezo scanning stage (maximum operating range: approx. 100 μ m \times 100 μ m).

The extraordinary advantage of this combined measuring system consists in the possibility of a direct *z*-calibration of the SFM. As soon as SFM and interference microscope are measuring at the same place of the sample, the interference-optical result can be used as calibration value for the SFM. Special step-height standards are suited to be used as precise standards for heights from a few nanometers up to some micrometers [11].

Comparison measurements performed at PTB with the newly established measuring system and the reference interference-optical microscope showed for stepheight measurements on 80 nm and 260 nm calibration standard deviations of less than 1 nm [20]. Figure 1.6 shows a comparison of the results of measurements performed on an 80 nm standard in the interference-microscope mode and in the SFM mode.

Another advantage of this combined device becomes obvious in the case of heterogeneous objects. As soon as the optical constants of substrate and measurement structure differ, the optical wave in the interference microscope experiences different phase jumps on reflection. This leads to a measurement error as long as the relevant optical constants are not taken into account in the interference-microscopic evaluation. Determination of these constants for thin layers in the nanometer range is, however, quite time-consuming and often imprecise, so this correction is only conditionally possible. This is different in the case of the device on hand: Here, the measured value of the interference microscope is corrected by the SFM module that had been calibrated before. It is worthwhile pointing out that the SFM calibration was, as already described, performed with the same interference microscope, although on a sample with homogeneous surface. This example shows the complementary properties of the two independent measuring principles combined in one measuring instrument [20].

1.3

Metrology Systems Based on Scanning Probe Microscopes

In addition to other development activities in the field of SPM metrology, two commercial SFMs have been extended by miniaturized homodyne laser interferometers and their data acquisition system has been improved in the past 2 years. The positioning system of a third device developed into a large range SFM at PTB has already been equipped with laser interferometers by the manufacturer. These laser interferometers were developed in cooperation with the Technical University IImenau and SIOS Messtechnik GmbH. In the case of all devices, special attention was already paid during the construction of the interferometer extension and the instrument design to the fact that principles as minimization of Abbe errors and tilting were complied with. At PTB, the SFMs described serve for the calibration of standards and the general characterization of microstructures. In the following, the SFMs equipped with laser interferometers will be referred to as metrological SFMs.

1.3.1

Scanning Force Microscopes of Type Veritekt

Since 1995, two metrological SFMs with integrated laser interferometers have been constructed on the basis of the commercial SFM Veritekt-3 of Carl Zeiss, Jena. These devices allow measurement objects to be characterized in "contact" SFM mode with a measuring range of $70 \times 15 \times 15 \ \mu\text{m}^3$ (*x*, *y*, *z*). Compared to other instruments, the advantage of these SFMs is that a precise flexure hinge stage is used as the basis for the positioning system and that position-controlled piezo actuators (with integrated capacitive sensors) are used for each axis of motion. A skilful geometry of the flexure hinges allows factors such as cross-talk of the axes and nonorthogonality of the directions of motion to be minimized.

The operating principle of the integrated laser interferometers and the procedure of how they are used to calibrate the capacitive sensors in the piezo actuators is described in detail in [21, 22]. Figure 1.7 shows the diagrammatic sketch of the two Veritekt SFMs. Veritekt B that has been completed in 1996 and optimized in the following years with respect to a minimization of the Abbe error, is used for calibrations at PTB. The results of international and internal comparisons [23, 24] have confirmed suitability of this SFM for calibration tasks.

On the basis of the experience gained with Veritekt B, another metrological SFM, Veritekt C (see Figure 1.8), has been developed in the years until 2002. Essential subassemblies of the commercial basic instrument were adopted and supplemented by modern measuring and evaluation electronics. The arrange-



Fig. 1.7 Sketch of the metrological scanning force microscope Veritekt with integrated laser interferometers (source: TK Ilmenau).



Fig. 1.8 View of the SFM Veritekt C.

ment of the laser interferometers was revised in such a way that it is now also possible to adapt measuring heads working in different SFM modes.

Contrary to the measuring strategy used for Veritekt B, in which the laser interferometers are used for calibration of the capacitive sensors at discrete measurement points ($\lambda/2$ zero points of the interferometer signals) and calculation of correction values, Veritekt C directly includes the interferometer values in the SFM's control loop. To allow the interferometers to be used as measuring and control systems, the data acquisition electronics were completely changed and signal processing realized on the basis of a fast signal processor [5]. Integration of these data acquisition electronics into Veritekt C allows the resolution of the interferometer values to be increased to 0.04 nm and the interferometers to be operated at a data rate of 20 kHz.

As nonlinearity of the interferometer signals (which amounts to approx. 3 nm in the uncorrected form) is a limiting factor when measurement uncertainties in the range of a few nanometers are concerned, diverse correction procedures for the nonlinearity were investigated when the measuring electronics was modified. Finally, a procedure that follows the principle developed by Heydemann [25] was embedded into the control loop of the interferometers. This procedure corrects the deviations of the interferometers' electrical signals u_1^d and u_2^d in amplitude, offset, and phase by an ellipse fitting method:

$$u_1^d = u_1 + p$$
 $u_2^d = \frac{1}{r}(u_2 \cos \alpha - u_1 \sin \alpha) + q$

In view of the calculation effort involved, this algorithm is usually not implemented as online method. The investigations performed on Veritekt C have, however, shown that the ellipse parameters p, q, r, and α can be assumed to be constant over a sufficiently long period of time and need not, therefore, be permanently determined during correction. This allows the procedure to be integrated into the interferometer's measuring circle without restriction of the data rate. The correction described allowed remaining nonlinearities of the interferometer signals to be reduced to 0.3 nm.

After the interferometer data rate had been successfully increased, the measuring principle of the SFM was revised to accelerate data acquisition of all signals. On this basis, a new measuring mode was developed for scanning of the sample. Central triggering of all measuring and control elements installed in the SFM then allows the measurement object to be scanned with constant velocity and to simultaneously determine the measurement data of both the positioning system and the SFM sensor acting as null indicator. This makes deceleration of the movement during acquisition of the measurement point data unnecessary; this "scan-on-the-fly" measuring principle allows the measurement velocity in the *x*-direction (fast scan axis) to be increased to up to 25 μ m/s as a function of the topography to be investigated. Because of the fast data acquisition, the influence of thermal drift and other environmental factors can be reduced.

Modernization of the data acquisition software, an automated sample positioning system, and the efforts taken to realize automatic measuring processes (batch processes) have further improved the handling of the device. Because of the use of laser interferometers as displacement measuring sensors, calibration of the measuring system so far required can be dispensed. This leads to a reduction of the whole measuring time.

1.3.2 Metrological Large Range Scanning Force Microscope

For an increasing number of practical applications of scanning probe microscopy – also in the field of SPM metrology – the measuring range of piezo scanning stages (x, $y < 100-200 \mu$ m) is too small. These applications comprise, for example, the determination of roughness in accordance with written standards and investigations on lateral standards whose evaluation requires measurements in the millimeter range. For the reasons mentioned, different concepts have been developed to extend the measuring range of SFMs with the aim of increasing the displacement range of piezo actuators [26] or using alternative positioning systems [27].

The PTB decided to develop and manufacture a positioning system on the basis of the so-called nano measuring machine [27] that meets the specific metrological requirements of industrial metrology. This device was combined with a measuring head based on a focus sensor known from the Veritekt SFM. A measuring instrument is thus available that combines a positioning range of $25 \times 25 \times 5 \text{ mm}^3$ with the detection principles of scanning force microscopy – the so-called metrological large range scanning force microscope (LR-SFM). Its operating principle is shown in Figure 1.9.

The object stage is moved via three linear driving systems that are position controlled by laser interferometers. Two angle-measuring systems have been included in the control unit to correct for guidance errors of the motion stage.

16 1 Metrological Scanning Probe Microscopes – Instruments for Dimensional Nanometrology



Fig. 1.9 Diagrammatic sketch of the metrological large range SFM (LR-SFM) (components such as drives and rails are not shown for reasons of clarity), (source: TK Ilmenau).

Similar to the Veritekt SFMs, the reference system is formed by plane mirrors; in the case of the LR-SFM, the mirrors have been combined to form a cube corner. The resolution of the measuring system amounts to 0.08 nm or 0.001, respectively. The construction of the device is aimed at achieving coincidence of measuring and reference plane to minimize Abbe errors.

To increase the dynamics of the positioning system, a compact vertically moving piezo stage was arranged on the sample stage of the NMM. This one allows fast scanning with a range of up to 2 μ m. Its compact and stiff design results in a high mechanical resonance frequency f_r of 20 kHz. The movement of this stage is measured and its position controlled via a capacitive sensor arranged in the middle of three symmetrically arranged piezo actuators. During scanning of the sample, the lateral movement is performed exclusively with the NMM, whereas the height adjustment results from a combined movement of the vertically adjustable z piezo stage and the NMM. The whole device is controlled via two signal processor systems. One is responsible for the NMM, the other realizes height adjustment and data acquisition. More detailed information about the measuring techniques used and the control systems implemented can be found in [28, 29]. The photo in Figure 1.10 shows the metrological LR-SFM.

After finishing the design of the measuring software for the complete device, extensive investigations into the metrological properties of the LR-SFM were carried out. As an example, the first results of measurements performed on a flatness standard and on a sinusoidal lattice standard are shown.

The topographic image of the flatness standard (Figure 1.11) can be used to estimate the quality of the motion (influenced by the guidance mechanism) and to evaluate the instrument's noise behavior. The image shows that the structure measured is very flat and that artifacts as they may, for example, be caused by the ball bearings, are not detectable. The residual instrument noise (3 nm p-v) 1.3 Metrology Systems Based on Scanning Probe Microscopes 17







Fig. 1.11 Investigations into the guiding properties and noise behavior of the LR-SFM Topography image of a flatness standard.

is mainly due to external influences such as building vibrations and acoustic excitations, and it should be reduced by optimizing the environmental conditions.

Suitability of the LR-SFM for measurements on lateral standards and determination of the structure period is illustrated by the example of a sinusoidal lattice. Figure 1.12 shows the scan image of a one-dimensional lattice that has been scanned in the *x*-direction with a measuring range of 1.35 mm (this corresponds to 20 times the scanning range of the Veritekt SFMs!). As calculation of the structure period is based on a statistical procedure, a larger number of structures allows us to improve the measurement uncertainty of the measuring procedure, provided the sample structure is homogeneous. Repeated measurements on this sinusoidal lattice showed an identical periodic value of 416.67 nm. This result agrees with the reference value from diffractrometric optical measurements within two decimal places.



Further measurements on nanostructures and step heights have confirmed the high spatial resolution of the measuring instrument and agreement of the measured values with reference values from international comparisons.

The investigations initiated to optimize the LR-SFM and extend it by alternative detection principles are permanently continued and are to demonstrate that the measuring system is also suitable for the measurement of structures with a topography up to the millimeter range. Measurement tasks such as calibration of tip geometries on indenters for hardness measurement, investigation of structures on photo masks from semiconductor industry, determination of dimensional parameters on parts in the field of microsystem technology and the like are already demanded by industry and represent potential fields of application for the metrological LR-SFM.

1.4 Summary

Special emphasis in the field of dimensional nanometrology at PTB is placed on the development and optimization of measuring instruments for SPM metrology. The development of sensor heads comprises, among other things, the concept of the "sensor objective" to combine conventional microscopy with scanning probe techniques. It is characterized by its extraordinary versatility that is due to the use of different measuring heads and detection principles. In the field of complete metrological SFM systems, the measuring properties of one of the existing Veritekt systems have been significantly improved by including laser interferometers directly into the position control loop and by a clear reduction of the nonlinearity of the interference signals. In addition, the application spectrum of metrological scanning probe microscopy has been considerably extended by the establishment of an SFM system with a measuring volume of 25 mm \times 25 mm \times 5 mm.

The experience gained in the past few years has shown that it is precisely the performance of development work in the field of SPM instrumentation at PTB that is of decisive importance for the quality and understanding required for subsequent use of these devices and their calibration. No study of operating instruc-

tions or training courses can replace the know-how gained in this work. Many development projects have produced innovative solutions to reply to metrological questions. In accordance with our philosophy, these activities are carried out almost exclusively together with partners from industry and are, if possible, based on commercially available components. Several examples of successful technology transfer (among others Physik Instrumente (PI) GmbH, SIOS Messtechnik GmbH, Surface Imaging Systems (SIS) GmbH) can be shown; they have been implemented in many industrial products in the whole world.

Because of the continuing miniaturization in many high-technology fields and the increasing number of metrological applications of SPMs, scanning probe microscopy will be of outstanding importance for the future work in the field of dimensional nanometrology at PTB.

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References

- O. Jusko, X. Zhao, H. Wolff, and G. Wilkening, Design and three dimensional calibration of a measuring scanning tunneling microscope for metrological applications, Rev. Sci. Instrum. 65, 2514ff (1994).
- 2 H. Geuther, K.-P. Schröder, H.-U. Danzebrink, and W. Mirandé, Rastermikroskopie im Nah- und Fernfeld an Strukturen im Submikrometerbereich, tm-Technisches Messen 61, 390–400 (1994).
- 3 S. Gonda, T. Doi, T. Kurosawa, et al., Realtime, interferometrically measuring atomic force microscope for direct calibration of standards, Rev. Sci. Instrum. 70(8), 3362ff (1999).
- 4 J. Haycocks and K. Jackson, in Proc. of the 2nd Euspen Conference, Torino, Italy, May 27th-31st (2001) 392ff.
- 5 G. Dai, F. Pohlenz, H.-U. Danzebrink, K. Hasche, and G. Wilkening, Improving the performance of interferometers in metrological scanning probe microscopes, Meas. Sci. Technol. 15, 444–450 (2004).

- 20 1 Metrological Scanning Probe Microscopes Instruments for Dimensional Nanometrology
 - 6 G. B. Picotto and M. Pisani, Ultramicroscopy 86, 247ff (2001).
 - 7 M. Bienias, S. Gao, K. Hasche, R. Seemann, and K. Thiele, A metrological scanning force microscope used for coating thickness and other topographical measurements, Appl. Phys. A 66, 837ff (1998).
 - 8 R. Dixson, R. Köning, J. Fu, and T. Vorburger, Accurate dimensional metrology with atomic force microscopy, SPIE Metrology, Inspection, and Process Control for Microlithography XIV, February 28 – March 2, 2000, Santa Clara, Ca, Vol. 3998.
 - 9 G. Garnaes, N. Kofod, A. Kühle, and C. Nielsen, Calibration of step heights and roughness measurements with atomic force microscopes, Prec. Eng. 27, 91ff (2003).
 - 10 F. Meli, and R. Thalmann, Long-range AFM profiler used for accurate pitch measurements, Meas. Sci. Technol. 9, 1087–1092 (1998).
 - 11 L. Koenders, T. Dziomba, P. Thomsen-Schmidt, and M. Senoner, Normale für die dimensionelle und analytische Nanometrologie, PTB-Mitteilungen 114, 16–24 (2004).
 - 12 C. Dal Savio, H. Wolff, T. Dziomba, H.-A. Fuß, and H.-U. Danzebrink, A compact sensor-head for simultaneous scanning force and near-field optical microscopy, Prec. Eng. 26, 199–203 (2002).
 - 13 H.-U. Danzebrink, D. V. Kazantsev, C. Dal Savio, K. Pierz, and B. Güttler, Optical microscope with SNOM option for microand nanoanalytical investigations at low temperatures, Appl. Phys. A 76, 889–892 (2003).
 - 14 M. Tortonese, R. C. Barrett, and C. F. Quate, Atomic resolution with an atomic force microscope using piezoresistive detection, Appl. Phys. Lett. 62, 834–836 (1993).
 - 15 R. Jumpertz, J. Schelten, O. Ohlsson, and F. Saurenbach, Bridge configuration of piezoresistive devices for scanning force microscopes, Sensors Actuators A 70, 88–91 (1998).
 - 16 C. Dal Savio, Th. Dziomba, D. V. Kazantsev, and H.-U. Danzebrink, Entwicklung eines kombinierten Nahfeldmikroskopie-

und -spektroskopiesystems, PTB-Bericht F-47, S. 1–94 (2003).

- 17 P. Güthner, U.Ch. Fischer, and K. Dransfeld, Scanning near-field acoustic microscope, Appl. Phys. B 48, 89 (1989).
- 18 J. W.G. Tyrrell, D. V. Sokolov, and H.-U. Danzebrink, Development of a scanning probe microscope compact sensor head featuring a diamond probe mounted on a quartz tuning fork, Meas. Sci. Technol. 14, 2139–2143 (2003).
- 19 H.-U. Danzebrink, J. W.G. Tyrrell, C. Dal Savio, and R. Krüger-Sehm, Combined optical and scanning probe microscopy, PTB-Bericht O-68, 112–123 (2003).
- 20 J. W.G. Tyrrell, C. Dal Savio, R. Krüger-Sehm, and H.-U. Danzebrink, Development of a combined interference microscope objective and scanning probe microscope, Rev. Sci. Instrum. 75, 4, 1120–1126 (2004).
- 21 M. Bienias, S. Gao, K. Hasche, R. Seemann, and K. Thiele, A metrological scanning force microscope used for coating thickness and other topographical measurements, Appl. Phys. A 66, 837–842 (1998).
- 22 K. Hasche, K. Herrmann, W. Mirandé, R. Seemann, L. Vitushkin, M. Xu, and G. Yu, Calibrated scanning force microscope with capabilities in the subnanometre range, Surf. Interface Anal. 33, S. 71–74 (2002).
- 23 L. Koenders, et al., Comparison on nanometrology – NANO2: step height, Metrologia 40, 04001 (2003).
- 24 K. Herrmann, W. Mirandé, K. Hasche, and F. Pohlenz, Investigations on gratings with period length in the nanometre range, in Proceedings of the 5th Seminar on Quantitative Microscopy and 1st Seminar on Nanoscale Calibration Standards and Methods, Bergisch-Gladbach, November 2001, PTB-Bericht F-44, S. 78–85 (2001).
- 25 P. Heydemann, Determination and correction of quadrature fringe measurement errors in interferometers, Appl. Opt. 20, S. 3382–3384 (1981).
- 26 F. Meli and R. Thalmann, Long range AFM profiler used for accurate pitch measurements, Meas. Sci. Technol., Special issue on Dimensional Metrology 9(7), S. 1087–1092 (1998).

References 21

27 G. Jäger, E. Manske, T. Hausotte, and H.-J. Büchner, Nanomessmaschine zur abbefehlerfreien Koordinatenmessung, tm-Technisches Messen 7-8, S. 319–323 (2000).

28 G. Dai, F. Pohlenz, H.-U. Danzebrink, M. Xu, K. Hasche,an d G. Wilkening, A metrological large range scanning probe microscope. Rev. Sci. Instrum. **75**, 4, 962–969 (2004).

29 G. Dai, F. Pohlenz, H.-U. Danzebrink, M. Xu, and K. Hasche, A novel metrological scanning probe microscope with a measurement range up to (25 mm)², PTB-Bericht F-48 (2003), 175–182.