The Scanning Force Microscope as a measuring tool

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Abstract: This paper presents the state of the art in scanning force microscopy for dimensional metrology. A description is given of the important factors affecting the major components of a scanning force microscope from the metrological point of view. Both instrument design and calibration are discussed together with an overview of industrial applications. Recent achievements by national metrology institutes and others to improve calibration procedures, traceability, and reduce measurement uncertainty are described.

1. INTRODUCTION

With the current rapid growth in micro and nanotechnology metrology is more important than ever. At the same time, SPM applications have expanded from nanoscience to many other areas where nanometre sized features or nanometre tolerances must be dimensionally quantified.

1.1 Measurement tasks

The measurement tasks to be carried out in the field of micro and nanometrology essentially correspond to those of the macroscopic world: determining the geometry and other physical and chemical characteristics. However, with decreasing feature size, measurements here must be seen in a broader context: the structures' dimensions begin to play an essential role in the overall properties of the structure; in nanotechnology, sometimes the dimensions even define the properties completely. Often the quality of components can only be assessed by performing measurements on both the nanometre and micrometre scales.

The fundamental measurement tasks in dimensional micro and nanometrology concern: distance, pitch, width, height, form, surface texture and roughness, volume, and layer thickness.

As stated above, there is a close connection between dimensional measurement and the determination of other physical or chemical quantities, and the measuring method must have an adequate dimensional resolution for resolving relevant features.

Scanning probe microscopes (SPMs) [1] fulfil this "holistic" approach that is undoubtedly of great use. Since their invention they have had a major influence on the development of nanotechnology and their metrological aspects have been treated in a number of reviews [2-5].

SPMs are regarded as typical nanotools as they have opened up the possibility to image and to manipulate nanostructures. However, SPMs have proven their suitability also in 'coarser' fields. Fig. 1 shows the measurement range of SPMs. It covers the needs of nanotechnology, IC technology, part of MEMS/MOEMS technology as well as the needs of surface roughness and particle size measurements.

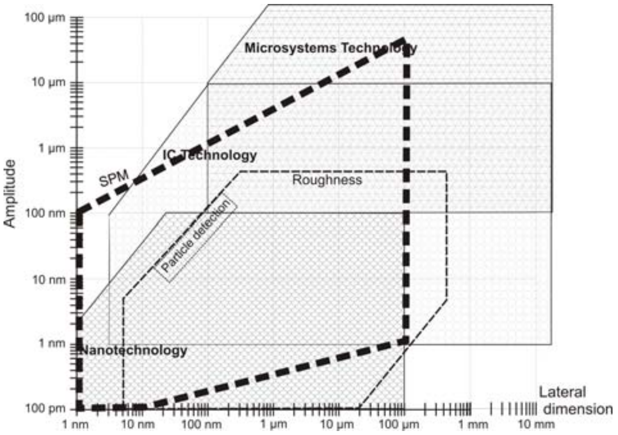

Fig. 1 *Extension of typical measurement features in new technologies and the measurement range that can be covered by SPMs.*

1.2 The family of scanning probe microscopes

A whole family of specific probing methods has been developed that are based on different interaction principles and allow a large number of quantities to be determined [5-7]; microscopes use force, optical, thermal or electrical interactions between tip and surface. While the tunnelling principle is mostly used in surface physics, the force principle is widely used in applied research and industry. The reproducible micro-mechanical manufacture of different cantilevers for a wide field of use [8,9] and the different probing modes, have made this principle the "workhorse" of scanning probe microscopy.

1.2.1 Scanning Force Microscopy (SFM)

Figure 2 shows a schematic diagram of the operating principles of an SFM. A cantilever with a sharp tip is mounted on the end of a piezo scanning tube.

Light from a laser diode is reflected from the upper surface of the cantilever onto a quadrant photodiode. The cantilever is moved towards the surface and when it is very close (a few tens of nanometres), the surface forces result in an interaction between the surface and the tip causing bending of the cantilever. The position of the light beam on the photodiode changes and the resulting change in signal from the photodiode can be used as the input to a servo system that ensures the force between the sample and tip (and hence distance) is kept constant.

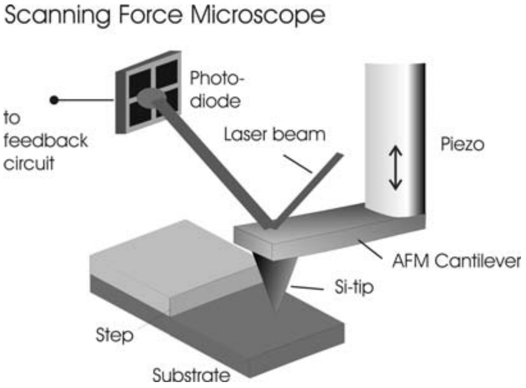

Fig. 2 *Sketch of a scanning force microscope (SFM) with cantilever probe and beam deflection detection. In this configuration the cantilever is moved up and down by the piezo tube in order to follow the topography of the sample that is scanned laterally.*

There are three modes of operation of an SFM, contact, intermittent and non-contact. In the ‘contact mode’ the cantilever is simply scanned across the surface of the specimen and since it is in contact the repulsive surface forces are used. In contrast to the contact mode, both the intermittent and non-contact modes are dynamic since the cantilever is oscillated close to its resonant frequency above the surface. In the intermittent mode, the tip’s average distance is in the region of the repulsive forces. When fully extended the tip ‘touches’ the surface atoms of the sample. The resulting interaction between the tip and sample causes a slight shift in the resonance frequency and changes the vibration amplitude and phase with respect to the driving frequency, which can be used in the feedback circuit. This mode is generally used for looking at soft specimens since it is less likely to cause specimen damage than the contact mode. The non-contact mode relies on the long-range attractive forces and is therefore more suitable for examining magnetic structures or those that exhibit high surface attractive forces. The achievable resolution is less than that of the contact and intermittent contact modes.

2 SFMs FOR DIMENSIONAL METROLOGY

An SFM suitable for measurement should be designed in a way, such that a) the measurement loop is as small as possible, b) the materials in the measurement loop are matched to get a zero thermal effect, c) the position of the tip is measured as close as possible to the interaction point (i.e. the “Abbe offset” is close to zero), d) the rotational errors of movement are as small as possible.

There are different approaches to achieving these goals and there is an increasing number of SFMs on the market that offer reasonable solutions. In general, the efforts of commercial providers address the stability (repeatability) issue and they equip their instruments with position monitors for x,y and z [10-13]. Great care is taken in the design of SFMs used by national metrology institutes since these instruments must be able to calibrate artefacts with the smallest achievable uncertainty. These instruments use laser interferometry for displacement measurements and advanced displacement mechanics. The basic idea is to measure the relative displacement of tip and sample in all three axes by laser interferometers whose measurement arms are adjusted in a way that they intersect at the interaction point.

2.1 Instrument classes

Existing types of SFMs may be divided into three categories with respect to their ability to measure dimensions quantitatively. These categories describe primarily the quality of the scanning apparatus of the instruments:

A Reference SFMs with integrated laser-interferometers, traceable directly via the wavelength of the laser used (often called “Metrological SFM”)

B SFMs with position monitoring by integrated position sensors, e. g. capacitive sensors, inductive sensors, strain gauges, encoders calibrated either by temporarily attaching laser-interferometers to the scan system or by using high-quality physical transfer standards. This category comprises both SFMs with active position control (**B1**: with a feedback-circuit: so-called “closed-loop” SFMs) and without (**B2**: “open-loop” SFMs with integrated position sensors for monitoring only).

C SFMs with positioning by simply using the voltage applied to the scanner (x & y) resp. with positions deduced from the voltage applied to the scanner (z) (**C2**). Often, the well-known drawbacks of piezo material – creep, hysteresis, non-linearity – are addressed by means of software correction (**C1**).

Instruments B and C have to be calibrated, which usually is accomplished by using physical transfer standards.

The properties of these instrument classes were clearly demonstrated in a comparison of measurements on different standards, using different instruments in different institutes or companies [14].
Measurement deviations of up to + 10% occurred without such measurements having to be clearly considered as outliers. Parts of the results are strongly dependent on the scan range used. This situation reflects the fact that lateral measurements depend strongly on the scanner properties. The great deviations occur only in the case of devices with tube scanners. Devices whose scanning principles guarantee small angular deviations and/or which are controlled in all axes by position sensors (type A and B) show very small deviations (< 0,5%). For type C devices, the smallest deviations occur when the greatest scan range is used. It is assumed that they were calibrated only in these ranges prior to the measurement. Metrology SFMs (type A) are clearly superior in lateral measurements: The deviations are in the range < 0,2% and compared with conventional devices, also the standard deviations are very small.
It should be noted here, that step height measurements showed smaller differences in all categories [29].

2.2 The tip

The most critical element in an SFM is the tip that senses the sample surface. For highest resolution the very end of this tip should ideally end in one atom interacting with the atoms of the sample via short-range repulsive/attractive forces [15]. For applications in dimensional metrology, the overall tip shape should be regular and as sharp as possible.
The overall tip shape is determined by the fabrication method; Si tips usually are very sharp. In some cases the tip shape is improved by additional processes [8,10,16-18]. Also, carbon nanotubes (CNT) attached to normal tips can be used to give ultimate imaging resolution [19].
For critical dimension (CD) metrology, where the measurement of sidewalls is necessary, special probes with horizontally protruding structures or flared ends have been developed and tested [3,20].

2.2.1 Tip characterisation

The uncertainties for step height and pitch measurements are now in the sub-nanometre range [21-23]. In both cases the measurements are independent of the tip shape as long as the structure is not too small and the tip is stable. On the other hand, for measurements of shape, width (CD) or roughness the tip directly influences the measured profiles. To evaluate the true shape, width, or surface profile it is mandatory to know the tip shape, or better, the effective tip shape.
Special artefacts known as tip characterisers have been developed for the in-line measurement of the tip shape. The tip shape is reconstructed from measuring data obtained when scanning the tip across the sharp edges of the characteriser. The measurement of a sample by a tip formally corresponds to a morphological operation and can be described as dilation [24-26]. Consequently, after performing erosion, the reconstructed surface is obtained, which is identical to the imaged surface in an ideal case. It has to be noted, that additional, unknown forces influence the effective tip shape.

2.3 SFMs for large range measurements

If a larger measuring range is required, or the measurement of small areas at selective regions within a large sample area, the SFM can be combined with a large-range positioning stage. This normally is an x-y-table, equipped with suitable position measurement sensors. Instruments of this kind are commercially available, and are used especially in the semiconductor and flat panel industry. In most cases, the positioning accuracy is limited to a few micrometres.
An interesting approach has been reported, where an SFM head has been mounted on a coordinate measuring machine (CMM) thereby achieving free positioning of the SFM in the space covered by the CMM [27]. The CMM is used to reposition the SFM probe in between measurements that are stitched together.
In contrast to instruments where mainly the positioning properties have been enlarged, there has been put considerable effort into SPMs with real large scanning ranges. The first SPM-based metrology instrument that is designed to achieve sub-nanometre resolution over a macroscopic area of 50 mm by 50 mm is called “Molecular Measuring Machine” (M^3) [28]. The “Long-range Scanning System” (LORS) has a measurement range of 25 mm x 25 mm x 0,1 mm [29]. A “Large Range SPM” (LR-SPM) has been developed with a measurement volume of 25 mm x 25 mm x 5 mm [30].

3 STANDARDS, CALIBRATION AND UNCERTAINTY

There is a number of artefacts available on the market that can be used for calibration purposes (see also Review of Standards for SPM [31]). They can be divided into two classes: standards that are used to calibrate the properties of the instruments, and reference samples that are used to check the instrument properties. The first class of standards consist of individual or regular structures of well-known geometry with calibrated dimension(s) in lateral and/or vertical direction. The second class is useful to check properties like out-of-plane motion of the scanner, the quality of the tip, and to check external influences which are acting on the device during the measurement.

Guidelines and standards have been published [32,33] and are under further development [34,35].

A number of NMIs have set up metrological instruments and offer calibration services. International key comparisons have taken place and have demonstrated uncertainties in the few nanometre range [21-23]

4 APPLICATIONS OF SFMS

The potential of the instrument was at first realised by the semiconductor industry that now uses SFMs on a routine basis. More generally the SFM has found its place in many industrial sectors that are actively involved with research. A recent survey in the optics, coatings and high precision surface manufacturing sectors showed that SFMs have become standard there as well [36]. Generally, SFMs are used to characterise manufactured surfaces and structures; they measure roughness and surface texture (e.g. crystallinity, edge roughness, tool wear), step heights and flank angles, grating constants (e.g. of diffractive optics) and are combined with nano-indentors for thin film characterisation. They are often used together with other instruments, such as interference or confocal microscopes, or profilometers and (micro)CMMs. Very often, these ‘wide range’ instruments give an overview, and the SFM is used for ‘zooming in’ on specific areas. Primarily SFMs are used for research and developmental purposes, or offline production control.

In the following, a few applications are shown in more detail.

4.1 Particles

Powder size, size distributions and particle numbers can be determined using numerous commercially available instruments. Such ‘particle counters’ have to be calibrated, which typically is accomplished by using mono-disperse reference particles such as gold colloids or polymer spheres with various sizes. An SFM in tapping mode has been used to determine the diameter of the reference particles in a traceable way. Two methods were applied: firstly, measuring the height of single particles adhered to an atomically flat surface, and secondly, measuring the pitch value of an array of closely-packed particles [37].

4.2 Roughness

Surface roughness of functional surfaces is an important factor affecting its behaviour. Smooth surfaces often consist of soft materials such as pure metals (aluminium, gold, copper). For roughness measurements on such surfaces conventional mechanical instruments cannot be used and optical methods have a limited lateral resolution. The measurement of roughness certainly is the most frequent application of SFMs. However, it has to be pointed out that the lack of relevant standards is especially hindering in this area of application. From the wide range of applications here an example from nanotechnology is discussed.

Ultra-hydrophobicity and hence self-cleaning of surfaces can be achieved by a large variety of surface structures differing in form and size. A new approach to achieve ultra-hydrophobic surfaces with optical quality utilises statistical nano-roughness of coatings [38]. Proper control of optimum roughness requires that the roughness analysis be extended over a wide spatial frequency range. Particularly important is a high spatial frequency roughness that does not induce optical scatter but significantly contributes to the desired functional effect.

4.3 Nano-indentation

Since the most significant source of uncertainty in nano-indentation measurement is the geometry of the indenter tip effort has been directed towards the characterisation of the three-dimensional tip geometry. The indenter area function - that is the relationship of the indenter area as a function of the indenter height seen from its tip - can be determined using an SFM [39]. The area function can be used for the correction of the measurement values from an indentation test.

5 CONCLUSIONS

SFMs are part of the measurement tool set in the respective industries, with the semiconductor industry as pathfinder. However, SFMs are not used for in-line metrology, and in most cases they are not used alone, but together with other optical or tactile instruments. SFMs are used when the highest resolution is required. At the moment, adequate standards are needed for calibration and proper measurement use. This situation is slowly changing; there are activities in international and national committees.

NMIs have set up metrological SFMs and offer measurement and calibration services. The uncertainty level is in the 'few nanometre range'.

SPMs are advancing, supported by user-friendly and application-oriented features, like automation, large sample volume and positioning, improved software, combination with other instruments. Industry is aware of quantitative aspects and is using calibration standards and proper software for the calibration of their instruments. Commercial instruments seem to have reached a sufficient degree of reliability and usefulness, the average age of SPMs in industry is around 5 years. Younger instruments often show larger positioning ranges and the direct combination with other measurement instruments.

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