Vol. **116** (2009) *ACTA PHYSICA POLONICA A* Supp.

Proceedings of the III National Conference on Nanotechnology *NANO 2009*

Measurements of Surface Properties in the Nano- and Microscale Using Optical, Mechanical, and Scanning Probe Methods

Ł. Rok[*]
Schaefer Technologie GmbH, Robert-Bosch-Str. 31, 63225 Langen, Germany

Modern science and technology requires advanced equipment to achieve further progress. In almost all fields scale of experiment was shifted to nanorange showing new challenges for researchers and scientific equipment design. This article concentrates on nanotechnology and surface engineering metrology solutions which are provided by the Schaefer Group of companies. Recent developments in the field of scanning probe microscopy, three-dimensional optical microscopy and of material properties characterization in the nano- and microscale are briefly described. Examples of possible applications of mentioned techniques are given as well.

1. Introduction

The Schaefer Group of companies supplies industrial and academic researchers in the field of nanotechnology and surface engineering with innovative surface metrology solutions. With constantly increasing activity in the area of scanning probe microscopy (SPM) since 1992, Schaefer is offering today a full selection of SPM systems made by Nanosurf AG, Park Systems and RHK Technologies. Based on a deep understanding of customer needs and our broad product spectrum range of scanning probe microscopes, we can suggest suitable SPM solutions for any situation: from academic education and portable atomic force microscopes (AFMs) or semi-automated systems for industrial and research applications to dedicated biomedical/life science AFMs and UHV STM/AFM (scanning tunneling — atomic force microscopes) systems for high-end research. In addition to SPM, Schaefer is also offering various optical 3D profilometers, using confocal, interference, or digital holography techniques, as well as mechanical micro- and nanotesting equipment.

2. Atomic force microscopy

Since its invention in 1986 [1] the atomic force microscope has proven to be a powerful tool for nanoscale science and technology. The most common configuration of the AFM uses a cantilever with a sharp tip on its end which is scanned over a sample surface with the use of piezoelectric tube scanner. The deflection of the cantilever is measured by detecting a laser beam reflected from its backside with a position sensitive photodetector (PSPD). The new generation of AFM microscopes uses flexure scanners. In comparison to tube scanners, the XY movement in a flexure scanner is completely decoupled from Z movement.

2.1. Park Systems XE-series AFM

The XE series of AFM microscopes uses two-dimensional flexure stages to scan the sample only in the XY direction and stacked piezoelectric actuator to scan cantilever probe in the Z direction only. The main advantage resulting from this decoupling is the possibility of making measurements in true non-contact mode. In this mode the cantilever oscillates with small amplitude very near to the sample surface. The tip–sample distance is maintained at few nanometres with fast Z-servo response, providing primary tip sharpness and best sample surface conditions during the whole measurement. Another important fact resulting from using flexure stage is complete elimination of the background scanner curvature, for example for the scan range of 50 μm out-of-plane motion is less than 1 nm. Thus images of the flattest samples show no bowing.

The true non-contact mode of the XE series provides the ultimate resolution which can be expected in ambient conditions. This resolution exceeds capabilities of both tapping and contact AFM modes as it is shown in Fig. 1. The fine details on the image presented in this figure demonstrate about 2 nm lateral resolution.

2.2. Nanosurf easyScan 2 AFM

Instead of piezoelectric scanners that work under high voltages and are vulnerable to creep, the easyScan 2 AFM uses a patented electromagnetic scanner that has a linearity mean error in XY direction of less than 0.6% with low noise and low power consumption. This and similar smart design features allow a significant reduction in cost and size without loss of precision performance. Moreover, this smart design includes modularity. User can start with simple STM microscope and add AFM later or begin with the AFM Basic Module which enables measurements in contact mode, and add dynamic modes capabilities later by installing the AFM Dynamic Module.

---

[*] e-mail: `l.rok@schaefer-tec.com`

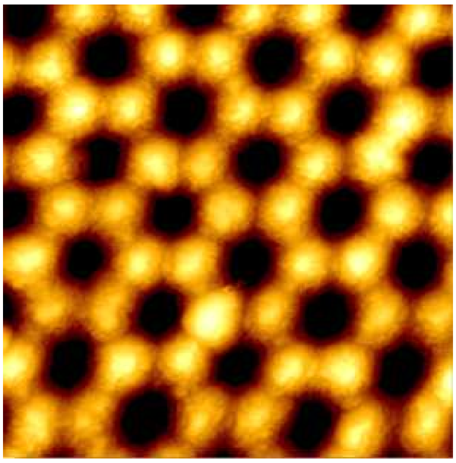

Fig. 1. The AFM image of nanostructures patterned on aluminium surface taken with true non-contact mode of XE-100 AFM microscope. Scan range of image is 0.5 μm × 0.5 μm [2].

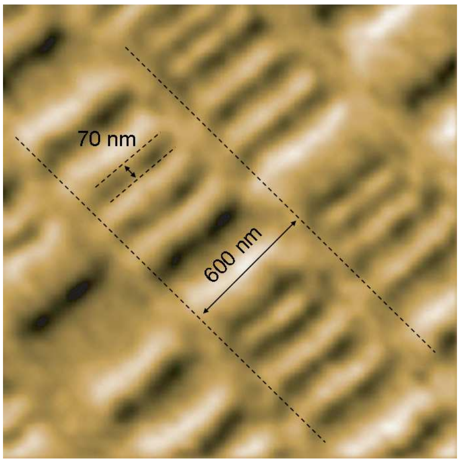

Fig. 2. The MFM measurement of the hard disk head (phase contrast mode). Bright and dark areas show magnetisation reversal, where white color stands for repulsive forces and black for attractive forces. Scan range 2 μm × 2 μm [3].

With the AFM Mode Extension Module measurements in phase contrast, force modulation, magnetic force and electrostatic force modes become possible. Three AFM scan heads are available: 70 μm designed for general purposes, 110 μm for large scan areas and 10 μm high resolution scan head. Due to modularity, the user can start with a very simple configuration and after some time have a fully equipped scientific tool. It is worth to mention that even in the most complicated configuration easyScan 2 microscopes are easy to use providing quick and reliable measurement results.

The idea of ease of use has been implemented in the new Flex AFM scan head as well. It is designed to extend the capabilities of easyScan 2 with measurements under liquids. This scan head uses an electromagnetically driven flexure scanner providing flat and linear scanning. Moreover, SureAlign$^{\text{TM}}$ laser optics eliminates laser adjustments, because the laser beam shift that normally occurs upon immersion into liquid is completely absent.

One of the possible measurement modes available in the easyScan2 AFM microscope is magnetic force microscopy (MFM) imaging mode. In the MFM mode magnetic force is detected by sensing sample surface with magnetically coated tip. Mentioned force introduces shift in the cantilever resonance frequency which causes shift in the phase of cantilever vibration. Example of MFM measurement is given in Fig. 2 where the sample was 10 GB hard disk magnetised with a track distance of 600 nm and a bit length of 70 nm.

3. The 3D optical microscopy

The three-dimensional optical microscopy is a very fast, non-destructive, and non-contact surface metrology technique.

3.1. Lyncee-tec's digital holographic microscopes

Digital holography uses the same principle as classical holography [4–6], but the hologram recording is made with the use of digital sensor, e.g. CCD camera. The reconstruction of recorded holographic image that contains information about the object wave is calculated with numerical algorithms [7] within a computer.

Lyncee-tec's digital holographic microscope (DHM) generates, in real time, high resolution three-dimensional digital images of a sample using the principle of holography. Holograms are generated by combining a coherent reference wave with the wave received from a specimen. They are recorded by a video camera and transmitted to a computer for real-time numerical reconstruction.

From a single hologram acquired in a few microseconds, DHM software procedures allow computation of the complete wave front emanating from an object. This provides intensity images with the same contrast as classical optical microscopy. The phase images provide quantitative data that are used for accurate and stable measurements. In reflection, the phase image reveals directly the surface topography with a sub-nanometric vertical resolution. In transmission, the phase image reveals the phase shift induced by a transparent specimen, which depends on its thickness and refractive index.

One of possible applications of the DHM microscopes is contactless and dynamic characterization of microdevices performed in conjunction with stroboscopic module. These measurements are performed with nanometric vertical resolution. The stroboscopic module has a frequency range up to 25 MHz; it has also selectable laser pulse freezing time down to 7.5 ns. With the use of this module any periodic or repeated signal can be generated to drive the device under investigation. Moreover, continuous frequency scan can be applied to approach resonance conditions. Example of possible use of the DHM microscope equipped with stroboscopic module is presented in Fig. 3. In this figure a precise measurement of displacements through extremely sensitive phase interpretation observed on an AFM cantilever vibrating with frequency equal to 79 kHz is shown.

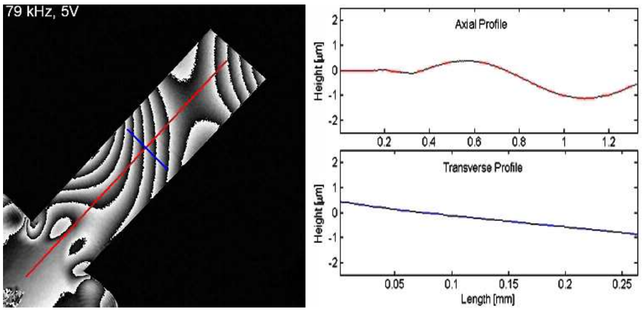

Fig. 3. Displacements on an AFM cantilever vibrating with frequency equal to 79 kHz measured with DHM stroboscopic module. On the right side profiles taken along two perpendicular directions are presented [8].

3.2. Sensofar PLu profilometers

Sensofar profilometers are unique solutions because they combine interferometry and confocal microscopy in one device. In an interferometer a light beam passes through beam splitter which directs the light to both sample surface and reference mirror. Light reflected from those surfaces recombines, resulting in an interference fringe pattern. Phase shift interferometry (PSI) has been developed to measure surface height of smooth and continuous surfaces. The sample is scanned in few steps that are a fraction of the wavelength. In the next step, algorithms produce a phase map of the surface which is then converted into a corresponding height map. On the other hand, white light vertical scanning interferometry (VSI) was developed to measure rough surfaces. In this mode the sample is scanned vertically in steps to pass every point of its surface through the focus plane. The height on given surface point is found by detecting the peak of the fringe pattern.

Confocal microscopy provides high contrast images by eliminating light that is out of the focus plane by using a spatial pinhole. The sample is scanned in steps along the vertical direction so every point on its surface passes through the focus plane. The height of the surface on a given point is found by detecting the intensity of the reflected light. Confocal profilers can measure the surface height of smooth to very rough surfaces with the highest lateral resolution that can be achieved by optical profilometry.

Sensofar PLu profilometers are successfully used for surface texture characterisation of various samples. Example of this application is given in Fig. 4, where silicon (111) surface was measured with PLu profilometer after process of wet etching with sodium hydroxide solution (monocrystalline solar cell). In this example the PLu profilometer offers possibility to control silicon surface texture, roughness and pyramid statistic during the manufacturing process. It should be mentioned that with the use of PLu profilometer three-dimensional measurements can be obtained in less than 10 s.

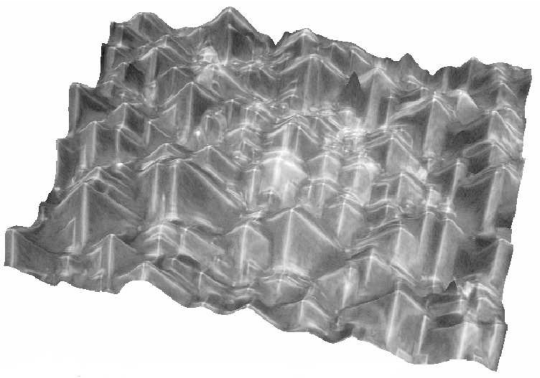

Fig. 4. Monocrystalline silicon (111) surface after wet etching made under sodium hydroxide solution. The pyramidal surface structure increases solar cell efficiency by increasing the amount of internal reflections resulting in higher light absorption [9].

3.3. Fogale interferometers family

All three-dimensional profilers made by Fogale are based on interferometry measurements. Interferometry principle was explained in previous paragraph, so we concentrate here on given application of the Fogale profilometers. They have very large field stitching capabilities, namely 3D reconstruction of large samples is made with high resolution. As an example the profile of a microcoil is presented in Fig. 5.

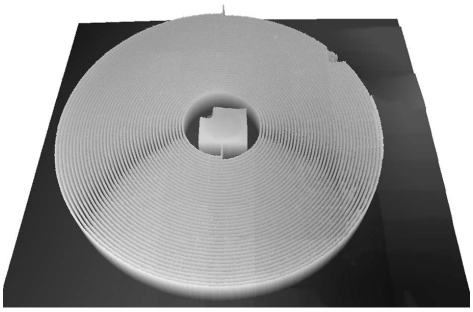

Fig. 5. Microcoil image combined from 9 × 9 profiles. The coil diameter is 2 cm [10].

3.4. PhaseView digital phase reconstruction

The digital phase reconstruction technology is based on methods of solving systems of differential equations that govern the light energy propagation that is described by Poynting's vector. In practice only the intensity, which is the average longitudinal component of the complex Poynting vector, of reflected light is measured by a camera. The phase of the incident wave is changed during reflection from a measured sample, so the three-dimensional information about its shape is imprinted on the reflected wave phase as compared to incident wave intensity. This phase difference is used to measure sample surface topography.

One of the various applications of PhaseView microscopes is roughness measurements in which calculation of the set of roughness parameters is performed in less than 5 s.

4. Mechanical properties characterisation in micro- and nanoscale

The universal mechanical tester (UMT) made by CETR is the most versatile tribometer in the world. It is a single platform with modular design for practically all common tribological tests which can be performed in different temperatures up to 1000°C, under different environmental conditions including vacuum and humidity control. It comes with different lower and upper sample drives, introducing rotational and reciprocating movements with different speeds. Moreover, it is designed in three configurations divided according to possible load ranges:

- UNMT1 for comprehensive nano- and micromechanical tests of thin films and nanostructured materials. The load range is from 1 μN to 10 N.
- UMT2 for micromechanical tests of coatings and materials, with the medium loads from 1 mN to 200 N.
- UMT3 for macromechanical test of lubricants, metals and ceramics in load range from 0.1 N to 1 kN.

Typical applications of the UMT testers include identification of the mechanical properties of thin film coatings [11], high temperature materials [12], semiconductor materials [11, 13] and many others. The UMT2 configuration with the lower sample rotary drive is presented in Fig. 6.

Fig. 6. CETR universal tribometer in the UMT-2 configuration.

5. Summary

The Schaefer group can provide scientific tools dedicated to a given research task. In each of our measuring system a user can find something outstanding which is not available in other solutions:

- True non-contact mode in Park atomic force microscopes providing ultimate resolution in ambient conditions.
- Modularity and ease of use design of all Nanosurf AFMs.
- Dynamic 3D optical characterisation performed with DHM stroboscopic module.
- The most versatile tribometer design of the UMT tester and possibility of performing material properties characterisation at temperatures as high as 1000°C.

Acknowledgments

I would like to thank the organizing committee of the NANO 2009 conference for the possibility of presenting our scientific equipment and for the great hospitality and help.

References

[1] G. Binnig, C. Quate, Ch. Gerber, *Phys. Rev. Lett.* **56**, 930 (1986).
[2] For more information see Park System application notes.
[3] Nanosurf application note.
[4] M. Wolfke, *Phys. Zeits.* **21**, 495 (1920).
[5] D. Gabor, *Nature (London)* **161**, 777 (1948).
[6] D. Gabor, *Proc. R. Soc. A* **197**, 454 (1949).
[7] E. Cuche, F. Bevilacqua, Ch. Depeursinge, *Opt. Lett.* **24**, 291 (1999).
[8] DHM stroboscopic module application brochure.
[9] Sensofar application note.
[10] Picture taken from Fogale brochure.
[11] Liu Cui, Li Guoqing, Chen Wenwu, Mu Zongxin, Zhang Chengwu, Wang Liang, *Thin Solid Films* **475**, 279 (2005).
[12] D.T. Wan, Y.C. Zhou, Y.W. Bao, C.K. Yan, *Ceram. Int.* **32**, 883 (2006).
[13] N. Satyanarayana, S.K. Sinha, *J. Phys. D, Appl. Phys.* **38**, 3512 (2005).