AFM – A TOOL FOR A STUDY OF SURFACES AND MICRO/ NANOSTRUCTURES

F. LOPOUR, D. ŠKODA, AND T. ŠIKOLA

Institute of Physical Engineering, Brno University of Technology, Technická 2, 616 69 Brno Czech Republic

ABSTRACT

Atomic force microscopy (AFM) has become a powerful tool for studies of topography of solids and micro/nanostructures. In the contribution the design principles and applications of an AFM/STM microscope developed at the institute will be presented. Particularly, the studies on AFM microscopy of thin films, micro- and nanostructures will be discussed. Additionally, nanostructures fabricated by AFM will be shown as well.

INTRODUCTION

Scanning probe microscopy (SPM) has undergone a vast development during last two decades. At present, several thousands of papers dealing both with this technique and relevant applications are published yearly. Its fundamentals have been laid by the invention of scanning tunneling microscopy (STM) by Binning and Rohrer [1] and later by its extension to observations of nonconductive surfaces [2], being known as atomic force microscopy (AFM). Since then, similar versions to AFM using probes sensitive to other sorts of interactions, e.g. magnetic [3] or electrostatic [4], have emerged. Generally, these techniques are together with AFM called scanning force microscopy (SFM).

In the Institute of Physical Engineering at Brno University of Technology (IPE BUT), an UHV compatible instrument for scanning tunneling microscopy and scanning force microscopy (STM/SFM) has been developed in close co-operation with TESCAN Ltd. These microscopic techniques will help us to investigate the initial mechanisms of thin film growth (e.g. nucleation), surface/thin film morphology (e.g. roughness), and atomic surface structures of solids in situ. In this contribution the main design features and some applications of the microscope under atmospheric conditions will be given.

PRINCIPLES OF OPERATION

Contrary to very well known STM [1], where the tunnelling current through a sharp tip as a function of the tip-to-surface distance is measured, AFM [2], [5] is based on the detection of forces between an observed sample surface and a sharp tip located at the end of a cantilever.

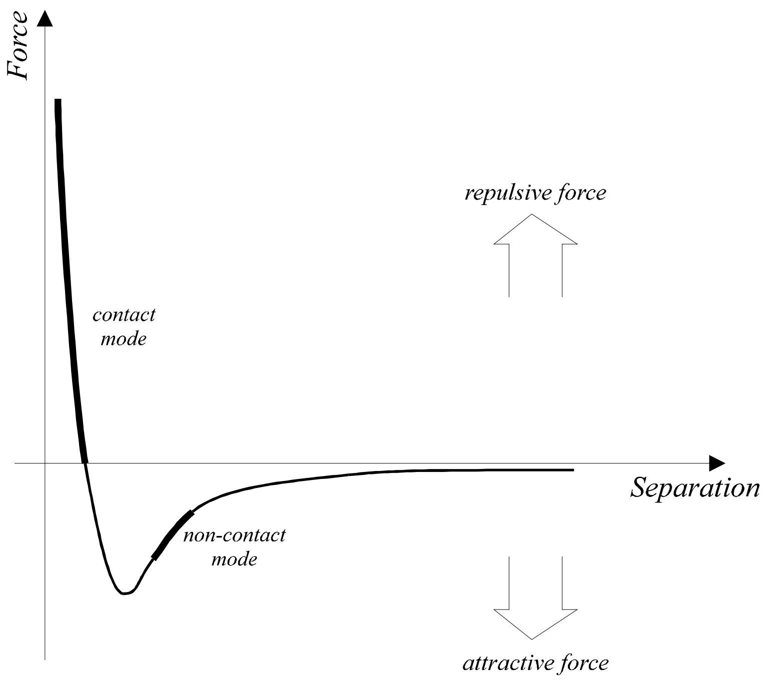

Fig. 1. Resultant force versus tip-to-sample separation.

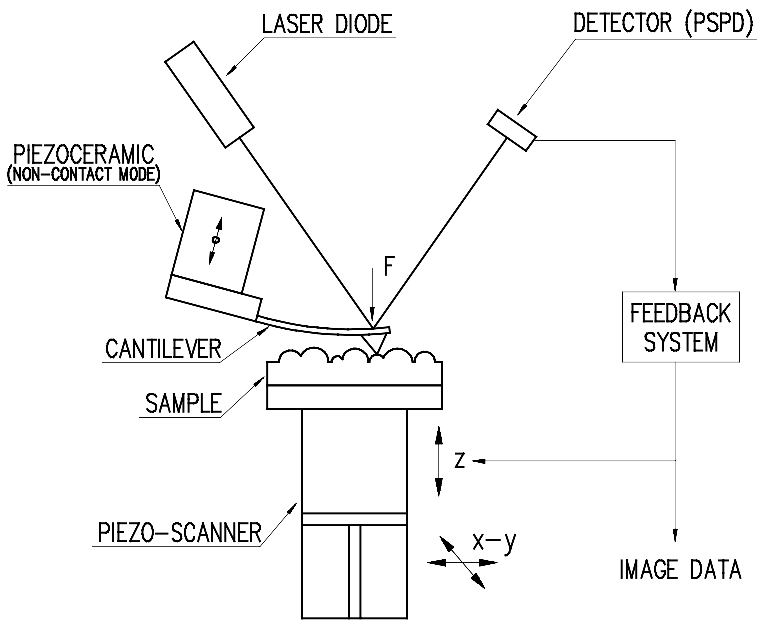

Fig. 2. Schematic view of an atomic force microscope.

The commercially available silicon or silicon nitride cantilevers are generally of a triangular shape with a typical height of 100 – 300 μm and a basis width of 40 μm [6], [7]. The diameter of the tip is as a rule in the order of tens of nanometers. For high resolution applications a specially sharpened tips with diameters close to 5 nm are used.

The general dependence of the resultant force on a tip-to-surface distance is shown in Fig. 1. If the cantilever is in the direct contact with a surface, the repulsive forces resulting from quantum mechanical principles prevail, and we speak about the *contact mode*. This method may also be used for the measurement of lateral forces acting on the cantilever and thereby for the determination of tribological properties (e.g. friction, adhesion) of surfaces [8]. On the other hand, the contact mode may lead to significant damages of the analysed surface due to scratches, defects and reconstruction caused by the tip. Hence, no atomic resolution can be obtained in the contact mode.

The *noncontact mode* of AFM, where the tip is not in a direct contact with a surface, significantly reduces this problem. The technique belongs to less destructive methods and is particularly suitable for surface analysis of soft, for example biological samples [9]. Quite recently, the true atomic resolution on selected surfaces (e.g. Si, GaAs, InP, NaCl) by means of the noncontact AFM has been demonstrated as cited in [10]. In this method, the tip interacts with surface atoms mainly by means of long-range attractive van der Waals forces and probably by an onset of covalent bonds [10], [11]. The principle of the measurements of surface topography is based on changes in the effective resonance frequency of the oscillating cantilever (vibrations of which are driven by an external generator via a piezoceramic element) with a tip-to-surface distance [12], [13].

The forces acting on a cantilever in AFM (SFM) are determined from the bending of the cantilever. The most sensitive and thus most frequent detection method of the cantilever bending is the *optical displacement method* [14]. In this method, depending on the bending of the cantilever, the laser beam after its reflection from the cantilever surface is deflected into different parts of the position sensitive photodiode (PSPD). The electrical signal from PSPD, proportional to a beam deflection, is processed via an electronic system identical with a STM electronics and might be used as a pilot signal for the feedback control of a tip-to-surface distance by a piezoscanner (constant force mode in SFM and constant current mode in STM). It is obvious that by a probe exchange the AFM (SFM) microscope might be easily modified for STM measurements.

The general scheme of an atomic force microscope based on the optical displacement method is shown in Fig. 2.

DESIGN OF THE MICROSCOPE

In Fig. 3 there is a cross view of the STM/AFM microscope assembly having been developed in IPE BUT. The whole assembly has a vertical configuration and is mounted on a CF flange

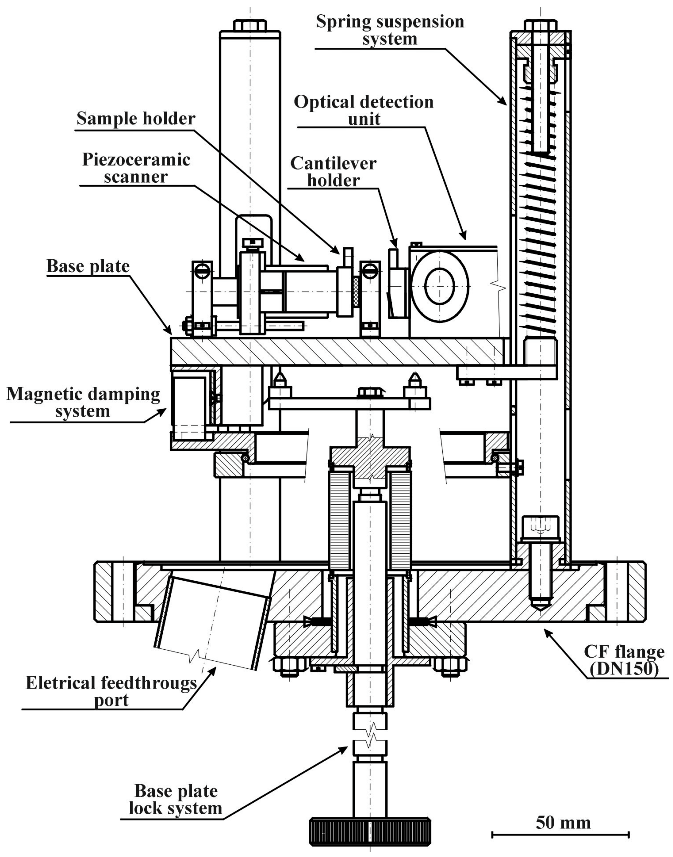

Fig. 3. A cross view of the microscope setup.

(DN 150). The base plate of the STM/AFM microscope is suspended on four springs to reduce the transfer of external vibrations into the microscope. The resonance frequency of the suspended system is 1.5 Hz. As the damping of resonance vibrations of the base plate would be under UHV conditions extremely low, this is enhanced by a magnetic damping system formed by a set of permanent magnets and copper sheets (eddy current damping effect).

The base plate bears a piezoceramic linear inchworm motor which enables a sample to approach the tip of a cantilever in a series of fine steps over a macroscopic distance. A piezoceramic scanner fixed to the inchworm motor provides scanning (maximum field ≈10 x 10 μm^2) and nanopositioning a sample over and to the tip, respectively. The optical detection system fixed to the base plate consists of a laser diode, cantilever, position sensitive photodetector (PSPD - quadrant diode) and two remotely adjustable mirrors for aligning a laser beam and for increasing a total optical path length (improving the resolution of the detection system). In the noncontact mode a piezoceramic plate, which the cantilever is fixed to, is supplied by alternating voltage from a generator in order to generate forced oscillations of the cantilever.

The control unit of the microscope and relevant software have been modified from the older version produced by TESCAN Ltd. for STM microscopes.

The samples or probes stored in a carousel placed together with the microscope in an UHV vacuum chamber can be transferred into the prismatic holders of the microscope by a vacuum tweezers. The samples and probes are delivered into the vacuum chamber via a magnetic transfer rod without breaking an UHV atmosphere. Details of the microscope design have been published in [15] and partially in [16].

APPLICATION OF STM/AFM UNDER ATMOSPHERIC CONDITIONS

The microscope was tested under atmospheric conditions for a large number of samples of different surface morphologies and chemical compositions. To verify the function of the microscope for smaller magnifications, various tests on samples prepared by microtechnologies (electron lithography, microprinting and wet or dry ion beam etching) have been carried out. The fabricated micro/nanostructures were in some cases (e.g. optical grids) applied for the calibration of the piezoscanner. It makes it possible to determine 3D

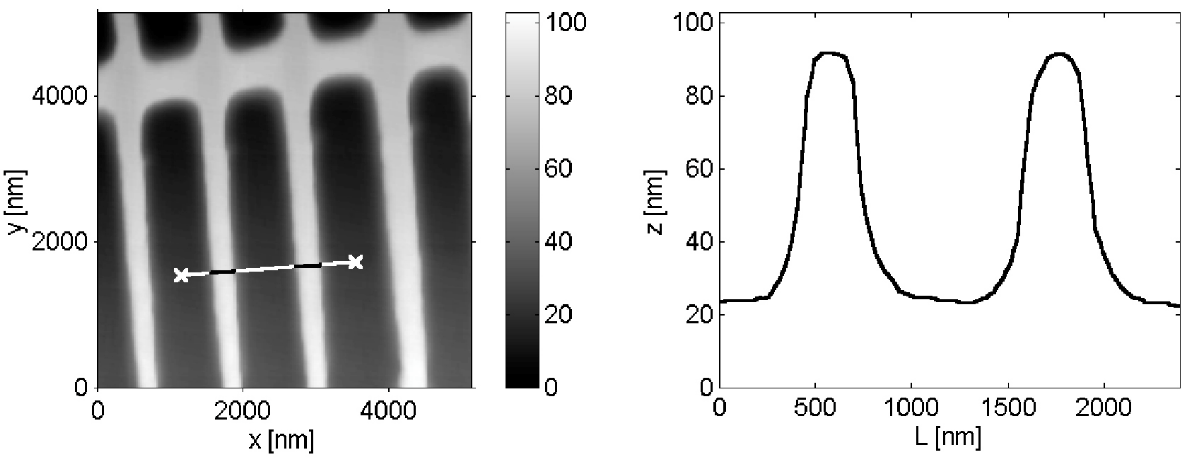

Fig. 4. A detailed view of the patterns etched by a 600 eV-argon-ion beam under normal incidence into Si substrate for 2:40 min (taken by AFM).

dimensions of the measured patterns accurately. In Fig. 4 an AFM image (contact mode) of the microstructure of Si (111) etched by Ar^+ ions (600 eV) is shown. Etching was made through a resist mask prepared by electron lithography. From the obtained structure profiles it was found that argon ion beam etching leads to the formation of silicon microstructures with a maximum pattern height of 100 nm. The height of patterns etched by Ar^+ ion beams into Ag thin films was approximately 200 nm (higher etching rate of Ag).

The tests mentioned above have proved the correct function of the microscope for maximum fields of view and small vertical magnifications. Additionally, it has been found the microscope also works properly for roughness measurements of very smooth surfaces of thin films (e.g. TiN, ZrN) prepared by ion beam assisted deposition (Fig. 5). In these experiments small fields of view and big vertical magnifications should be applied. The root mean square deviations of the measured vertical corrugations of deposited films for the field of view of 300 x 300 nm^2 were below 1 nm at any applied assisting ion energies (measured in the AFM contact mode).

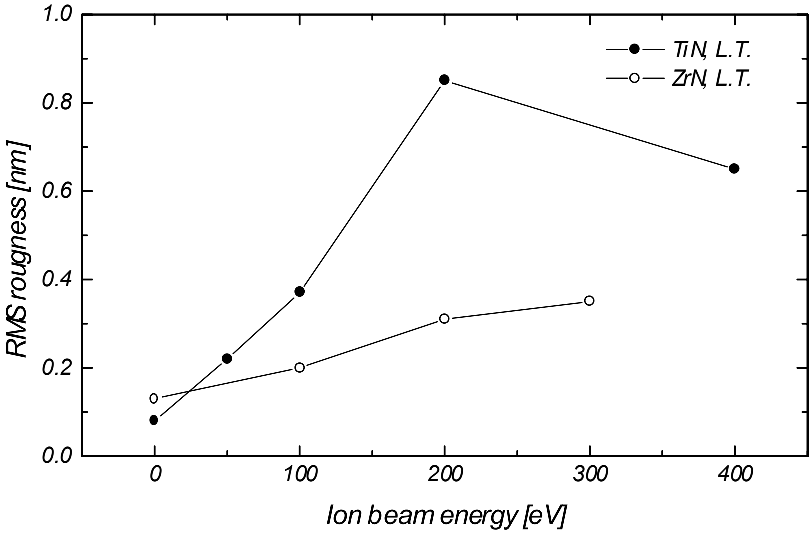

Fig. 5. Surface roughness of TiN and ZrN films as a function of the assisting nitrogen-ion energy (measured by AFM).

Recently, it has been shown, that similarly to STM, AFM can be also used for fabrication of nanostructures by local anodic oxidation of non-noble metal thin films (e.g. Ti, Al) deposited on $Si0_2$/Si substrates, silicon and GaAs surfaces. The oxides growth on these substrates by application of the voltage between the surface and the conductive AFM tip. There is a threshold voltage at which the anodic oxidation starts (typically about 5 volts). The width and height of lines changes with conditions of tip “writing”, such as tip-sample voltage, speed of tip writing, tip sharpness, humidity, etc. As water adsorbed on substrates acts as an electrolyte, lower humidity results in lower and narrower lines. In Fig. 6 a series of nanowires fabricated for a set of different writing speeds (from 20 to 120nm/s) at a constant tip-sample voltage of 6 V by the microscope described in previous sections is shown. The thickness and height of the individual nanowires grow as the writing speed decreases. A typical width of the nanowires was in the order of tens of nanometers. Decreasing the sizes of local oxide structures close to 10 nm, *single electron transistors* (SETs) working at room temperature might be designed in this way [17].

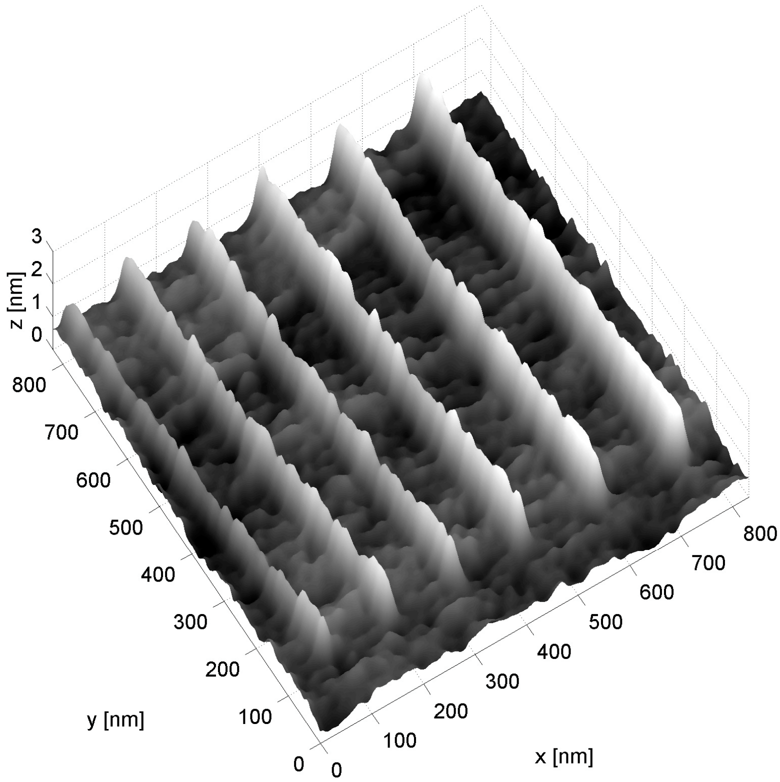

Fig.6. A series of nanowires fabricated for a set of different writing speeds (from 20 to 120 nm/s) at a tip-sample voltage of 6 V.

CONCLUSIONS AND NEXT DEVELOPMENT

In the contribution the principles and main design features of the UHV STM/AFM microscope developed in IPE BUT in a close cooperation with the company TESCAN Ltd. have been described. Experimental results in the field of micro- and nanotechnology performed by this microscope have been presented. Next research activities of the group will be aimed at applications of the microscope under UHV conditions, implementation of the noncontact AFM mode, and nanostructure fabrication via STM/AFM. The mesoscale systems will be prepared by a local AFM oxidation of the narrow ultra-thin metal wires (across or along the wires) deposited on SiO_2/Si substrates ($\approx$ 1 μm) and studied by concurrent resistivity measurements on the wires. In this way, related quantum mechanical phenomena will be studied.

ACKNOWLEDGEMENTS

Many thanks to Tescan s.r.o. for its help in the design of STM/SFM instrument. The work was supported by the Czech Grant Agency (Grant No. GA101/98/0781), and Ministry of Education (ME 334, SEZ:J22/98:262100002, Aktion No.27p13).

REFERENCES

[1] G. Binning, H. Rohrer, Ch. Gerber, E. Weibel, Phys. Rev. Lett. 49 (1982), 57.
[2] G. Binning, C. F. Quate, and Ch. Gerber, Phys. Rev. Lett. 56 (1986), 930.
[3] Y. Martin a H. K. Wrickramasinghe, Appl. Phys. Lett. 50 (1987), 1455.
[4] Y. Martin , D. W. Abraham, and H. K. Wrickramasinghe, Appl. Phys. Lett. 52 (1988), 1103.
[5] D. Sarid: *Scanning Force Microscopy*, Oxford University Press, New York, 1994.
[6] ThermoMicroscopes: www.thermomicro.com.
[7] Silicon-MDT Ltd.: www.siliconmdt.com.
[8] J. M. Neumeister, W. A. Ducker, Rev. Sci. Instrum. 65 (1994), 2527.
[9] A. Ikai, Surface Science Report 26 (1996), 261.
[10] R. Kalousek, F. Lopour, P. Dub and T. Šikola: Physical principles and resolution limits of the noncontact AFM mode – *simulation of cantilever oscillations (in Czech)*, Čes. čas. fyz., in print.
[11] R. Kalousek, F. Lopour, P. Dub and T. Šikola, Surface and Interface Analysis 30 (1), 2000, 292.
[12] T. R. Albrecht, P. Grütter, D. Horne and D. Rugar: J. Appl. Phys. **69** (1991), 668.
[13] L. Howald, H. Rudin, and H.- J. Güntherodt: Rev. Sci. Intsr. **63** (8), (1992), 3909.
[14] C. A. J. Putman, B. G. De Grooth, N. F. Van Hulst and J. Greve, Ultramicroscopy 42 - 44 (1992), 1509.
[15] F. Lopour: Design of the UHV STM/AFM for surface studies (in Czech), diploma thesis, IPE BUT Brno, 1997.
[1] F. Lopour, D. Škoda and T. Šikola: Design and Application of UHV SPM (in Czech), Čes. čas. fyz., in print.
[17] K. Matsumoto et al., Appl. Phys. Lett., 68 (1), (1996), 34.