University of Szeged

Department of Optics and Quantum Electronics

Investigation of Dynamical and Static Operation Modes of Atomic Force Microscope

PhD Thesis

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Szeged
2004

Introduction

The atomic force microscopy is a relatively new field of science. Despite its short history, the atomic force microscopy changed dramatically in the last two decades. While the construction of the first atomic force microscope required extreme technical effort in 1986, the first commercial instruments appeared in the middle of 90's already. Nowadays, atomic force microscope is widespreadly used in the molecular biology, material science and solid state physics.

The market of the atomic force microscopes exceeded the 500 million USD in 2002, and 60 % of instruments were sold for the industrial sector. These data clearly signify that the atomic force microscope is widely used not only in the basic research, but also in the applied research and in the quality insurance.

The increasing number of applications fuels a quest for new developments. The atomic force microscope should be faster and more reliable. To fulfill these aims, better understanding of AFM operation is necessary.

In the beginning of my doctoral work in 2001, I choose the atomic force microscope as research topic. In the first part of my thesis I investigate the origin of the observed topography artifacts using numerical simulations. Next, I study the dynamical properties of the probe operating in amplitude modulated mode in presence of surface forces. My aim was to find an optimal parameter setting which enables faster operation. To artificially modify the dynamical properties of the amplitude modulated probe, Q control can be used. Hence, I examined the properties of the Q control. Finally, I measured mechanical properties of soft samples.

Tools and methods

I mainly used a *Topometrix Explorer* atomic force microscope during the experimental work. The mechanical properties of soft samples were investigated by a *WITec α-SNOM* atomic force microscope. To realize amplitude modulated operation, I applied a *WITec AC Unit* and a slightly modified *DAS1414B* digital data acquisition system. Moreover, I employed a *Stanford SR-830* digital lock-in amplifier for describing function measurements and local mechanical spectroscopy. The Q control, I used during the experiments, was in-house built.

The simulation program of the AFM operation was written in *C++*. The program was compiled and linked by the complier and linker of the *GNU* project. The simulations were performed under *Solaris* and *Linux* environment.

The dynamics of the amplitude modulated probe was studied using step functions and describing function method. To determine the spring constant of the cantilever, I used the so called *Sader* method.

New scientific results

1. I presented a simple model for tapping mode imaging of weakly bound carbon nanoparticles standing on graphite surface. My results show that in case of high amplitude tapping mode the particles are displaced by the probe, and the hard surface appears in the topography signal only. On the contrary, in case of low amplitude tapping mode the particles are not displaced but imaged. These results are in good agreement with the experimental observations. [T1]

2. I investigated the origin of topography artifacts observed at atomic scale steps on graphite surface. I found that the increased surface energy can be accounted for the

topography artifacts. The magnitude of the artifacts changes with both the free and setpoint amplitude as well as with surface energy. [T2]

3. I studied the dynamical properties of the tapping mode probe in presence of surface forces with the help of numerical calculations and experiments. To study the dynamic response of the probe, I applied step function - like perturbations. I proved the nonlinear behavior of the probe and showed that both the maximal tip - sample force and the dynamical character of the probe depends on the relative drive frequency and quality factor. [T3]

4. Besides the previous method, I investigated the dynamical properties of the tapping probe using the describing function method. I established that the probe has a low pass character. My calculations show that in case of blunt tips a peak appears in the describing function which could lead to imaging artifacts. Furthermore, I demonstrated that the increase of the free amplitude significantly widens the probe bandwidth.

5. I compared the analytical and numerical solutions of the Q controlled tapping mode probe force equation which can be found in the literature. I ascertained that the numerical and analytical solutions are different. To elucidate the origin of the deviation, I repeated the numerical calculations. I found that an accurate simulation of the Q controlled probe requires significantly shorter step sizes than the conventional tapping mode probe does. The analytical and numerical solutions agree well provided short enough step sizes are used. [T4]

6. According to the conventional explanation, the enhanced topography pictures taken by Q controlled tapping mode AFM are caused by the increased slope of the amplitude - distance curve of the Q controlled probe. I proved that the Q control technique increases the slope of the amplitude - distance curves in case of soft samples only. Furthermore, there exists a maximal slope of the amplitude - distance curves which cannot be exceeded even with Q control. Hence, the conventional explanation of Q control mode of

action can be correct on soft samples only. In addition, I gave method to find the optimal value of the quality factor. [T4]

7. I proved that the Q controlled system is more sensitive to material parameters change which may cause the enhanced phase contrast and resolution. [T4]

8. I presented a new measurement procedure for determination of Young's modulus and viscosity of soft samples. In this procedure, a low frequency, small amplitude sinusoidal signal is applied to the Z piezo drive while the tip is in contact with surface. I determine the phase and the amplitude of the emerging vibration from the cantilever deflection signal. The surface force gradient and the viscosity coefficient can be calculated from the measured amplitude and phase values. By performing several measurements at different tip - sample force values, the contact part of the force - distance curve, the Young's modulus and the viscosity of the sample can be determined. The advantage of this procedure over the conventional force - distance curve measurements that the results are less influenced by noise. Meanwhile, special double cantilever system is not required like in case of differential AFM. [T4]

9. I proposed the idea that the Q control technique is suitable not only for the tapping mode, but also for pulsed force mode. In addition, I demonstrated experimentally the capability of the Q control technique to enhance the pulsed force mode frequency.

Publications that the theses are based on

[T1] Á Mechler, J. Kokavecz, P. Heszler: The observability of poorly bound powder – like material on hard surface by atomic force microscopy, Materials Science and Engineering C 15 pp. 29 (2001)

[T2] J. Kokavecz, P. Heszler, Z. Tóth, Á. Mechler: Effect of Step function - like Perturbation on Intermittent Contact Mode Sensors: a response analysis, *Applied Surface Science* **210** pp. 123 (2003)
[T3] Á. Mechler, J. Kokavecz, P. Heszler, R. Lal: Surface energy maps of nanostructures: Atomic force microscopy and numerical simulation study , *Applied Physics Letters* **82** pp. 3740 (2003)
[T4] J. Kokavecz, Z. L. Horváth, Á. Mechler: Dynamical Properties of the Q controlled Atomic Force Microscope, submitted to the *Applied Physics Letters* (2004)

Further publications

[1] I. Pócsik, M. Veres, M. Füle, M. Koós, J. Kokavecz, Z. Tóth, G. Radnóczi: Carbon nanoparticles prepared by ion-clustering in plasma, *Vacuum* **71** pp. 171 (2003)
[2] B. Hopp, N. Kresz, J. Kokavecz, T. Smausz, H. Schieferdecker, A. Döring, O. Marti, Z. Bor: Adhesive and morphological characteristics of surface chemically modified polytetra-fluoroethylene films, *Applied Surface Science* **221** pp. 437 (2004)
[3] M. Csete, J. Kokavecz, Z. Bor, O. Marti: The existence of sub - micrometer micromechanical modulation generated by polarized UV laser illumination on polymer surfaces, *Materials Science and Engineering* C **23** pp. 939 (2003)
[4] L. Landström, J. Kokavecz, J. Lu, P. Heszler: Characterization and Modeling of Tungsten Nanoparticles Generated by Laser-assisted Chemical Vapor Deposition, accepted for publication in *Journal of Applied Physics* (2004)

Posters

[1] R. Mingesz, Z. Gingl, J. Kokavecz, Á. Mechler, Z. Tóth: DSP-based extension to the IC mode Topometrix AFM, V. NC-AFM Conference, Montreál, Kanada (2002)
[2] J. Kokavecz, Á. Mechler, Z. Tóth, P. Heszler: Effect of step function – like perturbation on Intermittent Contact Mode sensors: A response analysis, V. NC-AFM Conference, Montreál, Kanada (2002)
[3] J. Kokavecz, A. Gigler, A. Döring, S. Hild, O. Marti: Novel Method for Characterization of Elastic and Viscoelastic Properties of Soft Samples Using Atomic Force Microscopy, III. SPM on Polymers Conference, Kerkrade, Hollandia (2003)