ELSEVIER

Ultrasonics 36 (1998) 499–503

Ultrasonics

Ultrasonic surface vibration investigation by atomic force microscopy

V. Snitka *, A. Ulcinas, M. Rackaitis, V. Mizariene

Research Center 'Vibrotechnika', Kaunas University of Technology, Studentu 65, LT-3031 Kaunas, Lithuania

Abstract

This study presents a novel method to study ultrasonic surface vibrations and ultrasound influence on the surface topography image obtained by atomic force microscope (AFM). The vibrations of piezoelectric plate in the frequency range of 10–200 kHz were investigated. The experiments show the possibility of using the AFM for the detection of acoustic waves and the measurement of the amplitude of vibrations and the distribution of electric field in a piezoplate. Influence of the ultrasonic surface vibrations on AFM tip–surface interaction was noticed. Lateral surface vibrations reduced the tip–surface adhesion down to zero. Experimental results show that the AFM cantilever in the presence of vibrations has a lateral vibration mode coupled with a normal vibration mode. It increased the contrast of an image obtained in the lateral force mode. © 1998 Elsevier Science B.V.

Keywords: Atomic force microscope; Surface vibrations; Dynamic measurements

1. Introduction

Atomic force microscopy, which originally was intended to image the surface and measure its topography, is also extensively used to probe different properties of the surface such as microadhesion, surface forces etc. Its principle of operation is based on sensing the interaction between the investigated surface and very sharp tip integrated on a flexible cantilever which is kept at a small distance from the surface.

Recently AFM started to extend its operation to the range of high frequencies, and the importance of precise measurement of the dynamics of tip–surface interaction is constantly increasing. Rabe et al. investigated vibrations of free- and coupled-to-surface rectangular AFM cantilevers in a spectral range of 100 kHz–10 MHz [1]. The same group used AFM at high frequencies to image surface areas with different elastic properties [2]. Burnham et al. employed a similar technique called scanning local acceleration microscopy [3]. Yamanaka et al. used ultrasonic surface vibrations for nanometer subsurface imaging [4].

In this paper we present the results of the study of dynamics of the tip and surface interaction. A new method for the determination of cantilever vibration was used. The influence of ultrasonic surface vibrations on the tip–surface adhesion is shown.

2. Experiment

The ultrasound influence on the surface morphology image was investigated with a Quesant Instrument Corp. Atomic Force Microscope Q-Scope 250®. Rectangular and triangular Si_3N_4 cantilevers with 0.05 and 0.01 N m^{-1} spring constants respectively were used for the experiments. The lengths of rectangular and triangular cantilevers were 180 micron and 320 micron respectively. Cantilever vibration was investigated using an oscilloscope mode of the AFM. It allowed us to plot the AFM sensor signal in a time domain or to plot a Lissajous figure which is derived summing the top–bottom and left–right signals of AFM sensor. To enable this, real time software was implemented in addition to the original software. For the investigation of the differences between the exciting signal and the cantilever signal we mounted an additional photodetector which registered the cantilever signal. The plot of the signal from the detector versus the exciting signal was registered using an oscilloscope. In addition, AFM images were obtained using different modes (lateral, z height, broadband) with applied vibrations, with amplitudes up to 0.25 micron.

Surface vibrations were excited by applying a high

* Corresponding author. Fax: +370-7-766652; e-mail: vsnitka@vb.ktu.lt

PII S0041-624X(97)00063-2

frequency voltage from a generator to a piezoplate. The investigated sample was glued on the plate. Normal, lateral and bimodal vibrations in the frequency range of 10–200 kHz were investigated during the experiments. Surface vibrations were detected and tip–surface interaction dynamics was investigated using the force calibration mode of the Nanoscope II and the Q-Scope. High frequency ultrasound influence on the force-displacement curves was measured using a commercial AFM Nanoscope II with a triangular Si_3N_4 cantilever with a spring constant of 0.06 N m^{-1}. The cantilever was 200 μm long and 36 μm wide.

In force calibration mode, the sample is scanned in the vertical direction involving contact and further retraction of the tip from the surface. The force acting on the cantilever is registered. The mode enabled us to obtain data representing the force dependence on tip-sample separation (called 'force–displacement curve'). All experiments were performed in air, under normal laboratory conditions.

3. Results and discussion

Force-displacement curves for vibrations of different modes are presented in Figs. 1–4. The interaction between the cantilever tip and the vibrating surface was observed at separation distances up to a few micrometers (Fig. 1). It is seen that vibrations of different modes (Fig. 2) can be clearly distinguished up to 70 kHz, and that the amplitude of the vibrations does not change appreciably with separation between cantilever and surface. It shows that the vibrations of the cantilever are activated not by molecular or electrostatic forces, or mechanical excitation of the system, but presumably by direct transmission of surface vibrations to the cantilever through an air gap. At the same time, non-linear interaction between tip and surface is seen as bending of the cantilever towards the surface. In addition, it was noticed that the amplitude of the cantilever vibrations is higher than that of the surface. This can be attributed to electrostatic interaction between the electric field of vibrating piezoplate and the charge on the tip. The experiments show that low frequency vibrations do not effect the tip–surface adhesion force.

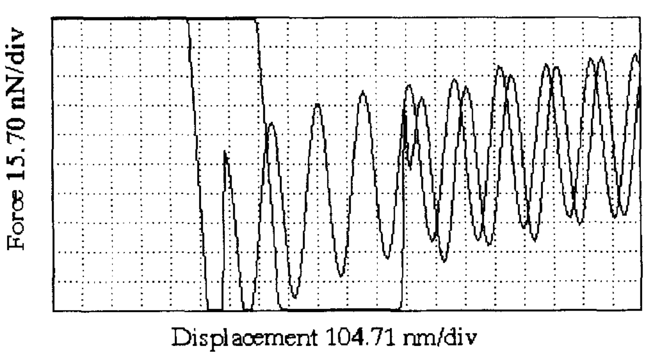

Fig. 1. Force-displacement curve of normal 10 kHz frequency vibrations measured by Nanoscope II.

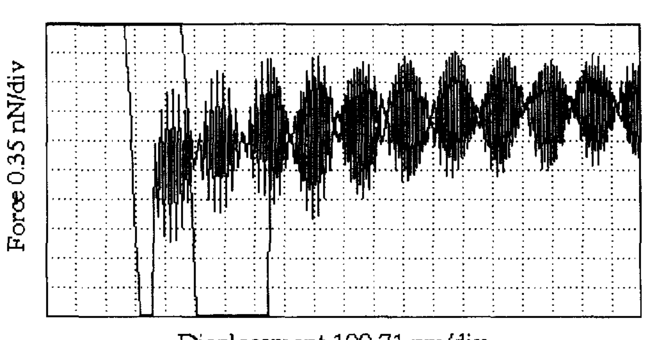

Fig. 2. Force-displacement curve of the bimodal 44 kHz frequency vibrations measured by Nanoscope II.

The different results were obtained with Q-Scope AFM. The frequency dynamic range of the force-displacement measurements was found to be significantly less and vibrations were observed up to only few kilohertz.

Fig. 3 presents the static mode (i.e. vibrations are not applied to the sample) of the force-displacement measurements. After the tip contact with the surface, the reverse motion of z-scanner is activated, and the cantilever has to overcome tip–surface adhesion force to separate from the sample. Because of that, discontinuities in the force–displacement curve are seen when the cantilever is separating from the sample. From the distance that the z-scanner has to travel to make cantilever overcome tip–surface adhesion force (adhesion distance), tip–surface adhesion force can be calculated, and was 240 nN in the case of Fig. 3.

The force-displacement curve presented in Fig. 4 shows that ultrasonic surface vibrations can significantly (down to zero) decrease the tip–surface adhesion. Cantilever motion was investigated using an oscilloscope mode. The results are given in Figs. 5–8. These figures show that the motion of the cantilever consists of coupled normal and lateral vibration modes. Fig. 5 shows the triangular cantilever motion at 5 kHz frequency. It is clear that even at a low frequency lateral vibration mode takes place in the cantilever's motion. According to the Lissajous figure method [5], one can notice that the normal vibration frequency is almost two times higher than the lateral vibration mode frequency. Changing exciting frequency in a wide range (20 Hz–10 kHz) does not significantly change the figure, only the phase shift between the normal and lateral vibrations is changed, which is seen as the Lissajous figure's rotation. The amplitudes of normal and lateral modes depend significantly on setpoint force of cantilever. When the cantilever is lowered down to the surface

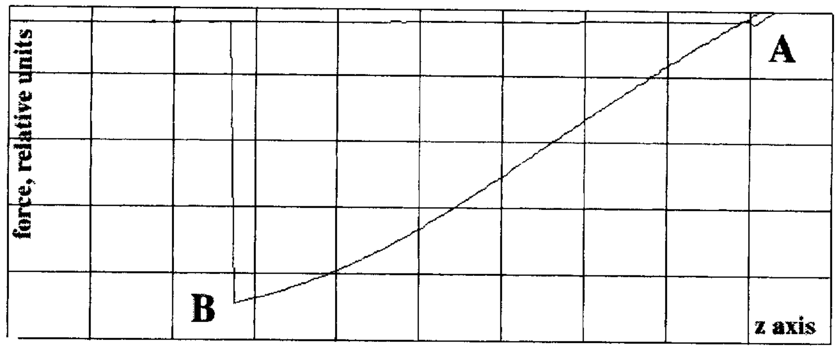

Fig. 3. Force–displacement curve of the glass substrate measured with Q-Scope. (A) tip–surface contact point; (B) tip–surface separation.

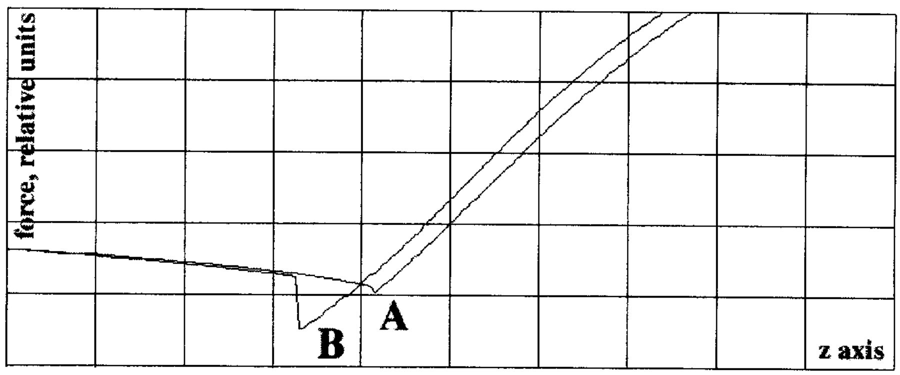

Fig. 4. Force–displacement curve of the same surface activated by 178 kHz frequency vibrations.

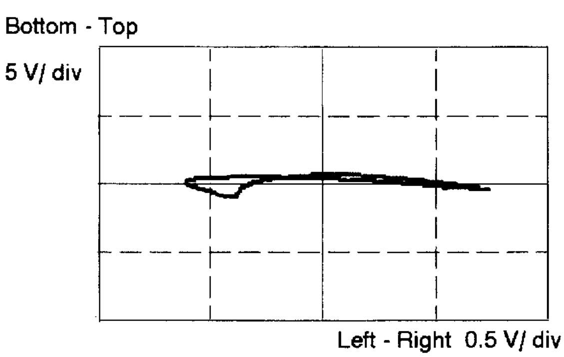

Fig. 5. Triangular cantilever vibration at 5 kHz frequency.

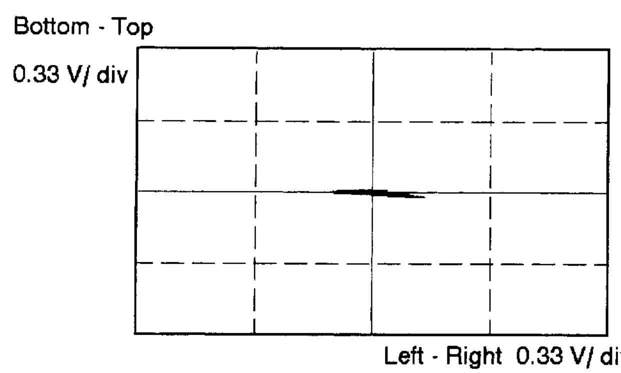

Fig. 7. Rectangular cantilever motion at 8 kHz frequency.

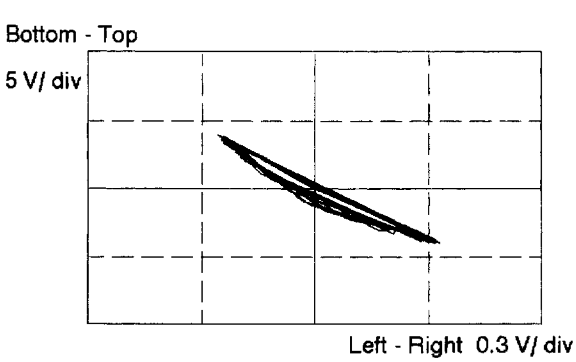

Fig. 6. Rectangular cantilever motion at 2 kHz frequency.

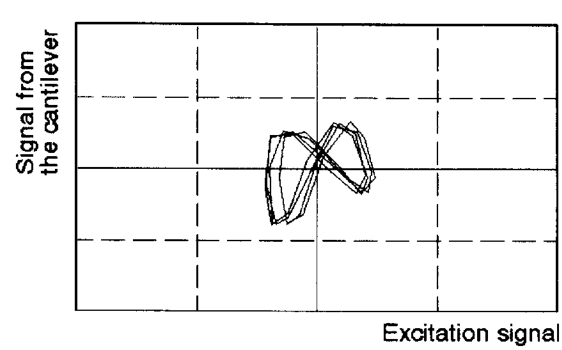

Fig. 8. Lissajous figure of the triangular cantilever at 10 kHz frequency.

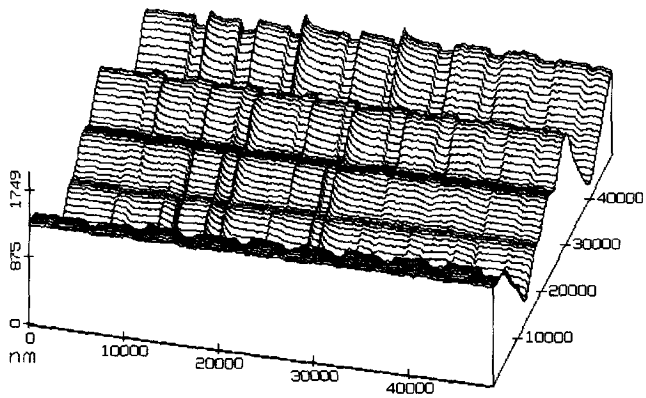

Fig. 9. 3-D image of single surface line scan extended in time with vibrations switched on and off.

so that elastic force acting on cantilever increases, the amplitude of normal vibrations decreases and the phase shift between normal and lateral modes changes. This occurs because of changes in damping and spring constant of surface-cantilever mechanical system.

The rectangular cantilever exhibits a slightly different kind of motion (Figs. 6 and 7). The lateral vibration has the same frequency as the normal, but there is a 30° phase shift between them. The phase shift changes when the frequency is changing. Fig. 7 shows the results of the rectangular cantilever vibration at 8 kHz frequency. It is clear that the lateral vibration mode has significantly higher amplitude than the normal vibration mode. Registration of the cantilever motion gives the sample's image. Vibrations significantly influence the cantilever motion. That has to induce changes in the AFM image.

Fig. 8 shows the oscilloscope plot where the horizontal axis represents the exciting signal and the vertical axis signal from the cantilever. As is seen from the figure, the cantilever vibrates in double frequency in comparison with the exciting signal. This frequency-doubling phenomena was observed for the triangular cantilever in a wide frequency range (10 Hz–20 kHz). The rectangular cantilever does not exhibit such behavior. It was noticed that the frequency doubling significantly depends on the tip surface distance. This is just the first result of the effect. It is necessary to investigate further.

In order to investigate the influence of the surface vibrations to surface topography, measured by AFM, experiments were performed scanning one surface line with vibrations periodically switched on and off. The 3-D image of the surface line extended in time is shown in Fig. 9. The measured surface shift in the vertical direction when vibrations were exited was 0.2 micron. The measured image cross section shows a significant shift in the lateral direction also. Vibration-activated surface attraction or repulsion of the cantilever was noticed depending on the vibration frequency and amplitude. In our case the cantilever was attracted to the surface. A similar shift in the effective position of the cantilever is reported in [2].

The higher contrast in the surface morphology is clearly seen from Fig. 10(a) in comparison with Fig. 10(b). Fig. 10(c) shows the new surface features (1, 2) related with the surface friction forces made clear at a frequency of 76.6 kHz, and the general image contrast is higher than in the case of Fig. 10(a),(b).

4. Conclusions

We have performed experiments measuring the interaction of the vibrating surface and AFM cantilever. We used three methods to investigate the influence of the surface vibrations to the AFM cantilever and image formation. The force–distance curves allow us to evalu-

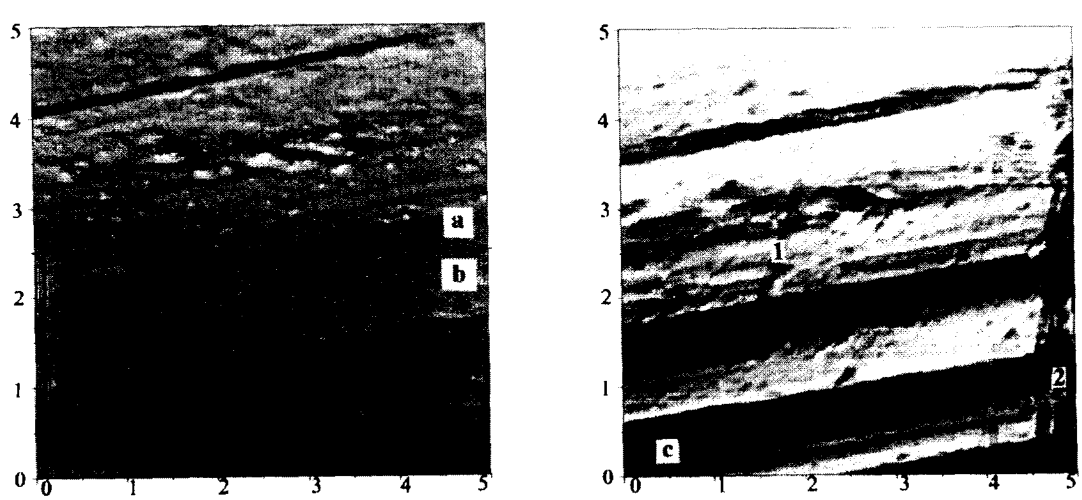

Fig. 10. The Q-Scope AFM lateral force mode image: (a) vibration frequency of 176 kHz, 0.2 micron amplitude; (b) without vibrations; (c) vibration frequency of 76.6 kHz, vibration amplitude 0.2 micron.

ate the amplitude of the vibrations. The Lissajous figures give us information about complex cantilever and cantilever-surface vibrations, whilst obtaining images with vibrations enables us to visualize areas of the sample with different properties. The investigations explore the complexity of the cantilever vibration phenomena.

Our results show that amplitudes of the normal and lateral modes and the phase shift between them strongly depends on setpoint force. Frequency doubling of the triangular cantilever vibration is noticed, but this effect needs further investigation.

References

[1] U. Rabe, K. Janser, W. Arnold, Rev. Sci. Instrum. 67 (9) (1996) 3281.

[2] U. Rabe, V. Scherer, S. Hirsekorn, W. Arnold, Proc. Nano IV, Beijing, 1996.

[3] N.A. Burnham, A.J. Kulik, G. Gremaud, P.-J. Gallo, F. Oulevey, J. Vac. Sci. Technol. B 14 (1996) 794.

[4] K. Yamanaka, H. Ogiso, O. Kolosov, Appl. Phys. Lett. 64 (1994) 178.

[5] R.W. Pohl, Mechanik, Akustik und Warmelehre, Springer, Berlin, 1964, p. 60 (Translation to Russian, Nauka, Moscow, 1971).