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Tapping mode and elasticity imaging in liquids using an atomic force microscope actuated by acoustic radiation pressure

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We report the use of the radiation pressure generated by a focused acoustic beam near 170 MHz to implement tapping mode and elasticity imaging using the atomic force microscope (AFM) in liquids. Since the acoustic radiation force can be localized on an area of a few microns in diameter, this method enables efficient excitation and spatial mapping of both higher-order flexural and torsional modes of AFM cantilevers in liquids. We exploit the sensitivity of the higher-order cantilever mode shapes to the tip–sample contact stiffness for elasticity imaging. We present higher-order flexural and torsional AFM cantilever mode shape measurements in the 1–250 kHz range, and initial results on elasticity imaging on a sample with 1.3 μm thick patterned photoresist layer on silicon obtained at 50 kHz.
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Dynamic imaging and measurement modes of the atomic force microscope (AFM) in liquids have been increasing both in number and significance in recent years.[1–3] In order to exploit the capabilities of the AFM in a wide range of applications from biology to surface science, researchers have used cantilevers with different shape, size, and materials in a variety of immersion fluids.[3–5] This variation in applications creates significant challenges for the actuation of AFM cantilevers in liquids. Furthermore, for accurate evaluation of the AFM data one needs to characterize not only the spring constant but also the higher-order modes and the vibration profiles of the cantilever in the liquid over a wide frequency range.[6,7] A piezoelectric transducer attached to the AFM system to vibrate the whole assembly is the most widely used technique for actuating AFM cantilevers.[8] However, this technique suffers from significant spurious mechanical resonances. Although thin piezoelectric and magnetic films deposited on cantilevers result in a ‘‘clean’’ excitation, these methods limit the type of cantilevers to a narrow range, prohibiting the use of very small, low-noise ones for force spectroscopy and fast imaging.[9–12]

The acoustic radiation pressure (ARP) generated by a focused acoustic transducer operating in the 100–300 MHz frequency range has recently been introduced as an AFM cantilever actuation mechanism in fluids.[13] This method does not require any special cantilevers and it is very suitable for array fabrication. In this letter, we show that this technique can be used for tapping mode imaging in liquids, avoiding spurious fluid–cell cavity resonances. We use the large bandwidth and point-like forces generated by the ARP actuator for active characterization of the flexural as well as the higher-order torsional modes of AFM cantilevers in liquids. We also demonstrate that the dependence of the cantilever mode shape on the contact stiffness can be exploited to form elasticity images. By positioning the acoustic radiation force on the AFM cantilever at a judiciously chosen location, the sensitivity to sample elasticity is optimized to obtain elasticity images in liquids similar to the ultrasonic AFM images in air.[14]

The experimental setup used in this study is shown in Fig. 1. It consists of a commercial AFM system with the addition of a rf signal generator to drive the ARP actuator and a lock-in amplifier to record the amplitude and phase information.[15] The ARP actuator is fabricated on a glass substrate and it consists of a piezoelectric transducer and a Fresnel lens to generate and focus the sound in the fluid. The ARP actuator is placed underneath the cantilever and the imaging sample is located on the actuator surface near the acoustic Fresnel lens. The cantilever and the sample–actuator assembly are immersed in water. The rf amplifier is used to drive the transducer with a carrier frequency of 174 MHz. The rf signal is amplitude modulated with the desired frequency to apply time harmonic forces. The lock-in ampli-

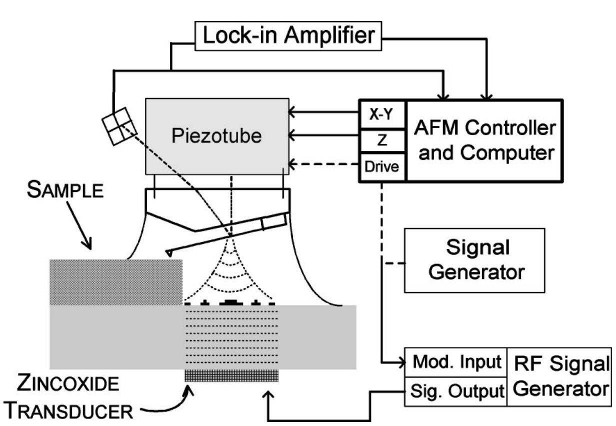

FIG. 1. Schematic of the experimental setup used to perform tapping mode, elasticity imaging, and resonant mode characterization.

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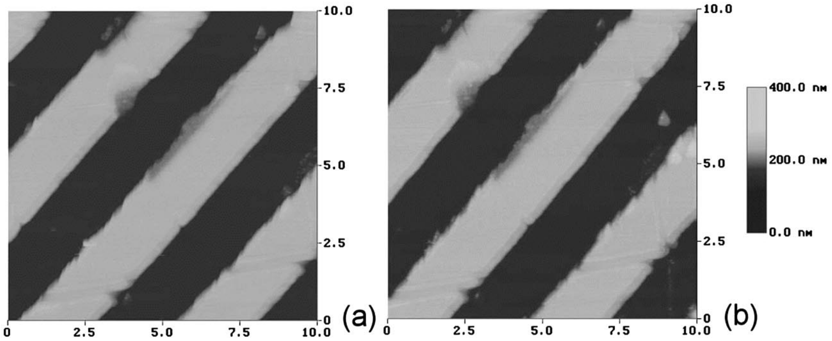

FIG. 2. AFM images of a 100 nm thick 2 μm wide aluminum grating on silicon substrate obtained in standard contact mode (a) and in tapping mode with ARP actuator (b). Image size is 10 μm square.

fier is synchronized with the amplitude modulation (AM) signal to the rf amplifier and the output of the lock-in is used to measure the cantilever vibration mode profile with high signal to noise ratio.

First, we used the ARP actuator to obtain regular tapping mode images in water. Our current experimental setup shown in Fig. 1 imposes certain restrictions on the choice of the sample and cantilever. The thickness of the sample needs to be close to the focal length of the acoustic Fresnel lens (360 μm) and the features on the sample must be close to the edge of the sample. In addition, the image size is limited by the width and length of the AFM cantilever and the size of the acoustic beam. Therefore, long and wide cantilevers are desirable to obtain reasonable-sized images. We used a 450 μm long rectangular silicon probe and a sample with large periodic features to satisfy these requirements.[16] The sample consisted of a 300 μm thick silicon substrate with 100 nm thick, 2 μm wide aluminum lines with 4 μm pitch. Figure 2(a) shows a standard contact mode image of this sample, whereas Fig. 2(b) shows the tapping mode image of the same region obtained by using the ARP actuator to vibrate the cantilever at 2.6 kHz, close to its first flexural resonance in water. During tapping mode imaging, a vibration amplitude of 200 nm is obtained in water with an input rf signal power of 10 dBm. The excellent correspondence between the contact and tapping mode images shows the viability of the actuation method for tapping mode imaging. We should also point out that integrating the ARP actuator to the cantilever holder will overcome the setup limitations imposed on the sample and cantilever selection.

A unique property of the ARP actuator is its ability to generate point-like forces on the AFM cantilever. It has been shown that the force generated by the radiation pressure of the acoustic field is confined to the size of the ultrasonic beam at the focal point.[17] At the 174 MHz frequency used in this study, the size of the focal spot is approximately 9 μm in diameter. The localized nature of the applied force can be used to characterize the flexural and torsional modes of the cantilever by moving the focal spot position over the cantilever and recording the oscillation amplitude and phase from the deflection signal. The ac excitation signal, regularly applied to the *Z* piezo by the commercial AFM system, is directed to the AM input of the rf signal generator to drive the ARP actuator (see Fig. 1). The tip of the cantilever is placed at the focal spot of the ARP actuator and the modulation frequency generated by the external signal generator is swept to find the vibration mode frequencies. The magnitude and phase of the vibration profile of the flexural modes are obtained by measuring the photodetector output using the lock-in amplifier while scanning the cantilever along its length. Note that the optical detection spot is not moved from the tip of the cantilever during these measurements. As an example, the measured vibration profile of the third flexural mode of a cantilever at 72.2 kHz is shown in Fig. 3.[16]

In order to verify the quantitative nature of the results, a simple analytical model is constructed assuming that the ideal, lossless modal shapes of the cantilever are unchanged in the liquid and the incoming acoustic beam creates a Gaussian force profile on the cantilever. The prediction of this simple model fitted to the measured data is also shown in Fig. 3. In these plots the free end of the cantilever is to the left of the graph. The good agreement between the experiment and the model shows that the ARP actuator can be used to obtain quantitative information for AFM cantilever characterization in liquids.

Although the torsional modes of AFM cantilevers are shown to provide elasticity and friction information, they have not been fully utilized because of the lack of an efficient actuation scheme.[18] Using the ARP actuator, the torsional modes can also be excited and characterized in fluids. Figure 4(a) shows the magnitude and Fig. 4(b) shows the phase of oscillation of the second torsional mode of a 100 μm long, V-shaped silicon nitride AFM cantilever at 250 kHz.[19] These images are formed by scanning the cantilever at the focal plane of the ARP actuator while recording the

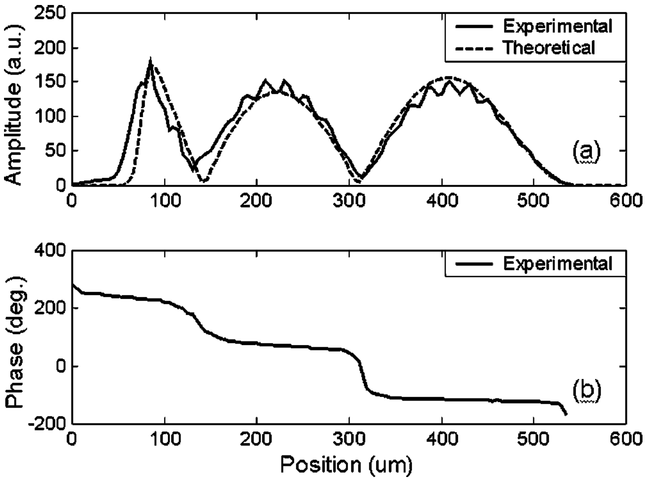

FIG. 3. Amplitude (a) and phase (b) profiles of the third flexural mode of a rectangular AFM cantilever at 58 kHz. Dashed line in (a) is the prediction of a simple theoretical model.

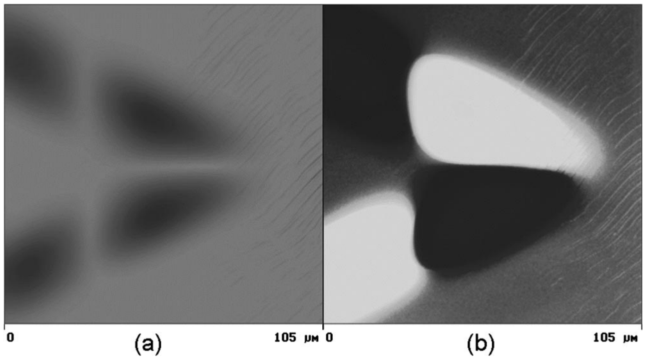

FIG. 4. Amplitude (a) and phase (b) ''images'' of the second torsional mode of a V-shaped silicon nitride cantilever at 250 kHz. Dark and bright regions in (b) represent a 180° phase difference. Brighter regions in part (a) represent the areas with larger oscillation amplitude.

magnitude and phase of the lateral tilt signal by the lock-in amplifier. Darker colors on the amplitude image correspond to larger magnitude of oscillation. The difference between the dark and light areas in the phase image is 180° so that two diagonal regions on the cantilever are moving in one direction while the other two are moving in the opposite direction.

The mechanical boundary conditions imposed on the cantilever when it is in contact with a sample change the resonant frequency and the resonant mode shape of a cantilever.[14] Since the quality factors of resonant modes are high in air, the shift in resonant frequency has been used effectively as a tool for elasticity imaging.[20] However, when the samples are immersed in liquids the quality factors are much lower and the resonant frequency shift method may not be adequate. In this case, one can take advantage of the ARP actuator by producing a point-like force on the cantilever and monitoring changes in the resonant mode shape, both in amplitude and phase, as shown in Fig. 3. This mode of imaging, which can be called ''mode shape imaging,'' provides images sensitive to local elastic properties of the sample. For elasticity imaging, the excitation frequency is ideally set to the proper flexural resonant mode, while the cantilever is in contact with the sample and the ARP actuator is positioned to a sensitive location on the cantilever such as a nodal point. Then the standard constant force mode imaging is carried out while the amplitude or phase of the deflection signal at the excitation frequency is filtered using a lock-in amplifier and is recorded as a separate image. This capability is demonstrated on a sample consisting of 1.3 μm thick photoresist patterned on silicon substrate. Figure 5 shows simultaneously recorded regular contact mode and the elasticity images obtained at 50 kHz. In this particular case, the amplitude information is used to form the elasticity image and in both images darker regions correspond to the silicon surface. It should also be noted that the 6 dB bandwidth of the ARP actuator is measured to be at least 5 MHz, so that it can be used to excite higher-order modes of even smaller cantilevers for low noise measurements and also to obtain elasticity images of hard samples.

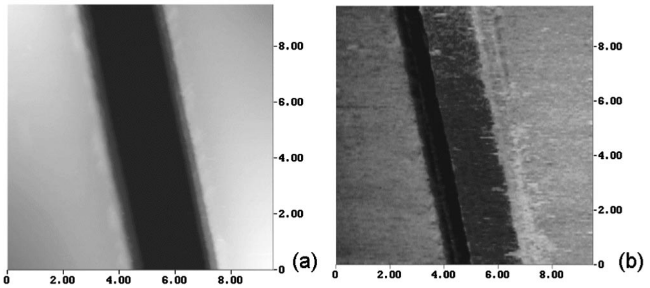

FIG. 5. Contact mode image (a) of a feature defined on 1.3 μm thick photoresist on silicon substrate. Image (b) shows the elasticity image obtained by using the amplitude of oscillation of the second flexural resonance mode at 50 kHz. Image size is 10 μm square.

In summary, it is shown that the ARP actuator can expand the capabilities of the AFM for applications in liquids. By applying point-like forces to the desired locations on a cantilever in a wide frequency range, it enables accurate characterization and excitation of flexural and torsional modes of AFM cantilevers. In addition to improving the tapping mode imaging performance, the ARP enables elasticity imaging based on monitoring the change in the cantilever mode shape due to contact stiffness. The wide bandwidth of actuation with the ARP offers the capability of high speed imaging in liquids. Future work will focus on integration of the ARP actuator with the cantilever holder to overcome sample limitations, as well as implementation of an array of actuators to vibrate an array of cantilevers.

[1] C. A. J. Putman, K. O. can der Werf, B. G. de Grooth, N. F. can Hulst, and J. Grece, Biophys. J. **67**, 1749 (1994).
[2] M. Bezanilla, B. Brake, E. Nudler, M. Kashlev, P. K. Hansma, and H. G. Hansma, Biophys. J. **67**, 2454 (1994).
[3] G. T. Paloczi, B. L. Smith, P. K. Hansma, D. A. Walters, and M. A. Wendman, Appl. Phys. Lett. **73**, 1658 (1998).
[4] I. Revenko and R. Proksch, J. Appl. Phys. **87**, 526 (2000).
[5] J. B. Thompson, G. T. Paloczi, J. H. Kindt, M. Michenfelder, B. L. Smith, G. Stucky, D. E. Morse, and P. K. Hansma, Biophys. J. **79**, 3307 (2000).
[6] D. A. Walters, J. P. Cleveland, N. H. Thomson, P. K. Hansma, M. A. Wendman, G. Gurley, and V. Elings, Rev. Sci. Instrum. **67**, 3583 (1996).
[7] M. V. Salapaka, H. S. Bergh, J. Lai, A. Majumdar, and E. McFarland, J. Appl. Phys. **81**, 2480 (1997).
[8] T. E. Schaffer, J. P. Cleveland, F. Ohnesorge, D. A. Walters, and P. K. Hansma, J. Appl. Phys. **80**, 3622 (1996).
[9] W. Han, S. M. Lindsay, and T. Jing, Appl. Phys. Lett. **69**, 4111 (1996).
[10] W. Han and S. M. Lindsay, Appl. Phys. Lett. **72**, 1656 (1998).
[11] T. Sulchek, R. Hshieh, J. D. Adams, S. C. Minne, and C. F. Quate, Rev. Sci. Instrum. **71**, 2097 (2000).
[12] I. Revenko and R. Proksch, J. Appl. Phys. **87**, 526 (2000).
[13] F. L. Degertekin, B. Hadimioglu, T. Sulchek, and C. F. Quate, Appl. Phys. Lett. **78**, 1628 (2001).
[14] K. Yamanaka and S. Nakano, Jpn. J. Appl. Phys., Part 1 **6**, 3787 (1996).
[15] Dimension III AFM system, Digital Instruments, Santa Barbara, CA.
[16] Etched Silicon Probes, Digital Instruments, Santa Barbara, CA. The resonant frequency in air and spring constant of the cantilever are given by the vendor as 6–20 kHz and 0.02–0.1 N/m, respectively.
[17] S. A. Elrod, B. Hadimioglu, B. T. Khuri-Yakub, E. G. Rawson, E. Richley, C. F. Quate, N. N. Mansour, and T. S. Lundgren, J. Appl. Phys. **65**, 3441 (1989).
[18] K. Yamanaka and S. Nakano, Appl. Phys. A: Mater. Sci. Process. **66**, S313 (1998).
[19] Silicon Nitride Probes, Digital Instruments, Santa Barbara, CA. The cantilever is 100 μm long and its spring constant is given as 0.58 N/m.
[20] K. B. Crozier, G. G. Yaralioglu, F. L. Degertekin, J. D. Adams, and S. C. Minne, Appl. Phys. Lett. **76**, 1950 (2000).