Actuation and characterization of atomic force microscope cantilevers in fluids by acoustic radiation pressure

F. L. Degertekin[a)]
G. W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332

B. Hadimioglu
Xerox Palo Alto Research Center, 3333 Coyote Hill Road, Palo Alto, California 94304

T. Sulchek and C. F. Quate
E. L. Ginzton Laboratory, Stanford University, Stanford, California 94305

(Received 24 October 2000; accepted for publication 10 January 2001)

An actuation method for atomic force microscope (AFM) cantilevers in fluids is reported. The radiation pressure generated by a focused acoustic transducer at radio frequency (rf) (100–300 MHz) exerts a localized force of controlled amplitude at a desired location on the AFM cantilever. This force can be used to measure the spring constant and other dynamic properties of the cantilever. Furthermore, by amplitude modulating the rf signal input to the acoustic transducer, the cantilever is actuated in the dc–MHz frequency range. This provides a broadband actuation and characterization method for AFM cantilevers with arbitrary geometry. The technique is demonstrated on AFM cantilevers with spring constants in the 0.01–10 N/m range using a micromachined acoustic transducer/Fresnel lens structure operating at 179 MHz in water. [DOI: 10.1063/1.1354157]

The versatility of the atomic force microscope (AFM) has turned this instrument into an essential measuring and imaging tool for a wide range of environments.[1–3] Many applications of the AFM take place in fluids and use both the quasistatic (force spectroscopy) and first and higher order dynamic modes (tapping mode, ultrasonic AFM) of the cantilever.[4–6] Characterization of AFM cantilevers is essential to optimize the experimental conditions and to obtain quantitative results. For example, the spring constant of the cantilever determines the force applied to the sample in the contact mode, and in noncontact and tapping mode operation it limits the imaging speed. Yet in some other applications, such as force spectroscopy, the accuracy of the measurement is directly determined by the accuracy of the spring constant. Therefore, most AFM cantilever characterization efforts have been in this area and several methods have been developed for cantilever spring constant measurement. Static methods use deflection of the AFM cantilever by another well characterized cantilever or by the effect of gravity when a known mass is placed at the tip of the cantilever,[7,8] whereas dynamic methods rely on the thermal noise spectrum, resonance frequency shift with added mass, or the hydrodynamic function of rectangular cantilevers.[9–11] These characterization methods are useful only for operation up to the fundamental resonance frequency, and the analysis of complex hydrodynamic forces in biological fluids like water is not trivial for arbitrarily shaped cantilevers vibrating at high frequencies.[12] These limitations can be overcome if one can apply static and time harmonic point-like forces at the location of the AFM tip on the cantilever in a noncontact manner and measure its deflection while it is immersed in a fluid. Although a localized electrostatic force applied to the AFM tip has been recently used for this purpose, its use is limited to conducting cantilevers immersed in gases or in dielectric liquid environments.[12]

In this letter, a method with which to actuate AFM cantilevers with localized forces generated by focused acoustic waves in fluids is described. It uses the second order force generated by sound beams, also known as the acoustic radiation force.[13] The method can be used to actuate AFM cantilevers with arbitrary shapes and materials, eliminating the requirement for magnetic and piezoelectric thin-film coatings and, since it is an ultrasonic method, it can be used in any fluid environment.[14,15] The technique is demonstrated on a commercial AFM system using a variety of AFM cantilevers immersed in water.

A plane target placed in the path of an acoustic wave beam in an unconfined medium experiences a time averaged force per unit area, which is known as the Langevin acoustic radiation pressure.[16] This pressure, given by the average energy density, U, at the target surface, can be localized at a desired location on the AFM cantilever by placing the cantilever at the focal plane of an acoustic lens as shown in Fig. 1. As a simple model, it can be assumed that at the focal plane a time harmonic acoustic pressure wave of amplitude, p_i, is incident on the cantilever immersed in a fluid and the wave is reflected with a complex pressure reflection coefficient, Γ, at the angular frequency, $\omega = 2\pi f$. This reflection coefficient can be considered as the weighted average over the incident spectrum of plane waves, which would be included in a focused beam. Then the time averaged energy density at the cantilever surface will be given by

$$U = \frac{p_i^2}{2\rho c^2}(I + |\Gamma|^2), \quad (1)$$

where ρ is the bulk density of the fluid, c is the speed of

[a)]Electronic mail: ldegerte@sununo.me.gatech.edu

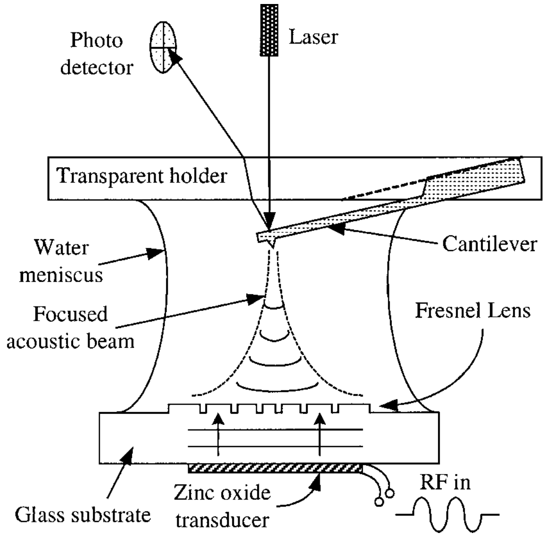

FIG. 1. Schematic of the experimental setup used to demonstrate acoustic radiation pressure actuation of AFM cantilevers.

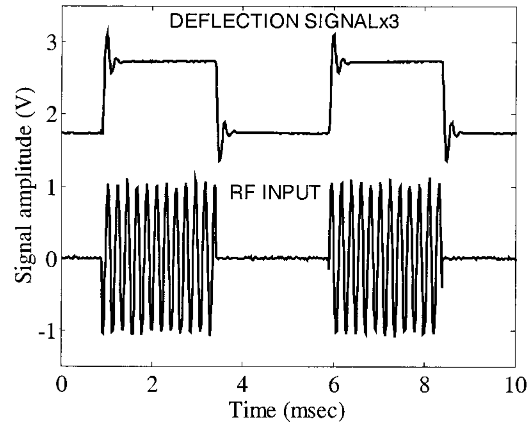

FIG. 2. The rf signal applied to the piezoelectric transducer and the resulting cantilever deflection signal. The rf signal is pulse modulated at 200 Hz and its frequency is 179.22 MHz. A rectangular silicon cantilever with a 0.148 N/m spring constant is used. The deflection signal is offset by about 1.7 V for clarity.

sound in the fluid, and $|\cdot|$ denotes the absolute value. Using the relation that the average intensity of the incident beam is given by $I_i = p_i^2/(2\rho c)$, the Langevin radiation pressure on the cantilever, Ω, can be expressed in terms of the intensity as

$$\Omega = I_i \frac{(1+|\Gamma|^2)}{c}. \qquad (2)$$

The total force applied to the cantilever can be found by integrating the radiation pressure, and hence it is proportional to the average power incident on the cantilever.

The localization of the radiation force can be estimated using the relations for diffraction limited focused acoustic beams.[13] For an acoustic lens with an F number equal to 1, the 3 dB diameter, d, of the diffraction limited beam at the focal plane is given by the relation $d = 1.02\lambda$, where λ is the wavelength of the time harmonic acoustic wave in the fluid. For example, in water ($c = 1.5\times 10^3$ m/s), the diameter of the beam at the focal plane will be between 10 and 5 μm when the rf frequency, f, is in the 150–300 MHz range. According to Eq. (2), for 150 μW incident average acoustic power at the focal plane of the lens, $\mathbf{P}_i = \pi d^2 I_i/4$, the force applied to the AFM cantilever will be 200 nN, if perfect reflection is assumed at the water/cantilever interface ($|\Gamma| = 1$). Both these frequencies and power levels are typically used for acoustic microscopy and acoustic ink printing applications.[17]

A schematic of the experimental setup used in this study is shown in Fig. 1. The AFM cantilever is mounted on a transparent holder, which fits into a commercial AFM scan head.[18] The cantilever is immersed into a drop of water to wet the entire holder–sample cavity. A glass substrate containing a surface micromachined acoustic Fresnel lens is placed below the cantilever as the sample. The Fresnel lens is designed such that when the zinc oxide transducer is excited with a sinusoidal signal at 179 MHz, the acoustic waves are focused to a diameter of approximately 10 μm at a focal distance of 360 μm. This lens structure is part of a two-dimensional array of micromachined acoustic lenses on the same glass plate, which was originally developed for acoustic ink printing purposes.[17] The deflection of the cantilever is measured using the standard optical detector of the AFM system.

Since the radiation force is proportional to the intensity, the acoustic waves can only ‘‘push’’ the AFM cantilever in the propagation direction. This is clearly seen in Fig. 2, where both the rf signal input to the piezoelectric transducer and the resulting cantilever deflection signal are shown. To obtain these data, the cantilever is first placed approximately in the focal plane of the acoustic transducer using the Z actuator of the scan head. Then it is moved in the X–Y plane to bring the cantilever tip to the focal spot of the acoustic beam. Finally, the Z level is adjusted for maximum deflection. The cantilever is made of silicon and has a spring constant of 0.148 N/m. The cantilever is moved upward from its rest position after a transient when the rf signal is turned on, showing its step response. This is consistent with the fact that the cantilever has a fundamental resonance of around 4.6 kHz in water. The low frequency appearance in the rf signal is due to the low sampling rate of the digital oscilloscope (100 kS/s).

Figure 3 shows the measured cantilever deflection as a

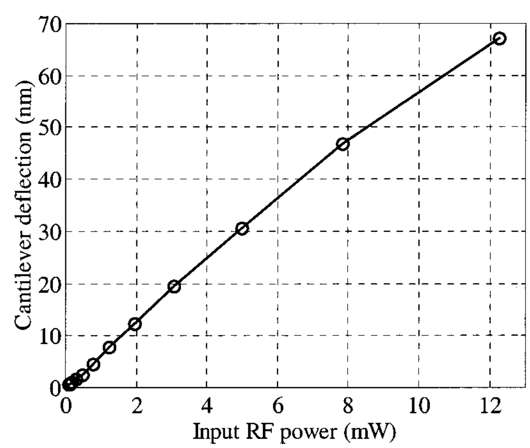

FIG. 3. Measured cantilever deflection as a function of the power level of the rf signal input to the piezoelectric transducer at 179 MHz. Data points are indicated by circles. The 205 μm long cantilever has a spring constant of 1.14 N/m.

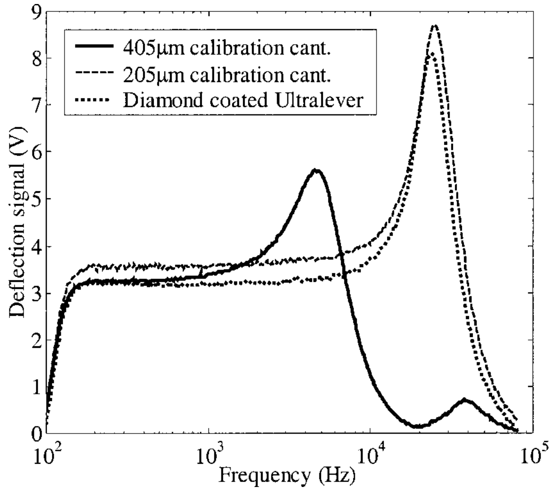

FIG. 4. Measured cantilever deflection signal at the lock-in amplifier output as a function of frequency for three different cantilevers. The signal levels are normalized by the autogain feature of the lock-in amplifier.

function of the rf power input to the transducer. The cantilever is 205 μm long, 2 μm thick, and 29 μm wide and its spring constant is specified as 1.14 N/m.[19] As expected from Eq. (2), the radiation force on the cantilever varies linearly with input power to the transducer over a wide range. Given the spring constant of the reference cantilever, the force applied to the cantilever can be calibrated. For example, for 4.98 mW electrical power input to the transducer we measure 31 nm cantilever deflection as shown in Fig. 3. Assuming that the calibrated spring constant of 1.14 N/m is accurate, we need to have a force of 35 nN applied to the tip of the cantilever by acoustic radiation pressure. An independent measurement of the insertion loss of the acoustic transducer and lens combination was performed to determine the incident acoustic power at the cantilever surface. This measurement yielded a loss of 26.5 dB. Therefore, for the 4.98 mW electrical power on the transducer, we find that the incident acoustic power on the tip is 11.1 μW. Using Eq. (2) the force on the cantilever due to acoustic radiation pressure is predicted as 13 nN, assuming an average reflection coefficient of 0.85 for the 2 μm thick cantilever immersed in water. This number is of the same order of magnitude as the value predicted from the cantilever deflection. Further measurements and theoretical analysis will be performed to explain the discrepancy in the numbers. Once the radiation force is calibrated, it is used to measure the spring constants of 405 and 105 μm long cantilevers on the same chip. The measured values are 0.132 and 10.18 N/m, which are 12% smaller and 19% larger, respectively, compared to the values quoted by the vendor.

The dynamic response of AFM cantilevers is also measured by the radiation pressure method. Time harmonic forces are generated by applying a sinusoidal amplitude modulation on the rf input signal. By choosing a modulation factor of less than 1, appropriate biasing force is applied to actuate the cantilever at the modulation frequency and its second harmonic. The deflection of the cantilever is then measured by a lock-in amplifier, which locks to the modulation frequency. Figure 4 shows the normalized magnitude of the lock-in amplifier output as a function of the modulation frequency for two of the calibration cantilevers and a V shaped, diamond coated force modulation cantilever.[20] The frequency sweep is limited to the 0.2–100 kHz range limitations of the lock-in amplifier.[21] The fundamental and second modes of the long cantilever are clearly seen around 4.6 and 38 kHz. Figure 4 also shows the ability of the radiation pressure method in actuating different cantilevers without any undesired effects of the cantilever holder and the fluid cell. It has to be noted that torsional modes of the cantilever can also be characterized by applying the radiation pressure at off-axis locations on the cantilever.

The frequency response of the actuation method is determined by the bandwidth of the acoustic transducer/Fresnel lens system around the center rf frequency. In this particular case, the 3 dB bandwidth is approximately 1 MHz, which is well above the resonance frequencies of common AFM cantilevers. It may be possible to use the method both in air and liquids, and microfabrication techniques can be used to implement it in the form of arrays for high speed imaging.

In summary, the radiation pressure generated by a focused acoustic beam is introduced as a method to actuate and characterize AFM cantilevers in fluids. The method is demonstrated on a variety of AFM cantilevers by measuring their static and dynamic characteristics in water.

The authors are grateful to Professor B. T. Khuri-Yakub of the E. L. Ginzton Laboratory, Stanford University, for his support during the experiments and to members of the Document Hardware Laboratory of Xerox-PARC for fabricating the acoustic lens.

[1] R. Lal and S. A. John, Am. J. Physiol. **266**, C1 (1994).
[2] L. Bottomley, Anal. Chem. **70**, 425R (1998).
[3] G. T. Paloczi, B. L. Smith, P. K. Hansma, D. Walters, and M. Wendman, Appl. Phys. Lett. **73**, 1658 (1998).
[4] M. B. Viani, T. E. Schaffer, A. Chand, M. Rief, H. E. Gaub, and P. K. Hansma, J. Appl. Phys. **86**, 2258 (1999).
[5] C. A. J. Putman, K. O. Van der Werf, B. G. De Grooth, N. F. Van Hulst, and J. Greeve, Appl. Phys. Lett. **64**, 2454 (1994).
[6] K. B. Crozier, G. G. Yaralioglu, F. L. Degertekin, J. D. Adams, S. C. Minne, and C. F. Quate, Appl. Phys. Lett. **76**, 1950 (2000).
[7] T. J. Senden and W. A. Ducker, Langmuir **10**, 1003 (1994).
[8] M. Tortonese and M. Kirk, Proc. SPIE **3009**, 53 (1997).
[9] J. L. Hutter and J. Bechhoefer, Rev. Sci. Instrum. **64**, 1868 (1993).
[10] J. P. Cleveland, S. Manne, D. Bocek, and P. K. Hansma, Rev. Sci. Instrum. **64**, 403 (1993).
[11] J. E. Sader, J. W. M. Chon, and P. Mulvaney, Rev. Sci. Instrum. **70**, 3967 (1999).
[12] M. P. Sherer, G. Frank, and A. W. Gummer, J. Appl. Phys. **88**, 2912 (2000).
[13] 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).
[14] T. Sulchek, R. Hsieh, J. D. Adams, S. C. Minne, C. F. Quate, and D. M. Adderton, Rev. Sci. Instrum. **71**, 2097 (2000).
[15] W. Han, S. M. Lindsay, and T. Jing, Appl. Phys. Lett. **69**, 4111 (1996); MAClever by Molecular Imaging Corp., made by depositing magnetic films on silicon nitride cantilevers.
[16] B.-T. Chu and R. E. Apfel, J. Acoust. Soc. Am. **72**, 1673 (1982).
[17] B. Hadimioglu, E. G. Rawson, R. Lujan, M. Lim, J. C. Zesch, B. T. Khuri-Yakub, and C. F. Quate, *IEEE Ultrasonics Symposium Proceedings* (1993), p. 579.
[18] Dimension III AFM system, Digital Instruments, Santa Barbara, CA.
[19] Force Calibration Cantilevers by ThermoMicroscopes Corp., Sunnyvale, CA.
[20] Diamond coated Ultralever cantilever by ThermoMicroscopes Corp., Sunnyvale, CA 94089.
[21] Model SRS830, Stanford Research Systems, Sunnyvale, CA 94089.