Sensor for direct measurement of interaction forces in probe microscopy

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We introduce a sensor for direct measurement of tip-sample interaction forces in probe microscopy. The sensor uses a micromachined membrane structure built on a transparent substrate with an integrated diffraction grating for optical interferometric detection, and a built-in electrostatic actuator. To demonstrate our concept for this sensor, we measured the force curves between an atomic force microscope (AFM) cantilever tip and a micromachined aluminum sensor membrane built on a quartz substrate. We also measured transient interaction forces exerted on the sensor membrane during each cycle of the vibrating AFM cantilever. These agree well with the temporal response of the sensor to a short force pulse applied by our integrated electrostatic actuator. With the addition of an integrated tip, this structure may be used for scanning probe microscopy with a bandwidth limited by the membrane dynamics. © *2005 American Institute of Physics*.
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Measurement and analysis of tip-sample interaction forces in dynamic applications of the atomic force microscope (AFM) has become an active area of research due to its importance in determining the viscoelastic properties of surfaces, as well as understanding other time dependent phenomena, such as adsorption and single-biomolecular interactions.[1,2] The mechanical force sensor used to monitor these processes is the AFM cantilever. Although the cantilever provides good control of applied force to the sample for static applications and imaging at low speeds, it has been a limiting factor for dynamic AFM applications. In tapping mode AFM, the resonant frequency of the cantilever limits the imaging speed and makes it difficult to recover the tip-sample interaction force. Similarly, pulsed force imaging and ultrasonic AFM applications suffer from the dynamic cantilever characteristics.[3,4] Although using smaller cantilevers with high resonance frequencies are shown to be effective, their use comes with increased system complexity.[5,6]

In this letter, we report direct time resolved measurement of the interaction forces in probe microscopy with a different sensor structure. As shown in the schematic of the experimental setup in Fig. 1, the main component of the sensor is an aluminum membrane microfabricated on a transparent quartz wafer. Under the gap beneath the membrane, there is an optical diffraction grating which also serves as a rigid bottom electrode for electrostatic actuation. To measure the membrane displacement, the reflective grating and the membrane are illuminated from the back side with a laser beam and the intensity of reflected diffraction orders is monitored to record the output signature. This provides a robust microscale optical interferometer structure.[7] The position of the membrane relative to the grating is controlled by applying a voltage between the grating electrodes and the conducting membrane. This enables us to bias the membrane for optimal displacement detection sensitivity. Moreover, with a sharp tip integrated on the micromachined membrane, the same actuator should be useful for applying static and dynamic forces to move the probe tip for various force spectroscopy and imaging applications.

The force sensing structure was originally developed as an acoustic transducer with optical receive and electrostatic transmission capabilities.[8] We demonstrated generation and optical detection of broadband ultrasonic vibrations in the MHz range, one-dimensional array operation, interferometric displacement detection with near-shot noise limited resolution, and more recently, the use of an asymmetric etalon structure with an embedded grating for improved sensitivity.[9–11] For operation in liquids, devices with sealed cavities and electrodes buried in dielectric membranes can be fabricated on transparent substrates.[12]

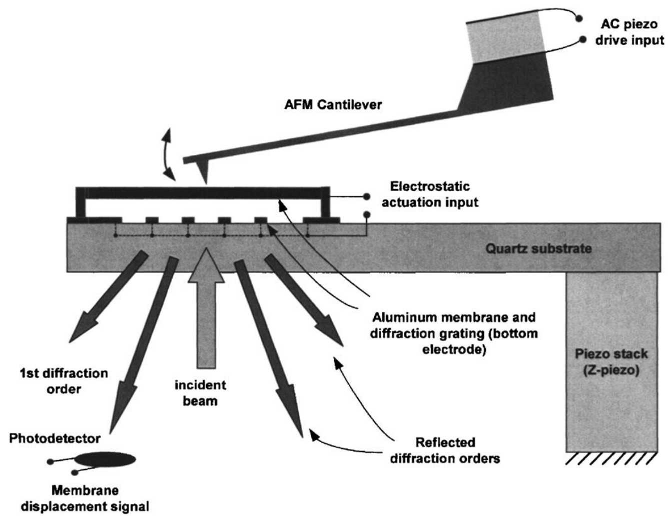

FIG. 1. The schematic of the experimental setup used to directly measure the dynamic interaction forces between an AFM tip and the micromachined sensor membrane.

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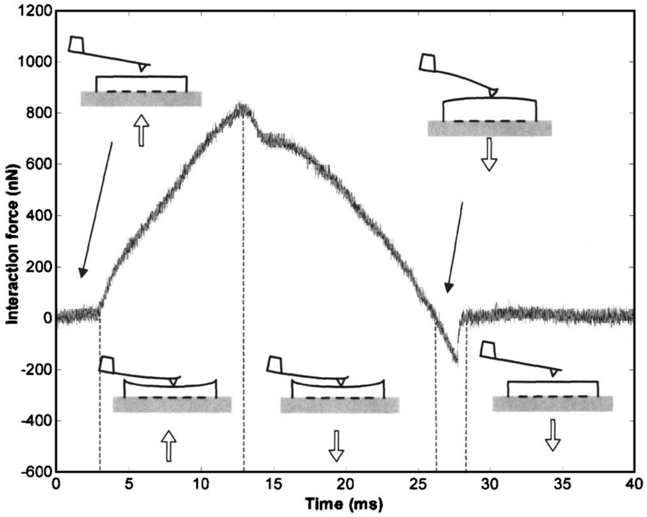

FIG. 2. Measured single shot force curve obtained by driving the AFM tip in and out of contact with the sensor membrane using the piezostack attached to the sensor substrate as shown in Fig. 1. The inset drawings show the cantilever and sensor membrane shape at different phases of the force curve.

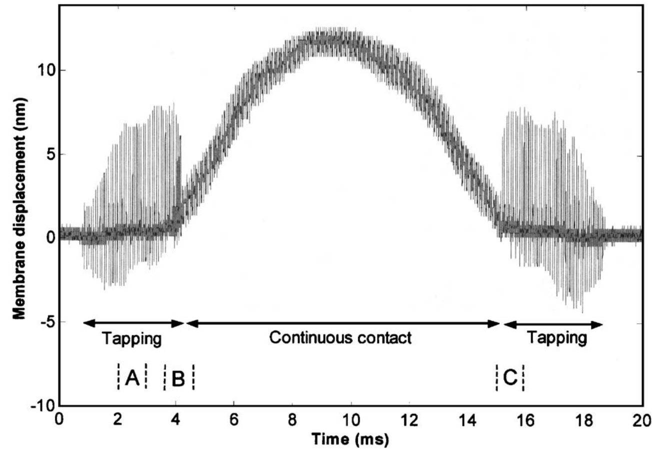

FIG. 3. Single shot membrane displacement curve obtained by driving the AFM tip in and out of contact with the sensor membrane while vibrating the cantilever with a sinusoidal signal at 67.3 kHz. The signal in time intervals A, B, and C are expanded in Fig. 4 to show individual tapping signals and the continuous to intermittent contact transitions.

As shown in Fig. 1, a quartz substrate with the sensor membrane is placed on a piezostack transducer, which was used to approach to the tip and obtain force distance curves. The particular aluminum membranes were circular with a 150 μm diameter, 1 μm thick, and are located over a 2 μm gap above the rigid diffraction grating electrode. The grating period is 4 μm for this particular case. The gap is open to air through several sacrificial layer etch holes. The grating was illuminated through the quartz substrate using a HeNe laser ($\lambda = 632$ nm) at a 5° angle. The output optical signal was obtained by recording the intensity of the first diffraction order beam. For measuring the AFM dynamic tip-sample interaction forces, the cantilevers were glued on a piezoelectric ac drive transducer which was used to drive the cantilever at its resonant frequency. The membrane stiffness of approximately 76 N/m was measured at the center using a calibrated AFM cantilever and the membrane resonant frequency was 620 kHz. The dc bias on the membrane was adjusted to 27 V to optimize the optical detection, and the sensitivity was calibrated as 16 mV/nm by contacting the membrane with a calibrated AFM cantilever and a calibrated piezodriver. The broadband root-mean-square noise level of the system was about 3 mV (0.18 nm) without much effort to reduce mechanical, laser, or electrical noise.

We obtained a force curve by moving the piezostack supporting the sensor substrate with a 20 Hz 850 nm triangular signal and making sure that there was tip-membrane contact during a portion of the signal period. The cantilever is FESP from Veeco Metrology ($k = 2.8$ N/m). This curve is shown in Fig. 2 where the inset drawings indicate the shape of the cantilever tip and membrane, and the arrow indicates the direction of motion of the piezostack and the quartz substrate. Tip-membrane contact happens at around 3 ms and the tip bends the membrane downward until about 26 ms while the piezomotion is reversed in the middle. Attractive forces due to adhesion pulls the membrane up for 2 ms and then membrane moves back to its rest position after a 180 nN jump at the end of the retract section.

For direct observation of time resolved dynamic interaction forces along the force curve, a similar experiment was performed while the cantilever was driven into oscillation by applying a sinusoidal signal to the ac drive piezo at 67.3 kHz. The single shot transient membrane displacement signal obtained during a cycle of the 20 Hz drive signal is shown in Fig. 3. As expected from dynamic interaction force measurements, this curve carries a wealth of information. We indicate the different interaction regimes during this measurement, and also expand the data in the initial tapping (A), intermittent to continuous contact (B), and continuous to intermittent contact transition regions (C) in corresponding parts of Fig. 4. Starting from the left, the cantilever tip is first out of contact with the sensor membrane. At around 1 ms, it starts intermittent contact (tapping) with the membrane as individual taps are easily detected as shown as the top trace in Fig. 4(a). As the cantilever gets closer to the sensor mem-

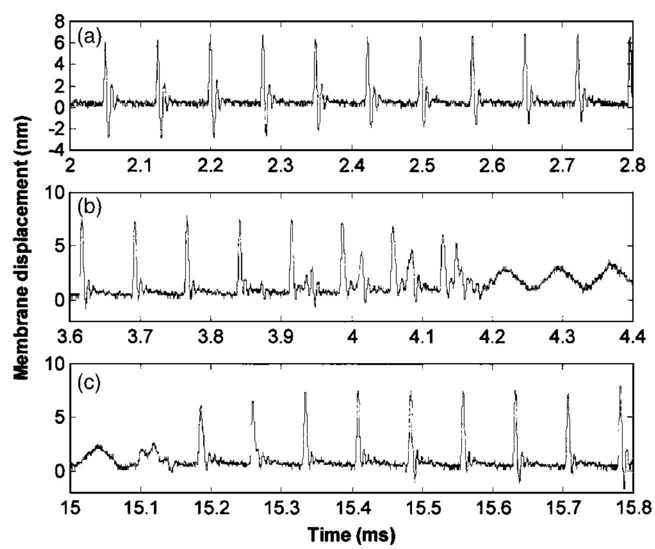

FIG. 4. Sections of the curve shown in Fig. 3 indicating different interaction force regimes during approach and retract of vibrating AFM cantilever. (a) Individual tap (intermittent contact) signals on the sensor membrane during approach. Positive displacement indicates that the membrane moves down. (b) The transition region from intermittent to continuous contact between the cantilever tip and sensor membrane. After 4.1 ms, the detected displacement is a distorted sinusoid when the tip is in contact during a full cycle. (c) Measured displacement signal in the transition Region C of Fig. 3, where the tip breaks off the membrane surface and starts tapping again.

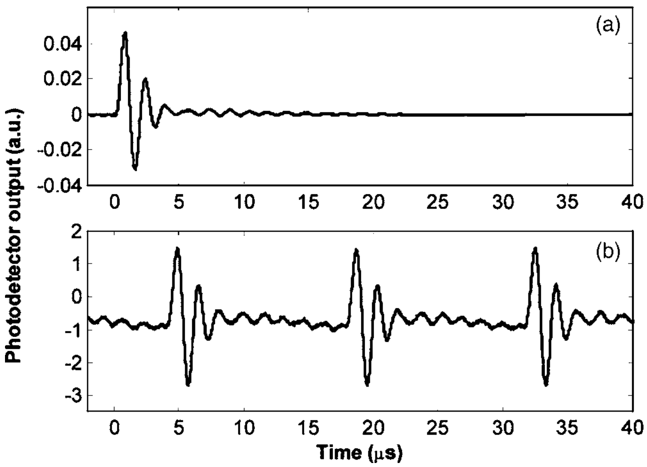

FIG. 5. (a) Measured membrane displacement when a short electrical pulse is applied to the sensor membrane using the integrated electrostatic actuator terminals shown in Fig. 1. (b) The individual tap signals (time averaged) detected by the sensor while an AFM cantilever vibrated at 72.5 kHz is tapping on the membrane.

brane, the pulses get unipolar and the distortion is more severe as there are double-peaked tap signals when the cantilever gets into contact due to nonlinear interaction forces as shown in the next trace [Fig. 4(b)]. When the tip is in continuous contact, which happens around 4.2 ms, the displacement signal has the periodicity of the drive signal in addition to distortion probably caused by contact nonlinearities and higher-order vibration modes of the cantilever with its tip hinged on the membrane. Similarly, around 15 ms, the cantilever starts breaking off the membrane surface and tapping resumes [Fig. 4(c)]. Between 7 ms and 12 ms, the curve is not linear. This may be because the sensor membrane-grating distance was not optimal in the beginning of the experiment, and the sensor enters the nonlinear region of the optical interferometer sensitivity curve.

Note that the individual tapping signals are still filtered by the response of the sensor membrane, but the information is more readily available as compared to several other techniques.[13–15] The device was not optimized for this experiment, and the sensor membrane acted as a lightly damped resonator rather than having a broadband frequency response that is ideal for fast interaction force measurements. Nevertheless, one can easily obtain the transfer function of the membrane using the integrated electrostatic actuator. Figure 5(a) shows the measured temporal response of the sensor membrane when a 2 V square pulse 100 ns in length was applied in addition to the 27 V dc bias at the actuator terminals. Comparing this wave form with averaged data from individual tap signals in Fig. 5(b), one can conclude that the stiff cantilever tap is nearly an impulsive force, which can be recovered by inverse filtering.

In summary, we present results from a proof of principle experiment for a microscale-integrated sensor-actuator structure for scanning probe microscopy. This system combines optical interferometric displacement detection and simultaneous force application through electrostatic actuation. This structure can be readily used as a sensor for material property measurement by applying thin films on the membrane and recording tip-membrane interaction forces. We are currently working on fabricating sharp tips on micromachined membranes and integrating them onto cantilevers for use in the AFM system illustrated in Fig. 1, to measure interaction forces during tapping mode imaging of samples on rigid substrates. This sensor structure can be tailored to meet the bandwidth and sensitivity requirements of a particular application, and the performance will ultimately depend on the displacement detection sensitivity.

We have demonstrated minimum displacement detection levels down to 10^{-4} Å/$\sqrt{\text{Hz}}$ with near-shot noise limited operation, and we envision mechanical structures with spring constants in the 1–10 N/m range that will allow us to monitor force levels in the pico-Newton range for this device.[9,10] These sensitivity levels will make it useful for a wide range of probe microscopy applications, including quantitative interaction force measurements, fast imaging in liquids and in air, and probe arrays for imaging, lithography, and single molecule force spectroscopy.

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