Spring constant tuning of active atomic force microscope probes using electrostatic spring softening effect

H. Torun, K. K. Sarangapani, and F. L. Degertekin[a)]
G. W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332, USA

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The authors describe a method to electrically adjust the spring constant of an active atomic force microscopy (AFM) probe using electrostatic spring softening effect. The probe consists of a clamped membrane with interferometric displacement sensing and integrated electrostatic actuation. Using the bias voltage on the integrated electrostatic actuator, the spring constant of the probe is reduced electrically. This increases the force sensitivity of the probe without significant dimensional change, therefore not affecting its noise level. The sensitivity improvement for force spectroscopy is demonstrated by capturing force curves using the membrane probe while it is in interaction with an AFM tip. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2827190]

In a conventional atomic force microscopy (AFM) system, a passive, microscale cantilever is used as the force sensing element to measure the interaction force between the sharp cantilever tip and the sample surface.[1] The spring constant of the cantilever depends on geometry and material properties, and it is permanently set once the cantilever is fabricated.[2] Although this is advantageous for stability purposes, it also limits the range of physical properties that can be effectively realized with a cantilever. For example, if one reduces the lateral dimensions of the cantilever to achieve low thermal noise in fluids, the thickness needs to be decreased to simultaneously keep the spring constant at its original value.

Recently, active AFM probes with integrated electrostatic actuators and interferometric readout have been introduced.[3] These devices, called force-sensing integrated readout and active tip (FIRAT), use surface micromachined membranes or clamped beams on transparent substrates as the force sensing structure. Membrane-type FIRAT devices without sharp tips have been already used for single molecule force spectroscopy.[4] With the addition of a sharp integrated tip to the membrane, fast tapping mode imaging, and material characterization through time-resolved interaction force measurements have been demonstrated.[5] The "active" nature of these probes is because of the integrated, parallel-plate-type electrostatic actuator. Due to nonlinear nature of the electrostatic forces, this type of actuator enables one to adjust or tune the stiffness of the coupled mechanical structure for small displacements around an equilibrium point determined by the bias voltage and the initial stiffness.[6] This effect has been used as a frequency tuning method for micromachined resonator devices.[7,8] Researchers have also studied this spring softening effect to model parallel-plate-type microstructures such as cantilever beams and membranes.[9]

In this paper, we use the integrated electrostatic actuator to adjust the spring constant of the active AFM probe based on the spring softening effect. This capability adds another knob to adjust the probe properties on demand which is not available in passive AFM cantilevers. We note that the variation of spring constant with bias voltage may not be desirable for some imaging applications. However, for applications requiring dynamic force measurements due to small displacements of the probe, such as molecular force spectroscopy and time resolved measurement of tap signals, this approach has certain advantages. One can adjust the sensitivity of force measurements and decouple the optimization of spring constant and the viscous damping coefficient of the probes for thermal mechanical noise limited force spectroscopy measurements.[10] Assuming a Hertzian contact model, one can shift the measurement range of sample elasticity linearly with the probe stiffness.[11]

To demonstrate the spring constant tuning capability for different applications, we used both polymer (parylene) membranes fabricated for single molecule force spectroscopy in liquids [Fig. 1(a)], as well as aluminum membranes similar to the ones with sharp imaging tips used for mechanical characterization [shown in Fig. 1(b)].[5] Parylene, a biocom-

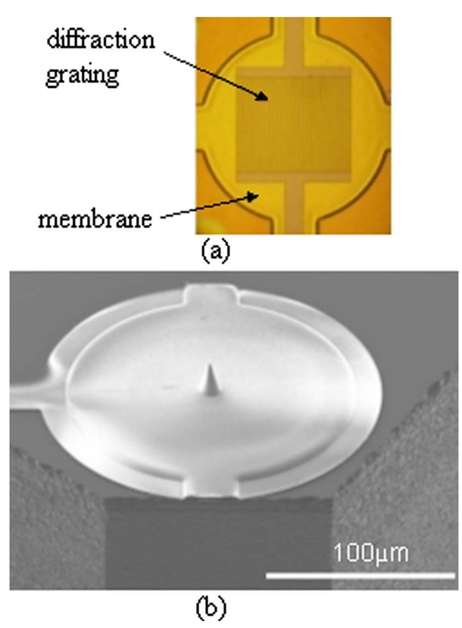

FIG. 1. (Color online) (a) Micrograph of a 200 μm diameter parylene membrane from backside through the quartz substrate. (b) SEM of a 150 μm diameter aluminum membrane with platinum tip for imaging applications.

[a)]Electronic mail: levent@gatech.edu.

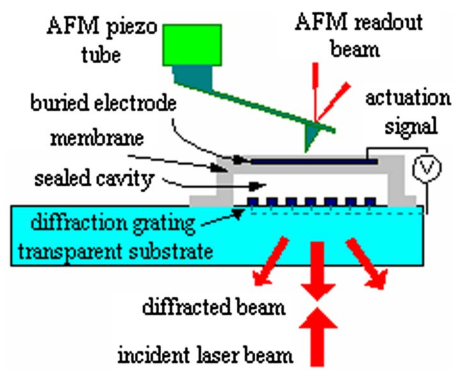

FIG. 2. (Color online) Schematic of a force spectroscopy probe membrane with self-sensing and self-actuation capabilities coupled with an AFM cantilever in a typical force spectroscopy experiment setup.

patible polymer with low modulus is suitable to build soft force spectroscopy probe membranes. The microfabrication process of the parylene membrane is similar to the one used to fabricate silicon nitride/dioxide membranes previously described.[4] It is a four-mask process carried out in room temperature. The aluminum membrane and tip fabrication process have been described elsewhere.[5]

Figure 2 shows the schematic of the probe coupled to a commercial AFM system (Veeco Metrology, Inc., Dimension AFM head). The probe is used as a force sensor while the cantilever is moved up and down using the piezoactuator. The force curves are simultaneously recorded by the optical lever detection mechanism of the AFM and the interferometric readout of the probe.

For probe characterization, a 250 μm diameter membrane with a diffraction grating period of 3.3 μm was used. The membrane was actuated by the integrated electrostatic actuator, and the intensity of light going to first diffraction order was recorded. Figure 3(a) shows this variation with respect to the voltage applied between the top electrode buried inside the parylene membrane and the bottom electrode

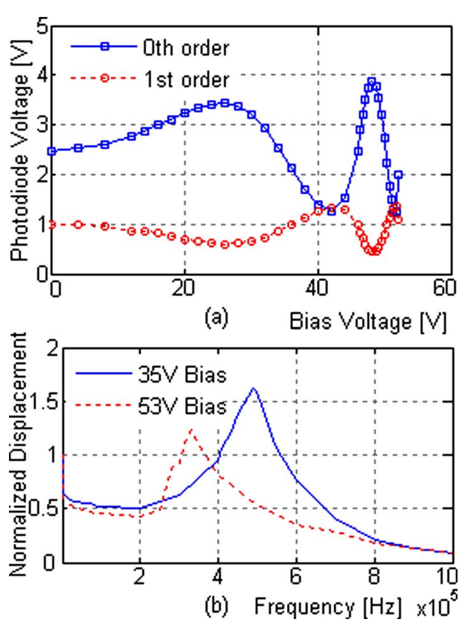

FIG. 3. (Color online) Experimental characterization of the membrane probe. (a) Variation of measured photodetector signals with respect to applied dc bias. (b) Frequency response when two different dc bias voltages are applied the electrostatic actuator terminals.

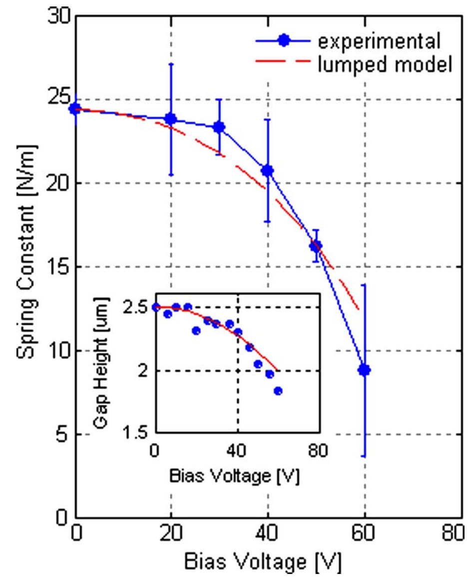

FIG. 4. (Color online) Measured and predicted variation of the spring constant of the membrane with increasing dc bias voltage. The inset shows the measured and predicted membrane displacement as a function of dc bias voltage.

patterned as a diffraction grating. The probe was excited with a small amplitude ac signal at a maximum sensitivity point for dynamic characterization. Measured dynamic response of the membrane for two different dc bias values is shown in Fig. 3(b). The results showed a shift of resonance peak and a change in quality factor which was expected due to spring softening effect.

The spring constant of the membrane vibrating in electrostatic field is reduced by increasing the electromechanical coupling coefficient (k_T) suggested by spring softening effect,[6]

$$k' = k(1 - k_T^2), \tag{1}$$

where k' and k are the softened and unbiased membrane's spring constants, respectively. The spring constant reduces as the dc bias voltage is increased and the membrane gap is decreased due to the relation between the coupling coefficient and membrane displacement,

$$k_T^2 = 2x/(d_0 - x), \tag{2}$$

where x and d_0 are the membrane displacement and initial gap height, respectively.

A spring constant of 24.4 N/m was measured at the center of the unbiased membrane by using a calibrated FESP AFM cantilever from Veeco Metrology. The measured variation of spring constant with respect to bias voltage is plotted, as shown in Fig. 4. The measurement is in agreement with the lumped model for the membrane. The model also predicts the membrane displacement within the measurement resolution of the white light interferometer used (Veeco Instruments, Wyko, Woodbury, NY), as depicted in the inset of Fig. 4. The softening of the specific membrane under consideration is approximately threefolds before it collapses when it moves one-third of the initial gap. We note that it is possible to increase stable displacement range by simply increasing the initial gap or using a multiple-electrode structure.[12]

A proof of principle experiment showing sensitivity improvement was carried out with the same probe. First, a force

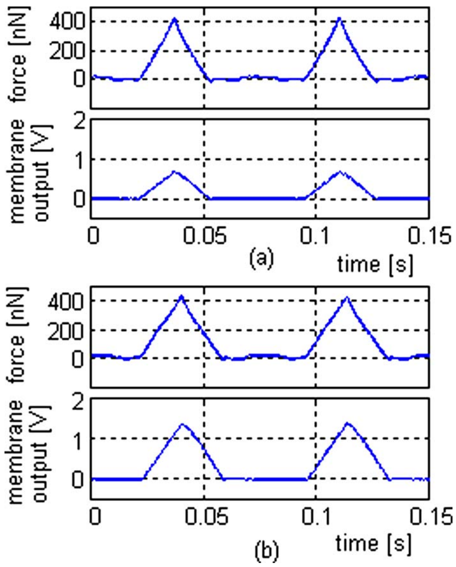

FIG. 5. (Color online) (a) Force curves obtained with the probe membrane biased at 35 V. The top curve recorded from the AFM cantilever shows a peak force of 400 nN. The bottom curve is recorded from the membrane with a spring constant of 22 N/m. (b) The peak force applied by the cantilever is kept constant at 400 nN, as shown in top trace. The bottom trace is from the same membrane with an electrically reduced spring constant of 11 N/m.

curve measurement was performed after the membrane was biased at 35 V. At this bias level, the spring constant of the membrane was 22 N/m. Then the AFM cantilever (TESP tip from Veeco Metrology, $k=40$ N/m) was driven in and out of contact of the membrane. The force curves obtained from the AFM system and the membrane probe are shown in Fig. 5(a). The maximum force applied by the cantilever was 400 nN, for which the probe output was 0.66 V. The same experiment was repeated after the bias voltage was increased to 53 V thus reducing the spring constant of the membrane to 11 N/m. The maximum force applied was kept constant while the probe generated 1.36 V, as shown in Fig. 5(b). Comparing the results, the reduction in spring constant of the membrane matches to 2.06-folds increase in the output signal of the active probe. Given that the current AFM cantilever spring constants are typically in the range of 0.1–40 N/m, further improvements are needed to achieve the full theoretical tuning range of the active probes so that one can cover a similar range with a single or several active probes.

Note that the force curve measurements in Fig. 5 were performed at discrete bias voltages, since the membrane needs to be biased at certain levels for optimum detection sensitivity. An improved probe structure with quadrature phase-shifted dual gratings that provides good detection sensitivity for small displacement for the whole range of membrane displacement has also been developed.[13] Using that structure, precise and continuous control of spring constant at any bias voltage with good detection sensitivity should be possible.

An important implication of adjustable spring constant for force spectroscopy is that, a small, relatively stiff membrane with smaller coefficient of viscous damping can be used for low force noise experiments. The large spring constant of such a membrane can be reduced electrically to obtain a larger signal for unit force, i.e., higher detection sensitivity, while still being limited by thermal mechanical noise dominated by the interaction of the probe structure with the surrounding liquid.

In summary, the use of spring softening effect for electrical tuning of the spring constant of active AFM probes is introduced. This method is used to soften the membrane-type active probes developed for force spectroscopy experiments to improve force sensitivity. In addition to force spectroscopy applications, we envision the effective use of this capability to optimize a single probe for quantitative characterization of different surfaces with different viscoelastic properties.[14]

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