Thermally actuated tapping mode atomic force microscopy with polymer microcantilevers

Bhaskar Mitra and Angelo Gaitas[a)]
PicoCal Inc., Ann Arbor, Michigan 48103, USA

(Received 5 November 2008; accepted 13 January 2009; published online 5 February 2009)

This paper demonstrates a thermally actuated tapping mode atomic force microscopy (AFM) with a polymer cantilever. The cantilever ($350\times250\times3\ \mu m^3$) is made from polyimide and includes an embedded resistive heater for thermal actuation. The oscillation of the cantilever is due to the stress gradient caused by alternating heating and cooling from the periodic ac excitation of the heater. The tip oscillation amplitude is 5–10 nm in air. The oscillation occurs at 2ω and is a linear function of the applied voltage. The maximum oscillation amplitude is seen at 0.8 Hz with a 3dB frequency of 26 Hz. The damping of the oscillation due to tip-sample interaction is used to image the sample without any optomechanical feedback. Scans with a 200 nm tall grating indicate a resolution comparable to deflection signal from the AFM in contact mode. © *2009 American Institute of Physics.* [DOI: 10.1063/1.3078010]

I. INTRODUCTION

Tapping mode atomic force microscopy (AFM) is a technique used for imaging surfaces that are easily damaged or adhere weakly to a substrate. In tapping mode, the cantilever is oscillated near the resonance frequency using piezoelectric actuators, and touches the surface periodically, producing a scan without scratching the surface. As the cantilever approaches the surface, the amplitude of its oscillations changes due to the tip-sample interaction. This measurement is used to map out topographical features. Typically the probe is operated in closed loop feedback (constant amplitude) to avoid nonlinear effects.

In conventional tapping mode the probe is oscillated via an external piezoelectric element. Alternative approaches include: probes with integrated piezoelectric actuation,[1] probes with integrated thermomechanical Al/SiO2/Si bimetallic actuation and integrated piezoresistive readout,[2] and probes with integrated thermomechanical actuation using doped silicon cantilevers.[3]

Unlike silicon or silicon nitride cantilevers, which have sharp resonant frequencies, polymer cantilevers are difficult to use in tapping mode. However, polymer cantilevers are attractive for scanning applications involving soft and biological samples because of their small contact forces. Applications of polymer cantilevers include mapping and subsurface imaging,[4] calorimetric applications,[5] maskless submicron thermochemical patterning of photoresist,[6] *IC*-hot-spot detection,[7] high-speed contact mode topography and lateral force scanning,[8] and low temperature scanning thermal microscopy.[9]

This article reports the first use of thermal tapping mode AFM scanning with polymer cantilevers that oscillate by the thermal bimorph effect of a polyimide/gold/polyimide sandwich layer. The device employs a resistive heater utilizing gold electrodes embedded into the polymer cantilever. The properties of polymer cantilevers are examined.

II. EXPERIMENT

The probe is shown in Fig. 1. The probe's tip diameter is <50 nm. The tip height is 8 μm, and the cantilever's dimensions are $350\times250\times3\ \mu m^3$. The cantilever is made out of polyimide with an embedded thin electrode of Cr/Au/Cr, which serves as an actuating element. The electrode is 90 μm wide and 150 nm thick. The tip is also made of Cr/Au/Cr. The probe offers a spring constant <0.1 N/m.

The probe is fabricated with the tip pointing toward the silicon wafer. After release the probe is flipped over, and held in place by a thermocompression bond. The probes are fabricated using six-mask complementary metal oxide semiconductor compatible surface micromachining process. Detailed descriptions of the device and fabrication have been reported earlier.[5]

The setup for experimental evaluation is shown in Fig. 2. An Agilent PicoMaps AFM along with a function generator and a lock-in amplifier (SRS830m) are used for these experiments. A function generator is used to apply sinusoidal alternating current (ac) to the probe's thin film gold resistor. ac driven intermittent heating and cooling causes the cantilever to vibrate due to the bimorph effect. The AFM laser aligned on the cantilever is reflected into the photodetector. The deflection signal is accessed from AFM's head electronics and fed into a lock-in amplifier. The probe is excited with an ac signal and vibrates at twice the ac signal's applied frequency. The probe oscillation modulates the signal of the photodetector.

The output from the first lock-in amplifier, that represents the cantilever oscillation amplitude signal, is connected to the auxiliary channel of the AFM to plot the measurements. The cantilever probe is mounted on the AFM scanner and it is positioned a few nanometers above the sample. This

[a)]Electronic mail: angelo@picocal.com.

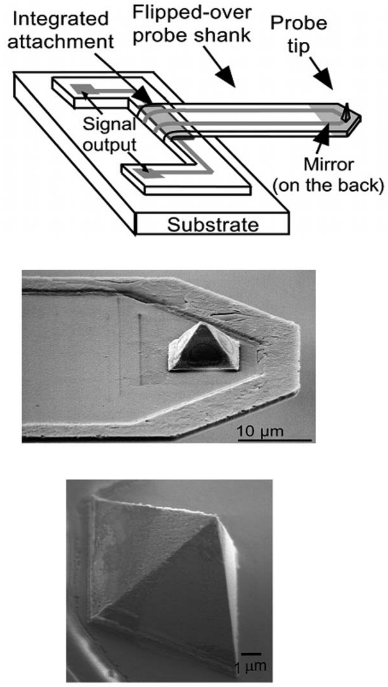

FIG. 1. (a) Schematic drawing of the probe die including the probe cantilever and tip (from Ref. 6). (b) SEM picture of the probe shank and (c) the tip.

is done by measuring the force-distance curve and positioning the probe at the edge of the linear region. The sample is scanned without the optomechanical feedback. The deflection signal provides topographical information, while the amplitude of the oscillation is related to the damping of the tip-sample interaction, and provides parametric information.

For the scanning measurements a 45 Hz excitation signal with a 4.5 V amplitude is employed. A second order low pass filter with a 10 ms time constant averages the lock-in amplifier signal. The amplitude of the probe oscillation is largest for $f < 1$ Hz, therefore a lower excitation frequency would provide greater resolution. However, the choice of excitation is limited by the scan speed of the AFM. As a result a higher (45 Hz) excitation frequency is chosen.

III. RESULTS

Figure 3 shows a plot of the amplitude of the probe deflection in air for applied voltages between 0.1 and 2.5 V at 20 Hz. The deflection amplitude is provided by the calibrated photodetector. A highly linear response, with a proportionality factor of 3.24 between the applied voltage and oscillation amplitude is observed. The amplitude of the oscillations is ~ 10 nm at the applied voltage of 2.5 V.

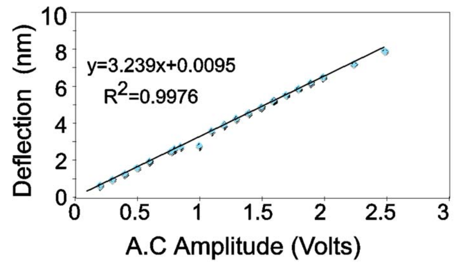

FIG. 3. (Color online) Plot of amplitude of the sinusoidal deflection (2ω) signal from the photodetector as a function of applied voltage.

Figure 4 shows the variation of the deflection signal as a function of applied frequency. The measurements were carried out for three different applied voltages: 0.5, 1, and 2 V. The normalized deflection is obtained by dividing the measured deflection by the excitation amplitude (the deflection is proportional to the applied voltage).

The three graphs are identical, showing the absence of pronounced second order effects. The maxima are seen at a frequency of 0.8 Hz, with a 3 dB frequency of 26 Hz. The broad peak and low frequencies suggest that the probe vibration is due to relaxation oscillations related to heating and cooling of the structure, instead of flexural mode oscillations.

Figure 5 shows the results of AFM scans of a 200 nm tall grating sample. The figure on the left is a reference topographical scan taken with feedback and without any electrical excitation to the cantilever. The figure on the right shows the plot of the 2ω ac deflection signal (AUX 1 in Fig. 2).

No optomechanical feedback is employed when the probe is thermally actuated. This is because feedback forces the probe oscillations to be damped. In addition, when the probe is in hard contact, with a large force applied between tip and sample, the oscillations are quenched. For these measurements the probe is kept in soft contact. In soft contact the microscopic forces between the tip and the sample provide the contact force with no external forces applied. The polymer probe, being highly compliant, does not apply large forces on the sample and the probe bends with changes in topography. Since the amplitude signal measures the damping of the oscillation, the signal is sensitive to the forces

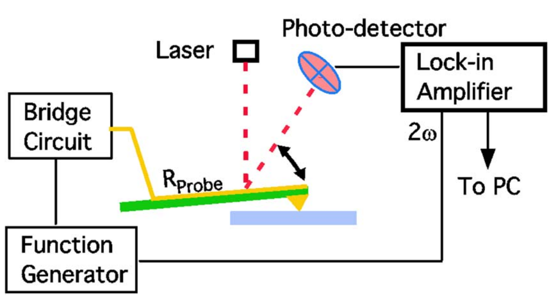

FIG. 2. (Color online) Sinusoidal alternating current (AC) oscillations produced by a function generator drive an AC current and cause the cantilever to vibrate. The AFM laser is reflected into the photodetector by the cantilever. The deflection signal is fed into a lock-in amplifier.

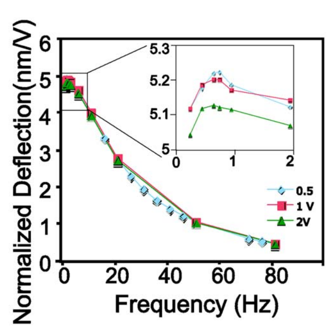

FIG. 4. (Color online) Plot of amplitude of the deflection signal (normalized to the applied voltage), versus the applied frequency. Inset: the plot in low frequency range showing a peak at 0.8 Hz.

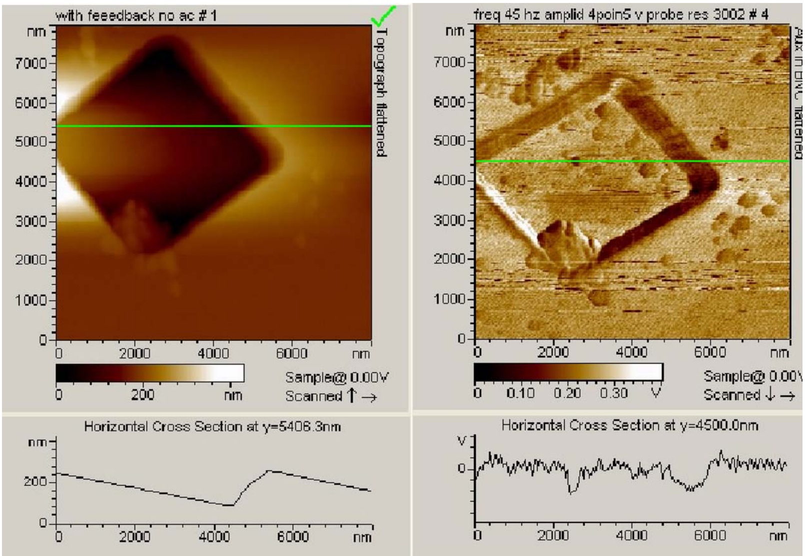

FIG. 5. (Color online) Scans of the 200 nm thick grating sample. Left: topographical image (without ac excitation). Right: plot of the amplitude of the deflection signal of the thermally actuated probe.

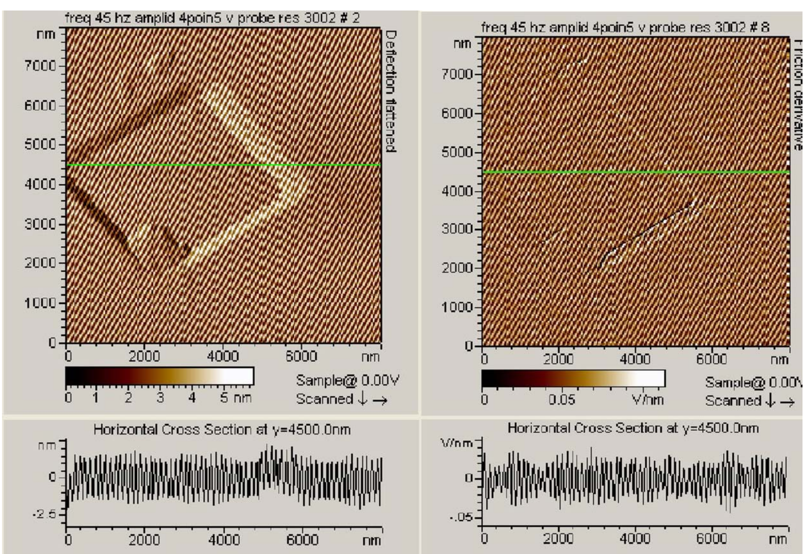

FIG. 6. (Color online) Deflection (left) and lateral (right) signals for a thermally oscillating cantilever measured as a difference in signal strength between different quadrants of the photodiode. The oscillation is superposed on the baseline deflection signal.

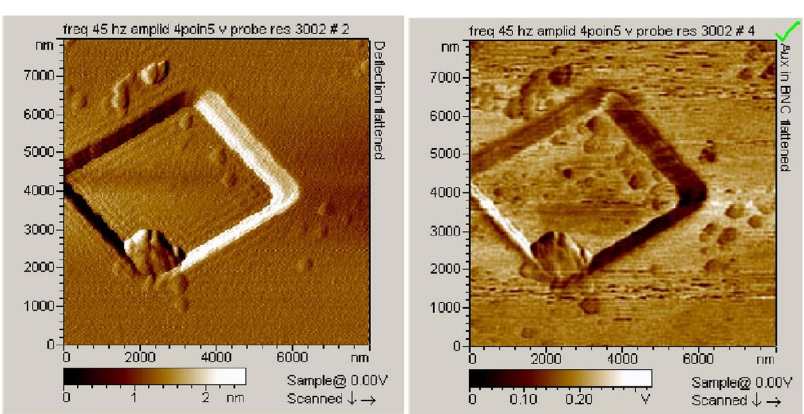

FIG. 7. (Color online) The scans from Fig. 6 after post-processing; deflection (left) and lateral (right).

between the tip and sample. Thus it is sensitive to variations in material properties in addition to topographical features. This is observed in Fig. 5, where the signal provides details absent from the topographical signal.

Figure 6 shows plots of the deflection and lateral signals measured simultaneously with the amplitude signal. The deflection signal measures the out of plane deformation of cantilever due to topography, while the lateral signal represents the torsional bending of the cantilever due to lateral forces. As seen in the figures, the signal due to the oscillating probe is superposed on baseline signals from deflection and lateral forces respectively.

Figure 7 shows the deflection and lateral signals after postprocessing. A fast-Fourier transform of the raw signal is performed, and the superposed oscillation signal is filtered out. The lateral scan in Fig. 7 is similar to the scan of the deflection oscillation signal in Fig. 5.

IV. CONCLUSION

This work demonstrates thermally actuated tapping mode AFM with polymer cantilevers. The thermal bimorph formed by a heated metal and an unheated polymer undergoes relaxation oscillations for applied voltages with $f < 100$ Hz. The oscillation amplitude change is a linear function of the applied voltage. The oscillation amplitude is damped by the tip-sample interaction. The resultant damping can be used to map topographical and material variations of the sample. Preliminary scans with a 200 nm tall grating show resolution comparable to that obtained by AFM deflection signals.

ACKNOWLEDGMENTS

The authors would like to thank Dr. Cheryl F. Albus for her support, and the National Science Foundation for funding this research project (Award No. 0822810).

[1] B. Rogers, L. Manning, T. Sulchek, and J. D. Adams, Ultramicroscopy **100**, 267 (2004).
[2] R. Pedrak, Tzv. Ivanov, K. Ivanova, T. Gotszalk, N. Abedinov, I. W. Rangelow, K. Edinger, E. Tomerov, T. Schenkel, and P. Hudek, J. Vac. Sci. Technol. B **21**,3102, (2003).
[3] J. Lee and W. P. King, Rev. Sci. Instrum. **78**, 126102 (2007).
[4] J.-H. Lee and Y. B. Gianchandani, Rev. Sci. Instrum. **75**, 1222 (2004).
[5] M.-H. Li, J. J. Wu, and Y. B. Gianchandani, J. Microelectromech. Syst. **10**, 3 (2001).
[6] A. S. Basu, S. McNamara, and Y. B. Gianchandani, J. Vac. Sci. Technol. B **22**, 3217 (2004).
[7] E. Hendarto, A. Altes, R. Heiderhoff, J. C. H. Phang, and L. J. Balk, *Proceedings 43rd Annual IEEE International Reliability Physics Symposium*, Piscataway, NJ, USA (IEEE, New York, 2005), pp.294–299.
[8] A. Gaitas and Y.B. Gianchandani, Ultramicroscopy **106**, 874 (2006).
[9] J. Wu, M. Reading, and D. Q. M. Craig, Pharm. Res. **25**, 1396 (2008).