2009 American Control Conference
Hyatt Regency Riverfront, St. Louis, MO, USA
June 10-12, 2009

High-speed intermittent-contact mode scanning probe microscopy using cantilevers with integrated electrostatic actuator and thermoelectric sensor

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Abstract*— Integrated sensing and actuation are pivotal for making high-throughput scanning probe microscopy based devices where a large number of probes are employed for parallel operation. A thermoelectric sensor and an electrostatic-actuation platform fabricated on a cantilever can provide a simple way to integrate sensors and actuators on probes of these devices. The electrostatic actuation facilitates an intermittent-contact mode imaging method which can be implemented for parallel operation of cantilevers. It can improve the durability of these devices and the rate of signal amplitude-loss over time by significantly reducing tip wear. The speed of this method scales with the operating frequency of the cantilever because the imaging data is captured directly from the deflection signal at the oscillation frequency of the cantilever. Traditional intermittent-contact methods are slow, and can not be implemented on a large number of cantilevers because they rely on the demodulation of the cantilever-deflection signal. In the intermittent-contact mode imaging using electrostatic actuation, small-amplitude oscillation is employed for increased speed and reduced tip-sample force. However, significant tip-sample adhesion from soft materials, such as polymers and biological samples restricts the operating frequency of the small-amplitude oscillation and subsequently the throughout of the device. In this paper, an intermittent-contact mode scanning probe microscopy method using electrostatic actuation with input-shaping is presented which can overcome the limitations posed by the adhesive forces. Intermittent-contact operation at the resonant frequency of the cantilever and small cantilever-oscillation within the adhesive region can be achieved by this method. The thermoelectric sensor integrated on the cantilever provides an off-contact signal that can be used in a feedback manner to ensure reliable small-amplitude intermittent-contact mode operation.

I. Introduction

Scanning probe microscopy (SPM) based techniques can be used to modify and interrogate materials down to the atomic scale [1], [2], [3]. Instruments exploiting these techniques can have many applications in pharmaceutical, materials and semiconductor industry. Moreover, currently available instruments despite their high-resolution capabilities are not suitable for many practical applications because of their low throughput. SPM-based instruments incorporating high-bandwidth components can be designed to increase their speed of operation [4], [5], [6], [7]. However, such approaches have not met the high-throughput requirements of many industrial applications. Note that the throughput of an SPM-based device scales with the number of cantilevers being employed. Current SPM systems employ a single cantilever along with a bulky and expensive optical detection system and a separate piezo-actuator for imaging. They are not suitable for incorporating a large number of cantilevers. For imaging with a large number of cantilevers, integrated sensors and actuators are essential on each cantilever [8], [9]. Using a similar principle, an ultrahigh-density storage device (shown in Fig. 1) was developed to write, read and erase data on very thin polymer films with densities beyond 1 $\mathrm{Tb/in^2}$ [10], [11]. In this device, a thermoelectric sensor and an electrostatic-actuation platform are fabricated on each of the cantilevers for parallel operation. The main component of the device is a micro-electro-mechanical-system (MEMS)-based cantilever array which provides a high-throughput scanning multi-probe microscope for imaging samples with relatively flat surfaces.

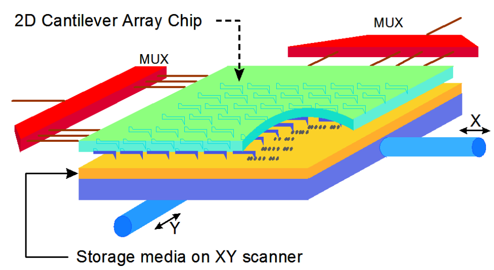

Fig. 1. Schematic of a MEMS-based scanning probe microscope where a large number of cantilevers are employed for parallel operation.

Note that high-speed operation at the individual cantilever level is also desired for high-throughput in a massive parallel configuration of cantilevers. An important challenge in high-speed imaging is the probing of the surface in a noninvasive manner with small tip-sample interaction forces. For example, lateral forces during imaging can be high for high scan-speeds which can wear the tip and damage the sample. Tip wear also results in a loss of image quality and decrease the lifetime of the SPM-based devices. The tip-sample forces are significantly reduced in intermittent-contact mode imaging methods. However, current intermittent-contact methods are slow because they rely on the demodulated deflection signal which has a low bandwidth; moreover, they are not suitable for parallel operation because of limitation from the electronic system [12], [13]. Intermittent-contact imaging using electrostatic actuation can provide a high-speed imaging method when the imaging signal is collected at the oscillation frequency the cantilever [14]. When a small cantilever oscillation amplitude is employed, the speed of operation is increased and the tip-sample forces are reduced. However, small-amplitude intermittent-contact imaging using small-amplitude inputs in [14] is limited to operating frequencies that are lower than the resonant frequency of the cantilever owing to significant tip-sample adhesion. In this paper, a high-speed intermittent-contact imaging method using elec-

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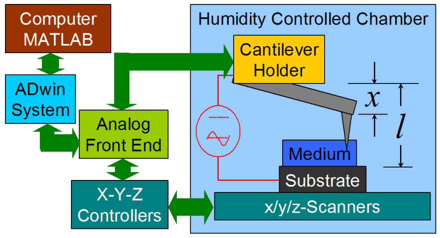

Fig. 2. Schematic of atomic force microscope setup

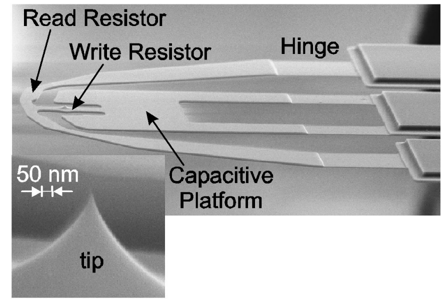

Fig. 3. Scanning electron microscope image of a cantilever with electrostatic actuation platform and thermoelectric sensors

trostatic actuation and thermoelectric sensing is presented that can overcome the tip-sample adhesion and capture imaging data at the resonant frequency of the cantilever (without requiring demodulation electronics). Input-shaping is employed to increase the speed of the intermittent-contact method presented in [14]. Using the new method presented here, the cantilever can be oscillated at small amplitudes within the tip-sample adhesion region of the soft materials.

The remainder of the paper is arranged as follows. First, in section II, our atomic force microscope setup is described. In section III, the cantilever model for electrostatic actuation and thermoelectric sensing, and tip-sample interaction model are described. In IV simulation results and the feedback scheme are presented. Finally, in section V, experimental results are presented.

II. Atomic Force Microscope Setup

An atomic force microscope setup (see Fig. 2) has been built to serve as test-bed for testing various functionalities of a MEMS-based scanning multi-probe microscope such as the data-storage system described in [15]. Commercial x-y-z scanners and controllers from Physik Instrumente (PI) are used in this setup. The scanners operate in closed-loop fashion using capacitive sensors. Each scanner is positioned with nanometer accuracy by supplying a precalculated voltage signal to the reference input of its controller. Various cantilevers, including the cantilevers identical to those employed in the MEMS data-storage device, can be used in this setup with all their functionalities (see Fig. 3). Electrical connections to the cantilever are made through a custom-designed cantilever holder. The holder is placed on a stage which can move with 5 degrees of freedom so that the cantilever can be positioned with x,y,z, pitch and yaw adjustments. The sample having an electrical connection to the substrate underneath it (by means of a pin) is placed on top of the scanners. The setup can be put on top of a vibration isolation table and inside an acoustic isolation chamber with humidity and temperature control. The operation of the AFM is automated by a Matlab based software which controls it using a digital real-time system (ADwin) and an analog front end (AFE) (see Fig. 2).

III. Modelling

In intermittent-contact scanning probe microscopy using electrostatic actuation and thermoelectric sensor, the effect of the tip-sample interaction force on the measured cantilever response can be analyzed by considering a linear model for the cantilever with nonlinear input due to electrostatic actuation, a first-order linear model for the thermoelectric sensor, and a static nonlinear model for the tip-sample interaction force. The cantilever dynamics is given by

$$m\ddot{x}(t)+c\dot{x}(t)+kx(t) = \frac{\epsilon_0 A}{2(\ell - x(t))^2}V(t)^2+\phi\left(x(t)\right), \quad (1)$$

where m, c, k and x are the effective mass, damping coefficient, spring constant, and the deflection of the cantilever, respectively. ϵ_0, V, ℓ and A are the electric permittivity of air, the voltage applied to the substrate underneath the sample for electrostatic actuation, and the effective separation and area between cantilever and substrate, respectively. The electrostatic force is modelled assuming that the cantilever and the substrate behave like two oppositely charged parallel plates. Note from Eq. (1) that when the cantilever is forced towards the sample by applying a voltage V, the mechanical restoring force on the cantilever increases linearly with x when the electrostatic pull-in force increases more rapidly as a nonlinear function of x. As a result, the cantilever snaps into the sample in an unstable manner if the cantilever deflection $x > \frac{\ell}{3}$ [16]. In this paper, the cantilever is operated within the stable region, i.e., $x(t) < \frac{\ell}{3}$ for all times t for smooth intermittent-contact imaging.

The nonlinear tip-sample interaction force $\phi(x)$ can be modelled as a piecewise linear system given by

$$\begin{aligned}\phi(x) &= 0 \text{ if } x \geq -g+d \\ &= -k_a(x+g-d)+c_a\dot{x} \text{ if } -g \leq x < -g+d \\ &= k_r(x+g)-k_a(x+g-d)+c_r\dot{x}+c_a\dot{x} \\ &\quad \text{if } x < -g, \end{aligned} \quad (2)$$

where k_a, k_r, c_a, c_r, g, and d are the equivalent adhesive (attractive) and repulsive spring constants and damping coefficients, the initial tip-sample separation, and the length of the adhesive region, respectively [17]. $\phi(x)$ is a nonconservative model of the tip-sample interaction force which takes into account the energy dissipation in the sample when c_a and c_r are nonzero. When imaging soft materials such as thin polymer films, the tip-sample adhesion force has a significant effect on the cantilever response. When the cantilever is forced to penetrate the surface, its response is dominated by the strong van der Waals repulsive force [18]. When the cantilever is forced to retract from the surface, its response is dominated by the strong adhesive force.

The thermoelectric sensor (see Fig. 3) is a micro-heater which is a low-doped region on a high-doped cantilever.

When a substrate is present near the sensor, a significant portion of the heat is conducted into the substrate through the air-gap between the micro-heater and the substrate [19]. The deflection of the cantilever is sensed by measuring the temperature-dependent resistance of the micro-heater, which changes because of the modulation of the air-gap length with the movement of the cantilever. The deflection sensing transfer function can be described by

$$G_{th}(s) = \frac{K_{th}}{1 + a_{th} s}, \tag{3}$$

where K_{th} and a_{th} are the gain and time constant of the above first-order transfer function, respectively [20], [21], [22].

All parameters in the Eqs. (1), (2) and (3) that describe the intermittent-contact operation can be obtained experimentally.

A. Intermittent-contact imaging with input shaping

For intermittent-contact imaging, the cantilever is permanently bent towards the surface to a desired position close to the surface by applying a DC voltage V_0 between the cantilever and the substrate (see Fig. 4). A shaped voltage signal $V_{ac}(t)$ is added to V_0 for small-amplitude intermittent-contact operation of the cantilever. The net voltage applied between the cantilever and the substrate is given by

$$V(t) = V_0 + V_{ac}(t), \tag{4}$$

where $V_{ac}(t)$ is chosen such that $V(t)$ does not change sign for all times t.

Typically, for intermittent-contact operation between cantilever positions x_c and x_o, which correspond to the contact and the off-contact state, an alternating voltage whose peaks correspond to the approach and retract voltages $V_a = \sqrt{2kx_c(\ell - x_c)^2/\epsilon_0 A}$ and $V_r = \sqrt{2kx_o(\ell - x_o)^2/\epsilon_0 A}$, respectively, is applied (see Eq. (1)). However, such an approach is slow because of small applied voltages and the large tip-sample adhesion force. The speed of intermittent-contact operation can be improved by shaping the input signal as explained below. Note that, when the cantilever is in contact with the sample, the restoring force at cantilever position x_c and retract voltage V_r is given by $F_r = kx_c - \epsilon_0 A V_r^2/2(\ell - x_c)^2$. The adhesion force can be overcome instantly and the retraction time can be reduced significantly if retraction voltage is set to zero volts. Zero retraction voltage can be applied for a short duration of time followed by the steady state retraction voltage V_r mentioned above, for small-amplitude oscillation. Likewise, the approach time can be reduced by applying the maximum allowed input voltage to provide an initial acceleration to the cantilever. Before the cantilever comes in contact with the surface, the approach voltage can be reduced to the steady-state approach voltage V_a mentioned above, after a short time in order to reduce the approach velocity to zero at the time of contact for small tip-sample forces. The duration of the retraction and the approach voltage-pulses can be calculated by considering a linearized model of the cantilever dynamics given by Eq. (1). The boundary conditions are that the velocity of the cantilever approaches zero at the in and out of contact positions x_c and x_o, respectively.

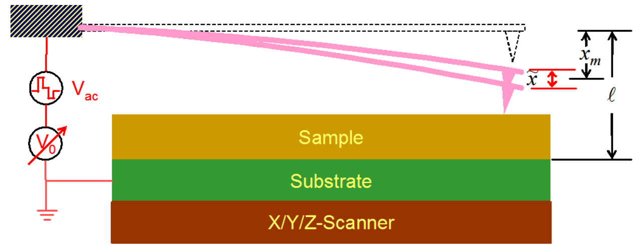

Fig. 4. Schematic of the small-amplitude intermittent-contact imaging with input shaping for electrostatic actuation.

Because the voltage-pulses shaping the input applied between the cantilever and the substrate for electrostatic actuation have a small duration of time, a linear model for the cantilever with electrostatic actuation can be obtained by assuming a mean voltage value V_m for $V(t)$ in Eq. (1). Suppose that the corresponding cantilever deflection can be written as

$$x(t) = x_m + \tilde{x}(t), \tag{5}$$

where x_m and $\tilde{x}(t)$ denote the mean deflection and the periodic oscillation of the cantilever. Substituting (5) in (1) and ignoring the second- and higher-orders terms, and considering the output sensor dynamics given by Eq. (3), the free cantilever dynamics (after ignoring the tip-sample force $\phi(x)$) in state-space form is given by

$$\begin{bmatrix} \dot{x}_1 \\ \dot{x}_2 \\ \dot{x}_3 \end{bmatrix} = \begin{bmatrix} 0 & 1 & 0 \\ -\left(\omega_0^2 - \frac{K_{esf} V_m^2}{(\ell - x_0)^3}\right) & -\frac{\omega_0}{Q} & 0 \\ \frac{K_{th}}{a_{th}} & 0 & 1 \end{bmatrix} \begin{bmatrix} x_1 \\ x_2 \\ x_3 \end{bmatrix} - \begin{bmatrix} 0 \\ 1 \\ 0 \end{bmatrix} \left(\omega_0^2 x_0 - \frac{K_{esf}}{2(\ell - x_0)^2} V^2(t)\right), \tag{6}$$

where $x_1 = \tilde{x}$, $x_2 = \dot{\tilde{x}}$, x_3 is the cantilever oscillation that is measured by the thermoelectric sensor, and $K_{esf} = \frac{\epsilon_0 A}{m}$. Note that $\frac{k}{m} = \omega_0^2$ and $\frac{c}{m} = \frac{\omega_0}{Q}$, where ω_0 and Q are the natural resonant frequency and the quality factor of the cantilever, respectively.

IV. Simulation

The simulation results are based on the system model given by Eq. (1) along with the tip-sample interaction model given by Eq. (2). The model parameters were obtained from the experiments described in Section V. In Fig. 5 the simulated cantilever deflection signals are shown in blue and red when a square-wave input and a shaped square-wave input are used for intermittent-contact imaging, respectively. The surface profile is shown in green. The contact and the off-contact voltages of the square-wave and the shaped-input signals are obtained from the electrostatic approach curve shown in Fig. 8. The off-contact voltage corresponds to a cantilever deflection which avoids the adhesion region. The contact voltage is chosen such that the tip can reach the surface at different scan positions (because the vertical position of the sample is fixed). Note that an imaging signal sampled at the oscillation frequency of the cantilever when the tip is in contact with the surface corresponds well with

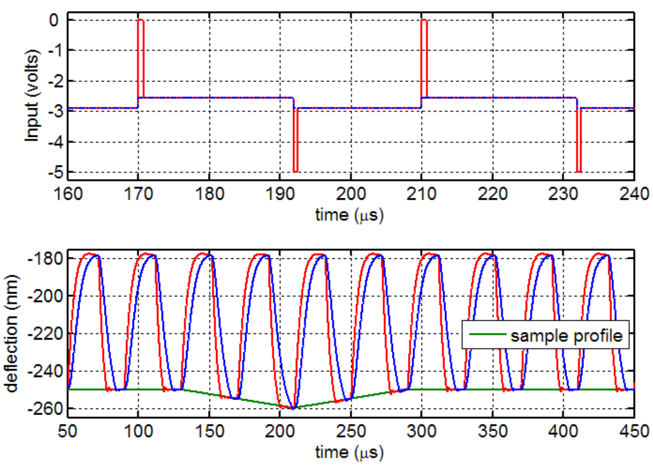

Fig. 5. Simulation: The input (top) and the corresponding cantilever-deflection (bottom) are shown when periodic square-wave-input (blue) and shaped-input (red) were applied.

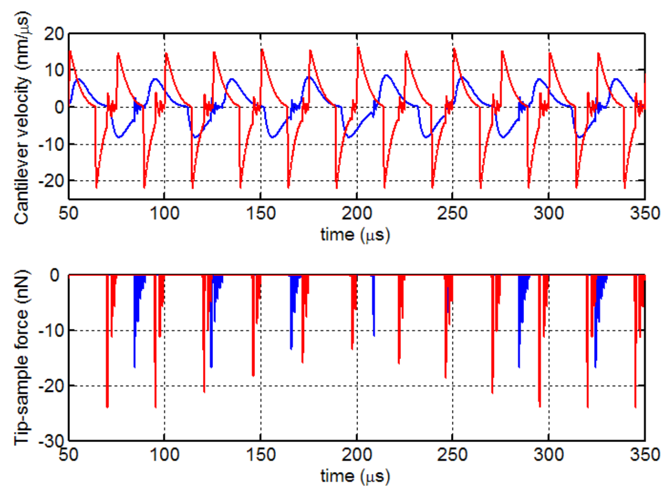

Fig. 6. Simulation: The cantilever-velocity (top) and the tip-sample force (bottom) are shown when square-wave-input (blue) and shaped-input (red) were applied.

the topography of the surface. To avoid artifacts in the imaging signal, the tip is required to stay in contact with the surface for a finite duration of time. In Fig. 5, when a small-amplitude input is used, the operating frequency is limited to 25 kHz (which is lower than the resonant frequency of the cantilever of 50.7 kHz). The limitation on the operating frequency is due to the large tip-sample adhesion force from the soft sample. When input shaping is used, the duration of approach and retraction improves significantly (see Fig. 5) and the cantilever can be operated at 40 kHz with the same steady state approach and retract voltages. The cantilever velocity and tip-sample forces are available from simulation and shown in Fig. 6. When input shaping is used, the initial approach and retract velocities are higher which improves the response time. At the same time, the contact and off-contact velocities are zero at their corresponding positions which maintains small tip-sample force and small amplitude oscillation. When input shaping is used, the tip-sample adhesion is overcome instantly and the limitation imposed by it on the operating frequency can be eliminated entirely. This enables intermittent-contact operation within the adhesive region of the surface, which is otherwise not possible with small-amplitude inputs. In Fig. 7, intermittent-contact operation at 50 kHz with a peak-to-peak amplitude of 25 nm is shown. For small-amplitude oscillation, the shaping pulse-width during retraction is reduced and a retraction voltage corresponding to the new steady state off-contact position is applied. Note that the imaging signals having spatial frequencies of up to the half of the resonant frequency of the cantilever can be captured by sampling the deflection signal at the oscillation frequency when the tip is in contact with the surface.

Higher mode dynamics of the cantilever may get excited because of input shaping. However, simulation shows that the intermittent-contact sensing with electrostatic actuation does not rely on the higher-mode dynamics of the cantilever for its operation. With prior knowledge of the complete modal dynamics of the cantilever, the additional short approach and retraction pulses which shape the input can be modified to prevent excitation of unwanted higher-mode dynamics of the cantilever.

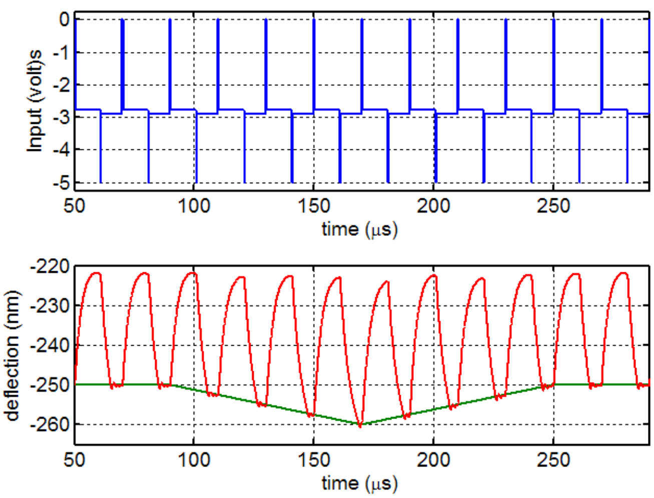

Fig. 7. Simulation: The input (top) at 50 kHz and the corresponding cantilever-deflection (bottom) are shown when shaped-input is applied for small-amplitude operation.

A. Feedback for reliable intermittent-contact operation

In intermittent-contact imaging, small-amplitude oscillation is desired for increased speed and small tip-sample interaction force. However, it is difficult to sustain in practice small-amplitude oscillations due to sample contamination and micro-scale surface irregularities. Note that in Figs. 5 and 7 the deflection signal when the cantilever is at the farthest point from the surface, is largely independent of the topography of the sample. For reliable intermittent-contact operation, this off-contact signal is captured and controlled by changing the retraction voltage V_r (or the offset voltage V_0) applied between the cantilever and the substrate. The feedback bandwidth is high because it is dictated by the bandwidth (resonant frequency and quality factor) of the cantilever [14]. Also, the off-contact signal from the

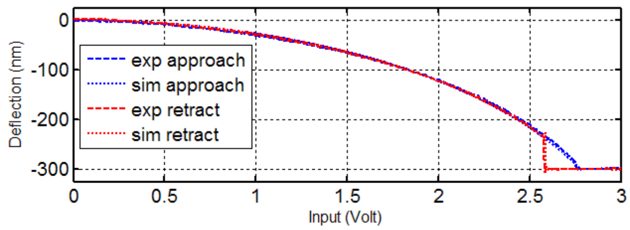

Fig. 8. Experimental approach curve obtained by applying electrostatic force on the cantilever and the corresponding fit with the parameters used in simulation.

thermoelectric sensor captures the vertical (z-direction) drift in the SPM system and the slope on the sample surface because it is a measure of the separation between the sensor and the sample underneath the sensor. It can be utilized to compensate for the vertical-drift in the system and to correct the slope during imaging by controlling the reference signal given to the z-scanner-controller for positioning the sample.

V. Experimental Results

Experiments on intermittent-contact imaging with input shaping were carried out using the AFM setup described in Section II. A relative humidity of less than 5% and steady ambient temperature were maintained during the experiments. Low humidity reduces the undesired squeeze film damping effect, which could hamper small-amplitude intermittent-contact imaging. Offset voltage feedback was employed for reliable imaging.

First, an approach experiment using the z-scanner was performed to set a desired tip-sample separation. The z-scanner was moved towards the cantilever until the tip-surface contact was observed in the thermoelectric sensor signal. Then the scanner was retracted by 300 nm and fixed at that position for the remainder of the experiment. Because of adhesion, the tip remained in contact with the surface during retraction for 55 nm of backward motion of the scanner from the first tip-sample contact-position that was established during the forward motion.

After setting the tip-sample separation at the desired value, the operating voltages for electrostatic actuation were obtained from an approach experiment performed by applying a saw-tooth voltage signal between the cantilever and the substrate underneath the sample (see Fig. 8). The cantilever was connected to the ground of the power supply and the substrate voltage was ramped up until tip-sample contact was observed in the thermoelectric sensor signal. The voltages required to pull the cantilever in and out of contact with the sample were different owing to tip-sample adhesion. The estimated adhesion in electrostatic voltage was 0.26 V.

The cantilever and thermoelectric sensor models for simulation were obtained from the frequency response of the cantilever by using an Agilent 33250A network analyzer. The cantilever was bent towards the surface by applying 2.6 V to the substrate. A 50 mV amplitude chirp signal from the analyzer was added to the substrate voltage. The thermoelectric sensor bias voltage was 2.25 V. The cantilever and the thermoelectric sensor model parameters given by Eq. (6) were directly obtained from the frequency response data. The frequency response data together with a third-order transfer function fit to it are shown in Fig. 9. The first-order component of the transfer function fit is taken as the model for the thermoelectric sensor. The -3dB bandwidth of the sensor is approximately 8.4 kHz. The second-order component of the transfer function fit is taken as the linear model for the cantilever near the surface given by Eq. (6). The equivalent resonant frequency and the quality factor of the cantilever were 50.7 kHz and 0.73, respectively. The electrostatic force constant K_{esf} and the tip-surface interaction model parameters given by Eq. (2) are obtained from the approach curve shown in Fig. 8 [23].

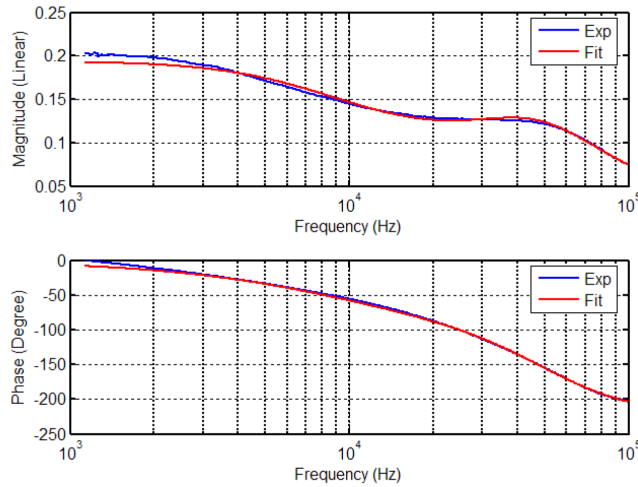

Fig. 9. Frequency response date along with a third-order transfer function fit is shown.

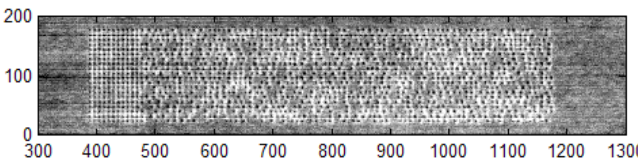

Fig. 10. An image of a soft polymer surface with nanometer deep bit pattern written at 1 $\mathrm{Tb/in}^2$ is shown.

An intermittent-contact image (with input shaping) of a soft polymer surface with nanometer-deep indentations written at 1 $\mathrm{Tb/in}^2$ (where the presence and absence of an indentation correspond to a data bit '1' and '0', respectively) is shown in Fig. 10. The symbol pitch and the track pitch are 14.67 and 29.33 nm, respectively. The scanner velocity was 146.7 μm/s. The cantilever oscillation frequency was 40 kHz, and the symbol frequency was 10 kHz. The in- and off-contact voltages were 2.9 and 2.5 V, respectively. The input shaping voltages applied during imaging were 5 V for 5.4 μs during approach and 0 V for 2.3 μs during retraction (see Fig. 12). The pulse widths were tuned during the experiment so that a good oscillation of the cantilever was achieved during intermittent-contact operation. The amplitude signal for one of the sections of the image is shown in Fig. 11. The proportional and integral gains for feedback based on offset voltage control were chosen as $K_p = -5 \times 10^{-2}$ and $K_i = -1 \times 10^{-6}$.

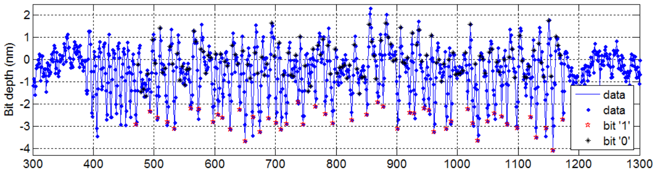

Fig. 11. Section view of an image of a soft polymer surface with nanometer deep bit pattern written at 1 Tb/in^2 is shown.

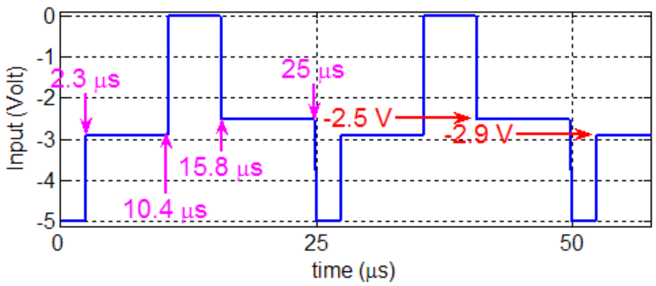

Fig. 12. The input pulse-shape used in imaging a soft polymer surface with nanometer deep bit pattern written at 1 Tb/in^2 is shown.

VI. Conclusion

Thermoelectric sensors and electrostatic actuators provide an convenient solution to integrate sensors and actuators in each cantilever for high-throughput scanning probe microscopy using a large number of cantilevers. The intermittent-contact sensing method presented in this paper can be implemented on such devices. It provides high-speed imaging capability at each cantilever level while improving the durability of these devices. Input shaping is used to increase the speed of operation by eliminating the effect of tip-sample adhesion. Using this method imaging data containing spatial frequencies of up to the half of the resonant frequency of the cantilever are captured. Off-contact signal feedback is used for reliable small-amplitude operation which improves the speed and reduces the tip-sample force. In experiment, nanoindentations on a polymer sample was imaged at a scan-speed corresponding to spatial frequency of 10 kHz with the cantilever oscillating at 40 kHz using input shaping. Simulation and experiment showed that this operating frequency could not be achieved without input-shaping because of large tip-sample adhesion. Assuming that the amplitude modulation imaging method using a tapping mode cantilever usually captures imaging signal below 1 kHz, an order of magnitude improvement in speed was shown in the experiment.

VII. Future work

In the future, the authors would like to demonstrate high-throughput intermittent-contact imaging by employing multiple cantilevers.

VIII. Acknowledgements

We are pleased to acknowledge stimulating discussions with our colleagues at the IBM Zurich Research Laboratory, and P. Agrawal and M. V. Salapaka from the University of Minnesota, Twin Cities. We are thankful to Charlotte Bolliger at the IBM Zurich Research Laboratory for her help in preparing the manuscript.

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