Thermal Noise Response Based Static Non-Contact Atomic Force Microscopy

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ABSTRACT

Micro-cantilever based devices have revolutionized imaging and they are the primary tools for investigation and control of matter at the nanoscale. In this paper a novel approach based on the thermal noise response of the cantilever is developed that makes non-contact AFM possible in static mode. This technique exploits the dependence of cantilever's resonant frequency on the tip-sample separation to maintain a small tip-sample separation by regulating the equivalent resonant frequency. The resonant frequency is estimated from cantilever's response to the thermal noise. The experiments performed in ambient room conditions have achieved tip-sample separations as small as 4 nm for time periods in excess of 20 min. Based on this control technique a new static non-contact mode operation of AFM has been demonstrated. This method has given rise to an extremely powerful non-contact imaging technique capable of detecting sub-angstorm features at a bandwidth of 200 Hz with a force sensitivity of a few pN.

Keywords: thermal noise response, cantilever resonance, non-contact force microscopy, static mode operation, tip-sample separation control

1 INTRODUCTION

In many putative studies a micro-cantilever based investigation of extremely small forces evolving over large time scales is of considerable interest. One such application is the study of conformational changes of proteins that is fundamental to investigating the function of proteins. Another application is the detection of single electron spin that is a key requirement for quantum computing. In these applications in order to detect the highly localized forces it is essential to maintain a tip-sample separation in the order of a few Å over extended periods of time.

In such applications the cantilever tip is too obtrusive to the observation if it encounters the repulsive region of the tip-sample potential. This necessitates maintaining the tip in the attractive regime of the tip-sample potential. In many samples this also achieves the objective of maintaining sub-nanometer tip-sample separation. A primary hurdle in achieving this goal is the drift of the system that becomes particularly detrimental due to the large time scales involved. These drift effects are due to uncertain factors like changes in the deflection detector [3], [6], thermal bending [5], [7] and drift in the piezo based sample positioner. Deflection based force detection cannot differentiate between attractive and repulsive interactions thereby making it unsuitable for maintaining the tip in the attractive regime. The dynamic modes of operation, viz., amplitude modulation (AM) [2] and frequency modulation (FM) [1], are not applicable due to the large amplitude oscillations of the cantilever tip that are needed in these methods.

In this letter we present an approach based on the thermal noise response of the cantilever that promises to meet the demands of maintaining sub-nanometer separations over large time periods and consequently enable a static non-contact mode operation of the AFM.

2 MODEL

The microcantilever modeled as a single spring-mass-damper system, as shown in Figure 1, is described by

$$m\ddot{p}(t) + c\dot{p}(t) + kp(t) = \eta(t) + F(t) \qquad (1)$$

where $p(t)$ is the cantilever deflection, m is the mass of the cantilever, c ($= \frac{m\omega_0}{Q}$) is the damping coefficient , k is the spring constant and $\eta(t)$ is the Langevin thermal noise forcing term and $F(t)$ describes other external forces acting on the cantilever. The noise power spectral density of a cantilever in thermal equilibrium in the absence of external forcing is given by

$$S_{pp}(\omega) = \frac{2k_BT}{m}\frac{\gamma}{(\omega_0^2-\omega^2)^2+\gamma^2\omega^2} \qquad (2)$$

where k_B is the Boltzmann's constant, T is the temperature, $\omega_0 = \sqrt{k_0/m}$ is the resonant frequency of the cantilever and $\gamma = c/m$ is the damping constant.

When the tip interacts with the sample, the tip-sample forces alter the effective spring constant thereby changing the resonant frequency. For small tip-sample forces the resonant frequency shift $\Delta\omega$ can be approximated by the relation

$$\frac{\Delta\omega}{\omega_0} = \frac{k_s}{2k},\ k_s = \frac{\partial F_s}{\partial l}. \qquad (3)$$

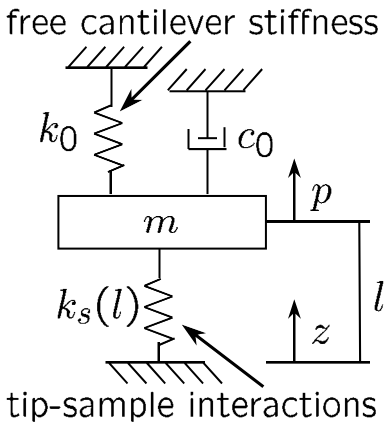

Figure 1: The cantilever is modeled by the spring-mass-damper system. The effect of the tip-sample interactions is modeled by a nonlinear spring whose stiffness depends on the tip-sample separation.

where k_s is the gradient of the tip-sample interaction force F_s with respect to the tip-sample separation l. The equivalent resonant frequency ω_{eq} $(= \omega_0 + \Delta\omega)$ decreases (increases) when the tip-sample interaction force is attractive (repulsive). Thus, by observing the equivalent resonant frequency the attractive and repulsive regimes of the interaction potential can be differentiated. The information about ω_{eq} is available in the power spectral density of the thermal noise response as a shift in the peak position of the power spectrum. The main contribution of this letter is to utilize this fact to estimate the equivalent resonant frequency from the power spectrum to control the tip-sample separation. One fundamental requirement is that the cantilever spring constant be large enough to overcome any jump-to-contact instabilities.

3 STATIC NON-CONTACT MODE OPERATION

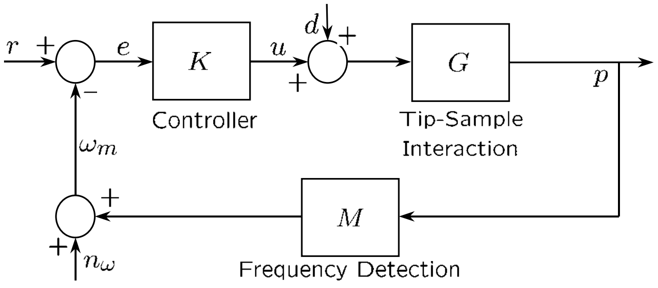

Figure 2: A schematic block diagram of the closed loop

As described above maintaining a constant tip-sample separation translates into a problem of regulating the resonant frequency of the cantilever at a desired value as illustrated in the proposed control scheme in Figure 2. The estimation of frequency from the cantilever's thermal noise response is performed by Pisarenko harmonic decomposition [4]. The deflection signal is assumed to be a single sinusoid buried in noise. The frequency of this sinusoid corresponds to the equivalent resonant frequency of the micro-cantilever.

The controller (K) should be designed such that it is capable of compensating for the disturbances afflicting the system due to drift. Any other disturbance that has a bandwidth greater than the closed-loop bandwidth of the system, will go unchecked by the controller and will potentially show up as a variation in the cantilever's resonance. This is the principle behind the use of this technique for imaging to monitor and observe variations in tip-sample interaction forces.

4 EXPERIMENTAL RESULTS

The thermal noise based non-contact mode operation has been demonstrated in a variety of experiments few of which are discussed below. The experiments are performed on a Digital Instruments Multimode AFM in ambient environment. The signal processing for frequency estimation and the controller are implemented on a TMS320C44 digital processing platform. The cantilevers used are of Silicon with a nominal $Q = 300, k = 40$ N/m and $\omega_0 = 350$ kHz and the sample surface was freshly cleaved HOPG.

Figure 3(a) shows the variation in the cantilever eigenfrequency as a function of tip-sample separation during approach and retraction. It is seen that the resonant frequency decreases due to the long range attractive tip-sample interactions. However, a similar effect is not observed in the deflection as the maximum observable deflection (see Figure 3(b)) estimated to be approximately 4 pm is much smaller than the deflection sensitivity of the instrument.

The following experiment demonstrates the feasibility of the proposed method to control the tip-sample separation. In this experiment a step change is given to the reference frequency and the recorded cantilever resonant frequency, control signal and deflections are shown in Figure 4(a), (b) and (c). In the initial stages of the control, the tip is not interacting with the sample and the measured resonance is 397 kHz (see Figure 4(a)), which is the free resonant frequency of the cantilever. The controller, therefore, acts to move the sample towards the tip as seen in Figure 4(b). Once the desired tip-sample separation is achieved, indicated by the resonant frequency being close to the reference, the control action counteracts the drift in the instrument. At approximately 1600 s into the experiment the step change in the reference is introduced and the controller is able to track this change. As the reference is reduced, implying a smaller desired tip-sample separation, the controller moves the sample towards the tip and is seen as

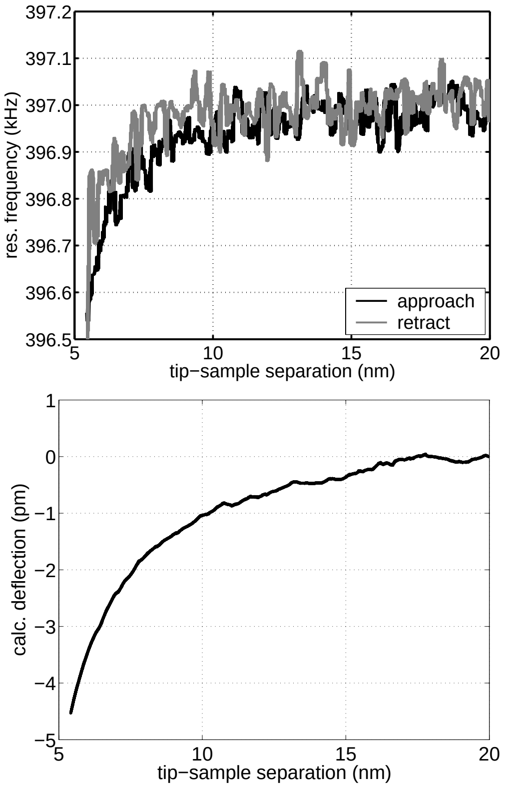

Figure 3: The variation in (a) resonant frequency with tip-sample separation. (b) The calculated deflection $p_{calc} = \frac{2}{\omega_0}\int \Delta\omega dl$ for the frequency observed in (a).

a small "jump" in Figure 4(b) at 1600 s shown by an arrow and magnified in the lower left inset. This control action results in a reduction in tip-sample separation of approximately 0.8 nm as seen in the upper right inset. This correlates well with the change required for a reduction in the resonance from 396.9 kHz to 396.75 kHz (see Fig 3(a)). The new reference is reached in approximately 7 s. As reasoned earlier, the variations in deflection in Figure 4(c) can be attributed to the drift in the deflection sensor as the tip-sample forces are too small to induce any perceivable change in the deflection. Figure 4(d) shows the estimated tip-sample distance is 6 nm in good agreement with the corresponding separation for a resonant frequency of 396.75 kHz in Figure 3(a). From the above discussion and Figure 4 it can be inferred that a tip-sample separation of approximately 7 nm is being maintained for over 15 min (from 600 s to 1600 s) and a separation of around 6 nm for time periods in excess of 5 min (from 1610 s to 1930 s). Similar experiments have yielded a tip-sample separation of under 3 nm for over 20 min.

Figure 5 demonstrates the non-contact imaging capability of this method. In this experiment the force gradient is modulated by moving the sample in a sinusoidal manner while the tip-sample separation in being

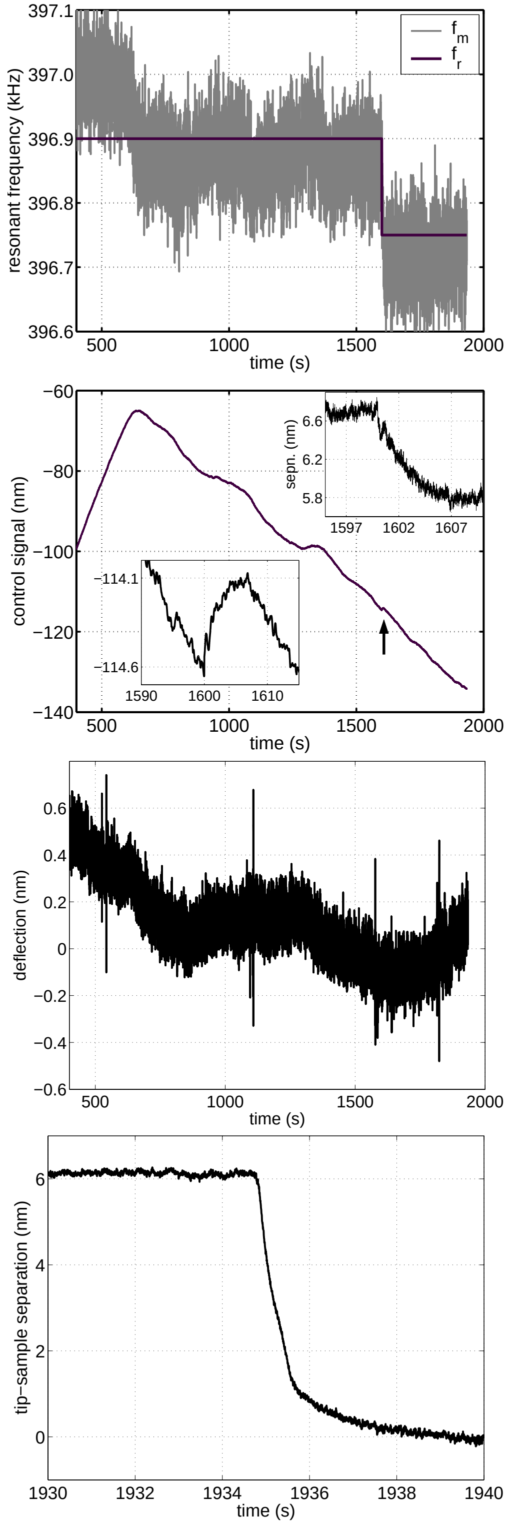

Figure 4: Time history of (a) estimated frequency (b) control effort and (c) deflection while tracking a step change in reference frequency shown in (a). The insets in (b) show the step response in tip-sample separation (upper right) and control effort (lower left). (d) Tip-sample separation just before tip-sample contact.

maintained. The resonant frequency of the cantilever in Figure 5(a) during control is lower than its free resonance, signifying the non-contact mode of operation. The drift compensation seen in Figure 5(b) indicates that the closed-loop bandwidth of 0.5 Hz is larger than the bandwidth of the drift processes in the system. During control the sample position is modulated by a 0.5 Å, 5 Hz sine wave such that the modulation frequency is greater than the closed-loop bandwidth and hence will not be acted upon by the controller. This results in a modulation of the tip-sample separation and consequently the cantilever's resonance modulates at 5 Hz as seen in Figure 5(c). No signature of this modulation is seen in the deflection signal (see Figure 5(d)) indicating an inferior sensitivity to the frequency based gradient detection. The changes in tip-sample forces induced by the modulation of the sample position have been estimated to be a few pN. Recent experiments have resulted in the detection of tip-sample modulations of 0.25 Å up to 200 Hz.

5 CONCLUSIONS

A novel static non-contact mode of operation of AFM based on the thermal noise response of the cantilever has been demonstrated. In this approach cantilever's thermal noise response is used to estimate the changes in its resonant frequency that is fed back for maintaining the tip-sample separation. This method enables an extremely powerful non-contact imaging technique in static mode with bandwidths up to 200 Hz and force sensitivity of a few pN observed in experiments performed in ambient room conditions. Tip-sample separations as small as 4 nm for periods extending over 20 min have been achieved. A better design of instrumentation and controlled experimental conditions promise improved performance of this technology.

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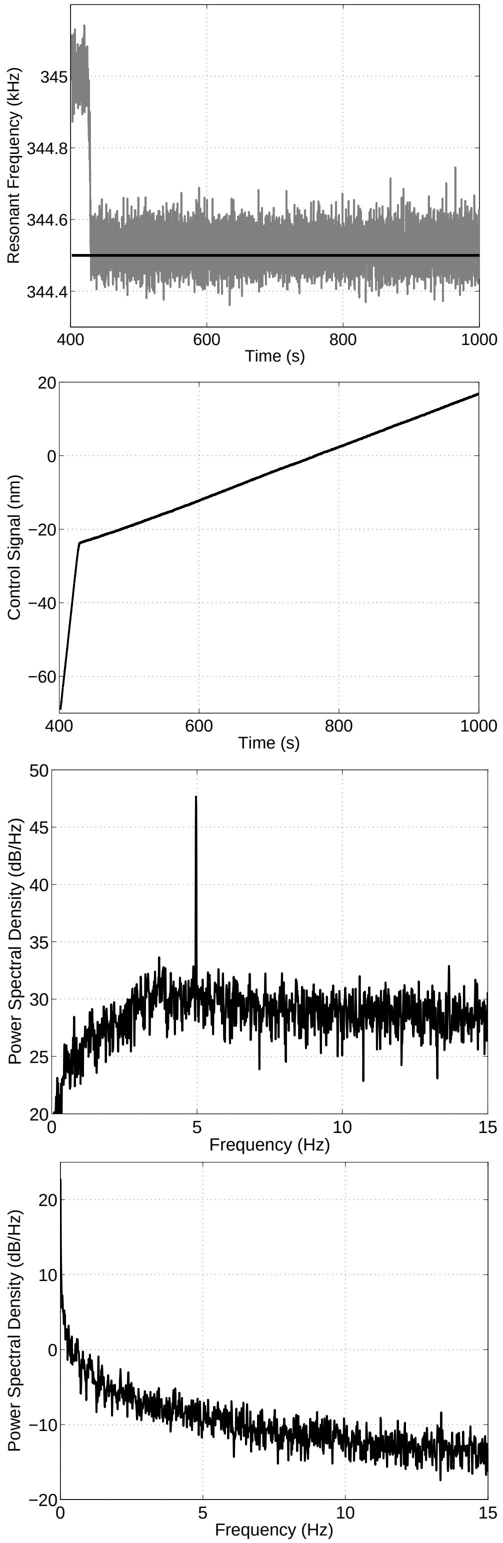

Figure 5: (a) Cantilever's natural resonance is at 345 kHz. A lower resonant frequency (344.5 kHz) during control signifies the tip in attractive region. (b) Drift compensation by the controller. (c) Power spectral density plot of the estimates of the resonant frequency - modulation of the resonant frequency at 5 Hz (d) Power spectral density plot of cantilever deflection. No modulation in the deflection is observed