Frequency modulation detection using high-Q cantilevers for enhanced force microscope sensitivity

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(Received 17 September 1990; accepted for publication 28 September 1990)

A new frequency modulation (FM) technique has been demonstrated which ennances the sensitivity of attractive mode force microscopy by an order of magnitude or more. Increased sensitivity is made possible by operating in a moderate vacuum ($<10^{-3}$ Torr), which increases the Q of the vibrating cantilever. In the FM technique, the cantilever serves as the frequency determining element of an oscillator. Force gradients acting on the cantilever cause instantaneous frequency modulation of the oscillator output, which is demodulated with a FM detector. Unlike conventional "slope detection," the FM technique offers increased sensitivity through increased Q without restricting system bandwidth. Experimental comparisons of FM detection in vacuum ($Q\sim50\,000$) versus slope detection in air ($Q\sim100$) demonstrated an improvement of more than 10 times in sensitivity for a fixed bandwidth. This improvement is evident in images of magnetic transitions on a thin-film CoPtCr magnetic disk. In the future, the increased sensitivity offered by this technique should extend the range of problems accessible by force microscopy.

I. INTRODUCTION

The atomic force microscope¹ (AFM) is frequently used to map force gradients near surfaces without surface contact. Force gradients are detected as shifts in the resonant frequency of the mechanical vibration of a cantilever, whose tip is positioned near the surface of interest. A raster scan of the tip over the surface in this mode provides an image of the force gradient variations above the surface. This mode of operation, which is often called the "ac" or "attractive" mode, has a variety of applications, including noncontact surface profilometry through the van der Waals interaction,^{2.3} study of fringing fields above magnetic samples,^{4.5} and imaging of localized charge.^{6,7} In the most commonly used detection scheme, the cantilever is driven at a constant frequency near resonance, and force gradients are detected as variations in the amplitude or phase of the cantilever vibration. In this scheme, the signal-to-noise ratio (S/N) for a given bandwidth can be increased by increasing the Q of the cantilever resonance. However, increasing the Q also decreases the maximum available bandwidth of the system. For example, operating in vacuum, the cantilever Q can be > 50 000, which offers excellent sensitivity, but the bandwidth may be < 1 Hz, which is clearly too slow for most applications.

Presented here is an alternative detection method which allows increased sensitivity through higher Q without placing any restriction on bandwidth or dynamic range. In this frequency modulation (FM) technique, the cantilever serves as the frequency-determining element of a constant amplitude oscillator. The frequency of the oscillator output is instantaneously modulated by variations in the force gradient acting on the cantilever. Dürig $et\ al$, have used a related approach to observe forces in tunneling experiments by measuring the frequency of thermal vibrations of a cantilever beam. ^{8,9} With FM detection, the S/N for a given bandwidth has the same dependence on Q as the conventional system;

however, the bandwidth is governed only by the characteristics of the FM demodulator, which can be tailored for different applications. The theory of operation of the FM and conventional methods are discussed below, and experimental results are presented which demonstrate the differences between the two techniques.

II. LIMITATIONS OF CONVENTIONAL DETECTION METHOD

The most commonly used detection method,²⁻⁷ which shall be referred to as "slope detection," involves driving the cantilever at a fixed frequency ω_d slightly off resonance (see Fig. 1). The resonant frequency of the cantilever is given by $\omega_0^2 = k_{\rm eff}/m$, where m is the effective mass of the lever. The effective spring constant is given by $k_{\rm eff} = k_L + \partial F/\partial z$,

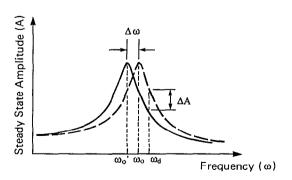


FIG. 1. In a slope detection system, the cantilever is driven at a fixed frequency ω_d slightly off resonance. A change in the force gradient causes the resonant frequency to shift from ω_0 to ω_0' , which results in a change ΔA in the steady-state amplitude.

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where k_L is the force constant of the lever and $\partial F/\partial z$ is the force gradient acting on the lever due to interaction with the sample. A change in $\partial F/\partial z$ gives rise to a shift in the resonant frequency $\Delta \omega$, and a corresponding shift ΔA in the amplitude of the cantilever vibration. The signal in slope detection is derived by measuring this change in amplitude.

Previous studies^{2,3,10} have shown that the minimum detectable force gradient is given by

$$\delta F'_{\min} = \sqrt{2k_L k_B TB / \omega_0 Q \langle z_{\rm osc}^2 \rangle},\tag{1}$$

where $\langle z_{\rm osc}^2 \rangle$ is the mean-square amplitude of the driven cantilever vibration, B is the measurement bandwidth, Q is the quality factor of the cantilever resonance, and k_BT is the thermal energy at the ambient temperature. Since it is possible to achieve very high-Q values (10^4 - 10^5) by careful design and reduction of air damping in vacuum ($< 10^{-3}$ Torr), it might appear advantageous to maximize sensitivity by obtaining the highest possible Q.

With slope detection, however, increasing the Q restricts the bandwidth of the system. The amplitude-versus-frequency curves shown in Fig. 1 are steady-state curves; only after a sufficient length of time will the vibration amplitude settle on a new steady-state value after a change in $\partial F/\partial z$ and ω_0 . The response of the system may be expressed in terms of a time constant $\tau=2Q/\omega_0$, which limits the available bandwidth.

The effect of this bandwidth limitation is illustrated in the following analysis of the theoretical response of the system to sudden changes in ω_0 . For example, consider the case of an instantaneous step in $\partial F/\partial z$ at time t=0 which results in an instantaneous shift in the resonant frequency from ω_0 to ω_0' . The cantilever is a driven damped harmonic oscillator whose equation of motion before the step is

$$m\ddot{z} + (m\omega_0/Q)\dot{z} + m\omega_0^2 z = F_0 \cos(\omega_d t), \qquad (2)$$

and the cantilever is in a steady state given by

$$z(t \leqslant 0) = A_0 \cos(\omega_d t + \vartheta_0), \tag{3}$$

where the values of A_0 and ϑ_0 are given by the well-known expressions

$$A_{0} = \frac{F_{0}/m}{\sqrt{(\omega_{0}^{2} - \omega_{d}^{2})^{2} + (\omega_{0}\omega_{d}/Q)^{2}}}$$
(4)

and

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$$\vartheta_0 = \tan^{-1} \left(\frac{\omega_0 \omega_d}{Q(\omega_0^2 - \omega_d^2)} \right). \tag{5}$$

After the change in $\partial F/\partial z$ occurs, the new solution contains a transient term and is given by

$$z(t\geqslant 0) = A'_0 \cos(\omega_d t + \vartheta'_0) + A_t e^{-\omega'_0 t/2Q} \cos(\omega_t t + \vartheta_t),$$
(6)

where A_0' and ϑ_0' are the new steady-state amplitude and phase, A_0 and ϑ_0 are adjusted to fit the boundary conditions, and ω_0 is the resonant frequency for free oscillations given by

$$\omega_t = \omega_0' \sqrt{1 - (1/4Q^2)}. \tag{7}$$

Since the signal is obtained by measuring the amplitude, it is instructive to rewrite Eq. (6) as

$$z(t \geqslant 0) = A(t) \cos[\omega_d t + \vartheta(t)], \tag{8}$$

where A(t) is the measured amplitude and is given by

$$A^{2}(t) = A_{0}^{\prime 2} + A_{t}^{2}e^{-\omega_{0}^{\prime}t/Q} + 2A_{0}^{\prime}A_{t}e^{-\omega_{0}^{\prime}t/2Q}$$

$$\times \cos[(\omega_{t} - \omega_{d})t + (\vartheta_{t} - \vartheta_{0}^{\prime})]. \tag{9}$$

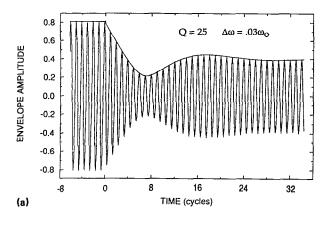
Here it becomes apparent that the measured amplitude has three components: the new steady-state term, a transient decay term, and a transient beat term.

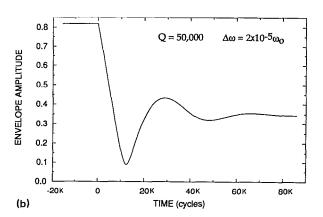
The behavior of the system is shown in Fig. 2. Low-Q values offer fast response, but low sensitivity, while high-Q values offer high sensitivity but slow response. For a high-Q cantilever in vacuum ($Q=50\,000$) and a typical resonant frequency of 50 kHz, the maximum available bandwidth is only 0.5 Hz, which is unusable for most applications. The dynamic range of the system is similarly restricted. Because of these restrictions, it is not useful to try to increase sensitivity by raising the Q to such high values. Moreover, if a vacuum environment is needed for other reasons (e.g., to prevent sample contamination), it may not be possible to obtain low enough Q for an acceptable bandwidth and dynamic range. Therefore, slope detection is unsuitable for most vacuum applications.

III. FM DETECTION

In the FM detection system, a high-Q cantilever vibrating on resonance serves as the frequency-determining component of an oscillator. Changes in the force gradient $\partial F/\partial z$ cause instantaneous changes in the oscillator frequency which are detected by a FM demodulator. The block diagram of the system shown in Fig. 3 reveals that the system is, in principal, no more complicated than a slope detection system. The cantilever is kept oscillating at its resonant frequency by applying positive feedback through the oscillator control amplifier. This amplifier is equipped with an automatic gain control (AGC) circuit which maintains the vibration amplitude at a constant level set by the user. The phase shift network is adjusted to insure maximum positive feedback on resonance. A bandpass filter is included to prevent oscillation on unwanted vibrational modes of the system, such as the resonance of the bimorph used to drive the cantilever.

A variety of methods may be used to measure the oscillator frequency, such as a digital frequency counter, gated timer, phase-locked loop, or various other analog FM demodulator circuits. We obtained best results with a tunable analog FM detector which measures the frequency-dependent phase shift in a dual *LC* filter as shown in Fig. 3. This demodulator has sufficient sensitivity to measure a frequency shift of 0.01 Hz at 50 kHz with 75-Hz bandwidth. The bandwidth may be adjusted as desired. A threshold circuit is used to provide a "pullback" signal if the cantilever vibration amplitude falls below some minimum value. In the case of a tip crash, this threshold circuit protects the system from ambiguous frequency measurements which may occur if the cantilever is not free to oscillate properly. The FM demodu-





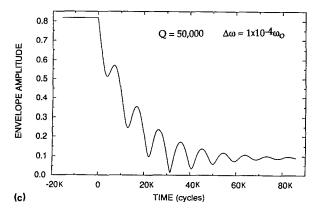
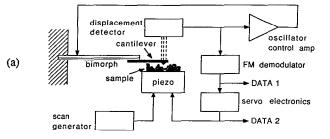
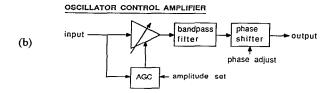


FIG. 2. Simulation of the amplitude response in slope detection for an instantaneous change in the resonant frequency at time t=0. For t<0, the cantilever is vibrating in a steady state. (a) For Q=25 (a relatively low value even in air), the amplitude reaches its new steady state value after approximately 25 cycles. (b) For $Q=50\,000$, a similar response can be measured for a much smaller resonant frequency shift, but the response time is increased to approximately 50 000 cycles. Because of the large number of cycles. only the envelope is shown. (c) With a larger frequency shift, strong transient beats are observed in the amplitude response.





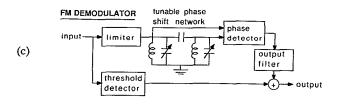


FIG. 3. Block diagram of the FM detection system. The complete system (a) consists of conventional force microscope components except for the oscillator control amplifier and FM demodulator which are shown in detail in (b) and (c).

lator is not sensitive to slow signal amplitude changes; thus changes in displacement sensor sensitivity, such as changes in laser power output in an interferometer, cause little disturbance to the system (unlike slope detection).

IV. NOISE AND SENSITIVITY CONSIDERATIONS

There are at least three sources of noise which limit the sensitivity of the FM detection system: (1) thermal vibrations of the cantilever, (2) noise in the displacement sensor, and (3) noise generated in the oscillation control amplifier and other electronics. With a low-noise displacement sensor, such as the all-fiber interferometer¹¹ used in these experiments, thermal vibrations of the cantilever are the dominant noise source under most conditions. Assuming no other noise sources, the minimum detectable force gradient due to thermal vibration of the cantilever is derived below.

According to the equipartition theorem, the thermal energy in the cantilever results in cantilever motion described by 12

$$\frac{1}{2}m\omega_0^2 \langle z_{\rm th}^2 \rangle = \frac{1}{2}k_B T,\tag{10}$$

where $\langle z_{\rm th}^2 \rangle$ is the mean-square displacement of the end of the cantilever due to thermal excitation. The spectral noise density $N_{\rm th}(\omega)$ and $\langle z_{\rm th}^2 \rangle$ are related by

$$\langle z_{\rm th}^2 \rangle = \frac{1}{2\pi} \int_0^\infty N_{\rm th}(\omega) d\omega,$$
 (11)

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and $N_{\rm th}(\omega)$ can be further described by

$$N_{\rm th}(\omega) = |G(\omega)|^2 \Psi_{\rm th}(\omega), \tag{12}$$

where $|G(\omega)|^2$ is the response function of the cantilever (damped harmonic oscillator) given by

$$|G(\omega)|^2 = \frac{1/m^2}{(\omega_0^2 - \omega^2)^2 + (\omega_0 \omega/Q)^2},$$
 (13)

and $\Psi_{th}(\omega)$ is the thermal white noise drive given by 12

$$\Psi_{th}(\omega) = 4m\omega_0 k_B T/Q. \tag{14}$$

Because the apparent strength of the thermal white-noise source decreases with increasing Q, high-Q cantilevers have less thermal noise off resonance, which has the effect of reducing the noise level in both slope and FM detection.

For a self-oscillating system with positive feedback, such as that used with FM detection, the spectral width of the oscillator output is decreased with increasing oscillator amplitude. This behavior can be described in terms of an apparent quality factor Q' in $G(\omega)$ which is larger than the actual Q by the ratio $^{12.13}$

$$\frac{Q'}{Q} = \frac{\langle z_{\rm osc}^2 \rangle}{\langle z_{\rm th}^2 \rangle},\tag{15}$$

where $\langle z_{\rm osc}^2 \rangle$ is the mean-square amplitude of the self-oscillating cantilever. The Q used in Eq. (14) for $\Psi_{\rm th}$ (ω) remains at the actual value. Under typical conditions, Q' falls in the range of 10^4-10^9 , resulting in an oscillator linewidth which is typically < 1 Hz. FM noise at a modulation frequency $\omega_{\rm mod}$ arises from thermal noise in sidebands of the oscillator at frequencies of $\omega_0 \pm \omega_{\rm mod}$. If we ignore noise components with a modulation frequency on the order of the oscillator linewidth and less, we may write an approximate expression for the spectral noise density $N_{\rm th}$ in each sideband in terms of the modulation frequency $\omega_{\rm mod}$ as follows:

$$N_{\rm th}(\omega_{\rm mod}) = k_B T / m \omega_0 Q \omega_{\rm mod}^2, \tag{16}$$

where $\omega_{\rm mod} = \omega_0 - \omega$ and the expression is valid for $\omega_{\rm mod} \gg \omega_0/2Q'$. The phase noise energy¹³ is given by $E_{\rho}(\omega_{\rm mod}) = k_L N_{\rm th}(\omega_{\rm mod})/2$ and the mean-square frequency modulation due to this noise source is given by 13

$$\langle (\delta\omega)^2 \rangle = \frac{1}{2\pi} \int_{\omega_{\text{mod}}} \frac{2E_{\rho}(\omega_{\text{mod}})}{E_c} \, \omega_{\text{mod}}^2 \, d\omega_{\text{mod}}, \quad (17)$$

where E_c is the oscillator energy given by $E_c = k_L \langle z_{\rm osc}^2 \rangle$. Integrating Eq. (17) over the bandwidth of measurement, we have

$$\langle (\delta\omega)^2 \rangle = \omega_0 k_B T B / k_L Q \langle z_{\rm osc}^2 \rangle, \tag{18}$$

where B is the bandwidth. The minimum detectable force gradient is then

$$\delta F'_{\min} = \frac{2k_L \delta \omega}{\omega_0} = \sqrt{\frac{4k_L k_B TB}{\omega_0 Q \langle z_{\rm osc}^2 \rangle}}, \qquad (19)$$

which is virtually identical to the expression for slope detection given in Eq. (1). Thus, with all parameters equal, slope detection and FM detection have similar sensitivity. However, in the case of slope detection (Sec. II), B and Q are not independent. With FM detection, the Q depends only on the damping of the cantilever, and B is set only by the character-

istics of the FM demodulator, which can be tailored for the desired application. Therefore, the FM detection method allows the sensitivity to be increased by using a very high Q without sacrificing bandwidth or dynamic range.

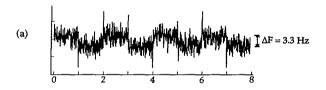
With very low damping $(Q > 10^4)$, noise in the displacement sensor can play a significant role in reducing system sensitivity. The spectral density of noise at the output of an interferometer may be approximately white in character (e.g., dominated by shot noise in a photodiode) which gives a spectral density of FM noise $N_{\rm FM}$ ($\omega_{\rm mod}$) [i.e., the integrand in Eq. (17)] which increases with frequency:

$$N_{\rm FM}(\omega_{\rm mod}) = (2E_{pd}/E_{\rm c})\omega_{\rm mod}^2, \tag{20}$$

where E_{pd} is the apparent white-phase noise energy due to the displacement sensor. Thus, the FM system is immune to slow changes in laser power in an interferometer-based displacement sensor, while rapid fluctuations can introduce considerable FM noise. The onset of domination by sensor noise can be recognized by the shape of the noise spectrum at the output of the FM demodulator. When sensor noise is the dominant noise source, the noise increases with increasing $\omega_{\rm mod}$. In our experiments, this onset occurred occasionally at very high-Q values ($>10^4$), depending on the noise performance of the interferometer.

V. EXPERIMENTAL RESULTS

The advantages of FM detection with high-Q cantilevers have been demonstrated in several ways experimentally. The displacement sensor used in these experiments was an





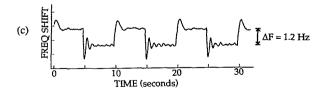


FIG. 4. Noise comparison of (a) slope detection in air with Q=115, (b) FM detection in vacuum with $Q=62\,000$, and (c) slope detection in vacuum with $Q=62\,000$. In (a) and (b), the force gradient was modulated with a 0.5-Hz square wave, and both have a 3-dB detection bandwidth of 60 Hz. In (c), a 0.1-Hz square-wave modulation was used, and the maximum available bandwidth is less than 1 Hz. Transient beats are visible in (c).

all-fiber interferometer¹¹ with a sensitivity of 10^{-3} – 10^{-4} $Å/\sqrt{Hz}$. The cantilevers used were single-crystal Si microcantilevers with integrated tips 14 with a variety of resonant frequencies (33-72 kHz) and force constants (1-20 N/m). Figure 4 shows a comparison of noise levels for three cases: (1) slope detection in air, (2) FM detection in vacuum, and (3) slope detection in vacuum. In all three cases, the force gradient $\partial F/\partial z$ acting on the cantilever was modulated by applying square wave and a dc bias to an electrode near the cantilever. The square wave generated a varying electric field and associated force gradient which caused the shift in resonance indicated next to each trace in Fig. 4. With identical bandwidth, the trace taken with FM detection shows an improvement in S/N of at least 10 times compared to slope detection in air, which is due to the increased Q of the cantilever in vacuum. If slope detection is used with the high Q obtained in vacuum, the sensitivity is excellent, but the maximum available bandwidth is < 1 Hz.

Figures 5 and 6 show that the amount of FM noise (measured at the output of the FM demodulator) behaves as predicted by Eq. (19). At very high Q, a small amount of excess noise due to the interferometer noise is observed in the particular measurements shown in Fig. 5. In other measurements, where the interferometer noise level was lower, thermal noise in the cantilever was the dominant noise source up to $Q > 50\,000$.

Figure 7 shows a comparison of two magnetic force microscopy images of the same sample, the first taken by slope detection in air, and the second by FM detection in vacuum. Both images were taken using the same microcantilever, which was coated with a thin film of magnetic material for magnetic sensing. ¹⁵ Both images are 256×256 pixels. Even though the FM image was acquired approximately 10 times more quickly than the image taken by slope detection (~ 30 s vs 5 min), the FM image shows considerably less noise. The sample is a thin-film CoPtCr magnetic recording disk designed for very-high-density magnetic recording. Because

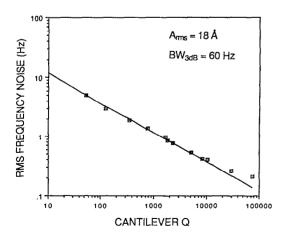


FIG. 5. RMS noise at the output of the FM demodulator decreases with increasing cantilever Q. The line shows the theoretical $Q^{-1/2}$ behavior given in Eq. (19), and the points shown are measured data.

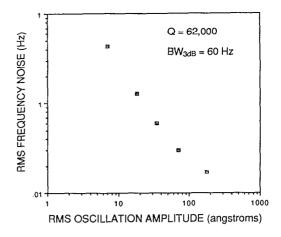


FIG. 6. RMS noise at the output of the FM demodulator decreases with increasing oscillation amplitude. The behavior is close to the expected behavior given in Eq. (19).

the magnetic films on both the disk and cantilever tip are unusually thin, the magnetic interaction is much weaker than for most magnetic force microscope images of magnetic disks. While the slope detection image shows a high noise level, no appreciable noise is visible in the FM image. The apparent noise visible outside the data tracks is actual magnetic information associated with randomly oriented magnetic domains of the unwritten (demagnetized) magnetic media. Within the data track, where the magnetic signal has been defined by the write head, the magnetic signal is smooth, and no appreciable noise is visible.

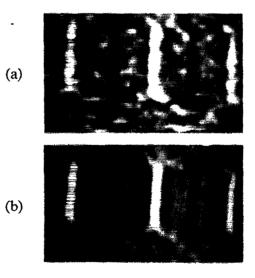


FIG. 7. Comparison of images taken on the same sample by (a) slope detection in air with Q=60 and (b) FM detection in vacuum (3×10 4 Torr) with $Q=40\,000$. Image (b) was taken at a rate 10 times faster than image (a). The magnetic transitions shown in the images are 2 μ m apart.

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Experience in using the FM system has shown that the FM system is simpler to calibrate and is disturbed less by drifts than a system based on slope detection. The FM demodulator has a linear output over a fairly large range, and the calibration of the demodulator (output voltage versus input frequency) remains constant. With slope detection, drifts in the system can cause the operating point to move along the slope of the response curve, which changes the sensitivity of the system. Thus, quantitative force gradient measurements are easier with the FM system.

VI. CONCLUSIONS

The FM detection system offers an alternative to conventional slope detection which allows increased sensitivity through increased cantilever Q. Operating in vacuum with Q values $> 10^4$, the sensitivity for a fixed bandwidth can be improved by more than 10 times compared to slope detection in air, where it is often difficult to obtain Q values much greater than 100. By taking advantage of this increased sensitivity, the FM technique allows the measurement of much weaker force gradients, opening new applications of ac force microscopy.

ACKNOWLEDGMENTS

The authors wish to thank G. McClelland and U. Dürig for helpful discussions and correspondence. The cantilevers used in these experiments were supplied by J. Greschner, O. Wolter, T. Bayer, and co-workers at the IBM German Manufacturing Technology Center, Sindelfingen, FRG.

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