

A magnetically driven oscillating probe microscope for operation in liquids

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Oscillating probe atomic force microscopy in fluids is simplified when an oscillating force is applied directly to a magnetized cantilever using a solenoid. The response of the detector is simpler than that obtained with acoustic excitation. Reliable operation over a broad frequency range is achieved with excitation amplitudes of a few nm. This lower amplitude appears to facilitate imaging by means of small asperities on the tip. Images of a DNA plasmid bound weakly to mica in water have a full width of 5 nm when scanned with tips of a nominal macroscopic radius of curvature of 50 nm.
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In tapping-mode atomic force microscopy,¹ a tip contacts a surface intermittently, offering the advantages of ac detection² and resulting in less shear distortion of weak samples (see, e.g., Leuba *et al.*).³ In ambient air, stiff cantilevers operated at large amplitudes are used in order to overcome adhesion.¹ In water, tapping mode^{4,5} gives much improved resolution over contact mode.⁴ Large amplitude oscillation is not required in this case because adhesion is greatly diminished.⁶ Nonetheless, a large acoustic driving amplitude must be used in order to overcome damping and inefficient acoustic coupling into the cantilever. For example, Chen *et al.* used an excitation of 300 nm amplitude.⁷

One characteristic of acoustically excited tapping mode is that not all of the signal reaching the detector is sensitive to the approach to the surface.⁵ This occurs, in part, because not all modes of the cantilever result in displacement of the tip. However, in our experience, a major contribution arises from mechanical excitations of the microscope that disturb the optical path of the detection system. Excitation at low amplitude (by thermal fluctuations, for example) removes many of these "resonances." This observation suggests that a substantial improvement will result if a driving force is applied to only the cantilever tip. Florin *et al.*⁸ observed a much simpler response when a cantilever was driven directly in ambient air.

Lindsay *et al.*,⁹ Florin *et al.*,⁸ and Cleveland¹⁰ have used a magnetic particle attached to an atomic force microscope cantilever in order to apply a force to the cantilever using a field generated by a solenoid. The most sensitive arrangement¹¹ is obtained by placing the magnetic field, \mathbf{B} perpendicular to the moment, \mathbf{M} on the cantilever, resulting in a torque, $\mathbf{N}=\mathbf{M}\times\mathbf{B}$, or a force on the end of the cantilever of magnitude N/L where L is the length of the cantilever. O'Shea *et al.* used this geometry to drive tips coated with a thin magnetic film, finding exquisite sensitivity to surface forces.¹¹

In this letter, we describe a new imaging mode for atomic force microscopy in fluids based on direct magnetic excitation of coated cantilevers. The instrument is similar to that used by Florin *et al.*⁸ to measure the local compliance of

materials in ambient air, differing mainly in the manner in which the cantilever is magnetized and driven. The enhanced sensitivity of this arrangement to changes in surface compliance⁸ is undoubtedly beneficial, but our goal is to avoid the spurious resonances associated with acoustic excitation in a liquid medium. The sensitivity to surface forces is shown to be substantially independent of driving frequency and a lack of a background signal permits the use of lower amplitudes of oscillation which, when combined with softer cantilevers, results in a substantial diminution of the energy deposited into the sample by the tip. This often leads to higher resolution, because fine asperities on the tip appear to be preserved during imaging.

We used a PicoSPM from molecular imaging (Tempe, AZ) in which a 1 mH solenoid was placed under the sample platen. A driving signal of 20 mA rms produced an rms field of 2×10^{-4} T at a distance of up to about 1 mm from the pole piece of the solenoid. The field increased by a small (<10%) amount as the frequency was increased up to the 20 kHz limit of the Gaussmeter we used. The microscope detection circuitry was modified to produce a 3 dB bandwidth of 200 kHz and the signal from the detector was diverted to a lock-in amplifier (Stanford Research Systems model SR820), the output of which was used to feed a standard SPM controller (NanoScope III from Digital Instruments, Santa Barbara, CA). Silicon nitride cantilevers (MicroLevers from Park Scientific, Sunnyvale, CA) were coated with a proprietary magnetic material and magnetized along the flexible axis of the cantilever by Molecular Imaging Corporation. We estimate that the magnetic moment in the plane of the cantilevers was about 5×10^{-10} A-m² based on the properties of macroscopic films deposited on test samples. We used the 140 and 85 μm cantilevers for which the free resonance frequencies were around 30 and 100 kHz. After coating, these resonant frequencies dropped less than 5%. Their spring constants increased slightly (less than 20%) as determined by measuring their deflection when pressed against calibrated glass fibers.

Measurement of the thermal response of similar cantilevers in water shows that it is well fitted as a single driven-damped harmonic oscillator¹² with the resonance frequency substantially lowered owing to mass loading of the cantilever

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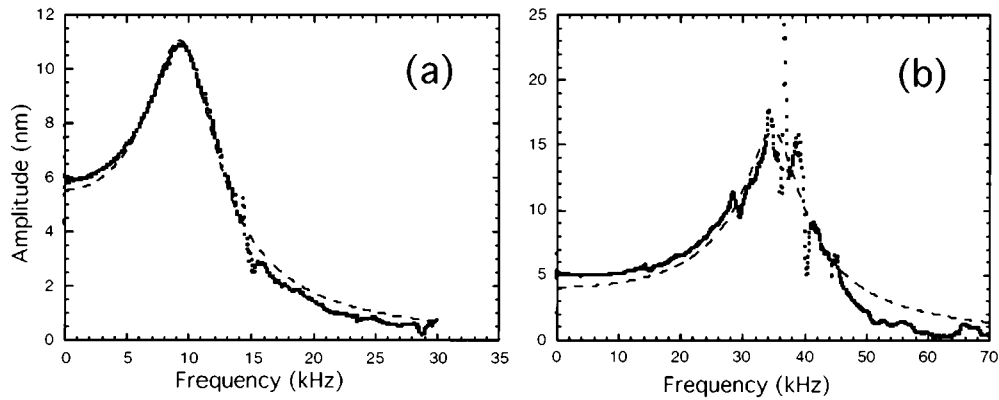


FIG. 1. Detected deflection of magnetically driven cantilevers of 140 (a) and 85 μm length (b) in water. The dashed line is a fit to a driven-damped harmonic oscillator model. Sharp features in the vicinity of 35–40 kHz have been traced to resonances of the cantilever substrate.

by the adjacent fluid.^{12,13} However, plots of the detector signal as a function of frequency are rather complicated when the cantilever is excited acoustically.^{5,8} This is not the case when the cantilevers are excited directly. Figure 1 shows typical plots of the detector output as a function of frequency for the 140 (a) and 85 μm (b) cantilevers operated in distilled water at a low-frequency oscillation amplitude of about 5 nm.

In air, we found that no signals were generated when nonmagnetic cantilevers were used up to the maximum drive signal of 160 mA. In water, spurious signals were detected for drives bigger than 40 mA, presumably because low-level acoustic signals owing to magnetic materials in the microscope were coupled efficiently by the liquid medium. Even at lower drive levels, magnetic cantilevers exhibit some spurious signals in the region of 35–40 kHz, and these have been traced to normal modes of the substrate onto which the cantilevers are fixed (it is also coated with a magnetic film). Aside from these modes, the amplitude of the cantilevers is well described by a single driven, damped harmonic oscillator, consistent with the results of Butt *et al.*¹² The resonant frequencies are reduced to 10 (140 μm cantilever) and 35 kHz (85 μm cantilever) and the damping constants are 5200

(140 μm cantilever) and 9200 s^{-1} (85 μm cantilever). These results are similar to those reported by Butt *et al.*¹² and Chen *et al.*¹³ The low frequency amplitudes are consistent with the driving-model outlined above, for which, with \mathbf{M} perpendicular to \mathbf{B} , the deflection, δ , of the cantilever is given by $\delta = MB/kL$, where k is the spring constant of the cantilever. The softer cantilever was driven at 20 mA corresponding to a 2×10^{-4} T field, yielding a predicted displacement of 6 nm, in good agreement with the data shown in Fig. 1(a).

One consequence of this simpler response is that the approach curves are almost insensitive to the operating frequency of the microscope as illustrated in Fig. 2. Figure 2(a) shows the change of amplitude as the tip is swept back and forth from a clean mica surface in distilled water. The sensitivity is characterized by the steepest slope midway through the approach, $(dZ/ds)_{1/2}$, and values for this quantity are plotted for the stiffer cantilever as a function of frequency in Fig. 2(b).

We have been able to obtain images with an amplitude, Z , as small as 1 nm and with a set point corresponding to a change in amplitude, ΔZ of 0.1 nm. Reliability is enhanced at higher amplitudes such as $Z=5$ nm and $\Delta Z=0.3$ nm, corre-

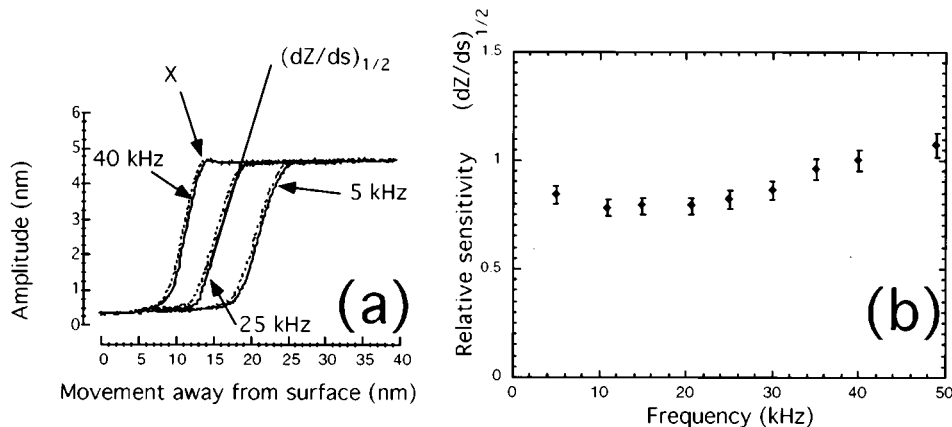


FIG. 2. (a) Approach curves for the 85 μm cantilevers and mica in distilled water at three frequencies showing how the amplitude decays as the surface is approached (dashed line) and is restored (solid line) as the tip is moved away again. Curves have been shifted laterally for clarity. The “X” points to a region where amplitude is increased owing to a change in the sign of force gradient. The sensitivity midway through the approach is plotted over a range of frequency in (b) (data averaged over 10 measurements).

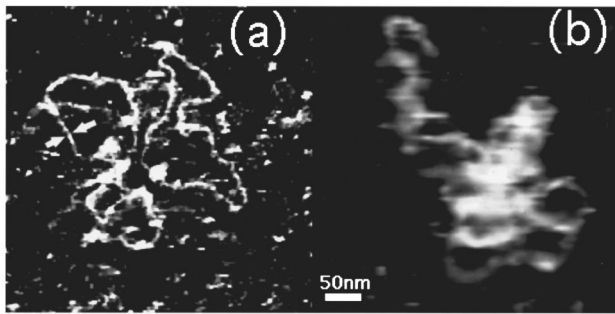


FIG. 3. (a) Images of two pMB9 (5300 basepairs) plasmids spontaneously adsorbed onto mica from a solution of $1 \mu\text{g/ml}$ in 20 mM MgCl_2 . The scan rate was 1.5 Hz/line with a 8 kHz oscillation amplitude of 5 nm and a set-point decrease of 0.3 nm , using a cantilever with $k=0.12 \text{ N/m}$. The full width averaged over the images is $5.3 \pm 0.15 \text{ nm}$ (arrows give an example of one measurement). An image taken in contact mode with the same tip (same scale) is shown in (b). Note that many loosely bound contamination particles are not seen in the contact-mode image.

sponding to an energy, E , deposited into the sample on each cycle of $1.7 \times 10^{-19} \text{ J}$ (with $k=0.12 \text{ N/m}$). By way of comparison, acoustically excited tapping⁵ with $Z=30 \text{ nm}$ and $\Delta Z=10 \text{ nm}$ with $k=0.38 \text{ N/m}$ gives $E \approx 10^{-16} \text{ J}$.

We have imaged the DNA plasmid pMB9 (Sigma Chemical Co.) spontaneously bound to mica in the presence of a solution containing Mg(II) . A typical image is shown in Fig. 3(a). For comparison, an image obtained with the same tip in contact mode is shown in Fig. 3(b). Hansma and Laney¹⁴ report that DNA bound to mica in the presence of Mg(II) cannot be imaged by fluid tapping mode without prior drying of the sample, so this present result suggests that the magnetically driven tip can interact with samples more gently. We have routinely obtained a full width of the DNA image of 5 nm (or better; Wenhai Han, M. Dlakic, R. E. Harrington, and S. M. Lindsay, unpublished data), similar to that reported by Hansma *et al.*⁴ using an e-beam deposited carbon tip. The nominal radius of curvature of the tips used in this work is 50 nm , so such high resolution is unexpected. However, we notice multiple images (double or triple) in the

majority of cases, and this suggests that asperities on the tip dominate the contrast. This is consistent with a tip model recently proposed by Putman *et al.*¹⁵ When the oscillation amplitude is increased or the microscope brought into contact, the width of the resulting images corresponds more closely to what is expected from the macroscopic radius of curvature of the tip, as illustrated in Fig. 3(b).

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