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## Direct torsional actuation of microcantilevers using magnetic excitation

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Torsional mode dynamic force microscopy can be used for a wide range of studies including mapping lateral contact stiffness, torsional frequency or amplitude modulation imaging, and dynamic friction measurements of various materials. Piezo-actuation of the cantilever is commonly used, but it introduces spurious resonances, limiting the frequency range that can be sampled, and rendering the technique particularly difficult to apply in liquid medium where the cantilever oscillations are significantly damped. Here, we demonstrate a method that enables direct torsional actuation of cantilevers with high uniformity over wide frequency ranges by attaching a micrometerscale magnetic bead on the back side of the cantilever. We show that when beads are magnetized along the width of the cantilever, efficient torsional actuation of the cantilevers can be achieved using a magnetic field produced from a solenoid placed underneath the sample. We demonstrate the capability of this technique by imaging atomic steps on graphite surfaces in tapping mode near the first torsional resonance of the cantilever in dodecane. The technique is also applied to map the variations in the lateral contact stiffness on the surface of graphite and polydiacetylene monolayers. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4894737]

Atomic force microscopy (AFM) has become an essential tool in a large variety of scientific research ranging from tribology<sup>1</sup> to biology<sup>2</sup> and even in geosciences<sup>3,4</sup> and food science,<sup>5</sup> providing unprecedented details of the structural and physical properties of materials at the nanometer or even atomic scale. The unique ability of this technique to work in liquid environment has enabled studies of soft surfaces such as polymers<sup>6</sup> and physisorbed molecular assemblies (e.g., DNA and membrane proteins).<sup>7-10</sup> Soft and weakly bound molecules are often measured using dynamic modes in order to minimize shear forces between the tip and the sample which can severely damage the sample.<sup>11</sup> However, the common method of oscillating the cantilever with a piezo transducer typically introduces a forest of spurious peaks which masks the cantilever resonance and introduces artifacts which complicates data interpretation.<sup>12-14</sup> It also severely limits the frequency range that can be sampled; this hinders the study of soft and biological materials, who have viscoelastic responses which require measurements to be conducted over many decades of frequency.<sup>15</sup> Various methods have been introduced to address these issues by directly driving the cantilever perpendicular to the sample surface, near its first normal resonance using Lorentz force actuation (idrive<sup>TM</sup>, Asylum Research, CA, USA),<sup>16,17</sup> magnetic alter-nating current (MAC) mode,<sup>12–14,18–23</sup> electrostatic,<sup>24</sup> and photo-thermal actuation,<sup>25</sup> which can achieve a highly uniform cantilever response vs. frequency to enable true molecular or even atomic resolution imaging capabilities under liquid environments. In another modification of this technique, the cantilever is actuated in lateral direction where the tip oscillates nearly parallel to the sample surface.<sup>26</sup> The cantilever amplitude in this mode of oscillation is not sensitive to long-range forces which act normal to the sample surface,<sup>27</sup> thus the change in cantilever amplitude and phase arises from short-range interactions and in addition, oscillation of the tip parallel to the sample surface enables studies of in-plane (i.e., shear) properties of material surfaces.<sup>28–38</sup> The latter method still remains challenging to implement in liquids as most of the proposed strategies employ piezo actuation at the base of the cantilevers or requires sophisticated instrumentation (e.g., for photothermal method) which is difficult to implement on commercial AFMs. Photothermal actuation method further suffers from its inability to achieve large amplitudes for stiff cantilevers and high laser power can locally heat the cantilever and sample.<sup>39</sup> In order to address these issues, Yang *et al.*, recently proposed an alternative method of direct torsional actuation using the Lorentz force technique.<sup>40</sup> In this method, the torsional twist in the AC current carrying cantilevers is produced by aligning the external magnetic field along the width of the cantilever. However, this technique involves timeconsuming and expensive microfabrication process, such as deposition of gold electrodes on the tip-side of the cantilever for current flow. Furthermore, tip side coating is undesirable for high resolution experiments where sharpness of the tip is critical and the exposed gold electrodes might cause electrochemical reactions in electrolytes or ionized buffers, leading to corrosion or delamination of the gold coating under high current flow.<sup>16,40</sup>

Here, we report a method for direct actuation of the cantilever in torsional mode, where a spherical magnetic bead is attached to the back of the cantilever and is actuated by a solenoid placed underneath the sample plate. The technique is capable of imaging at resonance (torsional tapping mode) as well as at off-resonance frequencies (lateral force

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modulation mode), which allows mapping of shear elastic and shear viscoelastic properties of surfaces in air as well as in liquid environments. We demonstrate the capability of this technique to study in-plane surface properties by obtaining high resolution lateral amplitude and phase contrast images of a graphite surface in resonance as well as in offresonance modes and by mapping lateral contact stiffness variations in molecular domains of polydiacetylene (PDA) monolayers.

All the experiments were performed using a commercial AFM (PicoPlus 5500, Agilent Technologies, Santa Clara CA, USA), equipped with a solenoid mounted underneath the sample plate to produce AC magnetic field. This AFM also allows the possibility to magnetically actuate the cantilever using a solenoid which is placed in the cantilever holder. This provides extra flexibility required for experiments where different type of sample plates are needed, e.g., for variable temperature experiments or for stand-alone applications where sample plates can not be used at all. This method requires the use of one of these two types of solenoids to magnetically actuate the cantilever. While this may not be feasible for all AFM systems, a number of commercial and custom AFM systems are compatible with or even include options for such solenoids. Silicon cantilevers (PPP-LFMR, Nanosensors, Neuchatel, Switzerland) were used in all the experiments. Ferromagnetic beads, made of a rare earth-iron-boron intermetallic compound (MQP-S-11-9, Magnequench, Singapore) were glued on the back side of the cantilevers under an inverted optical microscope (Alessi REL-4100A, Redwood City, CA, USA) by first applying a small quantity of epoxy (two-part epoxy, J-B Weld, Sulphur Springs, TX, USA) using a sharp tungsten wire (TGW0325, World Precision Instruments, Sarasota, FL, USA) and then placing the bead using a strand of hair.<sup>14</sup> This process takes approximately 10 min per cantilever. The cantilevers were stored overnight in a dry N2 purged dry box to allow the epoxy to fully cure. Later, the cantilevers were magnetized using a superconducting magnet (Physical Property Measurement System, Quantum Design, USA) under a magnetic field of 3 T such that the direction of the magnetic field was perpendicular to the long axis of the cantilevers and parallel to the cantilever surface. Samples were inserted and removed along the solenoid axis to ensure that magnetic reorientation did not occur during sample removal. This is a crucial step because lateral stiffnesses in torsion of these AFM cantilevers are typically one order of magnitude higher than normal stiffnesses. Thus, large bending deflections will occur with small misalignments of the bead's magnetic moment. This requires that the bead's magnetic axis be extremely well-aligned to achieve maximum selectivity of torsional bending. The diameters of the magnetic beads attached to the cantilevers were estimated using either bright field optical microscopy (Olympus BX51) or using field emission scanning electron microscopy (SEM, JOEL 7500F) as shown in Fig. 1(a). The thermal noise spectrum of the cantilevers was acquired before and after gluing the beads using a 16-bit high speed analog to digital converter (NI USB-6259 BNC, National Instruments Corp., USA). The resonance peaks were fit as described in detail in Ref. 41 to obtain resonance frequencies and quality factors (Q) to



FIG. 1. (a) SEM image of a cantilever with a 25  $\mu$ m diameter ferromagnetic bead attached to the back of the cantilever. (b) Schematic showing the experimental setup where the AC magnetic field (Bz) produced using a solenoid, generates a torque ( $\tau$ ) in the magnetic bead (magnetized along the width of the cantilever) which causes torsional deflection of the cantilever. The beads are typically 15–30  $\mu$ m in diameter.

calculate normal and torsional spring constants using the Sader method.<sup>41</sup> The cantilever deflection sensitivities along the normal direction were obtained by taking force curves on a clean silicon wafer or glass substrates, whereas lateral force sensitivities were obtained using the wedge calibration method.42,43 Cantilevers were magnetically actuated using the solenoid by sweeping the frequency of the AC drive signal (Fig. 1(b)). The normal or lateral cantilever amplitude and phase signals were recorded using a digital lock-in amplifier supplied with the AFM controller. Highly oriented pyrolytic graphite (HOPG) samples (SPI supplies, USA) were freshly cleaved using a scotch tape and were mounted in a fluid cell. The sample and the cantilevers were fully immersed in the pure dodecane (>99%, Sigma-Aldrich) solution during the measurements. PDA monolayers, deposited using Langmuir-Blodgett (LB) method on silicon and atomically smooth mica surfaces,<sup>44</sup> were also used to further demonstrate imaging capabilities of our technique in laboratory air.

We first discuss the influence of magnetic bead attachment on cantilever properties, namely, shifts in normal and torsional resonance frequencies and corresponding change in Q. The power spectral density of the thermal noise for a representative cantilever is shown in Fig. 2 where normal and torsional resonance frequencies decrease and corresponding Q increase as a



FIG. 2. Thermal noise spectra acquired in air of (a) normal and (b) lateral cantilever deflection before and after attachment of a  $21 \,\mu\text{m}$  diameter magnetic bead.

result of attaching a magnetic bead ( $\sim 21 \,\mu m$  diameter) to the cantilever. The data acquired in air for 10 different cantilevers using beads of diameters ranging from 18 to  $28 \,\mu m$  revealed that for the first normal mode, the resonance frequency  $(f_{0,N})$ decreased by 50%–70% and quality factors ( $Q_{0,N}$ ) increased by 200%–450%. For the first torsional mode, the resonance frequency  $(f_{0,\tau})$  decreased by 40%–70% and quality factors  $(Q_{0,\tau})$  increased by 60%–130% as a result of attaching the magnetic beads. The shifts obtained for normal mode oscillations are primarily due to the added mass and are consistent with a recent study by Hoof et al.<sup>13</sup> We note that the shifts in the torsional mode of the cantilevers show trends similar to those observed for the normal mode. However, a comparatively smaller increase in Q is observed for the torsional mode. This may be due to the difference in the hydrodynamic functions for the normal and torsional oscillation modes,<sup>41</sup> which are not known for the cantilevers with attached beads used in our experiments. One specific explanation for this effect could be that the finite width of the cantilever partially masks the spherical bead during normal cantilever oscillations, so hydrodynamic drag is not strongly affected. However, there is no such shielding for the torsional mode, so a larger contribution from hydrodynamic drag occurs. Thus, while Q is higher overall after adding the bead, the increase is not as significant for the torsional mode.

Figure 1(b) shows a schematic of the experimental setup where AC current (i) through the solenoid generates a fluctuating magnetic field. The magnetic bead attached to the cantilever is subjected to a torque  $(\tau) = m \times B_z$  (where *m* is the magnetic moment of the magnetic bead and  $B_z$  is the component of magnetic field produced by the solenoid along zaxis), such that the magnetic moment of the bead tends to align with the magnetic field produced by the solenoid resulting in actuated torsional bending of the cantilever. Varying the magnitude of frequency and the drive current through the solenoid, the frequency and amplitude of the torsional deflection of the cantilever can be controlled. Figure 3(a) shows the normal and lateral forces acting on the cantilever at a constant drive signal as a function of drive frequency. The torsional force acting on the cantilever dominates over the



FIG. 3. (a) Calibrated normal and torsional forces acting on a magnetically actuated cantilever as a function of drive frequency (measured in air). (b) Frequency response of lateral deflection and phase of the same cantilever when immersed in dodecane.

normal force by almost a factor of  $\sim 100$  over the entire sweep frequency range, and no spurious peaks are observed. The small cross-talk observed between the normal and lateral signals could be due to various factors such as a slight angular misalignment of the photodetector with respect to the cantilever chip, resulting in the photodiode axes being rotated with respect to the torsional and flexural deflection axes of the cantilever. This could also arise from a slight misalignment of the solenoid with respect to the direction perpendicular to the plane of the cantilever, which can give rise to an additional magnetic force acting normal to the plane of cantilever and is proportional to  $(\vec{m} \cdot \nabla)B_z$ . However, despite these factors, most of which may be further improved but may not be completely eliminated, and which will likely vary from one experiment to the next, similar amplitude vs. frequency response as in Fig. 3(a) was observed for all the measured cantilevers. Figure 3(b) shows the torsional amplitude and the phase response of the cantilever as a function of drive frequency when immersed in dodecane where no spurious peaks are observed.

Figures 4(a)-4(f) demonstrate the ability of the cantilever torsional actuation methods to perform stable and high resolution imaging of HOPG surface under liquid. Figures 4(a)-4(c) show tapping mode images obtained on a freshly cleaved HOPG surface immersed in dodecane near the first torsional resonance of the cantilever. An oscillation frequency of 73 kHz and a root mean squared (rms) oscillation amplitude of  $9.0 \pm 0.2$  nm were used during imaging. Monoand multi-atomic steps of the HOPG surface are resolved clearly in the topography image (Fig. 4(a)). Figure 4(b)shows sub-Angström level variation of the oscillation amplitude (feedback) signal during imaging, indicating high imaging stability of this method in liquids. In addition, strong phase contrast (Fig. 4(c)) is observed near the step edges of HOPG, which is consistent with the results obtained from the piezo-actuated torsional tapping mode imaging conducted by Huang and Su.<sup>30</sup> The phase contrast near step edges can be attributed to higher friction/energy-dissipation,<sup>45</sup> clearly demonstrating that in-plane properties can be mapped with  $20 \pm 5 \,\mathrm{nm}$  resolution using this method. Furthermore, the ability of our method to achieve mapping of lateral contact stiffness at off-resonance frequencies is demonstrated in Figs. 4(d)-4(f), where the HOPG surface was imaged in dodecane in contact mode with a 18 kHz modulation drive signal and rms lateral cantilever amplitude of  $0.48 \pm 0.01$  nm. The topography, amplitude, and phase signals shown in Figs. 4(d)-4(f), respectively, clearly reveal single- and multiple-atomic steps (atomic step height of  $0.35 \pm 0.05$  nm, which agrees well with the theoretical value) on the HOPG surface. The presence of dark and bright regions observed in amplitude and phase signals at the topographically flat regions of the HOPG, can be related to bonding of the top layer to the surface underneath, which can influence the mechanical properties of the topmost layer, particularly shear elastic and viscoelastic properties.<sup>46</sup> Lateral amplitude variations are resolved down to picometers (Fig. 4(e)), clearly indicating the ability of this method to map shear properties and its variations with very high sensitivity. The lateral amplitude signal can be converted to lateral contact stiffness maps to quantitatively estimate elastic

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FIG. 4. Topography (left), amplitude (middle), and phase (right) images of (a)–(c)  $2 \times 2 \mu m^2$  HOPG surface, acquired in dodecane in tapping mode near the first torsional resonance of the cantilever (~73 kHz), (d)–(f)  $2 \times 2 \mu m^2$  HOPG surface, acquired in dodecane in lateral force modulation mode (modulation frequency of 18 kHz and amplitude of ~0.5 nm), (g)–(i)  $15 \times 15 \mu m^2$  PDA surface acquired in lateral force modulation mode in air (modulation frequency of 11.15 kHz and amplitude of ~0.4 nm).

shear modulus of the film. However, this requires a very careful calibration of the lateral amplitude signal against a material with well-known shear properties<sup>20</sup> and an appropriate estimate of the lateral stiffness of the tip apex. Such detailed studies are beyond the scope of the present work and will be published elsewhere. To further demonstrate the ability of this method for mapping shear properties of thin films, images of PDA surfaces were obtained in air using off-resonance lateral force modulation mode at a drive frequency of 11.15 kHz and rms lateral amplitude of  $0.35 \pm 0.01$  nm. The topographic image of PDA (in Fig. 4(g)) is smooth and the different molecular domain boundaries are barely visible. The bright features which are localized near the domain boundaries, are contaminants. However, the amplitude (stiffness) and phase images show a clear contrast between different domains within the film, which is linked to the well-known anisotropy in shear properties (e.g., friction) of this system.<sup>44</sup> The lateral amplitude maps clearly resolve the domain boundaries themselves, as well as stripe-like features within the domains indicating the orientation of polymer backbones (the direction is specified by the blue arrows). Thus, the strong influences of these inplane features are readily resolved.

In conclusion, we demonstrate a simple method to directly drive the AFM cantilevers in torsional resonance as well as off-resonance modes in both air and liquid environments. The method is based on actuating the magnetic bead attached to the cantilevers using an AC magnetic field produced by a solenoid place underneath the sample. This method allows the actuation of the cantilevers over a wide range of frequencies, and all spurious peaks are eliminated even in liquid media. Torsional mode tapping and lateral force modulation results obtained on graphite surfaces in dodecane, and PDA surfaces in air, clearly demonstrate the versatility of this technique to map shear properties (lateral contact stiffness and dissipation) in liquid and ambient conditions, with a lateral spatial resolution of no more than  $20 \pm 5$  nm. This can be further improved significantly by working with sharper tips and smaller oscillation amplitudes.

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