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Dynamics of surface-coupled microcantilevers in force modulation atomic force microscopy – magnetic vs. dither piezo excitation

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Force modulation atomic force microscopy is widely used for mapping the nanoscale mechanical properties of heterogeneous or composite materials using low frequency excitation of a microcantilever scanning the surface. Here we show that the excitation mode – magnetic or dither piezo, has a major influence on the surface-coupled microcantilever dynamics. Not only is the observed material property contrast inverted between these excitation modes but also the frequency response of the surface-coupled cantilever in the magnetic mode is near-ideal with a clear resonance peak and little phase distortion thus enabling quantitative mapping of the local mechanical properties.
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I. INTRODUCTION

Force modulation atomic force microscopy (AFM) is widely used for mapping the local, nanoscale elastic properties of polymers, rubber, composites, and biological samples by superimposing a low fixed frequency excitation on the cantilever in a constant force, contact mode scan.[1,2] Variations in cantilever amplitude are rendered as a map of local elastic stiffness. Unlike the tapping mode, or other dynamic, intermittent contact modes,[3] the stiffness information derived in force modulation AFM is quantitative. Moreover, in comparison to the so-called force volume mode[4] the material property maps using force modulation AFM are high resolution and taken at relatively high speeds. Of course these advantages come at the expense of the fact that in force modulation AFM applies a continuous lateral force on the sample which makes the technique inappropriate for weakly bonded samples. On the other hand, force modulation AFM has emerged as the technique of choice for mapping the local elasticity of rubbers, polymers, and especially composite materials.[5]

The majority of force modulation AFM is performed using vibrations of a piezoelectric pad embedded inside the cantilever chip holder which leads to the dithering oscillation of the base of the cantilever or the sample. We refer to this as "indirect" excitation since the oscillating strain is created at the piezoelectric pad and propagates via other mechanical structures (piezo pad, chip holder, chip, etc.) to the base of the cantilever or sample. More recently, a case has been made for using *direct excitation force modulation AFM* where the excitation force is applied directly to the cantilever alone without exciting other mechanical structures say by magnetic excitation or by means of a deposited piezoelectric material on the cantilever.[6] However very little is understood or known about the differences between the dynamic responses of the cantilever coupled to a surface when excited using a dither piezo or when using direct magnetic excitation. These differences, if significant, could lead to a major reassessment of the optimal operating conditions and interpretation of data in force modulation AFM in its many applications in high resolution surface property characterization.

In this work, we investigate the fundamental differences in the cantilever dynamics and resulting material property maps using magnetic excitation (or any form of direct cantilever excitation) and use indirect dither piezo excitation (conventional excitation). While dither piezo excitation is commonly used in two forms, cantilever base excited or sample excited, we will mostly focus on the cantilever base excited version and highlight, whenever they occur, specific differences between the cantilever and sample excited versions. Specifically we find that (i) magnetic excitation enables a clean determination of the resonance frequency of the surface coupled cantilever, while a "forest of peaks" makes this very difficult in the dither piezo excited case, (ii) the observable in the dither piezo base excitation scheme is different from the others leading to an unusual inversion of the material property maps between the three methods, and (iii) unlike dither piezo excitation, the gain of the surface coupled cantilever using magnetic excitation is nearly constant over a large frequency bandwidth allowing the phase in magnetically excited force modulation AFM to be used to accurately measure the local material properties including viscoelasticity of the sample. All these issues are demonstrated experimentally using magnetically and dither piezo excited cantilevers on a carbon fiber/epoxy composite system.

II. EXPERIMENTAL SETTING

We choose as a sample a carbon fiber/epoxy composite system which can be considered a model system for studying force modulation AFM since the carbon fiber is a stiff surface on which calibration can be performed within an image. The specimen was prepared from a 0.25 in. thick unidirectional carbon fiber/epoxy plate by cutting a small (approximately 0.5 in. $\times$ 0.5 in.) sample from the plate using a diamond-tipped grinding wheel. The material system for the plate was the IM7 fiber (Hexcel Co., CA, USA) and the 977-3 resin system (Cytec Engineered Materials Ltd., United Kingdom). The plate was prepared by hand-layup of unidirectional pre-preg followed by

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an autoclave cure under vacuum and external pressure. The specimen was mounted for polishing in a 24 h, 2-part epoxy system such that the fiber direction was perpendicular to the polishing face. The sample was polished on 3 progressive grit (320, 400, and 600 grit) polishing wheels, followed by a 0.5 μm alumina particle polish. The sample was then cleaned in an ultrasonic bath consisting of 50% isopropyl alcohol and 50% de-ionized water. After the sample was dried, it was ready for analysis.

Experiments were performed with an Agilent 5500 AFM. AFM was operated in force modulation mode, wherein the microcantilever is always in contact with sample surface with a constant mean cantilever deflection (thus a constant preload force) over the scan. An additional low frequency (<50 kHz) vertical oscillation of a few Å amplitude is superimposed onto the microcantilever at the specific preload force. The excitation force is also kept constant over the scan. The amplitude and phase of the cantilever oscillation are recorded simultaneously with the topography image. The cantilevers used in our experiments are magnetically coated cantilever[7] so they can be excited either by a piezoelectric pad embedded inside the cantilever chip holder or by an external magnetic field (see Fig. 1). This AFM system allows us to switch from dither piezo excitation to magnetic excitation without changing any hardware or moving the chip, re-aligning the laser beam, thus ensuring a true comparison of different excitation mechanisms under identical conditions. Stiffness of each cantilever is calibrated by Sader's method.[8] Typical preload force used in our experiments is about a few hundred nanoNewtons.

III. RESULTS AND DISCUSSION

The first thing we want to emphasize here is that the observables in dither piezo and magnetically excited force modulation AFM are different. As shown in Fig. 1, a periodic displacement boundary condition is applied to the microcantilever in the dither piezo excitation mode, while in the magnetic excitation mode a periodic force is directly applied on the microcantilever. Most of AFM systems are based on the optical-beam deflection technique and their output signal is proportional to the cantilever slope at the laser spot location (usually at the tip position). Therefore, the observed cantilever deflection in dither piezo excitation mode $w(t) = Z(t) - u(t)$ is the bending of the cantilever, where $Z(t)$ is the cantilever base motion and $u(t)$ is the cantilever tip motion; while in magnetic excitation mode the observed cantilever deflection is just the tip motion $w(t) = u(t)$. In the discussion above, we considered dither piezo excitation applied to the base of the microcantilever. Of course when the dither piezo excitation is instead applied to the sample, the observed deflection becomes the same as in the magnetic excitation mode.

We next compare the frequency response of the cantilever coupled to the carbon fiber surface by magnetic excitation and dither piezo excitation in Fig. 2. The cantilever used here has a resonance frequency of 53 kHz and the frequency responses by magnetic excitation and dither piezo excitation are almost the same when the cantilever is far from the sample surface in ambient.[9] It is known that the resonant frequency shifts up when the effective tip-sample interaction stiffness increases,[10] which is clearly shown in the frequency response by magnetic excitation. Due to the vibrations from the dither piezo, the chip holder and the chip, there are "forest of peaks" which are not related to the real cantilever resonance in the amplitude response of the dither piezo excitation. The phase information by dither piezo excitation is also "contaminated" by these artificial resonances. Note that while the first contact resonance peak by magnetic excitation is a classic single degree of freedom (SDOF) peak, the big peak(s) around the first contact resonance frequency by dither piezo excitation is clearly not. Another observation is that there is a nearly constant amplitude over a large frequency bandwidth in the magnetic mode response allowing the phase in magnetically excited force modulation AFM to be used to measure the local viscoelasticity of the sample.[5] This forest of peaks and phase distortions are also observed when the dither piezo excitation is applied to the sample, although the distortions are lesser in comparison to when exciting the base of the microcantilever.[11] Lastly, we note that that the difference in frequency response of magnetically and dither piezo excited surface coupled cantilevers is

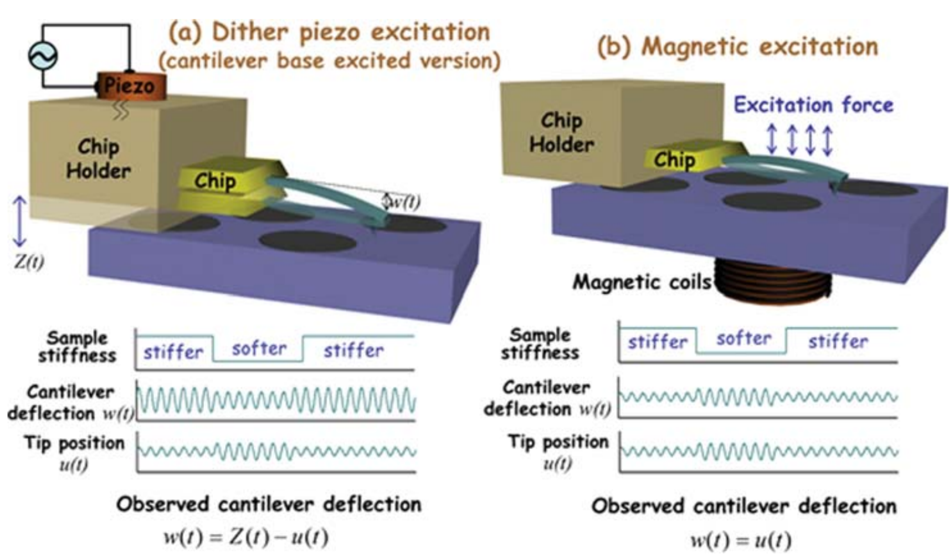

FIG. 1. (Color online) (a), (b) Schematics of force modulation AFM using magnetic and dither piezo excitation and corresponding waveforms (sample stiffness, cantilever deflection, and tip position).

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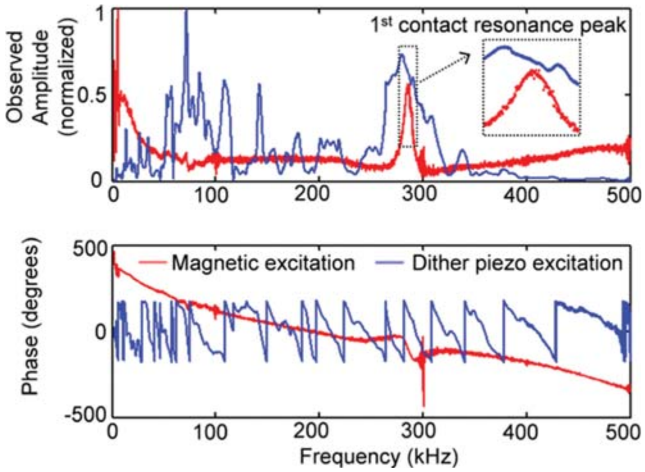

FIG. 2. (Color online) Observed amplitude and phase response of magnetically and piezoelectrically excited cantilevers while in contact with the carbon fiber surface under ambient conditions. Preload force is 80 nN.While the first contact resonance peak by magnetic excitation is a classic single degree of freedom peak, the big peak(s) round the first contact resonance frequency by dither piezo excitation is clearly not. Note there are circuit noises around 100, 300, and 500 kHz; those noises are not related to cantilever properties.

similar to what has been observed for cantilevers in liquid environments without surface coupling.[9,12–14] While it is possible to reduce the spurious resonances in the frequency response of dither piezo excited cantilevers by improving the cantilever holder design,[15–17] there is no published piezo/holder design that can completely eliminate them and produce a frequency response with a constant amplitude over a broad frequency bandwidth (1-500 kHz) by dither piezo excitation.

Now we compare the amplitude and phase images by magnetic and dither piezo excitations. Figure 3(a) shows an AFM topography image, and (b) shows a SEM image of the fiber/epoxy composite sample. Figures 3(c) and 3(d) show the amplitude and phase images by dither piezo excitation (applied to the cantilever base); and Figs. 3(f) and 3(g) show the amplitude and phase images by magnetic excitation. *Interestingly the contrast of amplitude images is inverted for the two excitation modes*. The stiffer fiber appears to be darker in the amplitude image by magnetic excitation but brighter in the amplitude image by dither piezo excitation. The inverted contrast is due to the different observables for the two excitation mechanisms. As we have discussed, the observable in dither piezo excitation mode is the bending of the cantilever $w(t) = Z(t) - u(t)$ and in magnetic excitation mode the observable is just the tip motion $w(t) = u(t)$. Obviously the observables for the two excitation mechanisms have different trends with respect to $u(t)$ which is directly related to the sample stiffness. Note that since the observed deflection is the same as in the magnetic excitation mode when the dither piezo excitation is applied to the sample, the contrast trends are also the same for these two cases. Another thing we want to emphasize here is that good material contrast can be obtained over a large frequency bandwidth by magnetic excitation, while by dither piezo excitation only selected spurious frequency peaks not related to cantilever resonance can give visible material contrast.

In order to further explain the above observations, we turn to the conversion of observed amplitude and phase images to quantitative local material properties. Previous work[5] shows that the viscoelastic properties can be mapped quantitatively from the amplitude and phase images. In this work we focus on the elastic map simply using the amplitude image.[1]

As we have seen, in force modulation microscopy, the local material property information appears as changes in cantilever amplitude (A) and phase (ϕ) over the scan area. For magnetic excitation, the drive force (F_{dr}) for the additional oscillation is kept constant, i.e., $F_{dr} = A \cdot k_{total}$ is constant over the scan, where k_{total} is defined as

$$\frac{1}{k_{total}} = \frac{1}{k_c} + \frac{1}{k_{contact}}, \tag{1}$$

where k_c is cantilever stiffness, $k_{contact}$ is the effective contact stiffness of the sample. Assume there is no deformation on the fiber, that is, $k_{contact}^{fiber} = \infty$, thus $k_{total}^{fiber} = k_c$. Because the drive force on the fiber and polymer are the same, we have

$$\bar{A}^{fiber} \cdot k_c = A^{polymer} \cdot k_{total}^{polyer}, \tag{2}$$

where $\bar{A}^{fiber}$ is the average amplitude on fiber. Combining equations (1) and (2), we have

$$k_{total}^{polymer} = \frac{1}{\frac{1}{k_c} + \frac{1}{k_{contact}^{polymer}}} = \frac{\bar{A}^{fiber}}{A^{polymer}} \cdot k_c. \tag{3}$$

From equations (1) and (3),

$$k_{contact}^{polymer} = \frac{1}{A^{polymer}/\bar{A}^{fiber} - 1} k_c. \tag{4}$$

Similarly, for dither piezo excitation, the effective stiffness map is converted from the amplitude map using the following equation:

$$k_{contact}^{polymer} = \frac{1}{\bar{A}^{fiber}/A^{polymer} - 1} k_c. \tag{5}$$

Notice the difference between Eqs. (4) and (5). From the effective stiffness map we can easily extract the effective Young's modulus map[2,18] by

$$k_{contact}^{polymer} = \frac{\partial F_{ts}}{\partial \tilde{\delta}} = 2E^* \sqrt{R} \left(\frac{F_0 + F_{ad}}{\frac{4}{3} E^* \sqrt{R}} \right)^{1/3}, \tag{6}$$

where F_0 is the mean force applied to sample and F_{ad} is the adhesion force, F_0 and F_{ad} can be obtained from the static FZ curve. $\tilde{\delta}$ is the instantaneous indentation, R is tip radius, and E^* is the effective elastic modulus of tip and sample which is given by $1/E^* = (1 - v_{tip}^2)/E_{tip} + (1 - v_{polymer}^2)/E_{polymer}$, where v_{tip}, E_{tip}, $v_{polymer}$, and $E_{polymer}$ are the Poisson's ratio and Young's modulus of the tip and polymer, respectively. By assuming the value of Poission's ratio of the tip and polymer (0.3), and assuming the value of the elastic modulus of the tip (130 GPa), it is possible to extract a value for local Young's modulus of the polymer.

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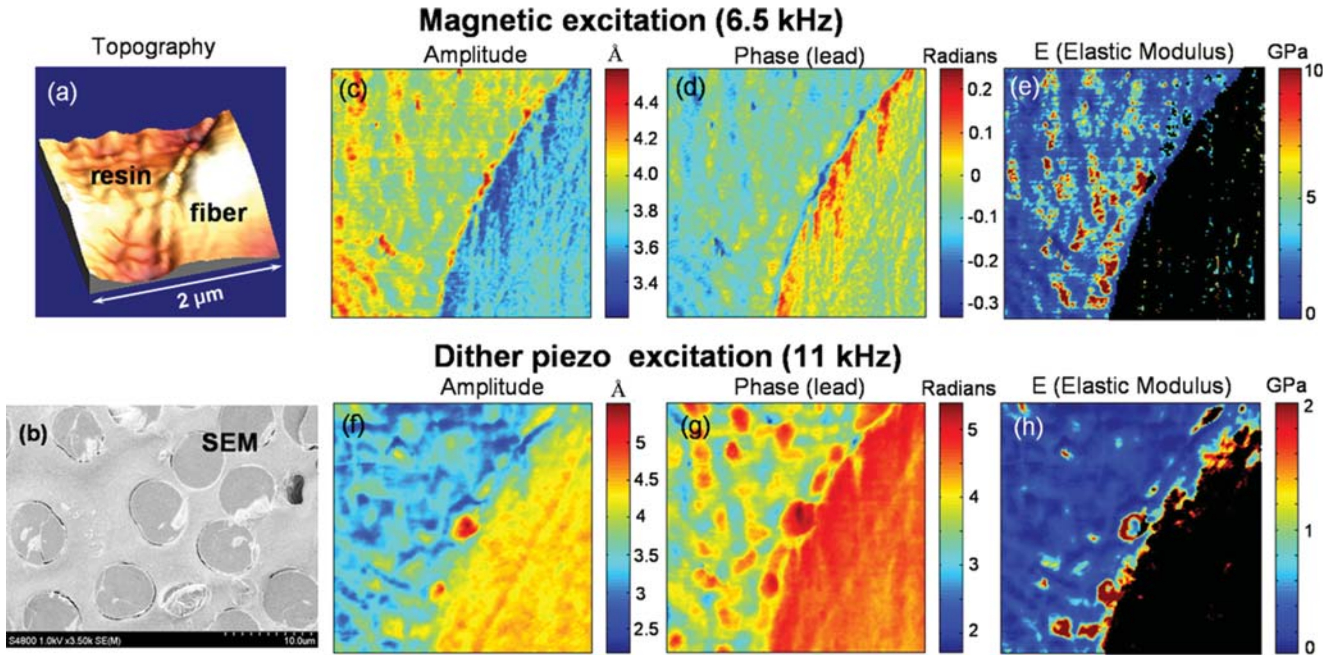

FIG. 3. (Color online) (a) AFM topography image, (b) SEM image of carbon fiber composite, (c) amplitude, (d) phase images, and (e) corresponding converted elastic modulus map by magnetic excitation, (f) amplitude, (g) phase images, and (h) corresponding converted elastic modulus map by dither piezo excitation. To show the local elastic property change on the polymer, (e) and (f) are plotted to the range of 0-10 GPa and 0-2 GPa, respectively; saturated pointed are plotted in black color. Note that we have assumed the carbon fibers to be rigid; those low elastic modulus points scattered over the carbon fiber surface are due to polymer debris or circuit noise.

The elastic modulus maps converted from the amplitude images by magnetic and dither piezo excitations are plotted in Figs. 3(e) and 3(h), respectively. In these two studies, we scanned roughly the same area using the same cantilever by same mean force (F_0); but the drive frequency of the superposed vibration is slightly different: 6.5 kHz for magnetic excitation and 11 kHz for dither piezo excitation. The average converted elastic modulus of the epoxy using the magnetic excitation is 2.52 GPa with 0.76 GPa standard deviation, which is close to the reported value.[19] The average elastic modulus calculated from dither piezo excitation mode images is only 0.21 GPa, which is far below the expected value.

The value of the measured effective stiffness near the fiber interface is affected by the structural compliance of the fiber. On the other hand, the local mechanical properties of the matrix may vary due to chemical reactions close to the fiber interface.[20–22] Both effects introduce an increase in the value of the effective elastic modulus close to the fiber interface. The increase in the effective stiffness due to the structural compliance of the fiber in this structure can be approximated using the analytical solution of a rigid indenter with displacements prescribed at the interface.[23–25] This solution shows a variation of the effective stiffness below 10% at a distance of 30 nm from the interface. However the measured elastic modulus plots shown in Figs. 3(e) and 3(h) in the manuscript do not show any significant variation as function of radial distance to the fiber. This is likely due to interface debonding in the composite system and the difficulty in measuring the local modulus with high resolution near the fiber edge.

Now we examine the consistency of the excitation methods. We collected the magnetically excited force modulation microscopy images of the fiber/epoxy composite sample at three different frequencies, 6, 8, and 14.1 kHz as shown in Figs. 4(a), 4(b), and 4(c), respectively. Because the effective sample stiffness is directly related to the ratio of amplitude $A^{polymer}/A^{fiber}$ instead of the absolute amplitude values, we normalized each amplitude image by the corresponding mean amplitude on fibers. It is shown that the three normalized amplitude images by magnetic excitation are very similar regardless of the excitation frequency. For comparison, we also plotted the normalized amplitude images by dither piezo excitation at its first three peak frequencies (from the forest of peaks), 14.1, 27.3, and 34.5 kHz as shown in Figs. 4(d), 4(e), and 4(f), respectively. Obviously the dither piezo excitation does not produce consistent results.

But why does the dither piezo excited force modulation microscopy not provide a quantitative evaluation of local material properties? The main reason is that the proposed method[1] has to be applied at excitation frequencies far below the cantilever resonance so that the amplitude change should only depend on the sample elasticity. However, as we mentioned earlier, in the dither piezo excitation mode only selected spurious frequency peaks can give visible material contrast. Those frequency peaks, though they are not necessarily related to the cantilever resonances, must be resonances of some mechanical substructures, such as chip holder, piezo, and chip. Therefore the vibration amplitude at those resonant frequencies depends on both the equivalent stiffness and damping of the resonance, which are extremely difficult to evaluate for such sub-structural resonances. As a result, the dither piezo excitation mode cannot be used to accurately, quantitatively evaluate the local sample properties. However it still gives a good idea of relative local material property changes.[25] On the other hand, the magnetically excited cantilever yields a nearly constant gain over a

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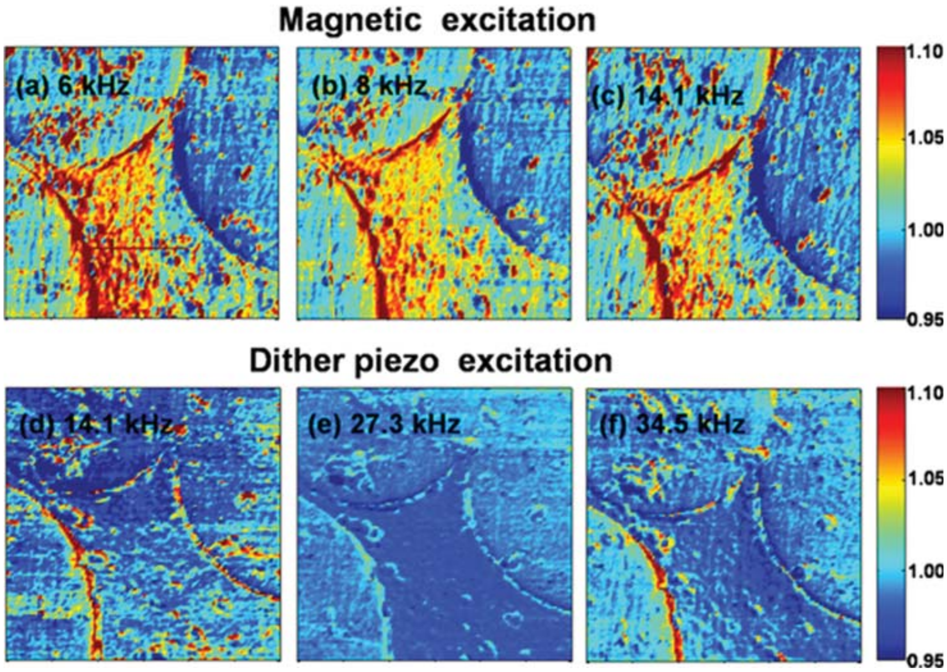

FIG. 4. (Color online) The normalized (by the corresponding mean amplitude on fibers) amplitude images by magnetic excitation at (a) 6 kHz, (b) 8 kHz, and (c) 14.1 kHz; and by dither piezo excitation at (d) 14.1 kHz, (e) 27.3 kHz, and (f) 34.5 kHz.

large frequency bandwidth allowing quantitative measurements of the local sample properties.

IV. CONCLUSION

In conclusion, we have shown that there is a major difference in the frequency response of surface coupled cantilevers in AFM when excited using direct means (such as magnetic excitation) versus using indirect means (such as dither piezo excitation). There is an inversion in amplitude contrast between the two and the extraction of quantitative material properties from indirect (dither piezo) excitation is problematic due to the spurious mechanical resonances of the underlying substructure. Magnetic or other direct excitations produce much more accurate, quantitative mapping of local material properties; however currently the choice of magnetic or other special directly excited cantilevers are very limited. Future development of quantitative force modulation AFM should focus on developing and improving directly excited cantilever technology.

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