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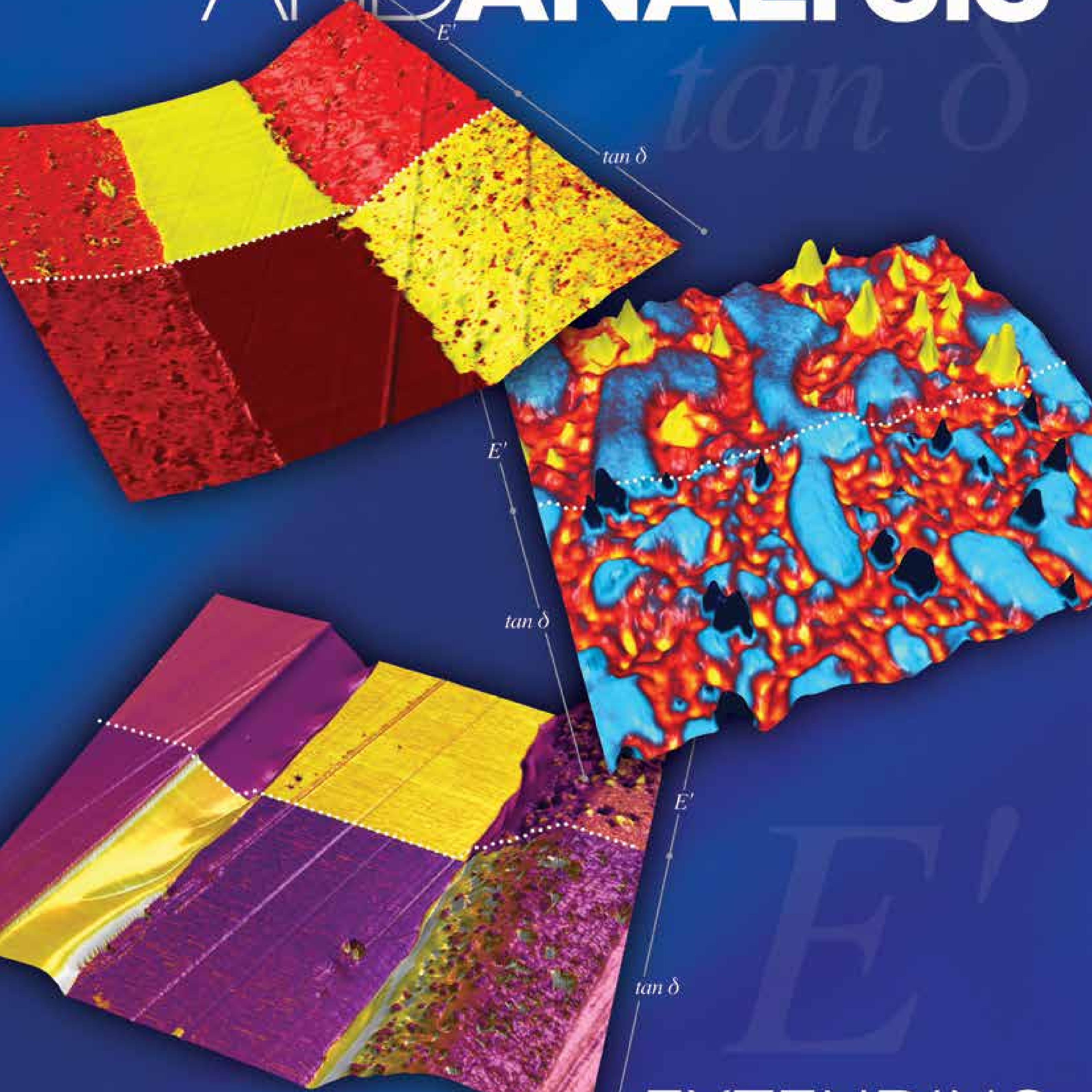
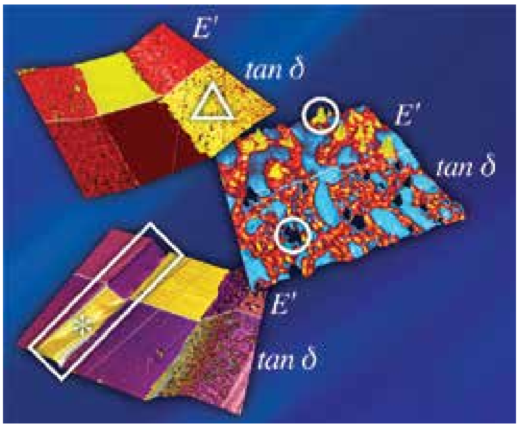


EXTENDING AFM NANOMECHANICS TO VISCOELASTIC MATERIALS

MICROSCOPYANDANALYSIS

INSIDE

COVER STORY



The cover shows examples of materials characterized by AM-FM Viscoelastic Mapping Mode on Asylum Research AFMs. The 3D surface represents the topography while the color scale represents the elastic modulus (E′) for the upper-half of each image and the loss tangent (tan δ) for the lower half of each image.

Many AFM techniques have emerged in recent years for measuring the elastic response of materials. Meanwhile, the viscous response, characterized by the loss tangent or loss modulus, has been relatively neglected. This is unfortunate because the viscous response of materials affects damping of vibration, noise, toughness, fatigue, impact resistance, friction, adhesion and bond strength at interfaces. The loss tangent of materials also provides one more criterion by which to distinguish and identify components that are very similar in elastic modulus. The examples shown here represent practical cases where knowledge of the full viscoelastic response helps to either identify the components and/or to understand their role in the composite material.

TOP-LEFT EPOXY BOND LINE BETWEEN TWO ELASTOMERS Bonding dissimilar materials is a common challenge in industry. Here, a thin epoxy layer joins two elastomeric materials with very similar elastic moduli. Though the epoxy is readily distinguished by its higher E′, only the higher tan δ identifies the elastomer selected for its damping characteristics (marked with the triangle). Scan size is 25 µm.

MIDDLE-RIGHT RUBBER BLEND The bulk properties of polymer blends are determined by the amount, distribution, and properties of their components. This sample is a blend of natural rubber, polybutadiene rubber, and zinc oxide. The elastic response distinguishes all three materials, but the zinc oxide inclusions (circles) stand out more clearly by their much lower loss tangent. Scan size is 5 µm. Images courtesy of Dr. Anna Kepas-Suwara, Tun Abdul Razak Research Centre, UK.

BOTTOM-LEFT MULTILAYER FOOD PACKAGING MATERIAL Food packaging materials are designed for strength, moisture and air impermeability, thermal sealing, and aesthetics. The film here consists of an aluminum barrier layer sandwiched between two polymer layers. To prevent delamination at the metal/polymer interfaces an additional polymer is present as a tie layer (rectangle). Though the elastic moduli of the polymer films and tie layers are similar, the tie layers have much higher loss tangent (asterisk), which helps dissipate stresses that lead to delamination. Scan size is 15 µm.

For more information on AM-FM Viscoelastic Mapping Mode, see our accompanying article in this issue: "Fast, quantitative AFM nanomechanical measurements using AM-FM viscoelastic mapping mode."

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Fast, quantitative AFM nano-mechanical measurements using AM-FM Viscoelastic Mapping Mode

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INTRODUCTION

Nanoscale mechanical properties impact the behavior and performance of countless materials and help inform both our theoretical understanding of these materials and their development for commercial applications. Examples can be found in many disciplines, for instance, in materials science, where elastic modulus affects the reliability of nanoporous dielectric films in semiconductor devices, and in biology, where relations have been observed between viscoelastic damping and the metastatic potential of cancer cells. As we continue to learn more about the critical role of nanomechanics, our need for techniques that more fully measure them grows in importance.

The atomic force microscope (AFM) is a powerful tool to meet this need, due to its superb spatial resolution and force sensitivity. As a result, nanomechanical techniques such as phase imaging in tapping mode [1,2,3], force curves [4], and contact resonance AFM [5] have been developed over the years. Nonetheless, research continues on improved methods. One reason is the sheer diversity of properties in today's materials. For instance, elastic modulus ranges from less than 1 kPa for cells and tissue scaffolding to over 1 TPa for diamond and graphene, while viscoelastic loss tangent varies from less than 10^{-6} for single crystals to greater than 1 for elastomers and biological materials.

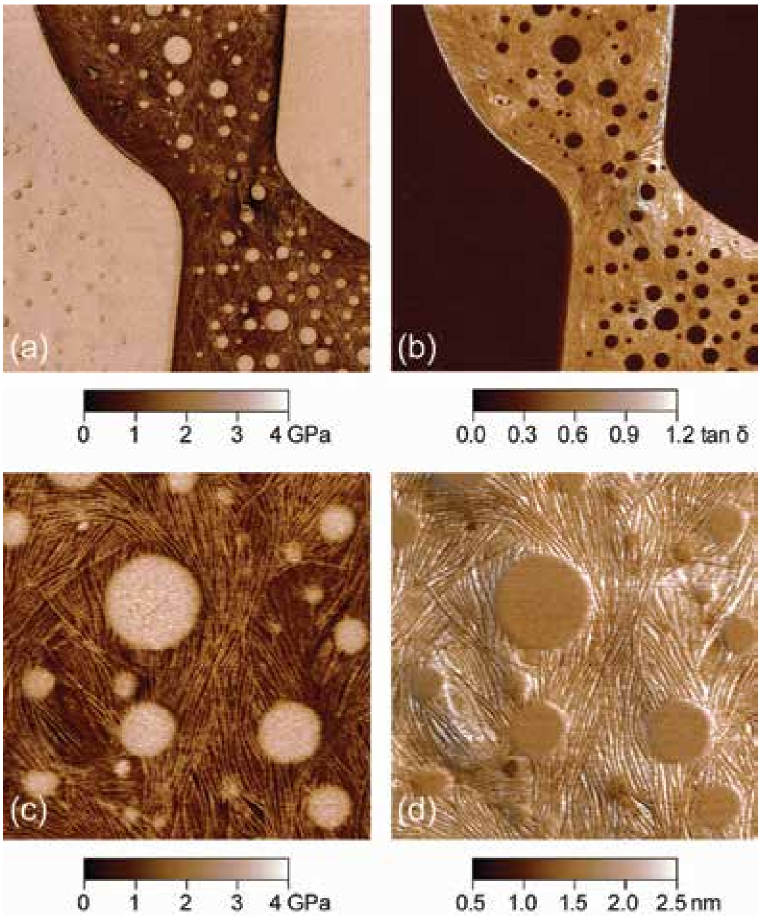

FIGURE 1 AM-FM Mode images for a polystyrene-polycaprolactone (PS-PCL) polymer film: (a) and (c) storage modulus E′, (b) loss tangent tan δ, and (d) indentation depth. Scan sizes are 5 µm in (a) and (b) and 1.5 µm in (c) and (d). E′ is higher in PS regions (light brown) than PCL regions (dark brown), while PCL regions exhibit higher tan δ than PS. The ability of AM-FM Mode to capture the fine fibrillar structure of PCL demonstrates its high spatial resolution and gentle nature. Images acquired on a Cypher S AFM at 2 Hz scan rate with blueDrive photothermal excitation.

An example of recent progress in AFM nanomechanical characterization is Asylum Research's AM-FM Viscoelastic Mapping Mode [6,7]. AM-FM Mode can be used to evaluate materials in terms of both elastic and viscous response including quantitative measurement of properties including Young's modulus, storage modulus, loss modulus, and loss tangent (Figure 1). Other notable features include high resolution, low-force operation compatible with easily damaged samples, and the ability to extend these capabilities to high-speed AFM. Here, we present an overview of AM-FM Mode. We explain its origins in other AFM techniques and describe its basic

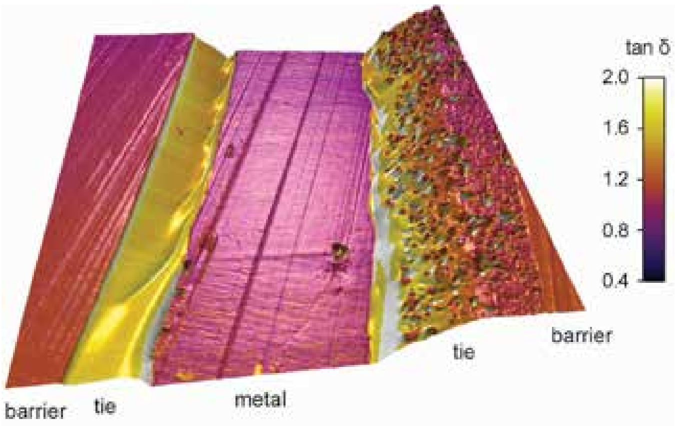

FIGURE 2 Loss tangent imaging of a commercial coffee packaging bag in cross section. Different sample components (vapor barriers, "tie" layers, and metal layer) are clearly distinguished. Scan size 15 µm. Image acquired on a Cypher S AFM

operating concepts. Example data are given to demonstrate its capabilities on a wide range of materials.

BACKGROUND

TAPPING MODE AND BIMODAL AFM

The roots of AM-FM Mode lie in tapping mode and bimodal AFM. Tapping mode, also known as AC mode or amplitude-modulated (AM) mode, is a ubiquitous technique for imaging nanoscale topography. In this mode the tip only contacts the sample during part of the cantilever oscillation cycle, reducing sample damage and tip wear while enabling high resolution and fast scanning. Sample topography is measured with a Z feedback loop that maintains constant tapping-mode amplitude signal. Simultaneously, qualitative nanomechanical information can be inferred from the tapping-mode phase signal [1,2,3]. Various approaches have been developed to relate the phase response to the mechanical properties of the sample surface [3,8,9,10], as well as to quantify tip-sample energy dissipation and storage [11,12]. However, the phase response depends not only on how the material stores elastic energy and dissipates viscous energy but also on other dissipative forces and factors such as cantilever vibrational parameters.

Recently, this situation has been addressed by applying the concept of loss tangent to tapping-mode phase contrast [13]. The loss tangent $\tan\delta$, defined as the ratio of loss modulus E' to storage modulus E'' or $\tan\delta = E''/E'$, also represents the ratio of a material's dissipated energy to stored energy during cyclic excitation. Equivalently, the angle δ describes the phase lag between an applied strain and the resulting stress in a material with time-dependent response. As a sensitive probe of polymeric phase transitions, loss tangent measurements provide valuable information on both fundamental (e.g., molecular motion) and practical (e.g., impact resistance) levels. AFM measurements of loss tangent use the amplitude and phase response in tapping mode to determine the ratio of dissipated energy to stored energy per cycle of the tip's periodic deformation. As seen in Figure 2, loss tangent imaging increases the capabilities of tapping mode for nanomechanical characterization.

As understanding of conservative and dissipative tip-sample interactions advanced, it was realized that the same effects in higher oscillatory modes afford enhanced sensitivity to material composition. This realization led to the development of multifrequency and bimodal AFM [7,14,15,16], in which multiple resonant frequencies of the cantilever are excited simultaneously. In bimodal AFM, two resonant modes are excited. The first (lower) mode is operated in regular tapping or AM mode, yielding the usual topography and phase data. The second (higher) resonance can also be operated in AM mode or with another feedback approach. Bimodal images can provide qualitative contrast that cannot be obtained with conventional tapping mode. Tremendous progress has also been made towards quantitative interpretation of bimodal amplitude and phase measurements in terms of the sample's nanomechanical properties.

PRINCIPLES OF AM–FM MODE

AM-FM Mode is one such approach for improved quantitative interpretation. As a bimodal technique, it operates simultaneously at two different cantilever frequencies. The cantilever's first and second flexural resonances are typically used, although operation with higher-order resonances is not uncommon. The lower mode operates in standard tapping (AM) mode, and the higher mode operates in frequency modulation (FM) mode with frequency feedback. The AM mode provides topography and loss tangent information. The FM mode, which operates at a much smaller amplitude, acts as a ride-along signal that sensitively probes the tip-sample interaction. By using the same imaging feedback as normal tapping mode, data acquisition in AM-FM Mode is reliable and rapid.

Figure 3 explains the concepts of AM-FM Mode in more detail. Signals from two voltage oscillators are summed and sent to the cantilever holder's piezoelectric actuator ("shake piezo") in order to excite two vibrational modes simultaneously. The photodiode signal containing the cantilever's response is then analyzed in two ways. First, the amplitude and phase of the lower mode at a fixed frequency are measured by a lock-in amplifier. As in regular tapping mode, the amplitude is used for feedback control. That is, the AFM adjusts the cantilever's vertical position to keep the amplitude at the setpoint value, which provides topography data. Combined, the amplitude and phase signals determine the loss tangent [13].

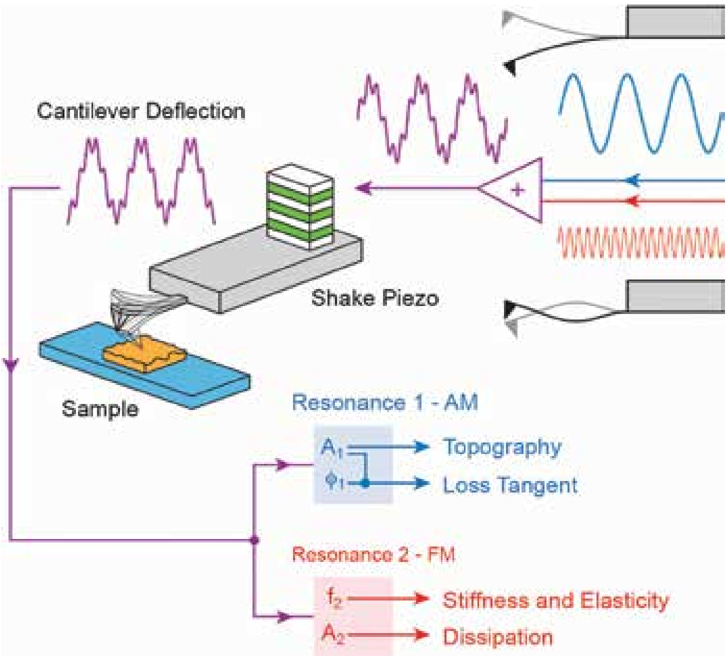

FIGURE 3 Schematic of operation in AM-FM Mode. Two separate excitation signals (blue and red curves, right) are combined to excite two cantilever resonances simultaneously (purple curve, center). The resulting cantilever deflection (purple curve, left) is analyzed to determine the response at each resonance. Resonance 1 operates in normal tapping or AM mode (blue box). The amplitude A_1 controls the vertical feedback loop for standard tapping mode topography, while A_1 and the phase ϕ_1 give values for loss tangent. Resonance 2 operates in FM mode (red box). Changes in resonance frequency determine stiffness and elasticity, while changes in the amplitude A_2 give viscous or dissipation information

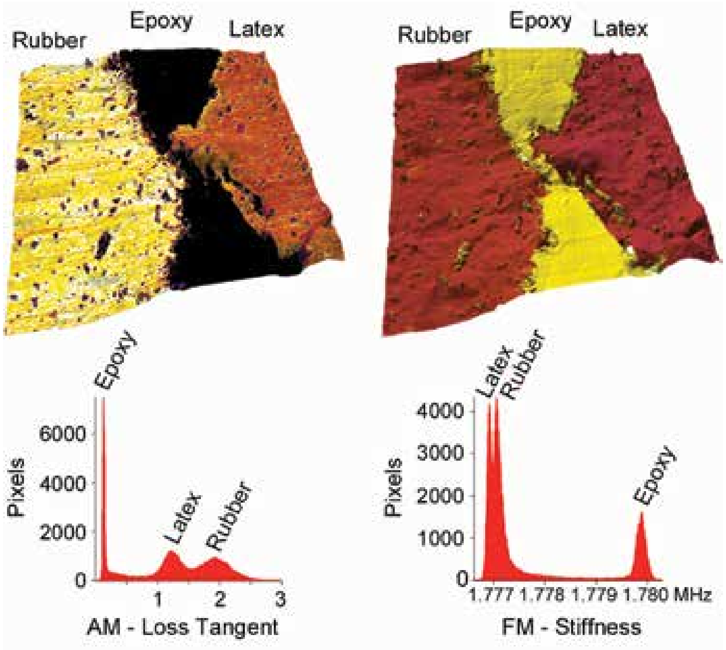

FIGURE 4, above, AM-FM Mode images of (left) loss tangent and (right) second mode frequency overlaid on topography for a rubber-epoxy-latex sandwich. Different sample components are clearly distinguished by the AM loss tangent of viscous damping. They are also resolved by the FM frequency, which correlates with elastic stiffness despite very similar modulus values for latex (~40 MPa) and rubber (~43 MPa). Scan size 5 µm; imaged with the Cypher S AFM

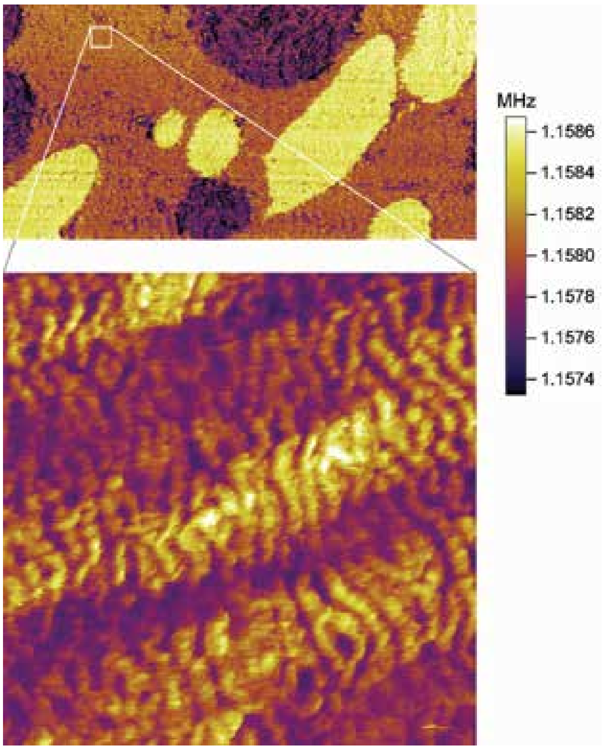

FIGURE 5, right, AM-FM Mode frequency images for a ternary polymer blend of polypropylene (PP), polyethylene (PE), and polystyrene (PS). In the top image with scan size 8 µm, regions of PS (yellow) and PE (purple) are clearly distinguished from PP (orange). The FM frequency also correctly ranks the relative stiffness of the components as PE < PP < PS. In the bottom image with scan size 300 nm, the lamellar structure of PP is clearly resolved

Sample courtesy Dalia Yablon and Andy Tsou, ExxonMobil Research and Engineering, Corporate Strategic Research.

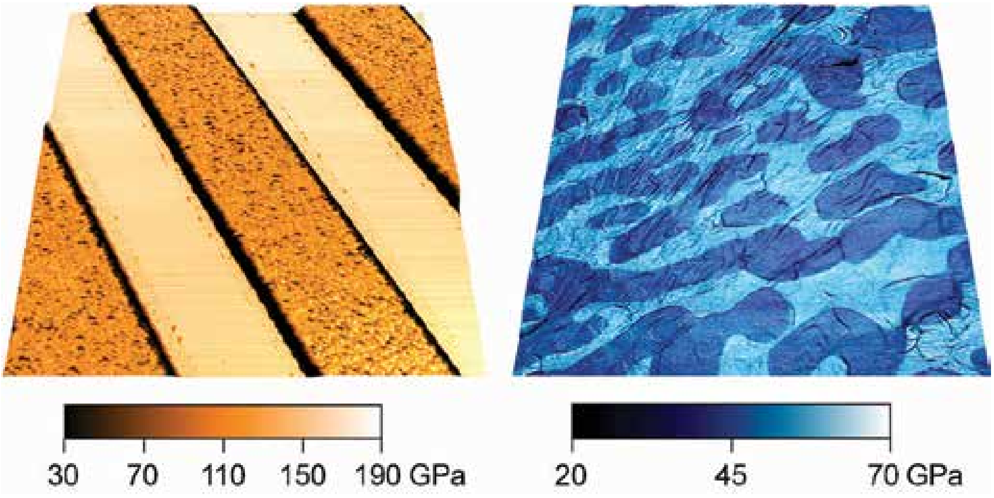

FIGURE 6, below, Modulus mapping of metallic samples with AM-FM Mode. (left) Elastic modulus overlaid on topography for a sample with a patterned titanium (Ti) film approximately 200 nm thick on silicon (Si). The image was calibrated with use of a literature value for the modulus of Si. The lower modulus values for the Ti stripes relative to Si are consistent with bulk literature values for Ti (110-125 GPa) and <001> Si (165 GPa). Scan size 10 µm. (right) Elastic modulus overlaid on topography of a tin/lead alloy solder. Tin-rich (lighter) and lead-rich (darker) regions can be identified in the modulus map. Scan size 8 µm; acquired at 1.5 Hz scan rate with blueDrive photothermal excitation. Both images were acquired on a Cypher S AFM

Meanwhile, a second lock-in amplifier measures the frequency and amplitude at the higher cantilever resonance operating in FM mode. An automatic gain control circuit monitors the amplitude and adjusts the drive voltage to keep the amplitude constant. A phase-locked loop monitors the phase and adjusts the drive frequency to keep the phase at 90°. The amplitude of the higher mode is always much smaller than that of the lower mode and is at a different frequency. With this approach the higher FM mode does not perturb the lower AM mode, ensuring that operation is highly stable and robust. Performance can be enhanced further with photothermal excitation [17], especially for operation in liquid environments.

The FM mode frequency signal is an exquisite probe of the elastic tip-sample interaction, with higher frequency corresponding to higher contact stiffness or elastic modulus. From a measurement standpoint, measurements of relative frequency shifts are always preferable to ones of absolute amplitude or displacement. Not only do relative measurements reduce common sources of systematic error, but smaller bandwidths in frequency-selective methods also mean lower noise. Both effects lead to higher measurement accuracy and precision. Figures 4 and 5 illustrate the sensitivity of AM-FM Mode. Individual constituents in heterogeneous polymer samples are readily identified, even those with only slight differences in elastic modulus. Like Figure 1, Figure 5 shows that AM-FM Mode has sufficiently high sensitivity and spatial resolution to discern even ultrafine structural details of polymers. Besides the elastic information provided by the FM mode frequency shift, the output drive voltage signal contains additional information on viscous or dissipative forces.

QUANTITATIVE VISCOELASTIC MODULUS MEASUREMENTS

As Figures 4 and 5 demonstrate, AM-FM Mode frequency imaging permits quick identification of sample components and qualitative assessment of relative stiffness. However, quantitative data on mechanical properties can also

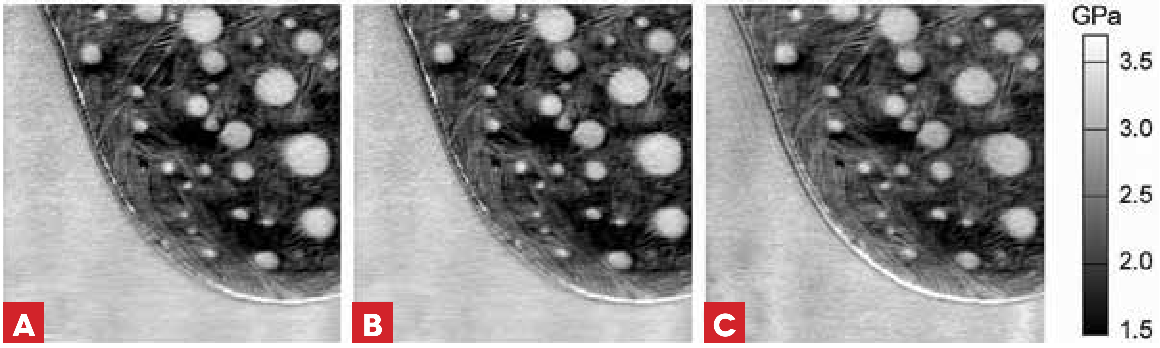

FIGURE 7 High-speed modulus mapping with AM-FM Mode on a polystyrene-polycaprolactone (PS-PCL) polymer film (see also Figure 1). The first mode of the cantilever had a resonant frequency of approximately 1.5 MHz and quality factor Q ≈ 100. Images were acquired at line scan rates of (a) 2 Hz, (b) 20 Hz, and (c) 26 Hz, corresponding to acquisition times of (a) 128 s, (b) 12.8 s, and (c) 9.6 s for a complete image of 256x256 pixels. Image quality remains virtually unchanged when the scan rate is increased by 10X from (a) to (b). Increasing the scan rate further results in only minor degradation, as evidenced by ripple at the PS-PCL step edge in (c). Scan size 2.5 µm; acquired on the Cypher S AFM

be obtained in AM-FM Mode from the measured amplitude, phase, and frequency. Contact stiffness and elastic modulus are determined from the FM mode frequency with use of a contact mechanics model [7]. The Hertz/Sneddon model is most commonly used to describe the interaction between the sample and a tip of specific geometry (e.g., hemisphere, punch, cone). Model parameters can be determined directly, for instance by measuring the tip shape with a tip-check sample. Alternatively, a reference sample with known modulus can be used to calibrate parameters such as contact area [6,7]. The same parameter values are then applied to the test sample images. Results are most accurate when the calibration and test samples have similar modulus. With different calibration samples, AM-FM Mode enables quantitative imaging on materials that span more that six orders of magnitude in modulus (approximately 50 kPa to 300 GPa). For example, AM-FM Mode has been used for modulus mapping in applications ranging from very stiff metallic samples (Figure 6) to highly compliant mammalian prions [18] and azo-polymer films [19].

For viscoelastic materials, AM-FM Mode determines loss tangent from the first mode amplitude and phase [13]. Because the measured loss tangent is the ratio of dissipated to stored power in the AFM tip-sample interaction, it is influenced by attractive forces and dissipative processes such as adhesion and plasticity. Thus, measurements represent an upper bound on the material's actual $\tan\delta$. Effects including long-range forces (electrostatic, van der Waals, etc.) and adhesive and capillary forces must be more fully considered for improved quantification. Nonetheless, the measured loss tangent serves as a useful estimate with which to characterize viscoelastic behavior more completely and provides valuable relative contrast. The loss modulus E'' can also be obtained by combining storage modulus and loss tangent, i.e., $E'' = E' \tan\delta$.

In addition, AM-FM Mode can map the tip-sample indentation (sometimes called deformation). Tapping-mode operation means that very gentle forces can be used in AM-FM Mode, with correspondingly small sample deformation. This both minimizes damage and maximizes spatial resolution. An example of an indentation map is shown in Figure 1(d). As is typical with AM-FM Mode, the sample deformation is only a few nanometers.

ONGOING ADVANCES TO AM–FM MODE

The basis of AM-FM Mode on tapping mode presents opportunities for further advances. For example, the recent introduction of commercial AFMs that support small cantilevers, such as Asylum's Cypher, has enabled dramatically faster imaging speeds, and this capability is directly extendable to AM-FM Mode. In tapping mode, the time per pixel is given by $\tau=2Q/f$, where f is the frequency and Q is the quality factor of the cantilever resonance. In AM-FM Mode, the f and Q of both resonances must be considered. Usually, the limiting factor for imaging speed is the first mode quality factor. The higher mode frequency relative to the photodiode detector bandwidth is an additional consideration, especially for very small cantilevers.

With resonant frequencies from hundreds of kilohertz to a few megahertz and quality factors in the hundreds, small cantilevers can achieve line scan rates of tens of hertz in AM-FM Mode. This high-speed imaging capability allows much faster quantitative nanomechanical imaging than possible with other techniques including force mapping [4], contact resonance AFM [5], and recent versions of fast force mapping based on pulsed force mode AFM [20]. As shown in Figure 7, imaging in AM-FM Mode has already been demonstrated at line scan rates over 20 Hz (equivalent tip velocity up to 300 µm/s), with image forces as low as 50 pN [7]. To our knowledge, these images are the fastest examples of AFM nanomechanical mapping to date. These increased imaging rates will enable new studies of many previously inaccessible dynamic processes (e.g., cellular processes, polymer thermal reconfigurations). In addition, they simply increase productivity by producing images in seconds, not minutes.

Tapping mode is commonly used for imaging samples in liquid, especially for biological samples. This suggests that AM-FM Mode imaging should also be possible in liquid. However, initial attempts were hampered by the complex frequency response of a piezoacoutically-driven cantilever in liquid. The recent development of blueDrive photothermal excitation [17] has resolved this challenge. As

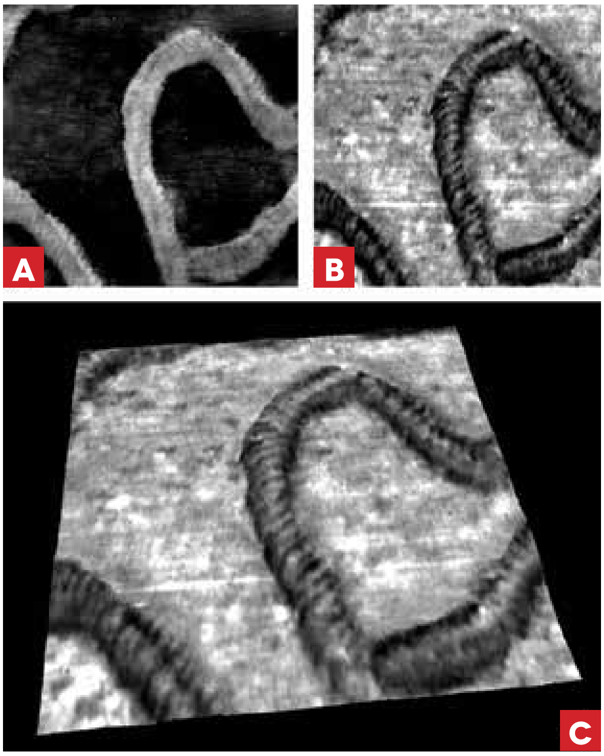

FIGURE 8 AM-FM Mode imaging of DNA in buffer. Both the major and minor groves of the double helix are clearly visible throughout both the (a) topography and (b) the second mode frequency images, demonstrating the exquisite sensitivity to elastic stiffness and high spatial resolution of the mode. The second mode frequency data has been overlaid on 3D topography in (c). As described previously, the observed frequency shifts in the second mode can typically be interpreted in terms of quantitative stiffness and elastic modulus variations. We have chosen to show the raw frequency data here because of the special case where the AFM tip is similar in dimensions to the DNA, which violates assumptions of common contact mechanics models. Qualitatively, however, the data shows what we expect, which is lower frequencies on the softer DNA and higher frequencies on the stiffer mica. Scan size 70 nm. Acquired on a Cypher S AFM with blueDrive photothermal excitation.

demonstrated in Figure 8 for DNA absorbed on mica, the combination of AM-FM Mode and blueDrive excitation enables nanomechanical mapping in liquid with high spatial resolution. While additional challenges remain, including convenient calibration standards for liquid operation, they are common to all nanomechanical techniques applied to samples in liquid.

CONCLUSIONS

Information on nanoscale mechanical properties is critical to successful development of many material systems. Nanomechanical characterization with the AFM has seen tremendous progress throughout its history and continues to be a major research goal. As an example of this progress, we have described a relatively new nanomechanical technique called AM-FM Viscoelastic Mapping Mode.

AM-FM Mode enables quantitative imaging of both elastic and viscous quantities including storage modulus, loss modulus, and loss tangent. Because it is based on tapping mode and bimodal AFM, AM-FM Mode also offers rapid scanning and gentle forces. The powerful capabilities of AM-FM Mode open the door to many exciting new avenues of research on advanced materials.

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BIOGRAPHY

Donna Hurley is a consultant in AFM measurement methods and their application to materials science. Previously she worked at the National Institute of Standards and Technology, where she led a team to develop and apply contact resonance AFM techniques for nanomechanical mapping of materials. She has a Ph.D. in Physics from the University of Illinois at Urbana-Champaign.

ABSTRACT

Information on mechanical properties is essential in many applications, and it is increasingly important to obtain this information on micro- and nanometer length scales. Since the invention of the AFM, nanomechanical characterization has been addressed in numerous ways. Here we describe the principles and features of a recently introduced technique for AFM nanomechanics, AM-FM Viscoelastic Mapping Mode. AM-FM Mode operates in tapping mode with simultaneous excitation of a second cantilever resonance in frequency feedback. It enables gentle, rapid imaging of nanoscale viscoelastic properties including storage modulus and loss tangent. Examples are given to demonstrate the capabilities of AM-Mode Mode on a wide range of materials.

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