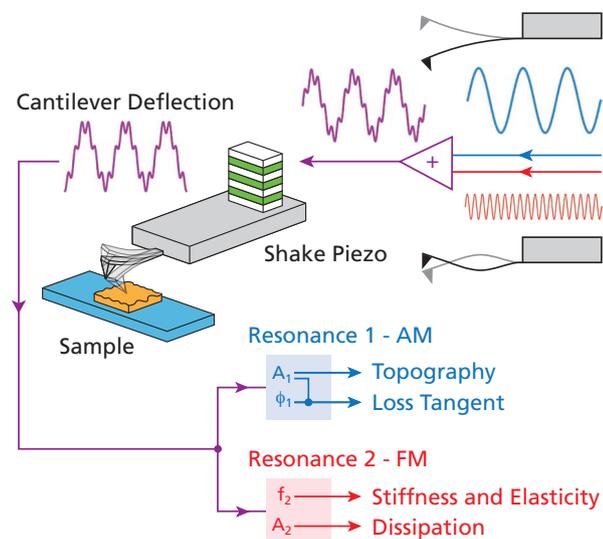


Information on mechanical properties is important in many applications. AM-FM Viscoelastic Mapping Mode lets you quickly and gently image viscoelastic properties including storage modulus and loss tangent with nanoscale spatial resolution. Its very wide operating range, from less than 1 MPa to hundreds of GPa, makes it a highly versatile technique. AM-FM Mode is available on all **MFP-3D™** and **Cypher™** family AFMs and is one of many options in Asylum's **NanomechPro™ Toolkit** for nanomechanical measurements.

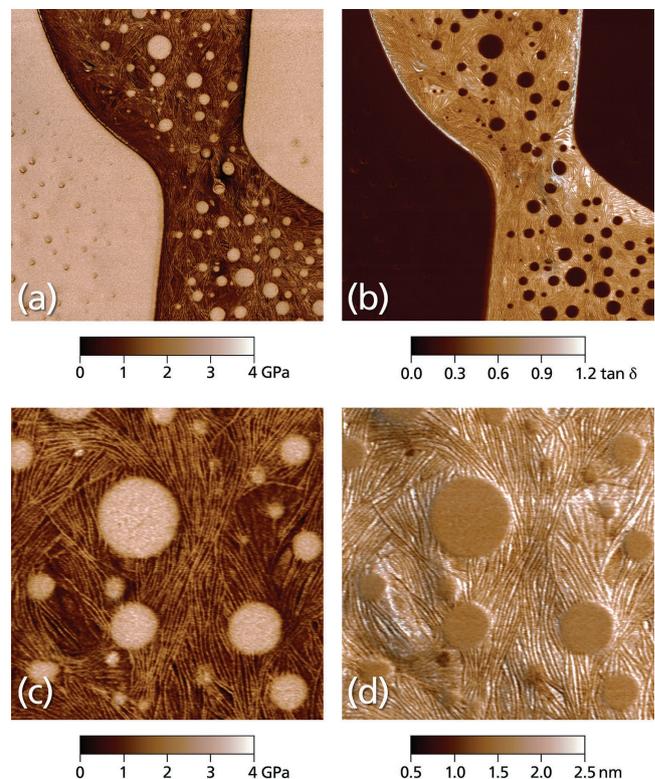
## Capabilities and Benefits

Asylum's exclusive AM-FM Viscoelastic Mapping Mode<sup>1</sup> is a flexible, convenient tool for nanomechanical characterization. With a range of applicability that spans a remarkable six orders of magnitude in storage modulus (from less than 1 MPa to hundreds of GPa), it is a general-purpose technique for anything from biomaterials and polymers to metals and ceramics. AM-FM Mode provides elastic information including storage modulus, Young's modulus, and contact stiffness and viscoelastic information including viscoelastic loss tangent and loss modulus.

AM-FM Mode gets results by operating at two cantilever resonances simultaneously. As the name indicates, the first resonance is used for tapping mode imaging, also known as amplitude modulation (AM), while a higher resonance mode is operated in frequency modulation (FM). At resonance, the cantilever frequency and phase respond sensitively to changes



In AM-FM Mode, two separate excitation signals (blue and red curves on 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 AM mode (blue box). The amplitude  $A_1$  controls the vertical feedback loop for standard tapping mode topography, while  $A_1$  and the phase  $\phi_1$  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.



AM-FM Mode images for a polystyrene-polycaprolactone (PS-PCL) polymer film on mica: elastic storage modulus (a and c), viscoelastic loss tangent (b), and indentation depth (d). Scan sizes  $5 \mu\text{m}$  (a and b) and  $1.5 \mu\text{m}$  (c and d). Storage modulus is higher in PS regions (light brown) than PCL regions (dark brown), while PCL regions exhibit higher loss tangent than PS. The ability to capture the fine fibrillar structure of PCL (c and d) demonstrates AM-FM Mode's high spatial resolution and gentle nature. Images acquired on a Cypher S AFM at 2 Hz scan rate with blueDrive™ photothermal excitation.

# AM-FM Viscoelastic Mapping Mode

in sample properties. Small frequency and phase shifts can be measured with very high precision and accuracy, reducing uncertainty and increasing sensitivity. You can use raw output signals to quickly visualize relative contrast and identify sample components; or you can use the observed amplitude, phase, and frequency data to make quantitative estimates of mechanical properties based on built-in or your own models.

Because AM-FM Mode works like tapping mode in the repulsive regime, it is familiar and straightforward to use. It also has the other advantages of tapping mode including fast scanning, high spatial resolution, and gentle forces. On high speed, low-noise systems such as Asylum's Cypher S and ES AFMs, modulus mapping in AM-FM Mode can routinely operate at line scan rates as fast as 20 Hz (equivalent tip velocity 300  $\mu\text{m/s}$ ) and forces as low as 50 pN.<sup>2</sup> Low forces mean less sample deformation, typically only a few nanometers, which both minimizes damage and maximizes spatial resolution. Because the FM amplitude is just a tiny fraction of the AM amplitude and is at a different frequency, topographic imaging operates the same as in standard tapping mode. This makes AM-FM Mode very stable and reliable to operate.

## How it Works

AM-FM Mode has its roots in research on multifrequency and bimodal AFM.<sup>4-8</sup> The diagram on page 1 explains the basic concepts of operation. The signals from two oscillators are summed and sent to the cantilever excitation or "shake" piezo, exciting two different vibrational modes of the cantilever simultaneously. Typically, the first and the second flexural resonances are used, although the third and even higher resonances can be used.

The cantilever's response is analyzed in two ways. The lower cantilever resonance operates in AM mode, exactly like standard tapping mode. The amplitude and phase at a fixed frequency are measured by a lock-in amplifier. Together, these signals determine the loss tangent.<sup>3</sup> The AFM also uses the amplitude for feedback control, adjusting the cantilever's vertical position

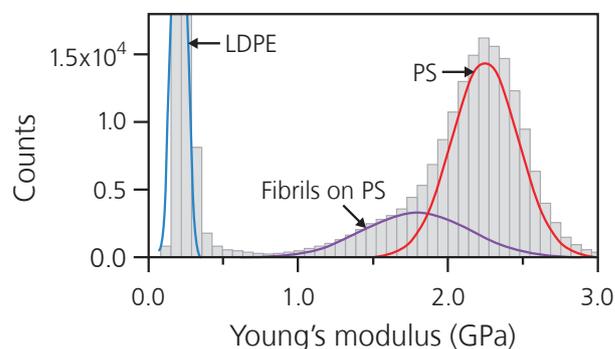
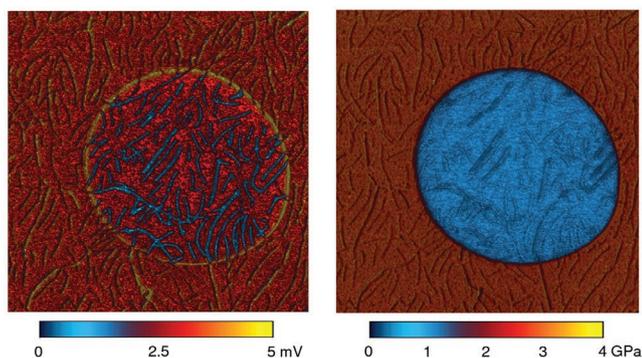
## What is Loss Tangent?

In materials mechanics, the loss tangent  $\tan \delta$  is the ratio of a material's dissipated energy to stored energy in response to cyclic excitation. Equivalently, the angle  $\delta$  describes the phase lag between an applied strain and the resulting stress in a material with time-dependent response. Loss tangent is defined in terms of the material's elastic storage modulus  $E'$  and viscoelastic loss modulus  $E''$  by  $\tan \delta = E''/E'$ . For many polymeric materials, loss tangent is a very sensitive probe of phase transitions, which can relate to performance-critical properties such as toughness and impact resistance. When measured on bulk samples with traditional techniques,  $\tan \delta$  typically ranges from less than 0.001 for metals and ceramics to more than 1 for elastomers and biological materials.

The loss tangent measured in AM-FM Mode is the ratio of dissipated to stored power in the AFM tip-sample interaction.<sup>3</sup> Attractive forces and dissipative processes such as adhesive rupture mean that the measurements are an upper bound on the material's actual  $\tan \delta$ . Further technique development and calibration protocols may improve agreement. Nonetheless, the measured loss tangent provides a useful estimate with which to assess viscoelastic behavior.

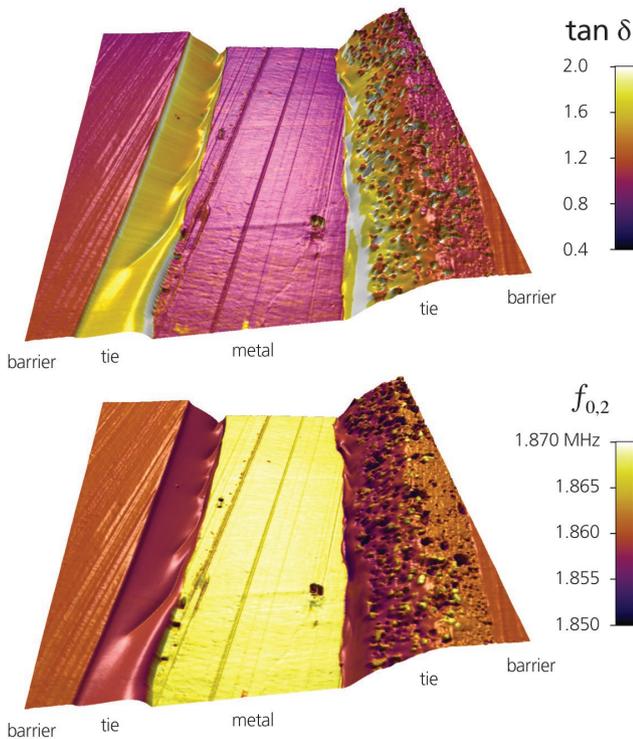
to keep the amplitude at the setpoint value. Just like tapping mode, this signal provides topography data.

Meanwhile, a second lock-in measures the phase and amplitude at the higher cantilever resonance, which operates 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 output drive voltage signal contains information on viscous or dissipative forces. The output resonance frequency describes the elastic tip-sample interaction; basically, higher frequency means greater stiffness or modulus.



AM-FM Mode images of dissipation (left) and elastic modulus (right) for mouse prion amyloid fibrils on a polystyrene (PS) substrate with a (LDPE) low-density polyethylene domain. Values for Young's modulus in the image and histogram (far right) were obtained by referencing to the PS-LDPE substrate. The dissipative signal shows greater contrast between the fibrils and the substrate than the elastic modulus. Image scan size 3  $\mu\text{m}$ . For more information see G. Lamour, K.C. Yip, H. Li, and J. Gsponer, *ACS Nano* **8**, 3851 (2014).

# AM-FM Viscoelastic Mapping Mode



(top) Loss tangent and (bottom) frequency for a commercial coffee packaging bag in cross section. Both images distinguish between the different sample components (vapor barriers, “tie” layers, and metal layer). Scan size 15  $\mu\text{m}$ . Acquired with a Cypher S.

Quantitative elastic modulus is determined from frequency, amplitude, and phase of the two modes with a contact mechanics model. Model parameters such as the cantilever tip radius can be determined either by directly measuring the tip shape with a tip-check sample or, much more easily, with use of a reference sample with known modulus.<sup>2</sup> In this case, model parameters such as tip radius are simply adjusted in software while scanning the reference sample to obtain the expected modulus value. The test sample is then imaged with the same parameter values. Results are most accurate when the reference and test samples have similar modulus. Loss modulus can be calculated by combining storage modulus and loss tangent, and tip-sample contact stiffness can be determined with use of the cantilever spring constant. In addition, maps of sample indentation (sometimes called deformation) are available for further insight.

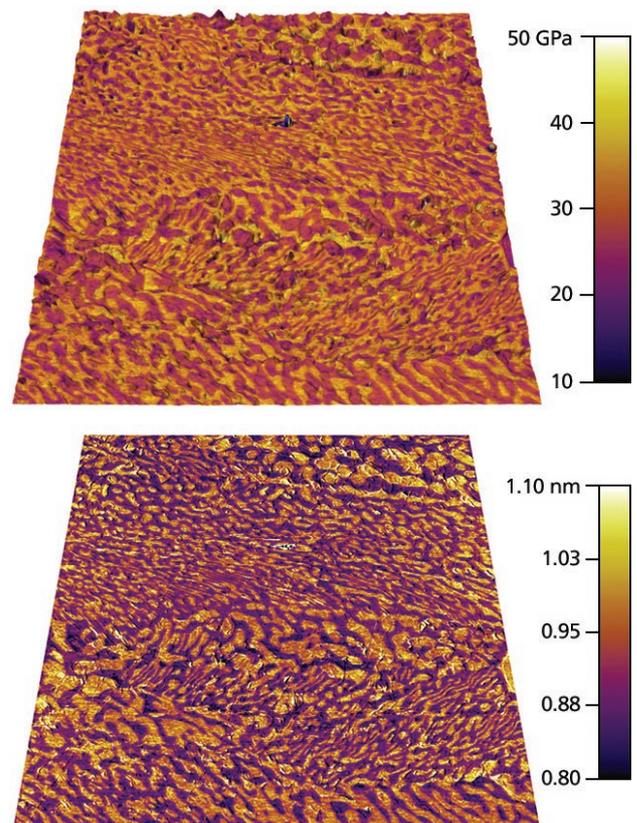
## Comparison to Other Techniques

AM-FM Mode is a widely applicable tool, but it is just one technique in Asylum’s NanomechPro Toolkit. You are encouraged to explore other options including Contact Resonance Viscoelastic Mapping Mode, instrumented indentation with the MFP Nanoindenter™ option, and force curves. Fast Force Mapping Mode is also available on MFP-3D Infinity™ AFMs. Comparing results from different methods is a valuable way to enhance measurement confidence.

Contact Resonance Mode is another exclusive Asylum technique for quantitative measurements of elastic and viscoelastic modulus. This makes it useful for direct comparison to AM-FM Mode. Both Contact Resonance Mode and AM-FM Mode exploit sensitive shifts in the cantilever’s resonance frequency to achieve high precision and accuracy. Contact Resonance Mode is best suited to materials with relatively high storage modulus (>1 GPa).

Force curves are a quasistatic indentation method for nanomechanical measurements. The sensitivity of force curve methods drops as the material modulus increases, so it is best suited for relatively soft materials. Also, image acquisition times with standard force curve methods are relatively slow. Fast Force Mapping Mode on Asylum Infinity MFP-3D AFMs overcomes this limitation with 300 Hz pixel rates. Like other techniques, force curves require analysis with contact models to achieve quantitative results.

Asylum’s MFP Nanoindenter option for MFP-3D AFMs gives true, ISO-compliant measurements of nanoindentation modulus. However, it makes much larger and deeper indentations than AFM techniques. This can decrease lateral spatial resolution and limit applicability to thin films.



(top) Storage modulus and (bottom) indentation depth overlaid on topography for tin/lead alloy solder. Tin-rich (yellow) and lead-rich (purple) regions can be identified in the storage modulus map. Scan size 23  $\mu\text{m}$ . Images acquired on a Cypher S AFM at 2 Hz scan rate with blueDrive photothermal excitation.

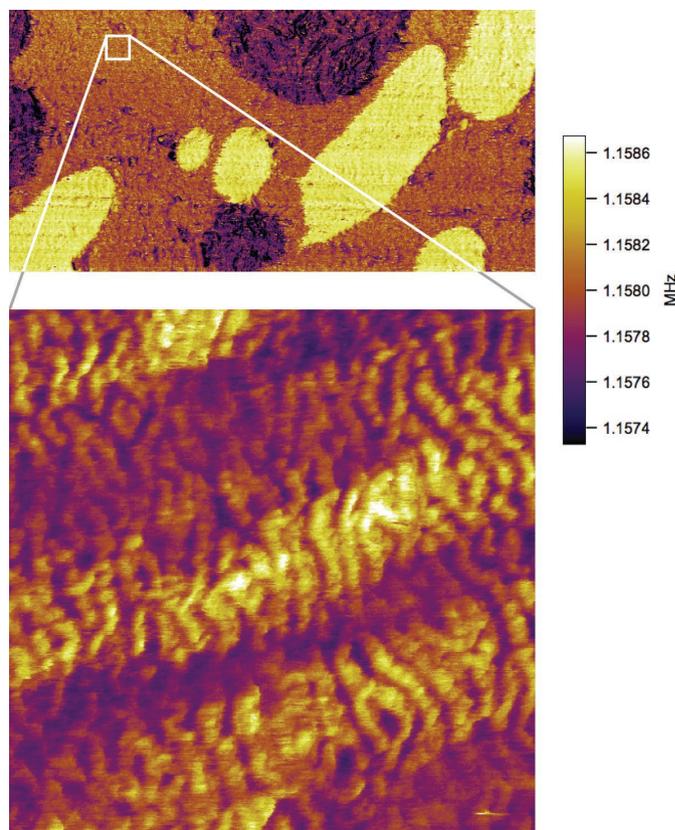
## Using AM-FM Mode on Your Asylum Research AFM

AM-FM Mode can be performed on all Asylum AFMs with standard software. ModeMaster™ simplifies AM-FM operation by automatically configuring the software and guiding you through the experiment. Setup time is minimized even more on Infinity MFP-3D AFMs with GetStarted™ software, which automatically sets tapping mode parameters. For absolute contact stiffness measurements with AM-FM Mode, cantilever spring constants are easily and accurately calibrated with GetReal™ software, free on all Asylum AFMs.

AM-FM Mode requires a cantilever excitation source with relatively flat response over a wide frequency range. These needs are met with the AM-FM Probe Holder for Cypher S and MFP-3D AFMs. Another option is the exceptionally clean and stable photothermal actuation of blueDrive, available on Cypher S and ES AFMs. Specialized cantilevers are not required with AM-FM Mode.

### References

1. Protected by certain U.S. patents assigned to or licensed by Oxford Instruments Asylum Research, Inc., including 8,555,711, 8,448,501, 8,024,963, 7,958,563, 7,603,891, and 7,921,466.
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Frequency images for a polypropylene-polyethylene-polystyrene (PP-PE-PS) blend. In the top image with scan size 8  $\mu\text{m}$ , regions of PS (yellow) and PE (purple) are clearly distinguished from PP (orange) despite relatively small differences in elastic modulus. The bottom image with scan size 300 nm shows the lamellar structure of PP, demonstrating AM-FM Mode's high spatial resolution and sensitivity. Sample courtesy of Dalia Yablon and Andy Tsou, ExxonMobil Research and Engineering, Corporate Strategic Research.

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