Contact Resonance Viscoelastic Mapping Mode

Asylum Research
an Oxford Instruments company

Introduction

Asylum Research's Contact Resonance Viscoelastic Mapping Mode option for the **MFP-3D**™ and **Cypher**™ **S** atomic force microscopes (AFMs) enables high resolution, quantitative imaging of both elastic storage modulus and viscoelastic loss modulus. It is just one of the many nanomechanical tools in Asylum's **NanomechPro**™ **Toolkit**. The contact resonance technique is particularly well suited for characterizing moderate to high modulus materials in the range of about 1GPa to 200GPa. Thanks to recent advances by Asylum and our collaborators, Contact Resonance Viscoelastic Mapping Mode is now faster, more quantitative, and easier to use than earlier implementations.

How it works

The contact resonance principle

The contact resonance technique, first developed in the 1990s by the Yamanaka and Arnold groups,[1,2] is based on the principle that the resonance of an AFM cantilever changes when it is in contact with a sample. As shown in Figure 1, the cantilever and sample in contact can be thought of as one spring coupled in series to a second spring and dashpot in parallel. Here, the first spring represents the elastic response of the cantilever and the second spring and dashpot represent the viscoelastic response of the sample. Therefore, as the stiffness of the sample contact changes, the frequency of the contact resonance changes (higher stiffness = higher frequency). Changes in the viscous response of the sample are reflected in the Quality factor (Q) of the contact resonance (more viscous = lower Q). Standard contact mechanics models can be used to then convert these stiffness and dissipation measurements to elastic modulus and loss modulus.

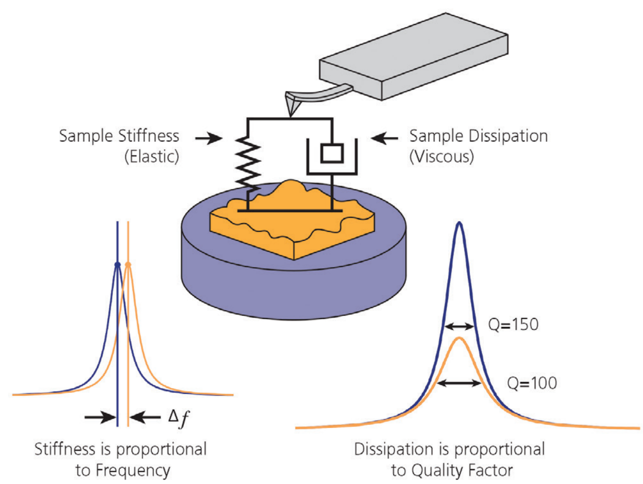

Figure 1: The cantilever-sample contact is modeled as a Kelvin–Voigt mechanical equivalent, where a spring represents sample stiffness and a dashpot represents sample dissipation. The frequency of the contact resonance is proportional to sample stiffness and the quality factor of the contact resonance is proportional to the sample dissipation.

Contact resonance imaging

The contact resonance technique is based on contact mode imaging. This means that the AFM cantilever scans along the sample surface at a constant force (the setpoint), measured by the cantilever deflection. As the tip encounters higher (or lower) features, the tip-sample force begins to increase (or decrease). A feedback loop continuously adjusts the height up (or down) to keep the force at a constant value (the setpoint). This motion is recorded as the sample topography.

While the tip scans the sample in contact mode, the contact resonance is continuously changing with the sample mechanical properties. In order to measure the contact resonance, a very low amplitude vertical modulation is introduced by driving either the cantilever or the sample. The drive is at a relatively high frequency, so this modulation does not affect the contact mode feedback loop, but the modulation nevertheless couples to the cantilever deflection and can be measured using a lock-in amplifier.

Contact resonance implementations

Various implementations of the general contact resonance technique have been developed, as summarized in the table on the next page. Two of these, Dual AC™ Resonance Tracking (DART) and Band Excitation (BE), will be discussed in greater detail in following sections. Note that it is also possible to make contact resonance measurements at single points rather than during continuous imaging or point-wise mapping, though this is less popular since the introduction of faster imaging techniques.

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Methods	What it does	Benefits	Disadvantages
Fixed frequency[2]	The cantilever response is measured at a fixed frequency, which varies as the contact resonance frequency shifts.	Simple to implement and produces elastic contrast images.	Produces only qualitative results since the frequency shift itself is not measured. Contrast is lost if the peak shifts too far from the selected frequency.
PLL frequency tracking[1]	A phase-locked loop (PLL) uses the phase of the cantilever response to track the contact resonance frequency.	The actual contact resonance frequency is tracked.	Difficult to tune the PLL to achieve stable frequency tracking due to spurious phase shifts in the response. Does not measure the Q of the resonance.
Frequency sweep (chirp)[3,4,5]	A frequency sweep (chirp) is done at each point. The cantilever response is Fourier analyzed to recover the full frequency response.	Measures the entire frequency response, so both the frequency and Q are obtained. Additional analysis is possible based on more complex models.	Mapping is quite slow when collecting large numbers of pixels. Each sweep must be done slowly enough for the cantilever to respond (rate limited by Q).
DART[6,7,8]	The amplitude and phase response at two frequencies (bracketing the contact resonance) is measured, which enables the contact resonance to be tracked.	Provides both the contact resonance frequency and Q. The tracking is extremely fast, so DART imaging can be done at normal imaging rates.	The full response is not measured, so analysis is more limited than frequency sweep or band excitation methods.
Band Excitation[8,9]	A continuous band of frequencies is excited. The cantilever response is Fourier analyzed to recover the full frequency response.	The entire frequency response is measured. By exciting the entire band at once, it is much faster than other full spectrum techniques (e.g. sweep).	Data transfer bandwidth limitations make the current implementation significantly slower than DART. Future speed improvements are possible.

DART

Fastest contact resonance frequency and quality factor imaging

Dual AC Resonance Tracking (DART) is an exclusive technology invented by Asylum Research in collaboration with Oak Ridge National Laboratory[10] that tracks the contact resonance as it shifts in response to changing material properties (see Figure 2). It provides both the frequency and the Q of the contact resonance, to characterize both stiffness and dissipation. It accomplishes this while operating at normal imaging rates or even at fast scanning rates with small cantilevers on the Cypher AFM. Figure 3 shows a DART contact resonance image of a 80/20 polypropylene / polystyrene blend. Since both the resonance frequency and the quality factor are measured with DART, we can detect differences in both the elasticity and dissipation of the two materials.

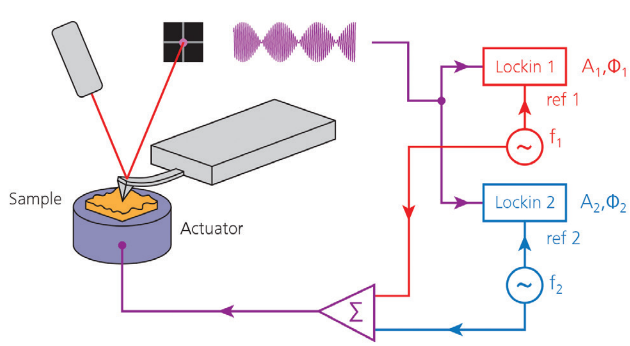

Figure 2: DART drives the actuator at two frequencies that bracket the contact resonance. By measuring the amplitude and phase of the cantilever response at both frequencies, the equivalent simple harmonic oscillator can be calculated, yielding both the contact resonance frequency and Q. The two frequencies are continuously adjusted to track the contact resonance peak.

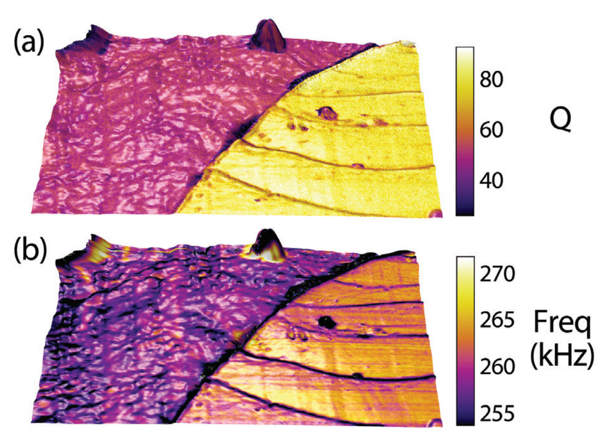

Figure 3: DART images of the PP/PS blend show high dissipation (Q) contrast, but much less stiffness (frequency) contrast. This is consistent with bulk measurements on PP and PS, which indicate that their elastic modulus is very close (2.8GPa vs. 2.4GPa) but their loss modulus is quite different (134MPa vs. 49MPa). Adapted from reference 7.

Band Excitation

Full spectrum contact resonance mapping

Band Excitation (BE) is a unique technology developed jointly by Oak Ridge National Laboratory and Asylum Research[10] and available exclusively on Asylum AFMs (see Figure 4). It enables faster acquisition of the full contact resonance spectrum at each pixel compared to sweep techniques. Though DART is faster in the present implementations and can provide both the contact resonance frequency and Q, it does not provide the full spectrum. This makes BE a complementary technique, which may be used to confirm DART results or to enable analysis with more complex models. Figure 5 shows one example of dissipation mapping with BE. Note that the pixel resolution is lower than normal in order to keep the acquisition time shorter.

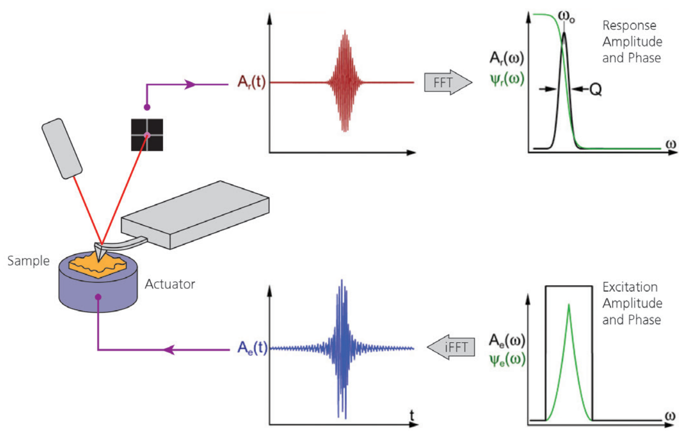

Figure 4: BE drives the actuator with a signal containing all frequencies in a continuous band. The cantilever response is Fourier analyzed to obtain the full contact resonance spectrum.

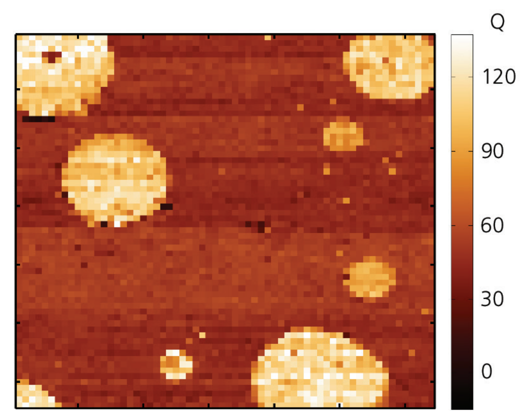

Figure 5: BE image of polystyrene spheres suspended in a polypropylene matrix. The Q image is shown, indicating strong contrast in dissipation between the two materials. Sample courtesy of D. Yablon and A. Tsou, ExxonMobil Research and Engineering, Corporate Strategic Research.

Contact resonance actuators

Contact resonance techniques have very stringent demands on the excitation source. The ideal actuator would have a flat response (free of resonances), a linear response (linear with drive), and a high bandwidth (up to 10MHz). Asylum has developed sample actuators (Figure 6A,B) that deliver the best performance that is commercially available. The actuators are highly damped, so the small deviations from an ideal, frequency independent drive are unlikely to interfere with quantitative measurements.

Sometimes it is not possible to actuate the sample. Large or oddly shaped samples may interfere with the response. Some accessories, like heaters, already take the place of the sample holder. For these cases, Asylum offers cantilever actuators (Figure 6C,D). Though based on the same technology as the sample actuators, design constraints limit the performance of these cantilever actuators. For this reason, the sample actuators are highly recommended if your experimental conditions allow.

Asylum's exclusive blueDrive™ photothermal cantilever actuation option for Cypher AFMs shows promising early results (Figure 7). Because it uses light instead of a mechanical actuator, the drive response is nearly ideal – flat, linear, and high bandwidth.

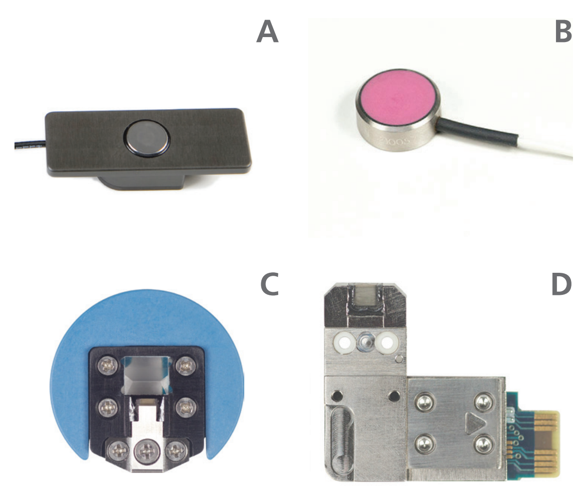

Figure 6: Contact resonance sample actuators for: (A) the MFP-3D, and (B) the Cypher S, and contact resonance cantilever actuators for: (C) the MFP-3D, and (D) the Cypher S.

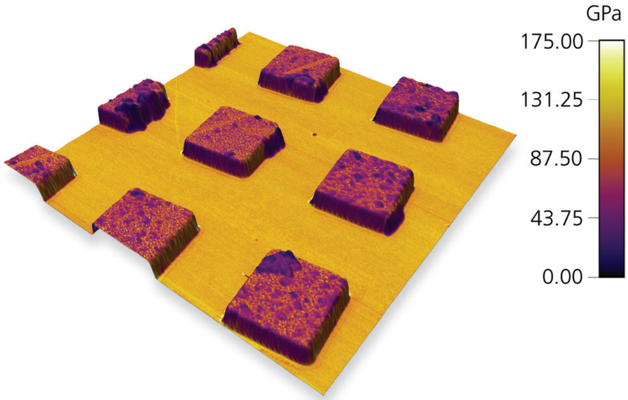

Figure 7: Elastic modulus image of a patterned titanium film on silicon, rendered on topography. blueDrive photothermal excitation on the Cypher AFM was used for DART contact resonance imaging. Note the strong contrast between these two very high modulus materials.

ModeMaster™ software interface

Simpler quantitative results

ModeMaster is an exclusive Asylum software feature that automatically configures the software for the desired measurement type and guides the user through the experiment. For DART contact resonance, the ModeMaster panel assists in setting up both the stiffness measurement and calibrating contact mechanics models using a reference sample with known modulus. This makes it possible to rapidly and simply start making measurements, all based on well-established methods,[7] but without the full complexity. Experts are, of course, free to delve into as many advanced operational and analysis options as they like.

Where does contact resonance fit in with other techniques?

Contact resonance is just one tool in the NanomechPro Toolkit. One should understand and explore the full range of options, including AM-FM Viscoelastic Mapping Mode, instrumented nanoindentation, and force curves. Asylum believes that the ability to compare results from multiple techniques is valuable and adds confidence to the results.

AM-FM Viscoelastic Mapping Mode is another exclusive Asylum technique for quantitative nanomechanics. Like contact resonance methods, AM-FM can measure both the elastic and loss modulus over a very wide range. This makes it a useful direct comparison to contact resonance results.

Asylum's MFP NanoIndenter™ allows users to readily switch between true, ISO-compliant nanoindentation modulus measurements and AFM techniques. However, it makes much larger indentations than AFM techniques, which limits its applicability on thin films and limits it to relatively coarse lateral resolution for modulus mapping applications.

Force curves, measured individually or with force mapping, are also widely used for indentation measurements. However, on moderate to high modulus materials the curves rapidly begin to approach the constant compliance case that one would measure on an infinitely stiff sample. That is, the difference in contact slope in the two cases becomes vanishingly small and the uncertainty grows large. In contrast, the frequency shift associated with contact resonance can be measured with far greater accuracy and precision.

References

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10. Protected by certain U.S. patents assigned to or licensed by Oxford Instruments Asylum Research, Inc., including: 8,448,501; 8,024,963; 7,603,891; 8,448,502; 7,775,086

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