Fast Force Mapping (FFM)

OXFORD INSTRUMENTS

ASYLUM RESEARCH

Atomic force microscopy (AFM) is mainly known for its ability to measure high resolution 3D topographical images. Due to its inherent spatial resolution and force sensitivity, AFM can also measure a myriad of nanomechanical properties such as hardness, adhesion and viscoelasticity. Nanoscale mechanical properties impact the behavior of many materials and are useful in both theoretical understanding and commercial deployment of such materials. However, the diversity of material properties limits any single AFM technique from providing the most relevant and accurate data for every application. Asylum Research provides multiple techniques to measure a broad spectrum of nanomechanical properties, providing flexibility for different samples and greater insight through comparison of results. Here, we discuss the nanomechanical technique known as Fast Force Mapping (FFM).

Nanomechanical Imaging

Imaging modes that provide a samples nanomechanical properties can be divided into two main types: 1) force curve-based modes such as force curves, force volume and fast force mapping and 2) tapping mode-based modes which include bimodal imaging and AM-FM. Both types of modes excel depending on the type of sample and material properties of interest. See table 1 for a summary of the differentiating properties for each mode.

Force Curve-based Imaging Modes

Force curves are a well-known method of probing surface forces. During a force curve, the AFM tip approaches the sample until it contacts the surface. Once in contact, the tip indents the sample until a trigger threshold is reached. The tip is then withdrawn until it loses contact with the sample. The measured cantilever deflection versus Z sensor position during this load-unload sequence is converted to a force curve with measurements of the cantilever sensitivity and spring constant. The Asylum Research GetReal™ proprietary software automatically calibrates, the cantilever spring constant and deflection sensitivity via a single mouse click.

Force curves are relatively simple to perform and data interpretation for material properties is a well understood curve fitting process based on several theoretical models that have been widely used and documented.

Force curves can also be acquired in 2D arrays called force maps or force volumes to provide visual representation of the sample surface and its mechanical properties.

Although force curves and force volume mapping are powerful techniques for correlating functional and structural information, both can suffer from slow acquisition times.

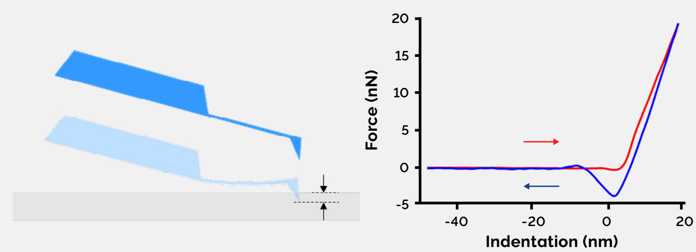

Figure 1: Schematic representation of sample indentation and the corresponding force curve (approach in red and retraction in blue).

Asylum Research Fast Force Mapping (FFM) Mode

Fast Force Mapping Mode, FFM, which has been designed to overcome the relative slowness associated with traditional force curve-based acquisition, can acquire force curves at much faster ramp rates, depending on the AFM, and reach up to 2.5 kHz on Jupiter XR AFMs. As with regular force curves, during FFM, the distance is always measured using the Z sensor to provide accurate distances without worrying about piezo calibration.

Using a continuous sinusoidal motion from pixel to pixel (Figure 2), Fast Force Mapping Mode can capture a complete array of force curves in just a few minutes.

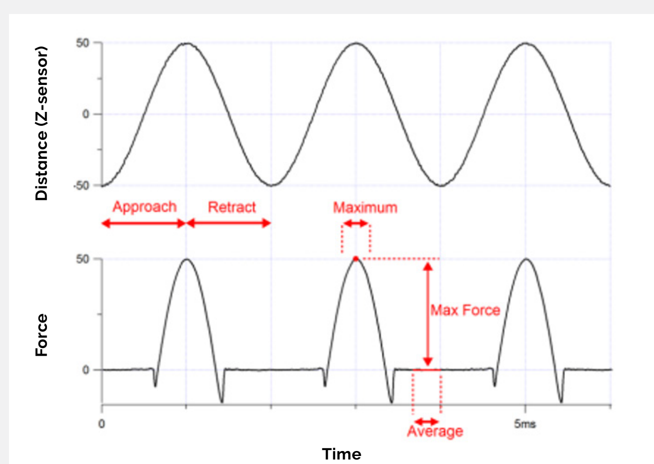

Figure 2: FFM sinusoidal force curves.

For example, a 256 points × 256 lines image of standard force curves takes over 18 hours to acquire at 1 second per pixel. By contrast, it takes less than 10 minutes with the Asylum Research Fast Force Mapping Mode. Such dramatically reduced acquisition times enable images with higher pixel density, for improved lateral resolution.

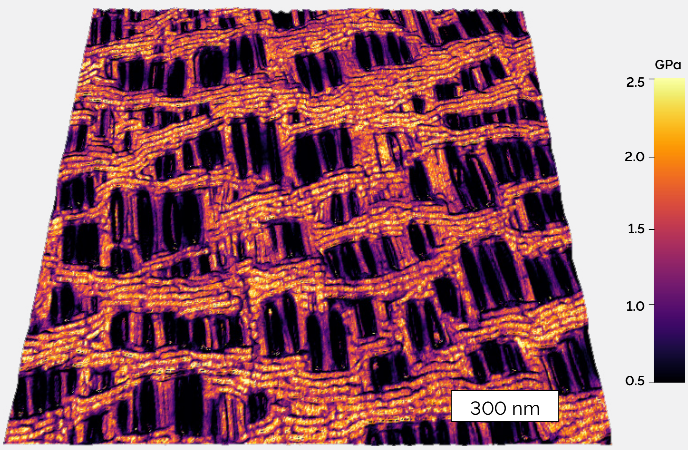

Figure 3: FFM of Celgard© polypropylene membrane. 512x 512 pixels, acquisition time: 18 min.

Figure 3 shows FFM of Celgard© polypropylene sample where modulus is overlaid on topography, features as small as 20 nm are resolved.

Fast Force Mapping – Flexibility to Choose and Customize Different Theoretical Models

Quantitative data on mechanical properties, including elastic modulus, hardness, and adhesion, are obtained from AFM force curves by fitting the experimental data to a model for the tip-sample contact.

The diversity of materials and many possible experimental conditions means that no single model can correctly apply to all samples. For this reason, Asylum Research software provides a variety of analysis models that offer user-friendly flexibility and convenience. In addition to the standard Hertz/ Sneddon and Derjaguin-Muller-Toporov (DMT) models, Johnson-Kendall-Roberts (JKR) and Oliver Pharr models add the possibility of modelling adhesion inside the contact area and plastic deformation, respectively. Moreover, various parameters in each model can be modified from the main software interface, while the software's open architecture makes all routines accessible for customization, if desired.

The Asylum Research software also has an integrated Model Selection Guide, a helpful tool which, based on several force curve parameters (calculated plasticity factor, force ratio and Tobor coefficient) suggests the model may best describe the data.

Asylum Research's FFM software automatically saves all the force curves performed during imaging, offering the customer the possibility to analyze the nanomechanical data post acquisition. This capability adds flexibility to data analysis so that many models can be used to analyze the same data set, while parameters, such as tip radius, can be re-adjusted post data acquisition.

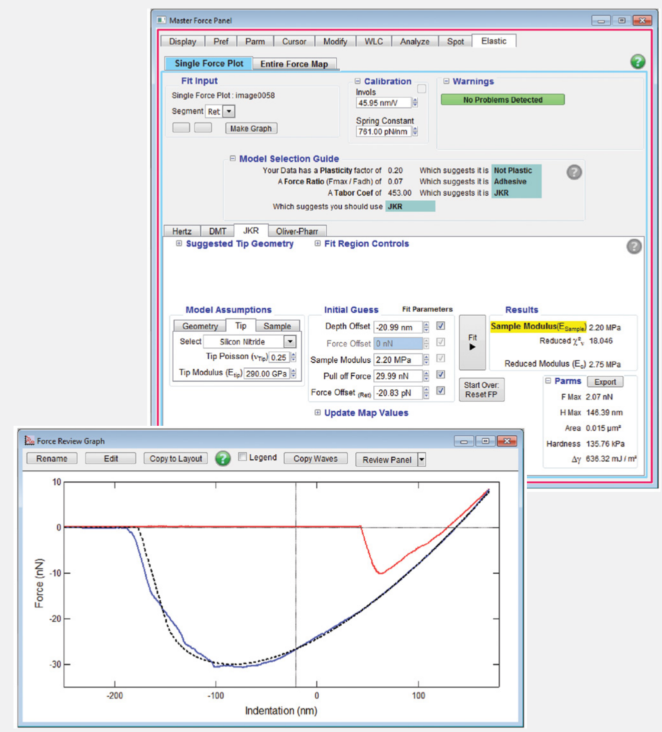

Figure 4: Elastic Panel with various analysis models and a force curve fit.

Fast Current Mapping

Fast force mapping can also be used in conjunction with conductive mode imaging resulting in fast current maps (Figure 5), where topography, nanomechanical information and the current is acquired simultaneously.

The benefit of acquiring conductive data in fast current mapping mode lies in the intermittent tip-sample contact. During regular conductive mode imaging, the AFM tip is in constant contact with the sample which may displace a weakly attached sample and dull the tip faster, which in turn will reduce resolution. Fast current mapping does not perturb samples as

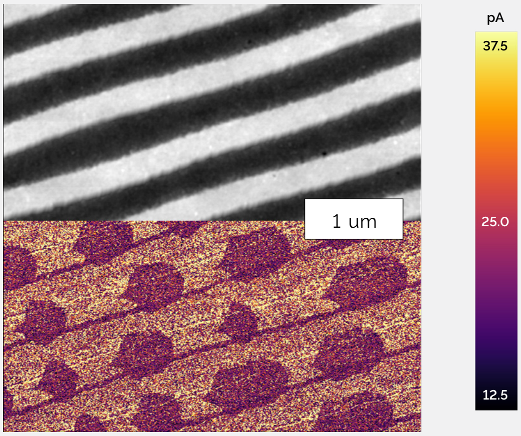

Figure 5: DVD imaged in fast current mapping mode. Top half of the image shows sample topography and the bottom half show the current signal.

much, since the tip “jumps” from one pixel to the next during data collection. Carbon nanotubes deposited on an electrode are challenging to image in standard conductive mode, however, they can be imaged effortlessly and with high resolution in fast current mapping mode (Figure 6).

High resolution imaging in FFM

Both tapping mode-based and force curve-based nanomechanical modes can produce complimentary high resolution data. In the case of FFM, the resolution is closely related to the control of the force feedback. The greater the force control, the smaller the indentation which results in gentle probing of the surface. In figure 7, molecular level resolution was achieved: darker (softer) areas of the sample correspond to the amorphous regions of the sample and the brighter (stiffer) region correspond to the highly ordered (aligned), crystalline regions of the sample.

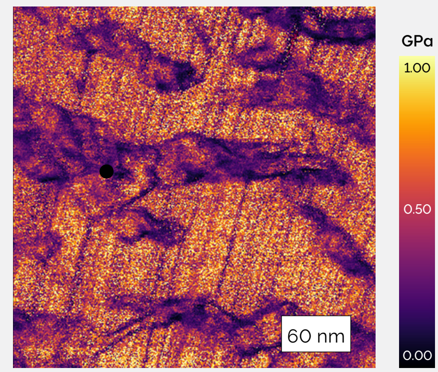

Figure 7: Teflon sample. Modulus is higher on the regularly aligned regions (crystalline) and is lower on the disordered regions (amorphous) (acquisition time: 6 min).

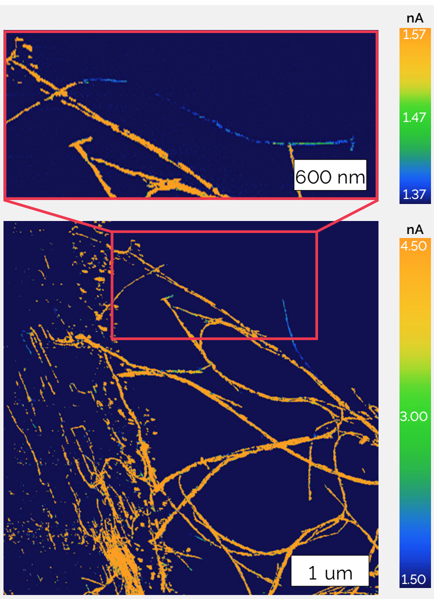

Figure 6: Fast current map of carbon nanotubes on an electrode. Inset shows a single nanotube.

Advantages of Asylum Research FFM

- Every force curve in the image is captured and saved automatically with no hidden data manipulation providing the user the flexibility of data analysis after acquisition.
- The distance is always measured using the Z sensor to provide accurate results and eliminating the need for Z piezo calibration.
- Force measurements are limited only by Brownian (thermal) motion. Combined with its ultra-low-noise Z sensors, this means that both axes of force curves are measured with the highest possible sensitivity and accuracy.
- Realtime and offline analysis models can be applied to calculate modulus, adhesion, and other properties. Models, including JKR and Oliver Pharr are fully accessible for user inspection or modification, allowing for ultimate analysis flexibility.
- The software-integrated Model Selection Guide simplifies data analysis by suggesting which model best matches the data.
- GetReal™ proprietary software automatically calibrates the cantilever spring constant and deflection sensitivity of the probes.

Table 1: Advantages of Asylum Research FFM.

Comparison and summary nanomechanical modes

Table 2 shows a summary of various imaging considerations together with advantages and disadvantages of force curves-based FFM mode in comparison to tapping mode-based modes such as AM-FM. Depending on the type of sample (thick, thin, sticky) and the nanomechanical properties of interest (adhesion, modulus, viscoelasticity) both modes are complimentary. Asylum Research understands the intricacies of nanomechanical characterization and provides solutions that take into account the sample types, user needs and scientific integrity.

Conclusions

Asylum Research Fast Force Mapping mode is a nanomechanical imaging mode that offers unique benefits and when coupled with other nanomechanical modes, such as AM-FM, provides a comprehensive suite of tools for a wide range of samples.

Considerations	FFM	AM-FM
Speed of image acquisition	Generally slower	Fast
Quantitative sample properties	Yes, fitting of indentation force curves	Yes, mathematical analysis of tapping mode signals
Sample thickness allowed due to indentation depth requirements	Generally best on thicker samples to reduce surface effects (large indentation depths)	Thin and thick samples can be imaged (indentation depth is very low, < 1 nm)
Resolution	Indentation depth vs. imaging resolution trade-off	Highest resolution is achieved in tapping mode techniques
Sample types especially well-suited for the technique	Sticky samples, high aspect ratio samples, weakly bound samples	Small features that require molecular resolution, high modulus samples
Properties measured	Adhesion, hardness, Youngs modulus, current (in Fast Current mapping)	Loss tangent, Youngs modulus stiffness, dissipation

Table 2: Imaging considerations of force curve-based vs. tapping mode-based imaging modes.