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A brief overview of ImAFM
with some applications

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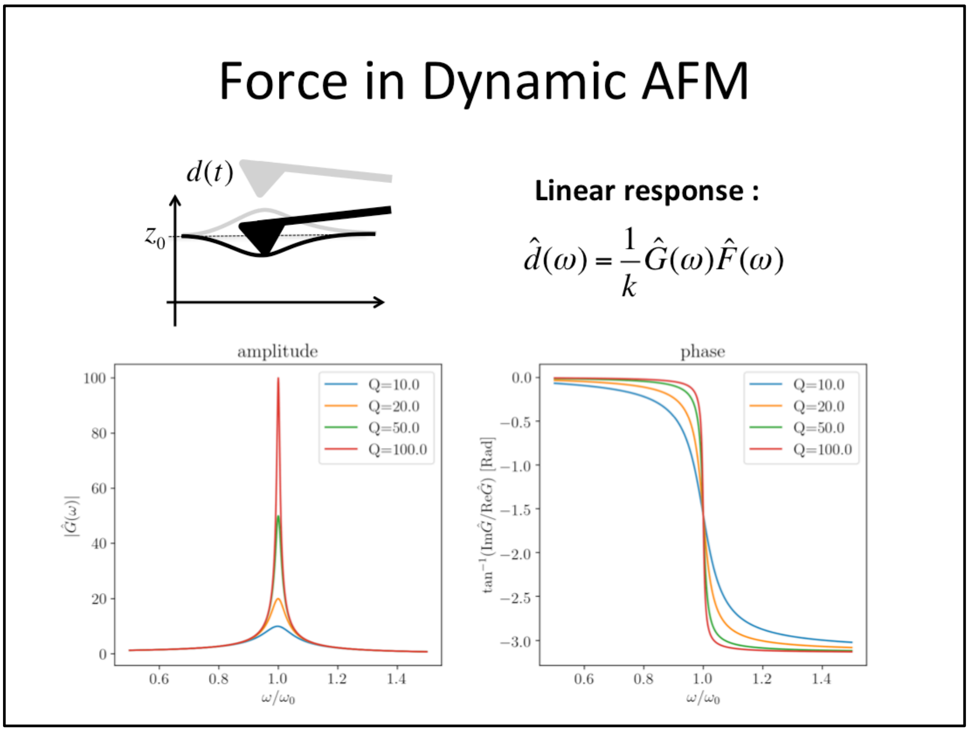
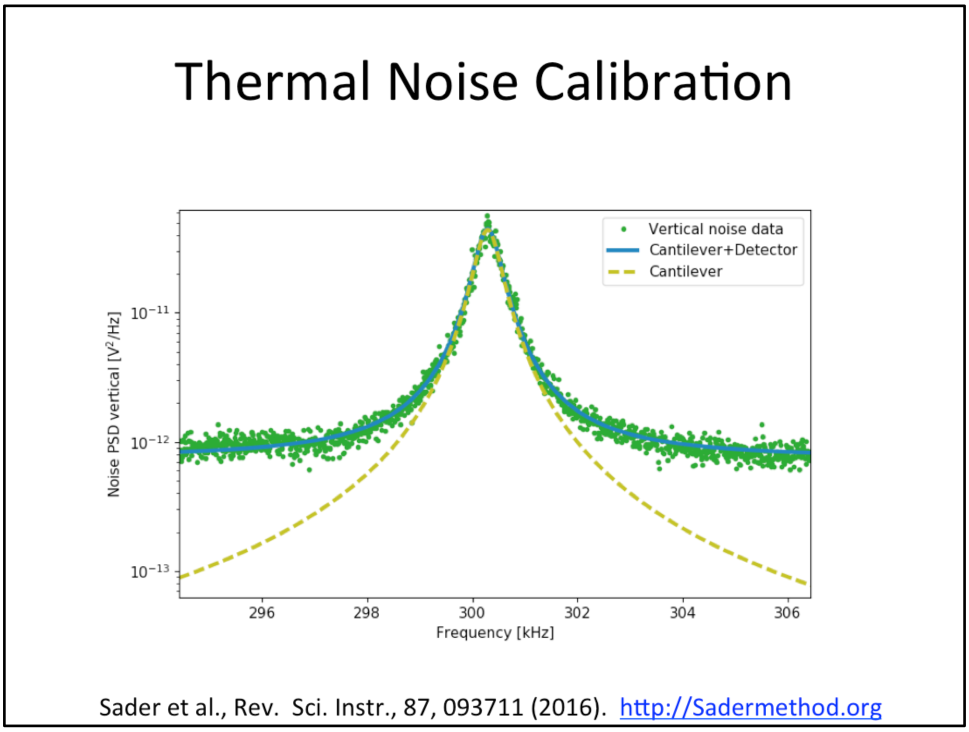
The slides give a brief overview of Intermodulation AFM with examples of some of its applications.

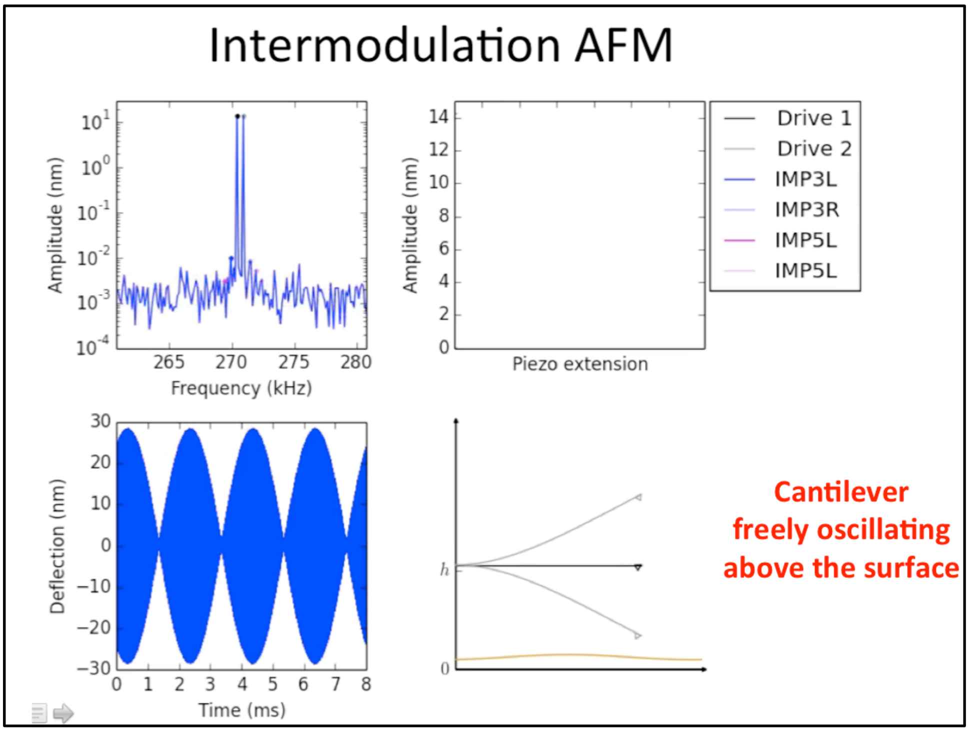


The cantilever is a linear transducer of force. In the frequency domain there exists a linear response relation between Fourier components of the Force (F 'hat') and deflection (d 'hat'). In the narrow frequency band around a mechanical resonance, the linear response function of the cantilever (G 'hat' / k) is very well approximated by a simple harmonic oscillator model. The transfer gain (G 'hat') is very large on resonance, giving a factor Q enhancement, or the responsivity of the force transducer.

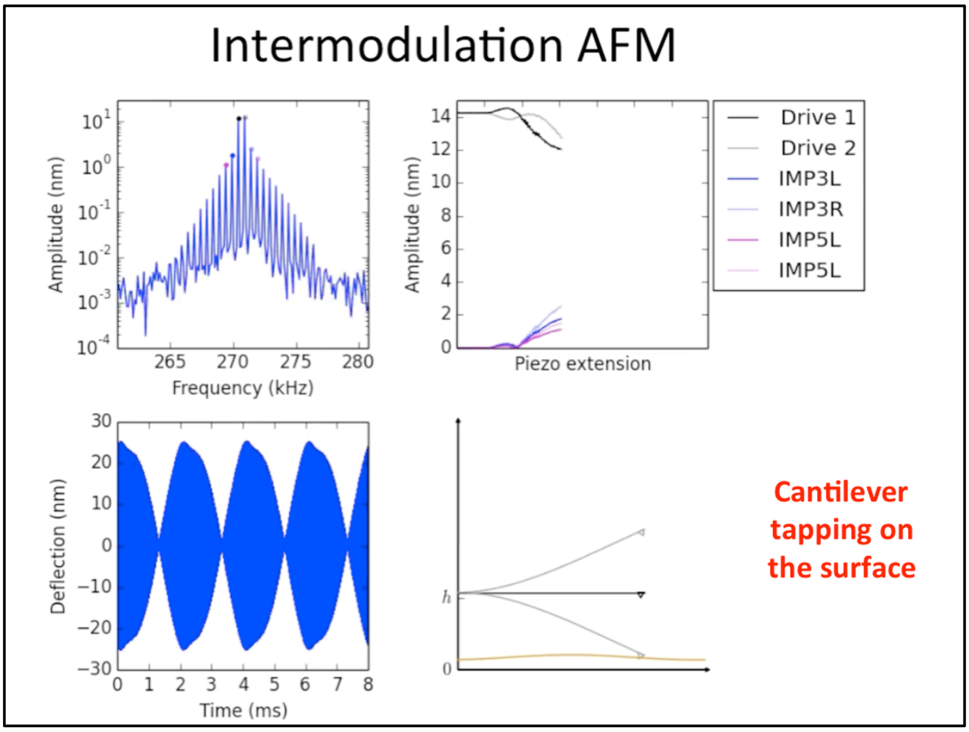


This enhanced responsivity near resonance means the the force has increased sensitivity. Near resonance we can see the net effect of many random force pulses of individual molecules of air hitting the cantilever, giving rise to the thermal Brownian motion of the cantilever. In this regime, the sensitivity of the measurement is limited only by the thermal noise. ImAFM works in this regime by measuring many frequency components of the force, all near resonance.

The best method of calibration is a dynamic method, allowing you to get the full linear response function of the cantilever. Using a theory of hydrodynamic damping, together with the relationship between damping and fluctuation force (fluctuation-dissipation theorem), we can fit a measurement of Brownian motion to calibrate the linear response function of the cantilever.

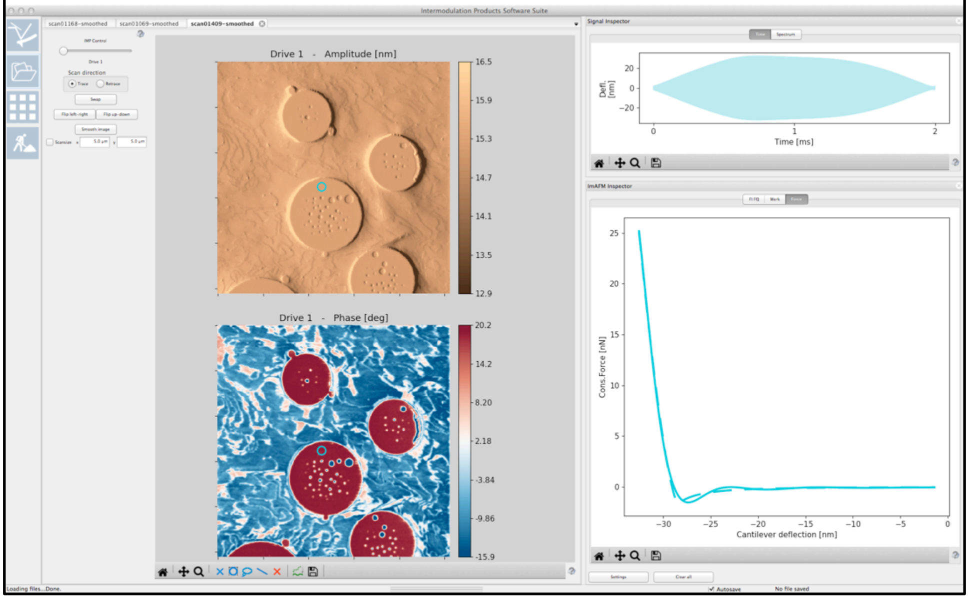


After calibration, the cantilever is driven with two pure tones at closely spaced frequencies, centered on resonance. The frequency separation of the tones is roughly the width of the cantilever resonance. In the time domain this type of drive gives a 'beating' waveform.



When the sample is engaged and the tip starts to interact with the surface, the nonlinear nature of the oscillator causes strong intermodulation (frequency mixing) of the two drive tones, producing many intermodulation products (mixing products) of the two drive tones. A special multifrequency lockin technique is used to measure amplitude and phase at these many frequencies at the same time, or in the same time window corresponding to one beat period.

Reconstruct force curve from intermodulation spectrum (amp. + phase) – at each pixel

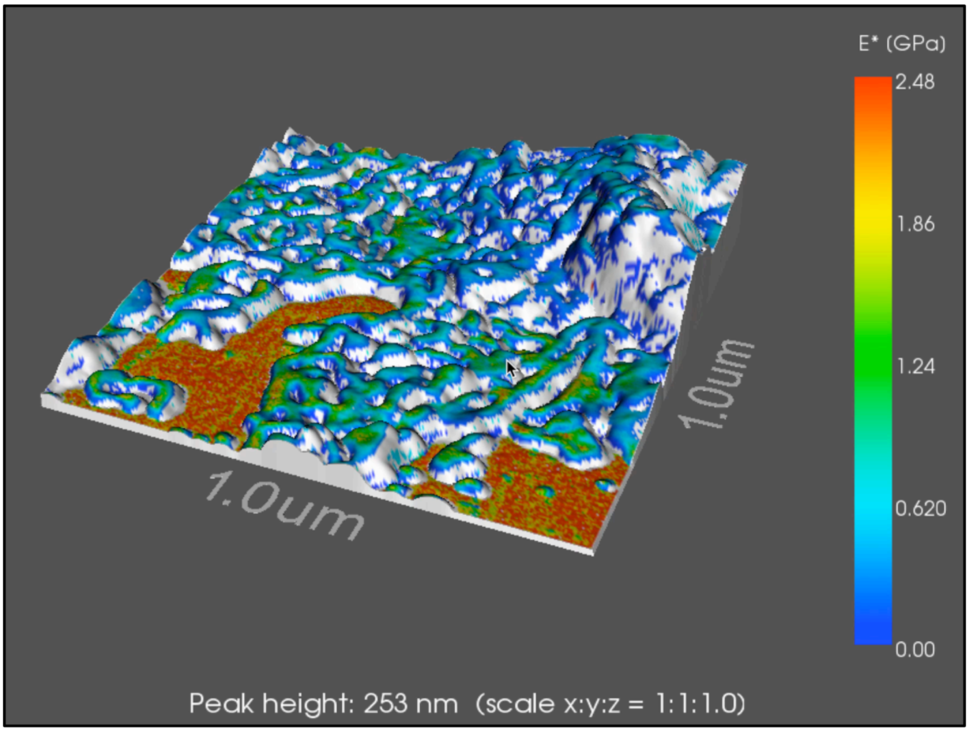


It is possible to reconstruct the tip-surface forces (the nonlinear part of the oscillation) using different mathematical methods. Here we show two examples. At the pixel marked with a small blue circle in the center of the scan, we find the best-fit polynomial which describes the force curve (solid line) and we also fit it to a DMT model (dashed line).

You can point and click on the image and instantly see force curves at that pixel – even while scanning in the image.

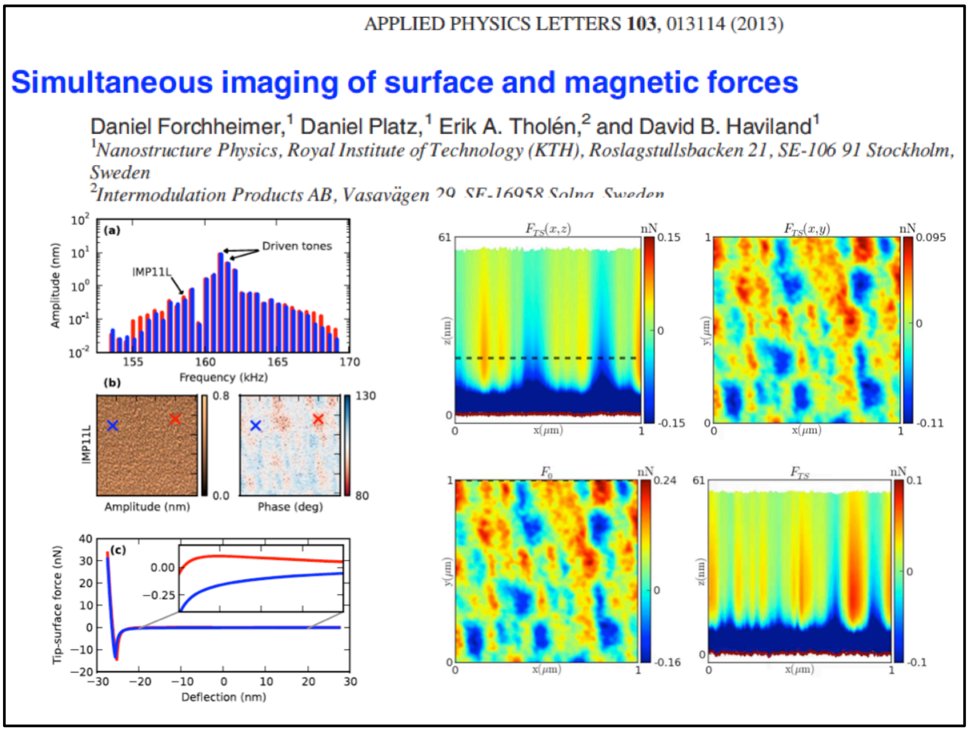
The scan speed is typical for tapping mode AFM (2ms per pixel, 1 sec per scan line @ 256 pixels/line, 4.2 minutes per image @ 256 scan lines.)

This sample was a blend of Polystyrene (PS) and Polycaprolactone (PCL). The PS forms more solid islands (red) in a sea of PCL (blue).



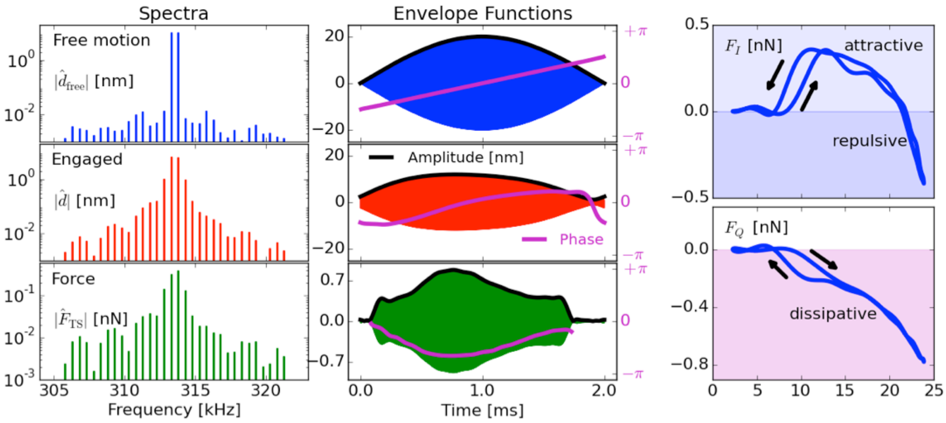
Fitting the parameters of a model to every pixel of an image, we create color coded maps of the parameter values. Here we see the Youngs modulus, found by fitting the DMT model to the data. The parameter maps can be projected on to a 3D rendering of the surface topography. The software has IMP Software Suite has functionality for all this fitting and displaying of the data, including animating the 3D image by rotation about the azimuth.

The sample is an electro-sprayed polymer material on a Si substrate. The white shade on the steep side-slopes are regions where the model could not be fit reliably.



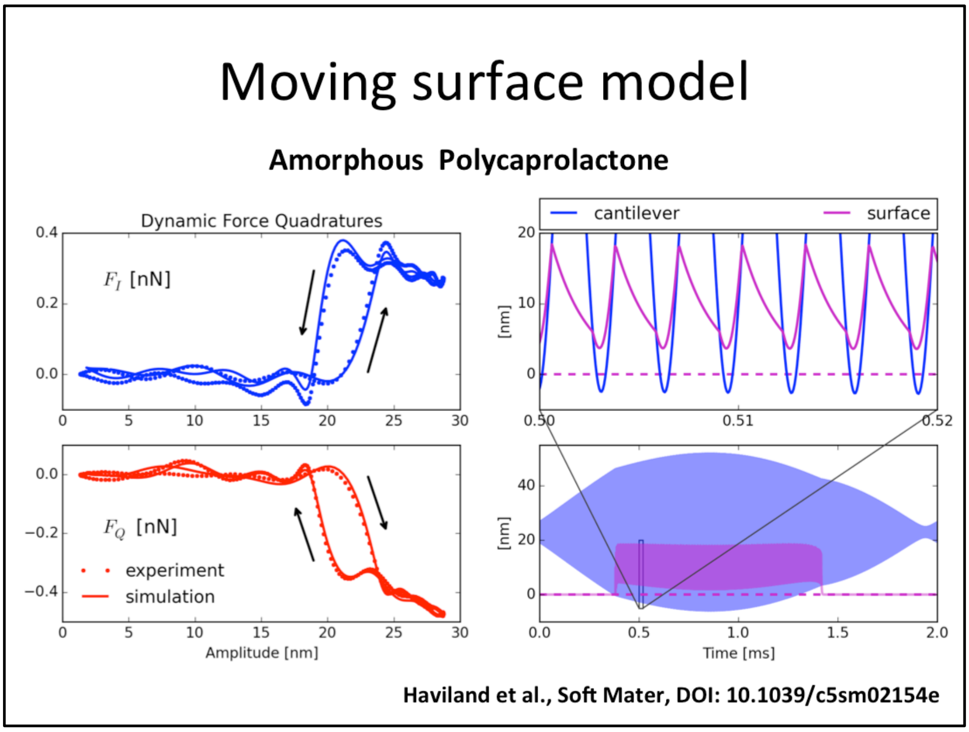
Here we explore a commercial hard disk with a magnetic tip. We constructed a force model that includes both short-range surface forces and long range magnetic forces. Processing the data offline we extract a force – volume data set, F_z(x,y,z). The movies show different 2D cuts of the data set. One can see how the magnetic forces appear at larger distances.

Model-free dynamic force curves



Dynamic Force Quadratures are a new way of looking at tip surface forces. They give the integrated force on one fast oscillation cycle of the tip, as the amplitude of this fast oscillation is slowly modulated. The curve FI(A) is the integrated force which is in-phase with the motion, and this tells us about the conservative part of the interaction. The other quadrature FQ(A) is in phase with the velocity, giving the dissipative or viscous part of the interaction. These curves are determined without using a force model. They are a direct transformation of the measured multifrequency response.

Through analysis of the force quadrature curves we gain a deeper understanding of the viscoelastic nature of the tip-surface interaction. For example, the hysteresis in the force quadrature curves can be understood to be the result of the finite relaxation time of the viscoelastic surface. Fitting these curves to a a new type of moving-surface model, we can extract this relaxation time.



When tapping on soft materials, the surface can be significantly lifted due to the adhesion force. After the tip releases the surface, the viscoelastic surface requires a finite amount of time to relax back to its equilibrium position. If the oscillation is rapid enough such that the tip hits the surface before it can fully relax, it will result in an time-average lifted position surface. The moving surface model takes in to account the dynamic oscillations of the surface, allowing for extraction of the viscoelastic time constants of the surface and bulk. .

The sample is amorphous Polycaprolactone. Simulations were fit to the experimental data with the following parameter values.

surface stiffness ks = 5.130e-02[N/m]
surface time constant eta_s / ks = k/(w0 ks Qs) = 1.837e-06[s]
bulk stiffness km = 1.648e-01[N/m]
bulk time constant eta_i / km = k/(w0 km Qi) = 6.983e-07[s]

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Imaging high-speed friction at the nanometer scale

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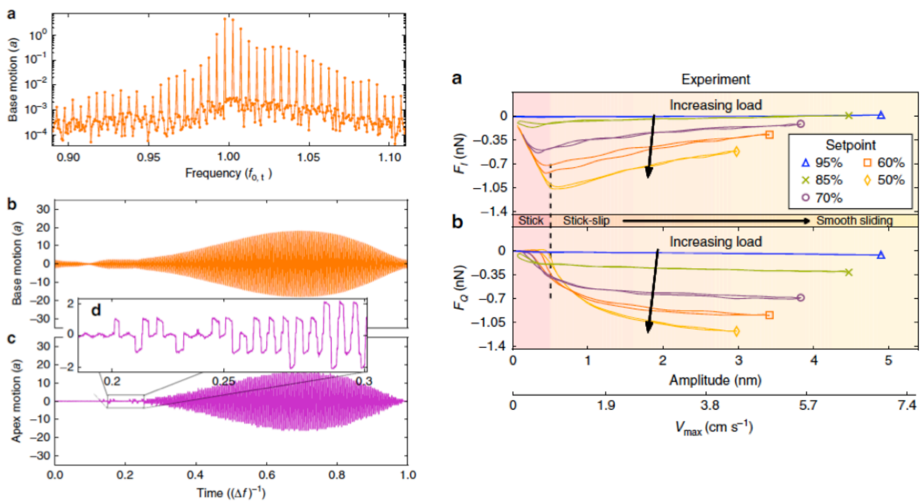
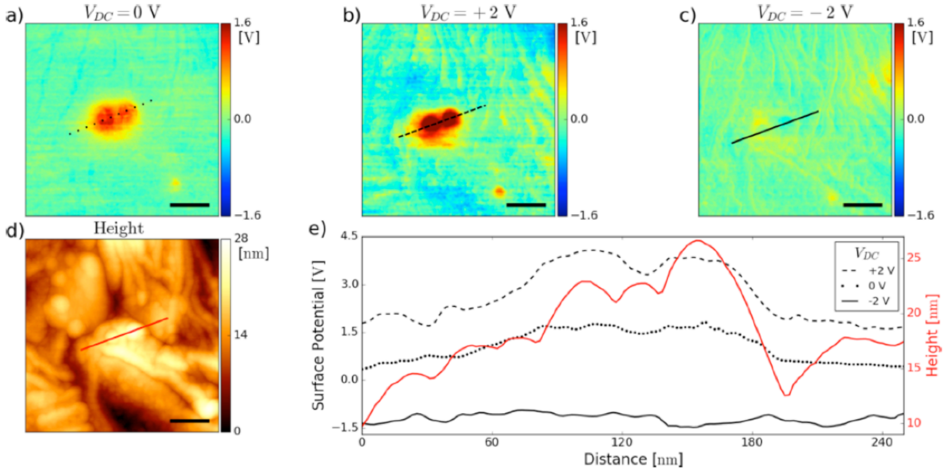


The intermodulation method also works with torsional resonance, allowing for the measurement frictional forces at very high speed. Although the tip slides on the surface with very small oscillation amplitude (5 nm), the maximum velocity can be very high (cm/sec.) due the the high frequency of torsional resonance. The force quadrature curves show the conservative and dissipative forces due to friction. We observe a crossover from static to free sliding friction which depends on strength of load force.

Intermodulation Electrostatic Force Microscopy (ImEFM)

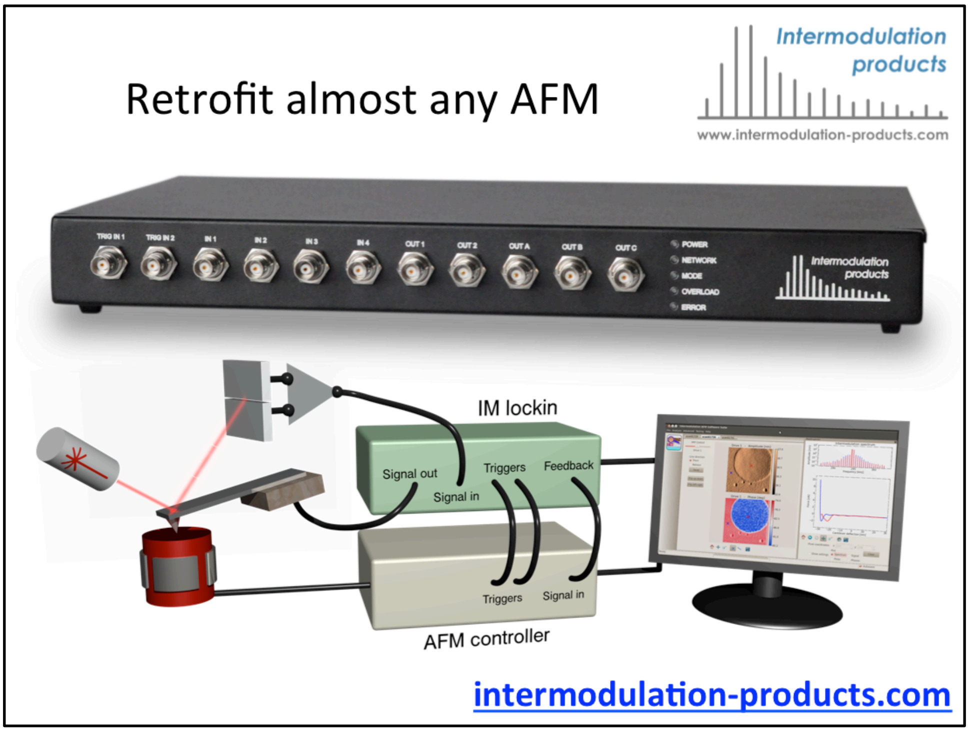
Measure surface potential for different DC bias on the tip



Borgani et al. Nano Lett. 16, 5934 (2015)

ImEFM is a multifrequency method for extracting the surface potential (Borgani et al. Appl. Phys. Lett. 105, 143113 (2014)). The method works without voltage feedback, allowing one to study how the surface potential changes as a DC, or 'gate' voltage is applied to the tip.

The images above shows AlOx nanoparticles in a Polyethylene matrix. The particles are charged and discharged by the tip, depending on the sign and magnitude of the DC voltage. With this method we could verify that the nanoparticles create hole traps in the insulator. This nano-composite material is interesting for electrical insulation in high voltage DC transmission cables.



Intermodulation Products sells a kit which to retrofit to nearly any AFM. The kit consists of a special multifrequency lockin, and a complete software suite for cantilever calibration, measurement by the various modes of ImAFM, on-line, and a host of different analysis and plotting methods. The system does not require an expensive upgrade of the AFM controller, and it works very well on older DI Multimode and Dimension systems.