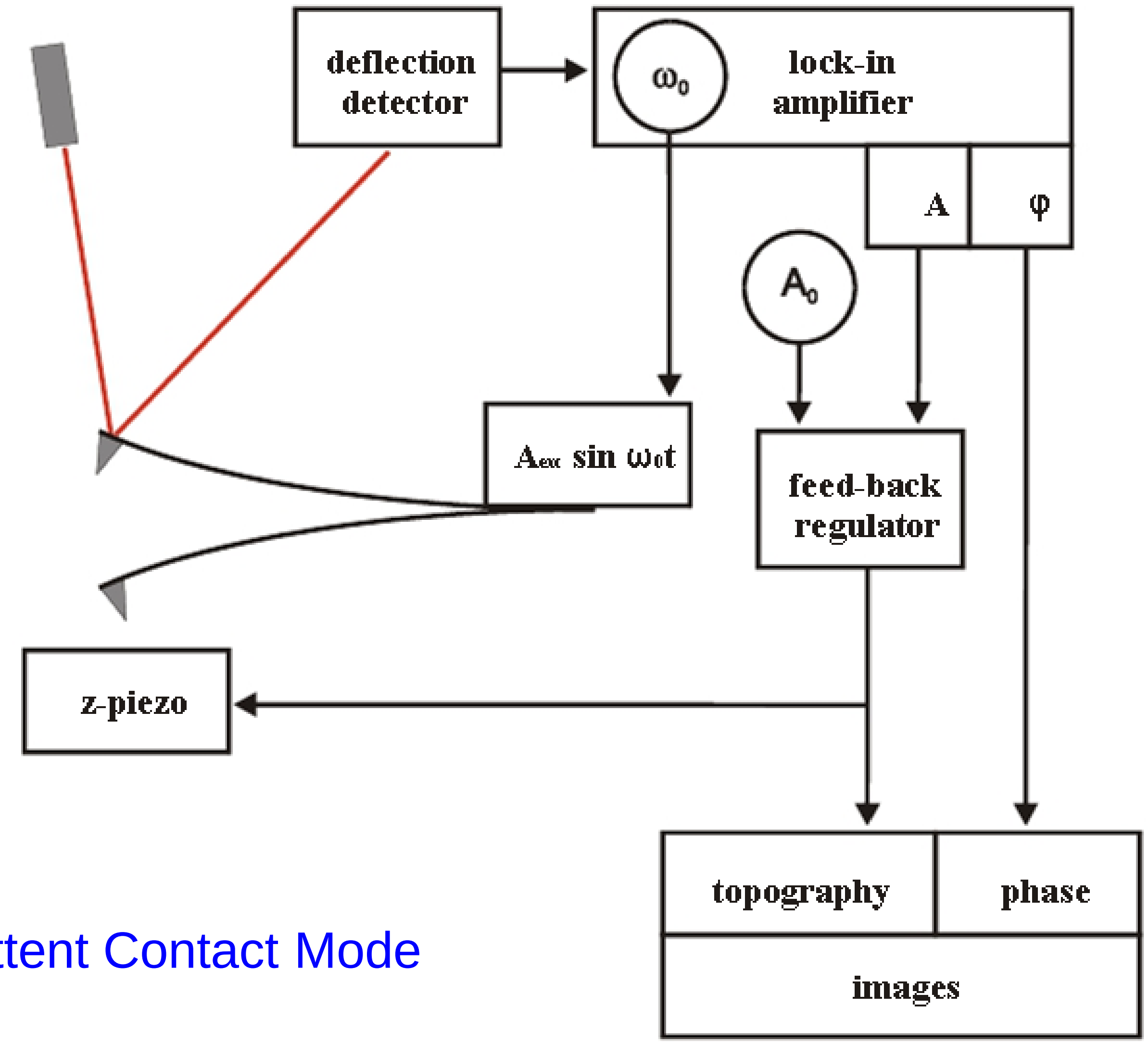
Intermittent Contact Mode SFM
Tapping Mode
Pulsed Force Mode SFM



Setup for Intermittent Contact Mode

Principles of intermittant Contact Mode

• The tip is oscillated with an amplitude of 20 to 100 nm close to the cantilevers resonance frequency, and the root-mean-square value of the amplitude is used as regulation parameter for the tip-sample distance.

• The amplitude is reduced due to an intermittent contact during each cycle and (*tapping mode)*

• The advantage is greatly reduced lateral force (compared to the *contact mode)* while the resolution is similarly limited only by the tip shape.

• Large oscillation amplitudes or relatively hard cantilevers are prerequisite in *tapping mode* in air to overcome attractive capillary forces by the restoring force of the cantilever spring.

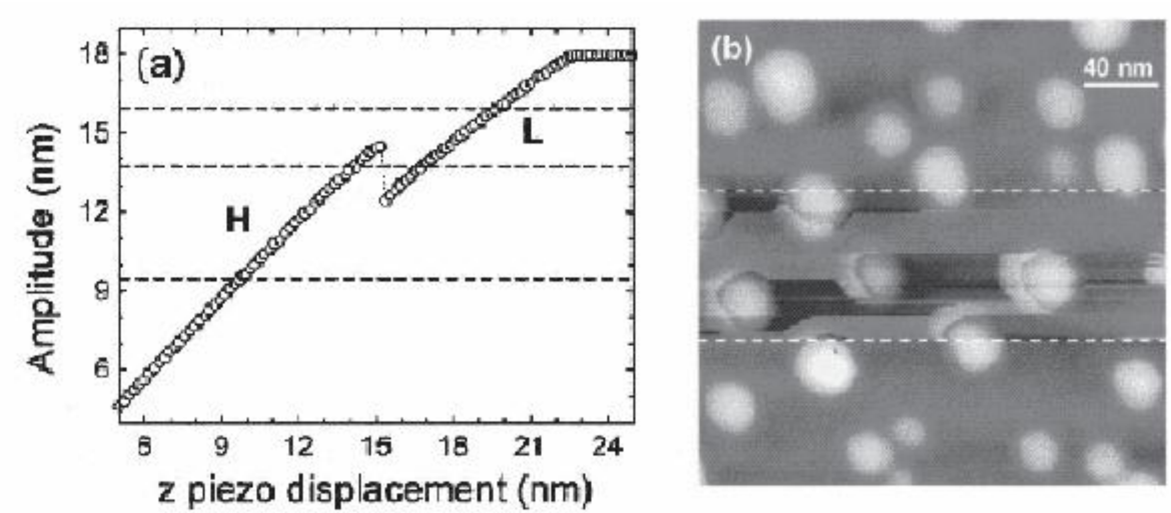
• If the detection of the oscillation amplitude is done with a lock-in amplifier additional information can be obtained from the phase shift between excitation and oscillation of the cantilever.

• The use of the harmonic approximation can be justified by experimentally verifying that the tip oscillation is staying sinusoidal. In this case, the phase shift can be related to the tip-sample energy dissipation normalized to the internal cantilever dissipation.

• Generally, phase imaging can resolve material inhomogeneities at surfaces in great detail, however, a quantification is often hampered by non-linearities.

• The distance dependence of the tip-sample force is highly non-linear, particularly when the tip comes into repulsive contact with the sample. It is a basic of mechanics that for such non-linear systems more than one state of oscillation can be stable. The amplitude detected in *tapping mode* force microscopy can show abrupt jumps for certain sets of experimental parameters. Consequently, artifacts like sudden height jumps or changes in the overall contrast appear in topography images.

Instabilities of intermittant Contact Mode - Scanning Force Microscopy



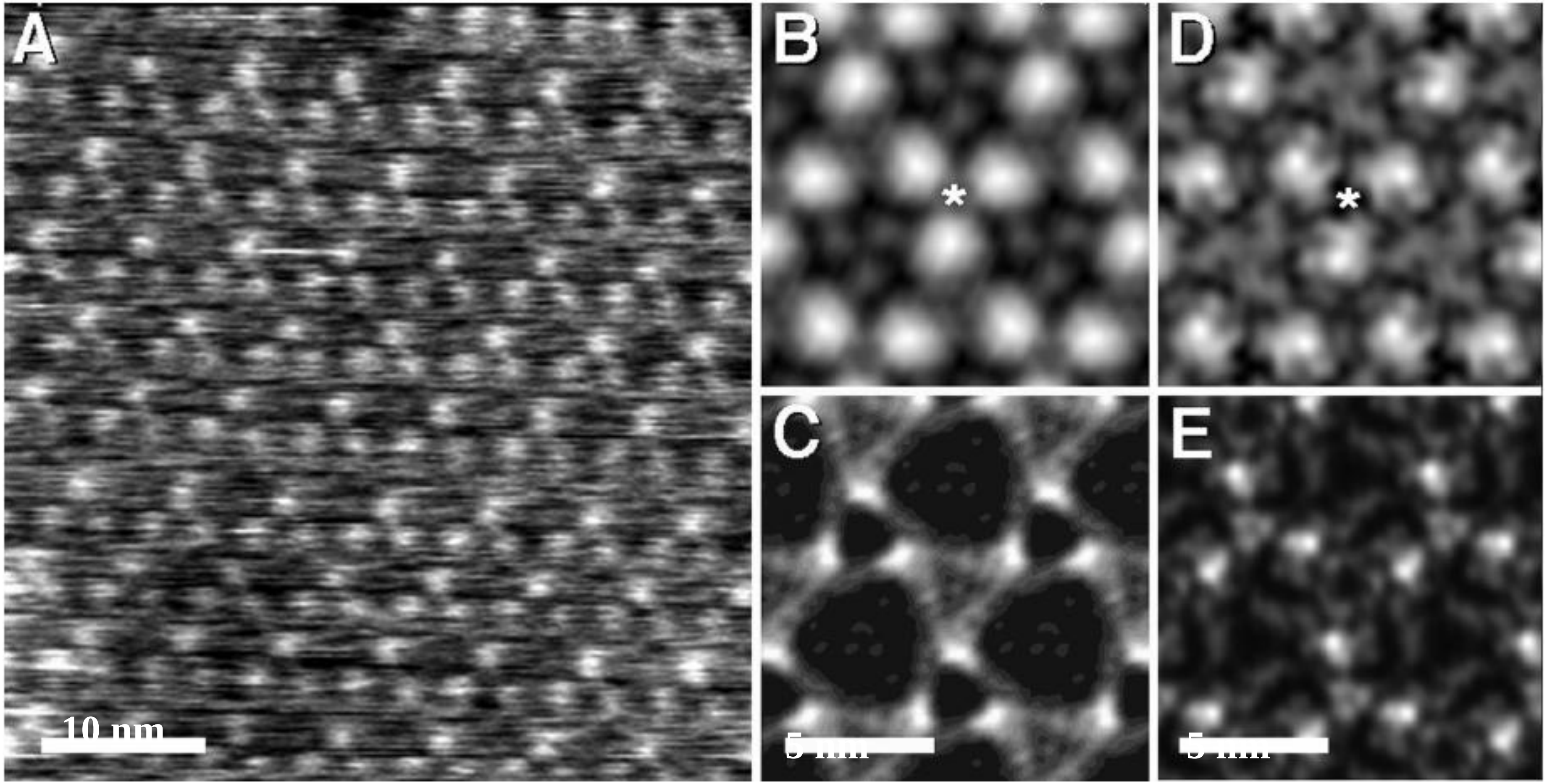
Instabilities in *tapping mode* force microscopy due to the non-linear tip-sample interactions. (a) The amplitude vs. distance curve exhibits a jump between two stable oscillations states labeled L and H. (b) The imaging process becomes unstable for amplitude set-points close to the value of the discontinuity in (a). Imaging with larger or smaller amplitude results in stable oscillations.

From the viewpoint of an experimentalist it appears desirable to control the occurrence of bistable oscillations. Experimental and theoretical results indicate that situations where either attractive or repulsive forces dominate the imaging process have a minimum risk of bistabilities: For example, small oscillation amplitudes more likely result in a purely attractive interaction without repulsive contact while for large amplitudes the repulsion during contact dominate.

Avoiding condition of bistabilities:

- For a strong potential of the cantilever spring, accomplished by large amplitudes or use of stiff cantilevers, the tip-sample interaction is a weak perturbation and the oscillation will be monostable.
- adjusting the driving frequency exactly to the resonance can help to minimize bistabilities.
- The best contrast in phase images is found for situations, where attractive and repulsive interactions contribute similarly to the total interaction. Even slight changes of adhesion or stiffness will greatly affect the oscillation parameters. Unfortunately, this is exactly the regime of bistabilities, resulting in sudden changes of the phase contrast and a complicated quantification.

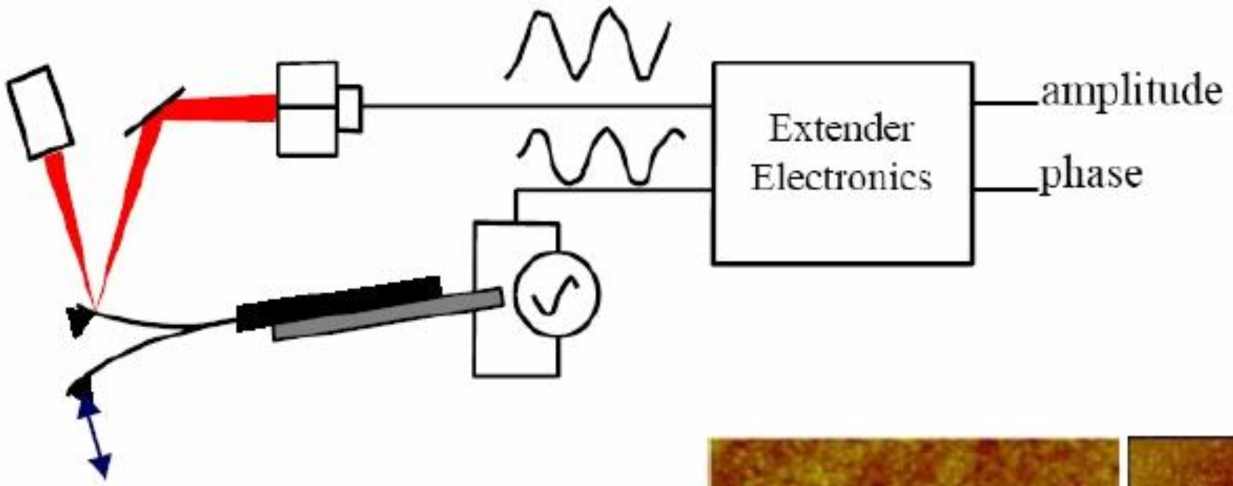
Image of a membrane surface

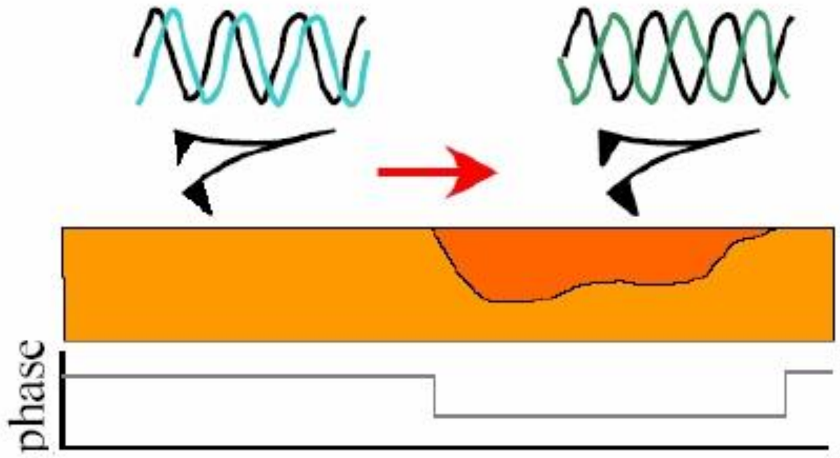


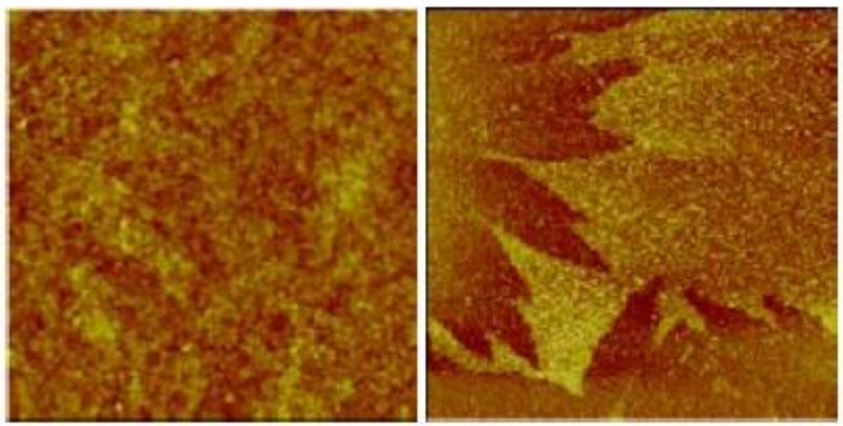
High-resolution tapping mode images (A). Correlation analysis (B) in comparison with contact mode images (D). C. Möller et al., Biophys. J. 77 (1999) 1150

Tapping Mode Phase reveals

Local Stiffness







Two-phase structure of a semi-crystalline polymer

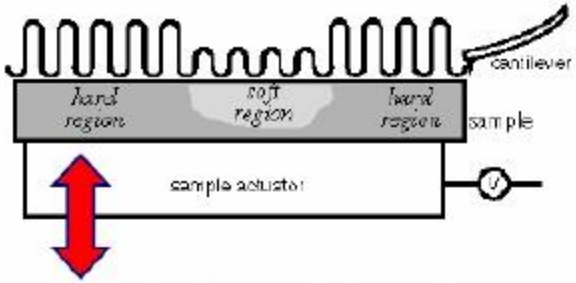
left: topography

right: phase image

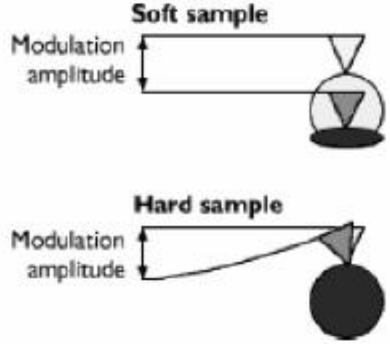
Force Modulation

Scanning Force Microscopy

Imaging Local Stiffness

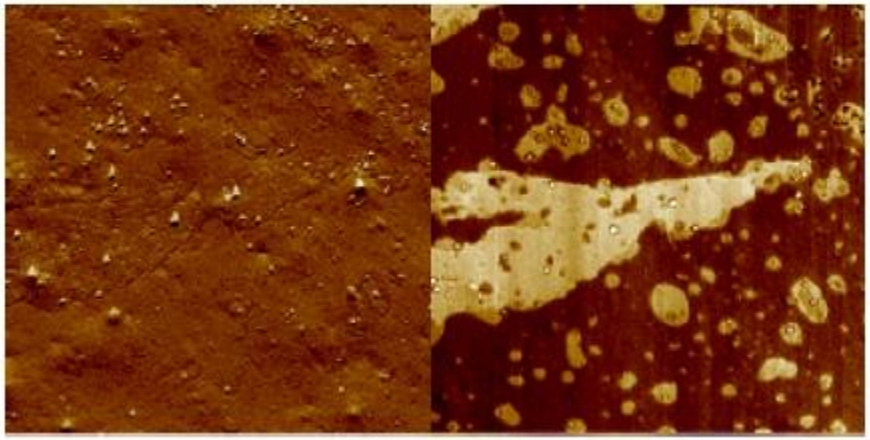


cantilever amplitude is monitored:



sample is modulated with constant amplitude

topography | surface stiffness



Tapping | Force modulation

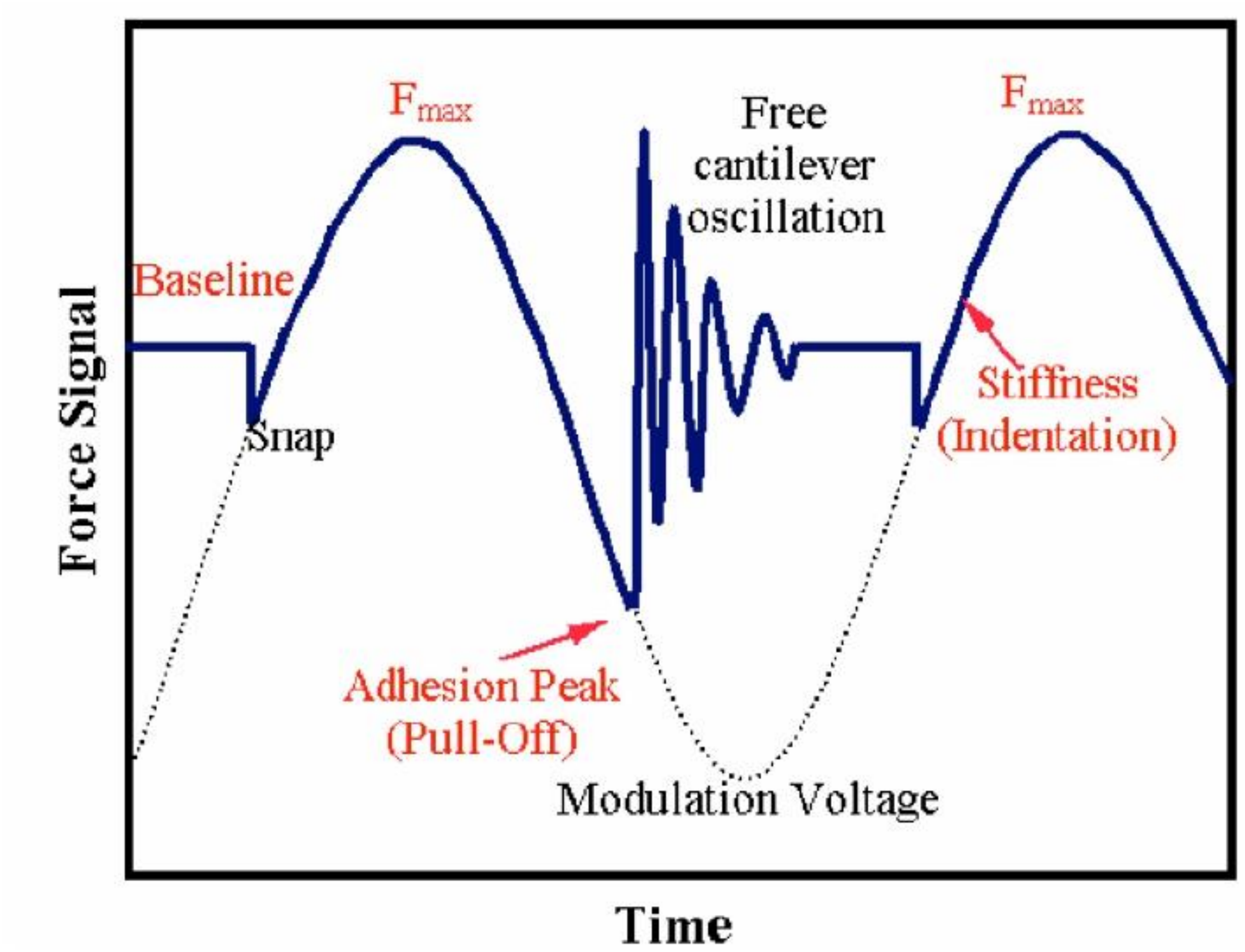
Impact modified polymer

Topography (left):
light colored regions are higher

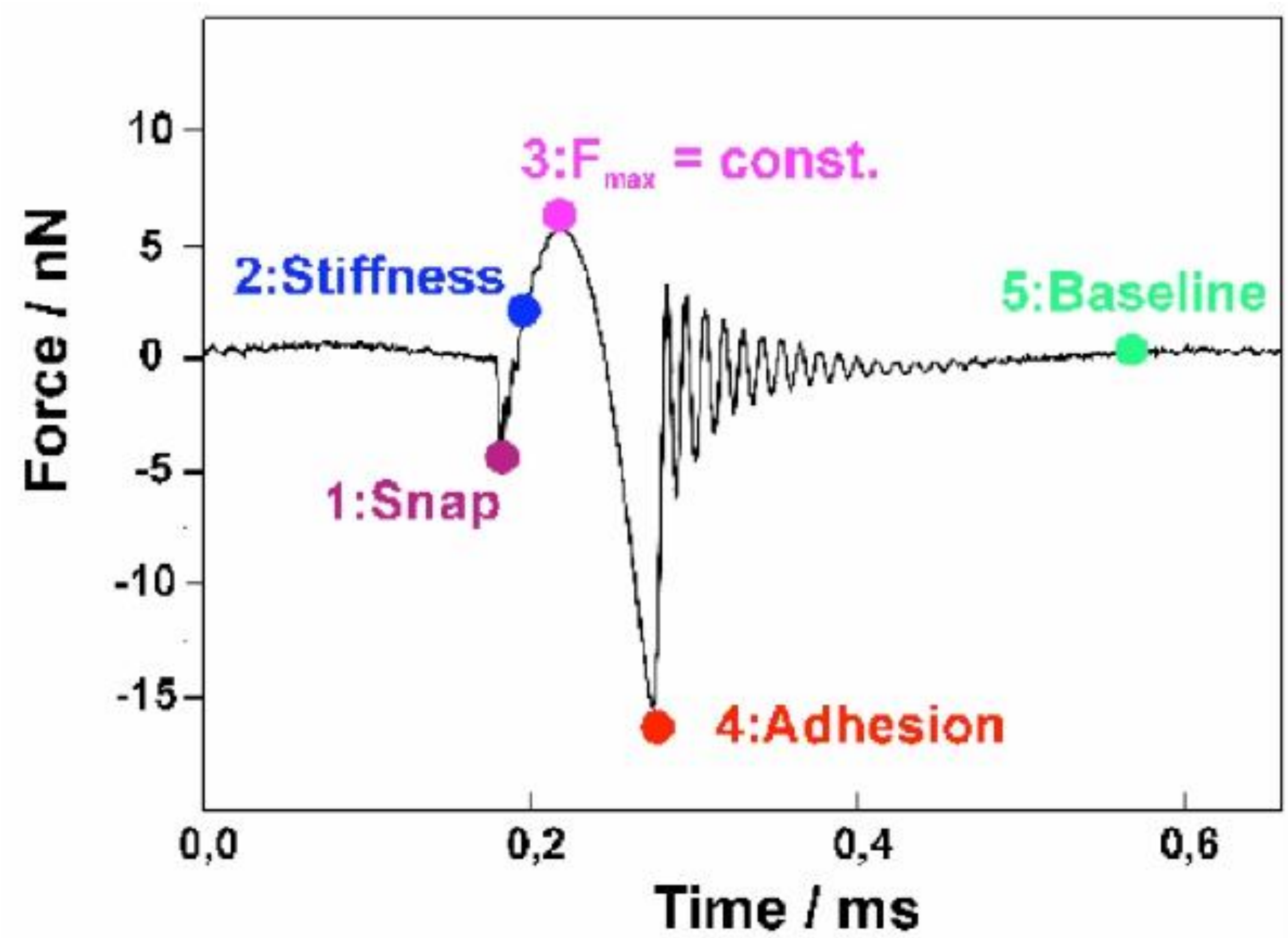
Surface stiffness (right):
light color corresponds to higher surface modulus/stiffness

Pulsed Force Mode

Pulsed force mode (PFM) is based on a standard AFM set-up with a sinusoidal modulation of the z-axis piezo. The tip starts above the surface of the sample, approaches, makes contact, indents then is pulled away from the surface. This makes possible a force-distance curve measurements as illustrated below:



In this way images of topography, 'stiffness' from the indentation measurement and 'adhesion' from the pull off measurement are obtained as shown below.



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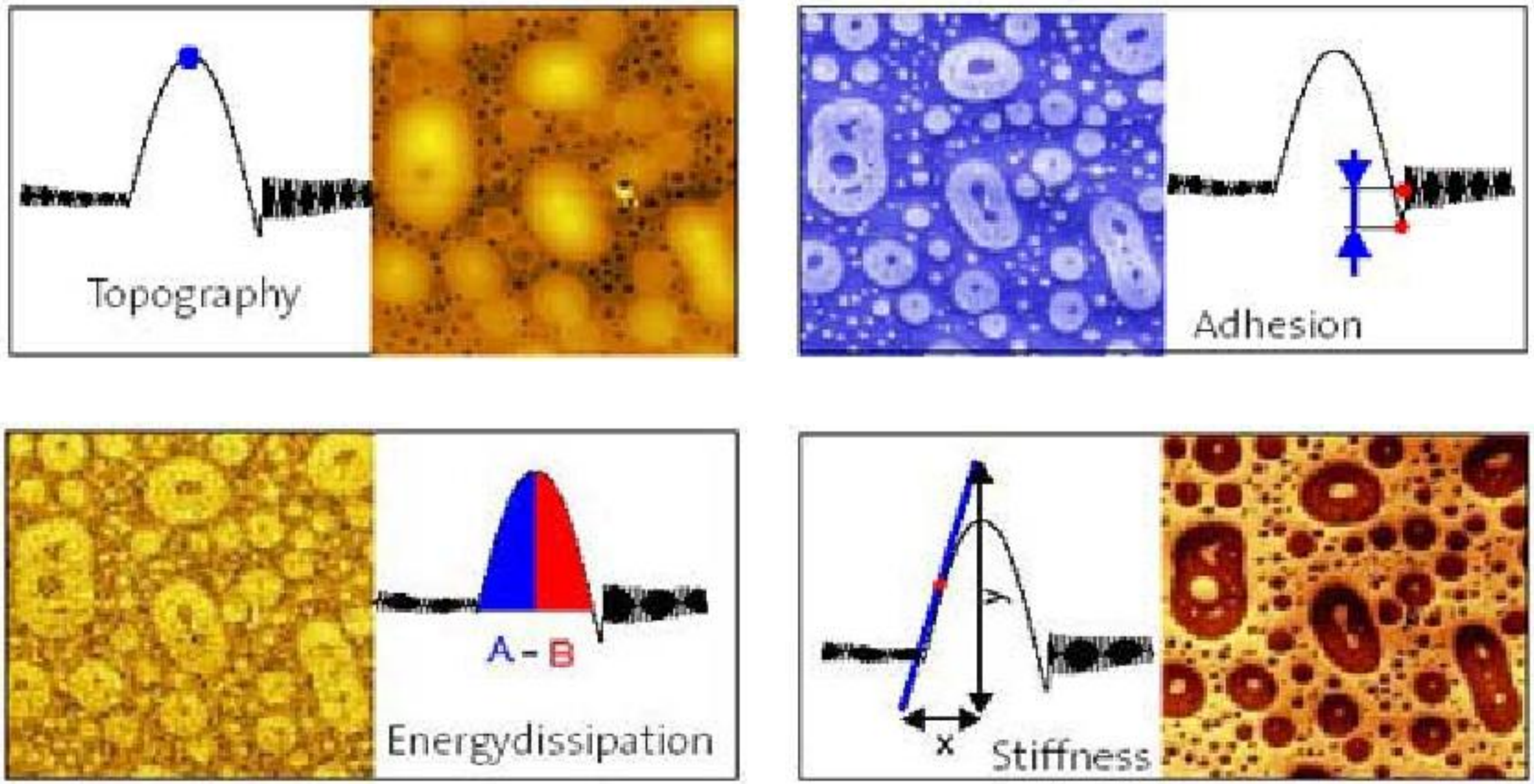
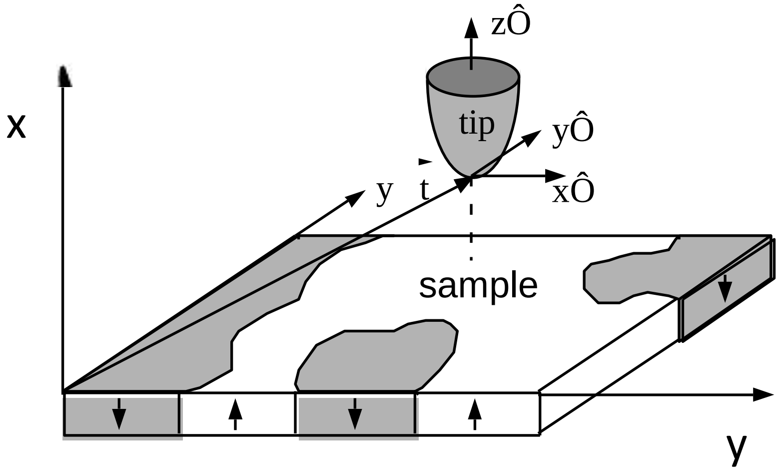


Magnetic Force Microscopy (MFM)

principle

in reciprocal space



$$F_z(\vec{t}) = -\mu_0 \int \vec{M}_{tip}(\vec{r}) \cdot \frac{\partial}{\partial t_z} \vec{H}_{sample}(\vec{r} + \vec{t}) d^3r$$

force acting on tip
=
convolution of tip magnetization
with sample field

$$F_z(\vec{k}) = \mu_0 q_{tip}(\vec{k}) \cdot \vec{H}_z(\vec{k})$$

force acting on tip
=
product of tip transfer function
with sample field

H.J. Hug et al. J. Appl. Phys. 83, 5609 (1998)

P.J. A. van Schendel et al., J. Appl. Phys. 88, 435 (2000)

Quantitative Magnetic Force Microscopy (MFM)

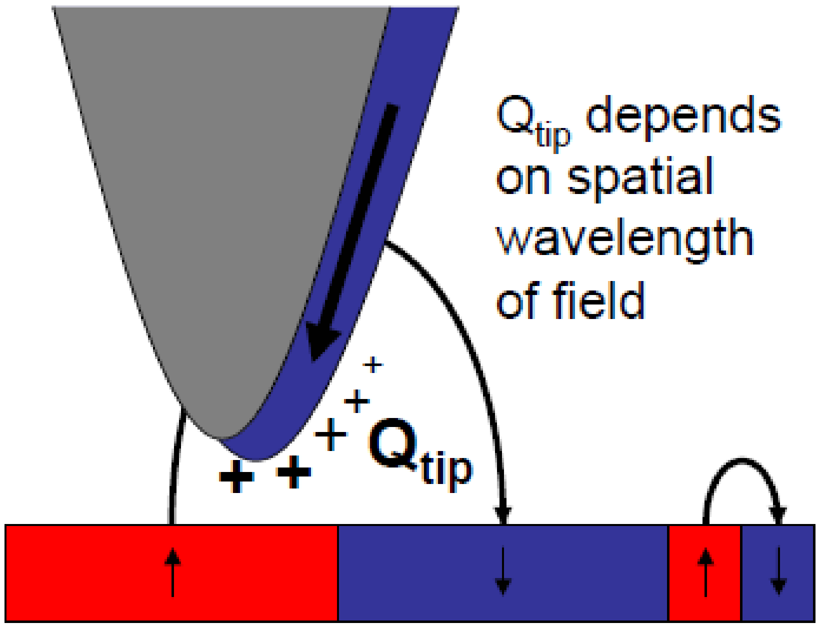
Why Simple Models DO NOT work!

- Point-pole tip models

$$F_z(\vec{r}) = q_{tip} \cdot H_z(\vec{r}) + p_{tip,z} \cdot \frac{\partial}{\partial z} H_z(\vec{r})$$

Quality of point-pole tip models not sufficient, because q_{tip} and p_{tip} depend strongly on spatial wave-length of H-field!

Quantitative image interpretation not possible!



In Fourier space, a convolution becomes an simple multiplication AND the monopole tip model becomes valid because *qtip* depends on the spatial wave-length.

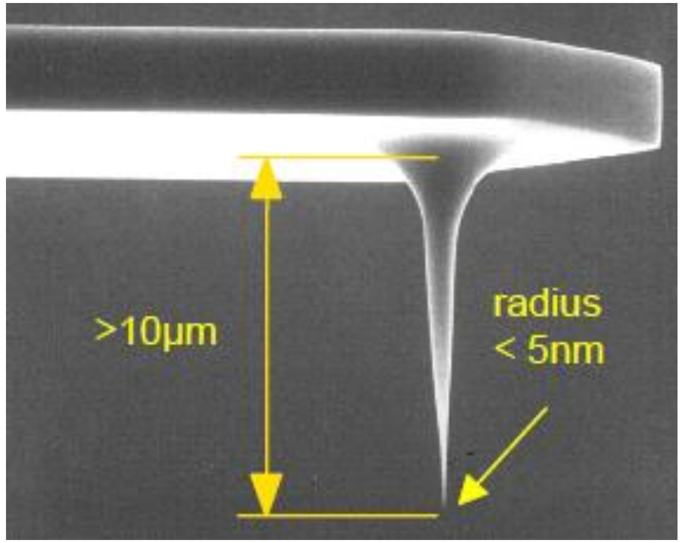
An independent calibration of q_{tip} is required, then quantitative MFM is possible !

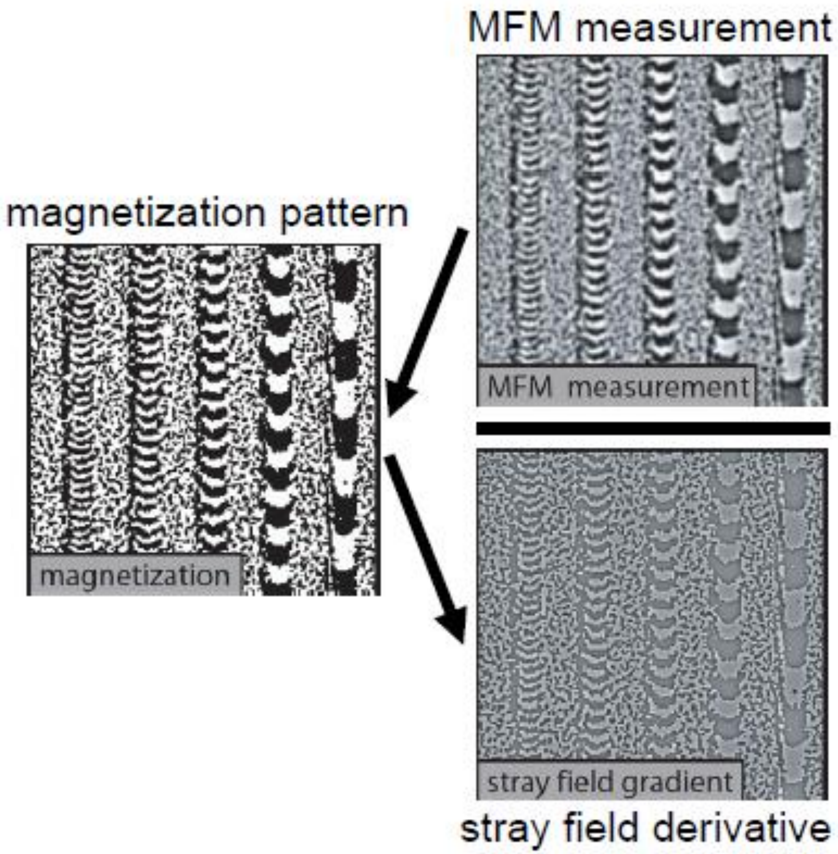
Calibrate & Tip Transfer Function

$$\Delta f(\mathbf{k},z) = q_{tip}^{\ *}(k)\cdot \mathrm{rest}(\mathbf{k})\cdot \frac{\partial}{\partial z} H_z(\mathbf{k},z)$$

$$\Downarrow$$

$$q_{tip}^{\ *}(k) = \frac{\Delta f(\mathbf{k},z)}{\mathrm{rest}(\mathbf{k})\cdot \frac{\partial}{\partial z} H_z(\mathbf{k},z)}$$





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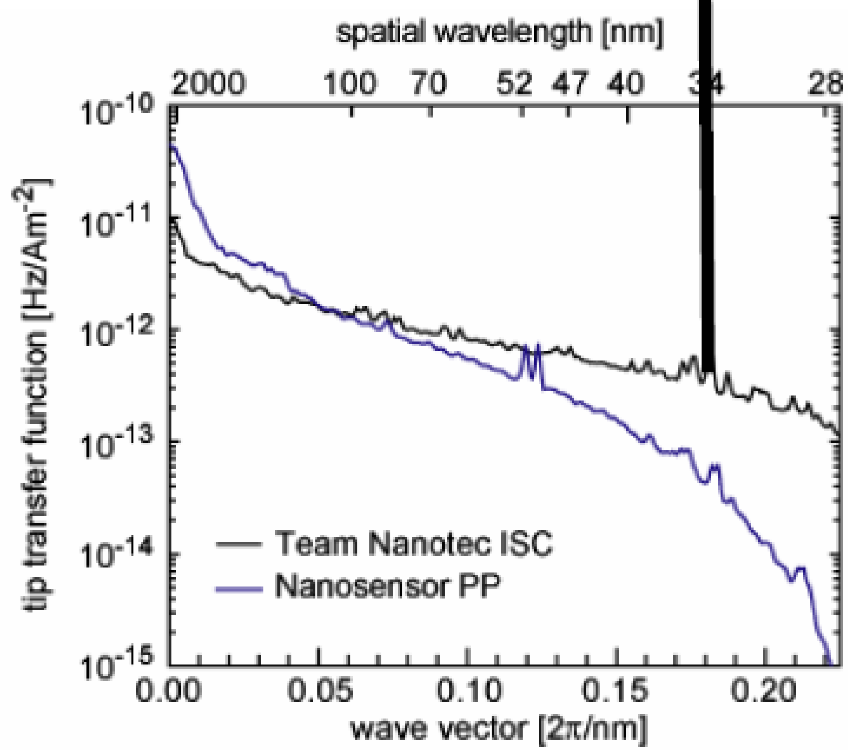
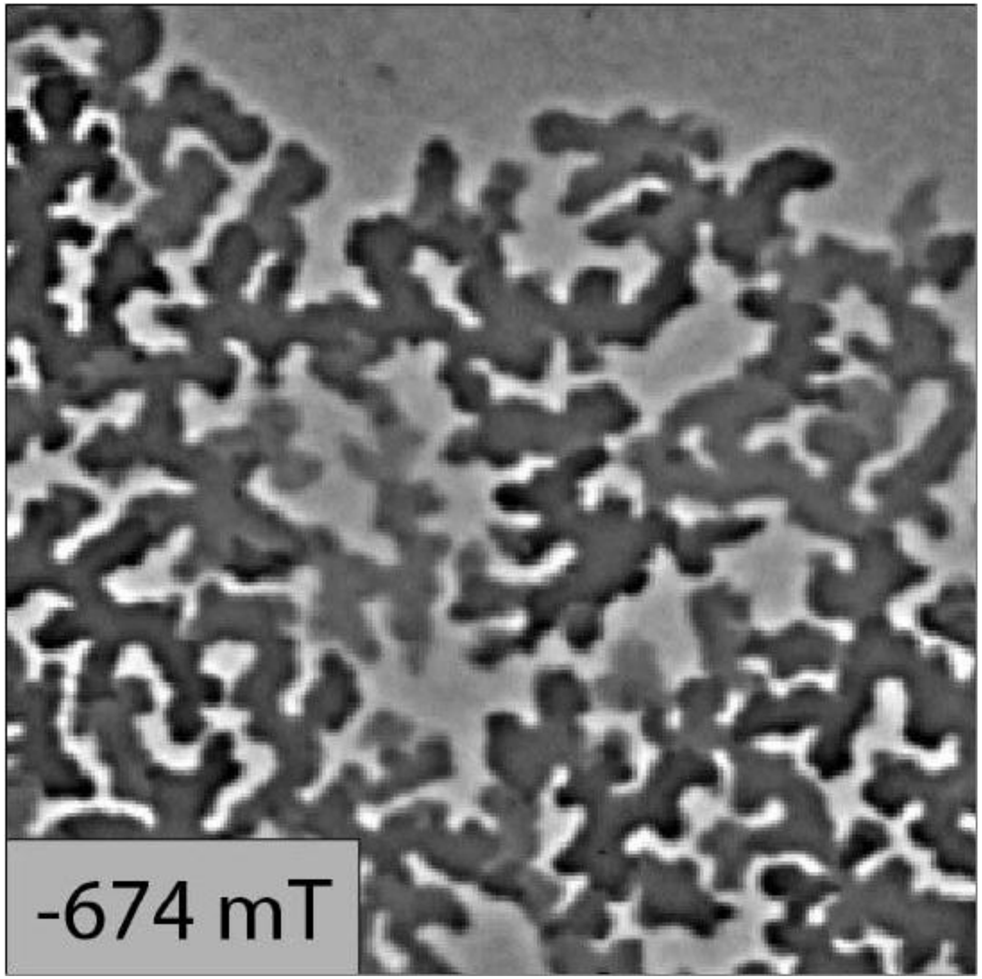
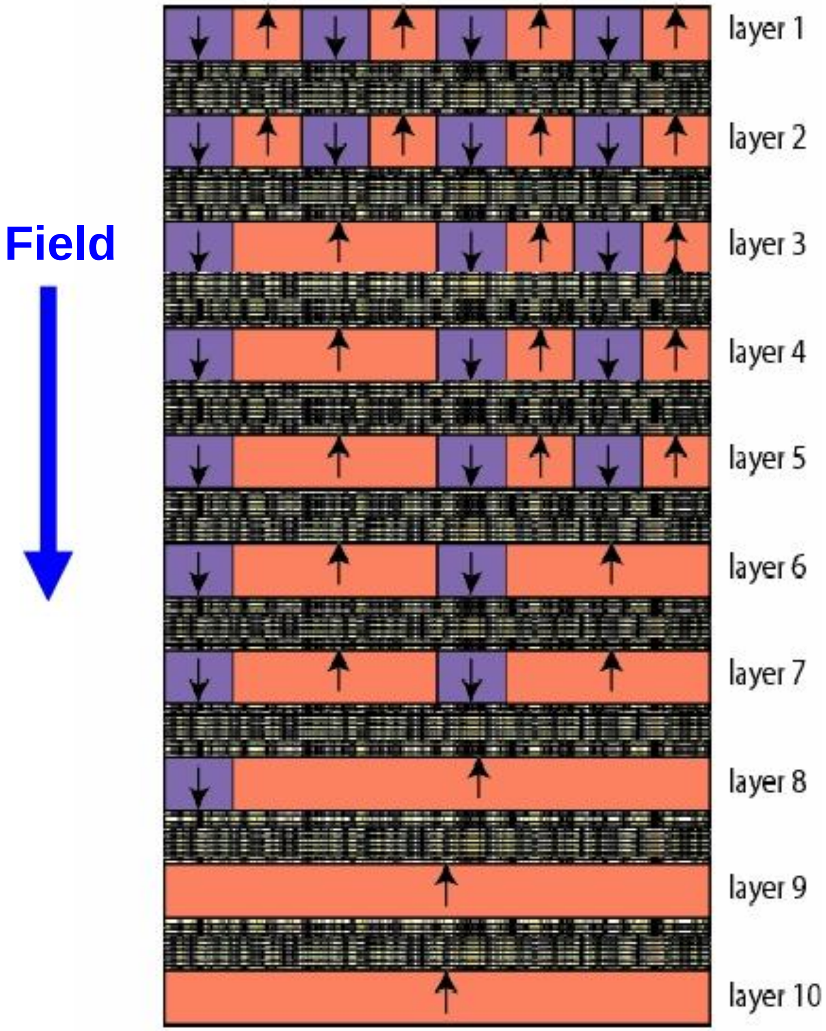
tip-transfer function



Field Cooled: The Hysteresis Loop

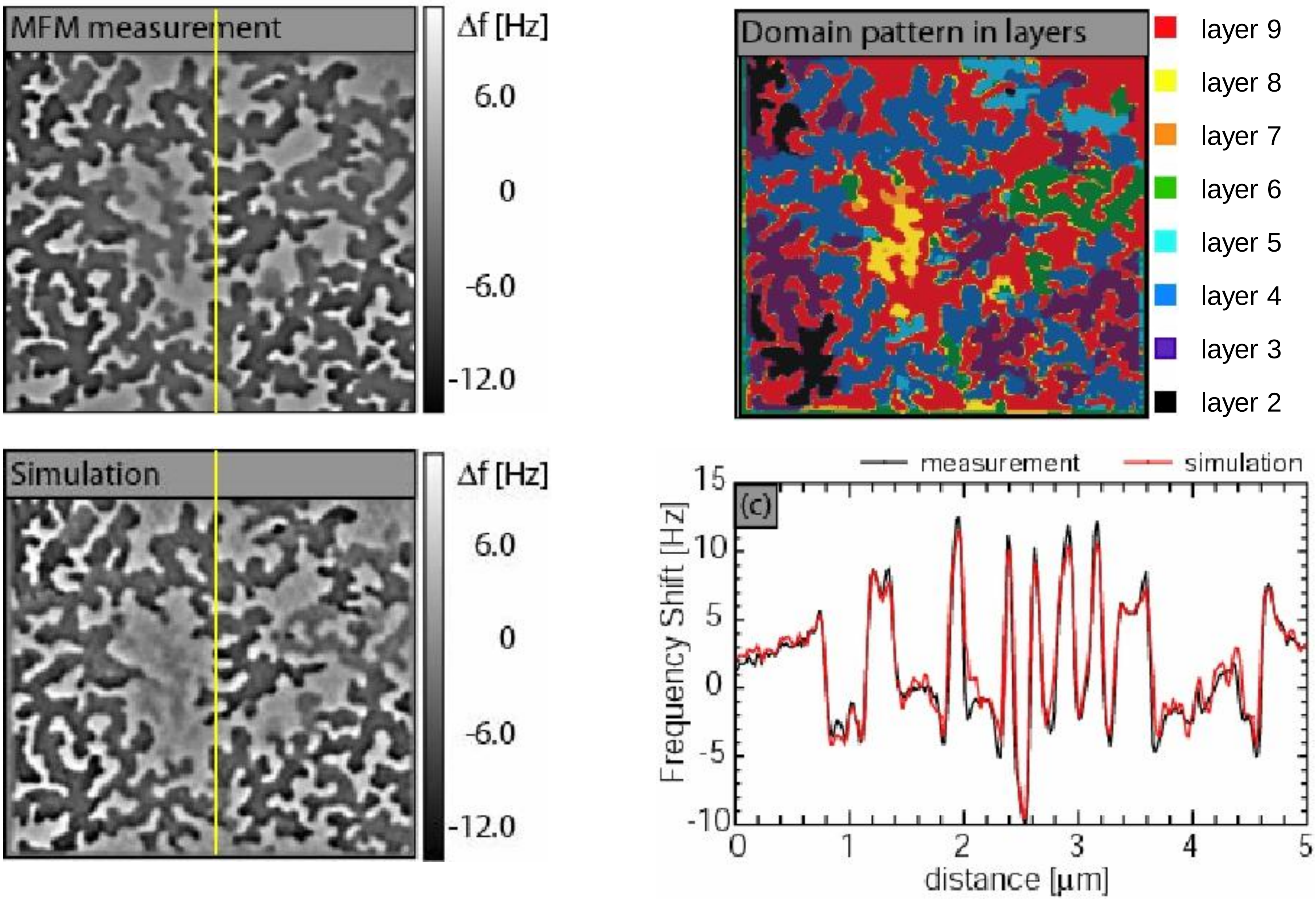




Magnetization of layers from top to bottom & lowest two layers last indicates that exchange biasing is stronger in lower layers !

But how do we know which grey level corresponds to which layer ?

Quantitative Evaluation of Contrast using Calibrated Tip



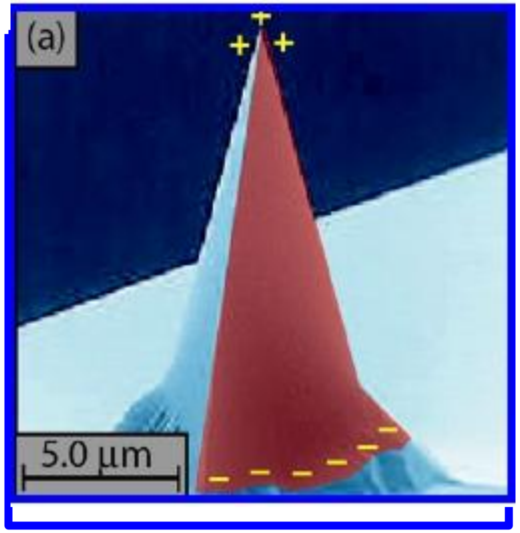
Principles of high-resolution Magnetic Force Microscopy (MFM)

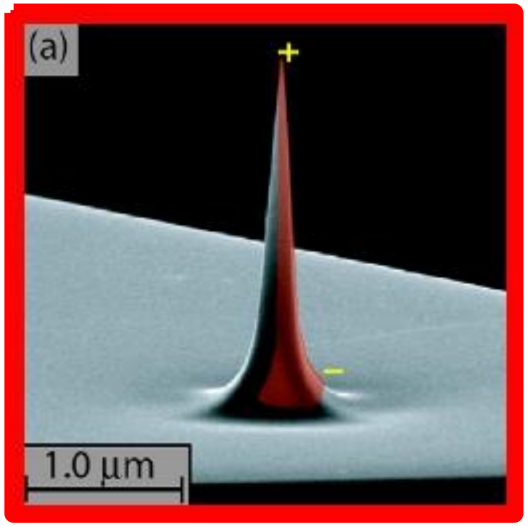
Resolution Factors

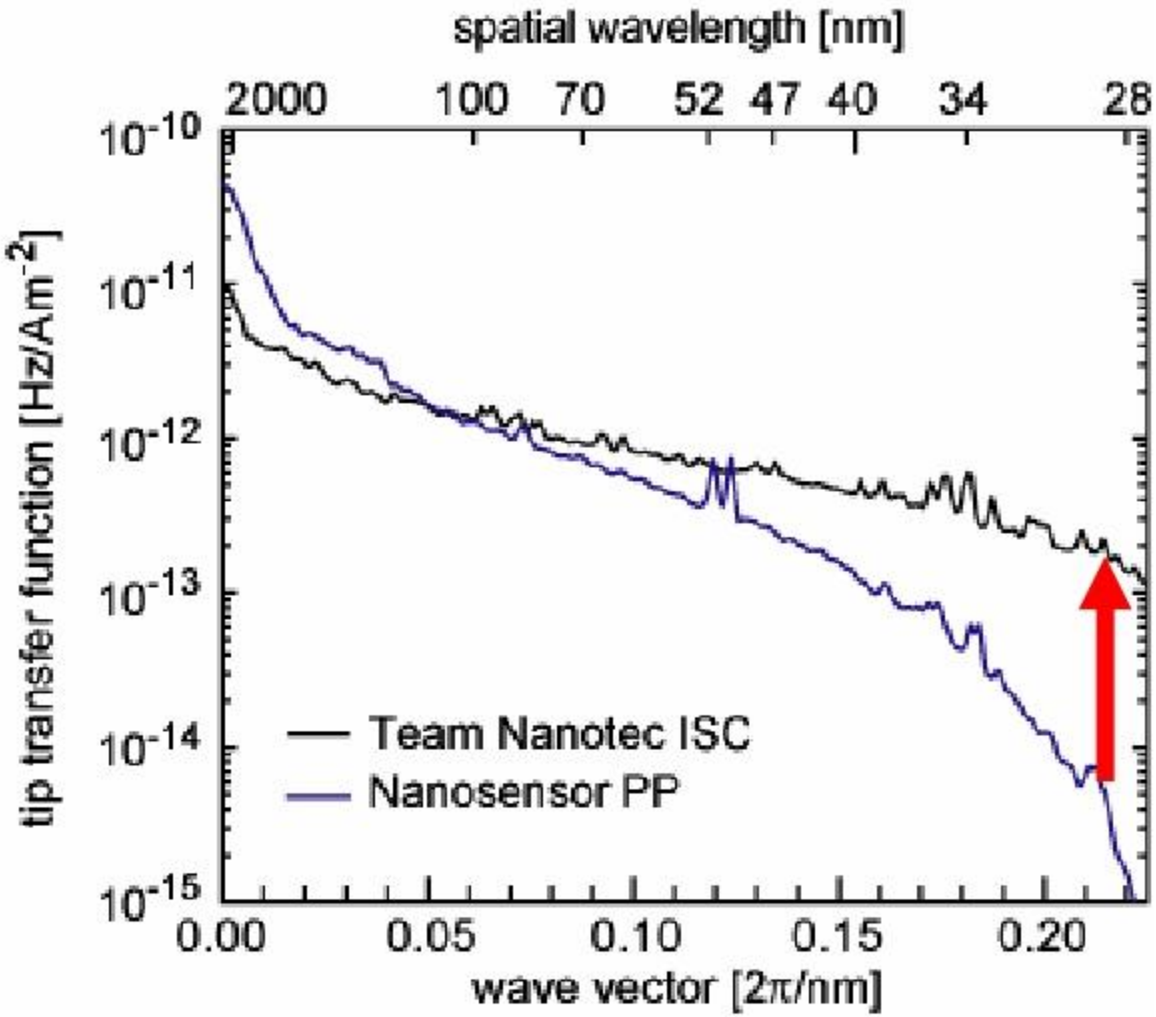
distance loss	$\frac{1}{2}\exp(-kz)$	use small tip-sample distance, but problems with topography
thickness loss	$[1-\exp(-kt)]$	thick samples are easier
amplitude loss	$\frac{LCF(\vec{k})}{kA_0}\cdot \mathrm{I}_1\left(kA_0\cdot LCF(\vec{k})\right)$	optimize amplitude for maximum sensitivity for small spatial wavelengths
cantilever	$\frac{f_0}{2c_L}$	use sufficiently soft cantilevers with high resonance frequency
instrument noise	$\left.\frac{\partial}{\partial z}F_z\right\vert_{min,rms} = \frac{1}{A_{rms}}\cdot\sqrt{\frac{4k_bTBc_L}{2\pi f_0 Q}}$	limited by thermal noise of cantilever, use high-Q cantilever in vacuum
signal recovery		amplifiy small spatial wavelengths by image processing
tip transfer function	$\mu_0\cdot q_{tip}(\vec{k})$	optimize tip transfer function

… and use numerical recovery of supressed small spatial wave-lengths !

Optimization of Tip Transfer Function



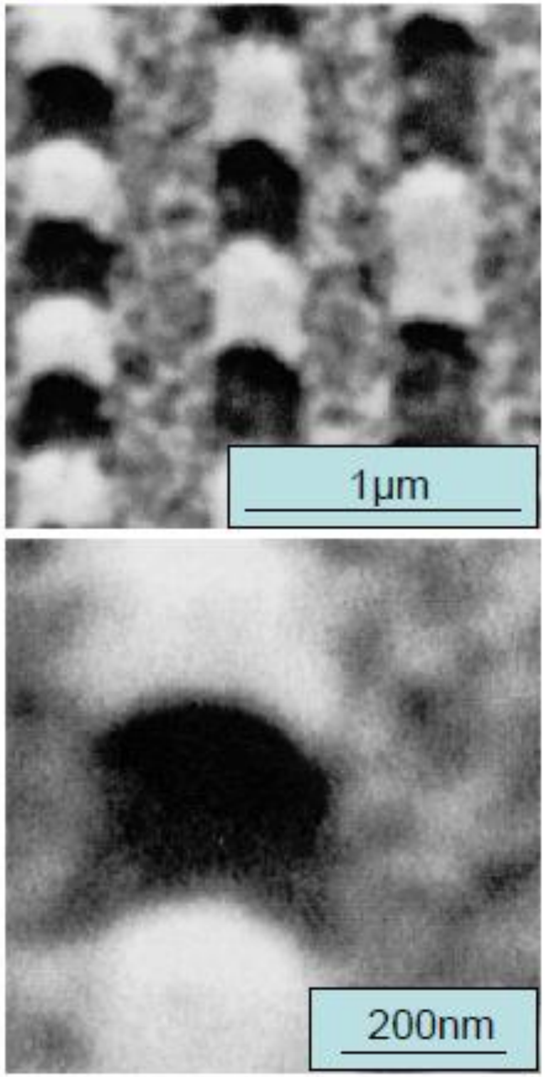




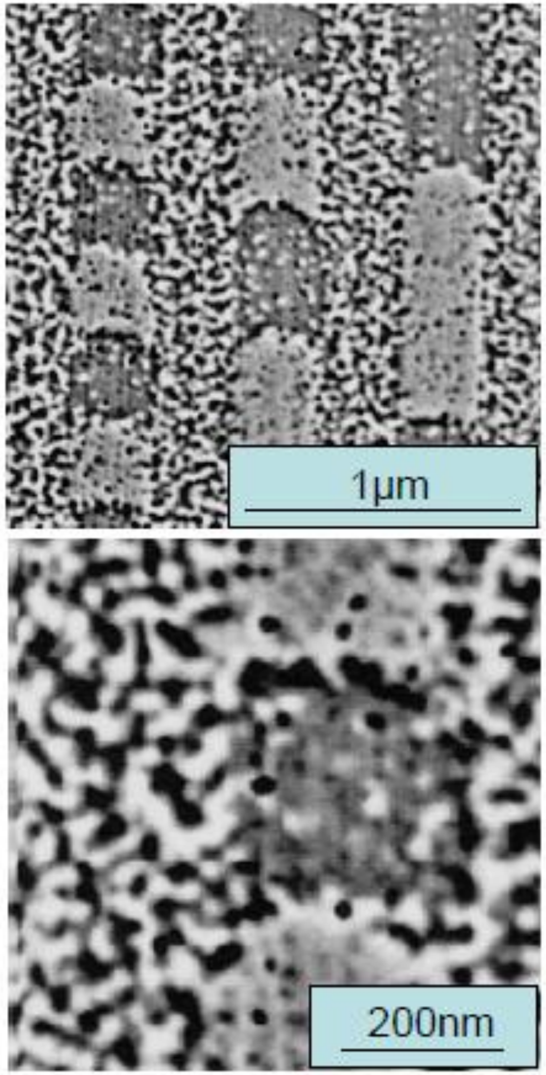
- use high aspect ratio tip,
- thin & optimized coating
- do not destroy tip by tapping, use true non-contact mode of operation

Comparison

Todays MFM
@ HGST, San Jose



Ultra-high resolution MFM
SwissProbe's hr-MFM



SEMPA
Hitachi CRL, Dr. T. Kohashi