NT-MDT Spectrum Instruments

ScanT™

a Shortcut to Reliable AFM results

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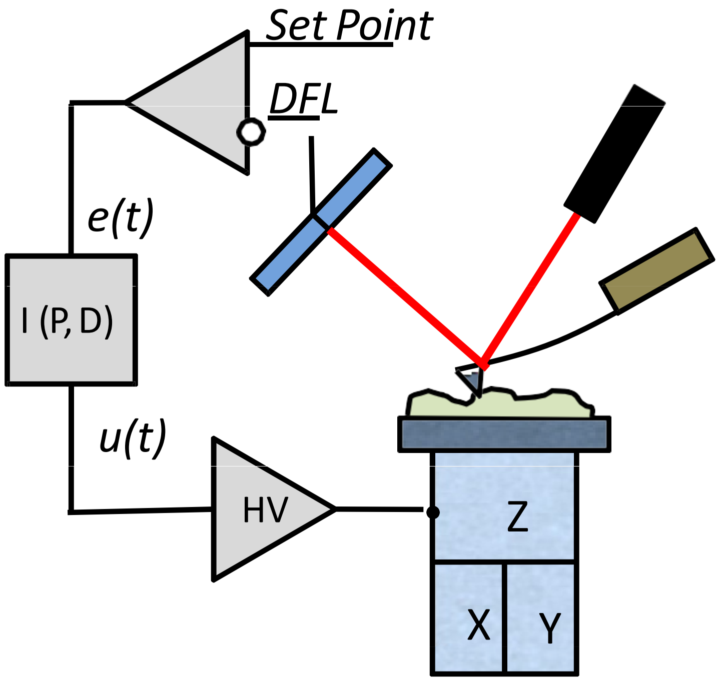
Agenda

- Introduction
- Motivation for the development of ScanT^{TM} and RapidScan^{TM} technologies
- Automated optimization of scanning parameters in tapping mode AFM: physical background, basics of the algorithms and examples of application
- Rapid scanning
- Summary

Intro: Atomic Force Microscopy



- 3D-imaging of surface topography with (sub-)nm spatial resolution
- Imaging of nanomechanical, electrical, magnetic and other surface properties with nm-scale spatial resolution *("more than 50 AFM modes")*
- Can be combined with optical techniques in UV, visible, IR and THz ranges (AFM-Raman, nano-IR AFM, IR and THz s-SNOM)
- Can be used under different environments (vacuum, liquid, controlled atmosphere, temperature variations, etc.)
- Field of view: up to $\approx$100 μm in X and Y; $\approx$ 10 $\div$ 20 μm in Z
- Scan rate: 1 $\div$ 2 Hz

Intro: NT-MDT SI Product Line

AFM

2011

2009

SOLVER NANO

- Compact desktop AFM/STM for both education and science
- Full set of AFM/STM modes
- High AFM/STM performance
- Closed-loop Scanner

NEXT/ TITANIUM

- AFM/STM with exceptional level of automation
- Fast, precise and low-noise closed-loop scanner
- High resolution imaging due to extremely low noise and high stability
- Full set of standard and advanced AFM/STM modes
- HybriD Mode™

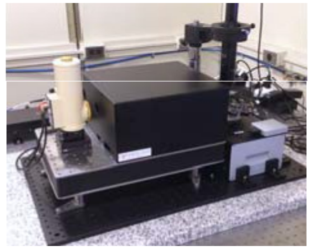
NTEGRA

- Modular high performance AFM/STM for wide range of applications
- Low noise and high resolution
- Full set of standard and advanced AFM/STM modes
- HybriD Mode™

VEGA

- Automated high-resolution AFM for up to 200x200 mm samples
- Ultra stable AFM
- Full set of standard and advanced AFM modes
- HybriD Mode™
- ScanT™

AFM-Raman / IR / TERS



NTEGRA SPECTRA II

- SPM
- Automated AFM laser, probe and photodiode
- Confocal Raman / Fluorescence / Rayleigh Microscopy
- Tip Enhanced Raman Scattering (TERS)
- TERS optimized system for all possible excitation/detection geometries
- HybriD Mode™

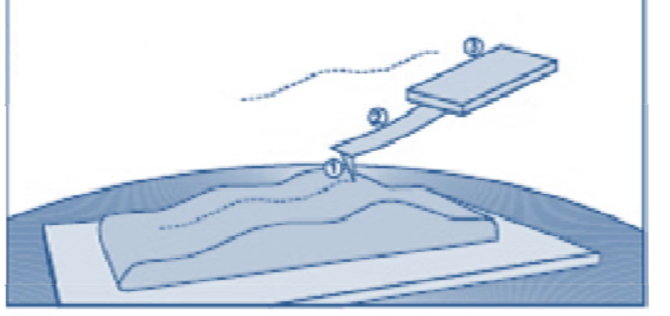
NTEGRA Nano IR

- IR sSNOM system
- High resolution AFM
- Stabilized CO_2 laser
- HybriD Mode™

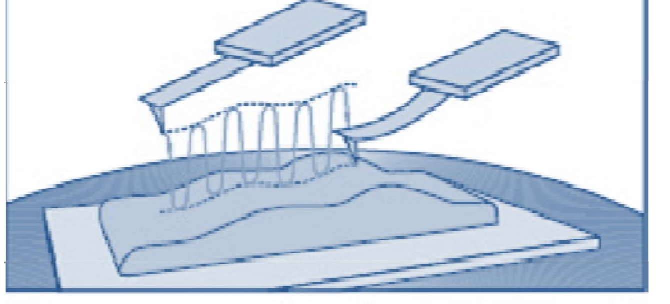
Intro: Basic AFM Modes

AFM modes
- Contact AFM
- Non-resonant oscillatory AFM
- Resonant oscillatory AFM

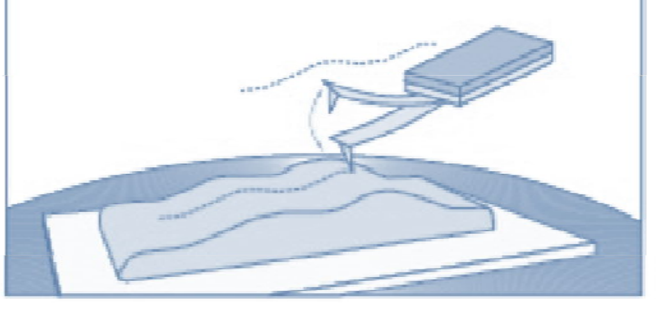
Contact AFM



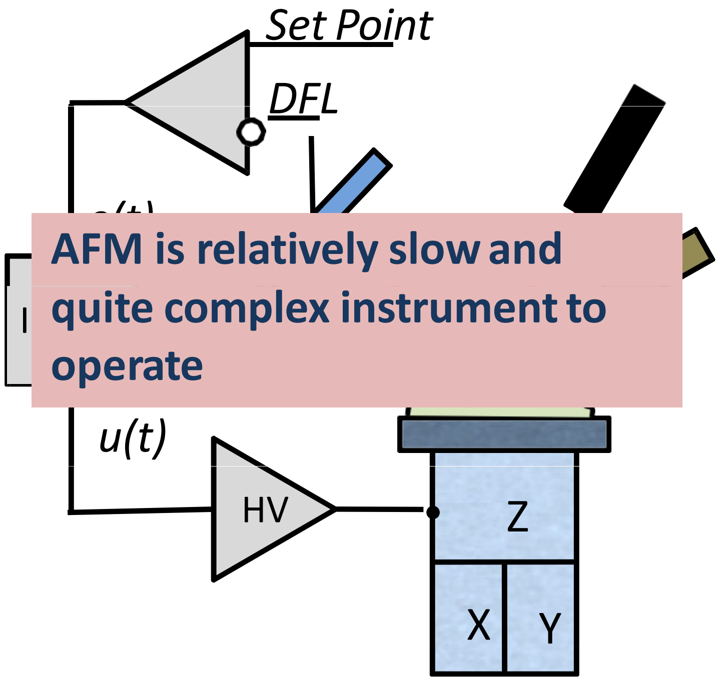
Non-resonant oscillatory AFM



Resonant oscillatory AFM



Motivation: Atomic Force Microscopy

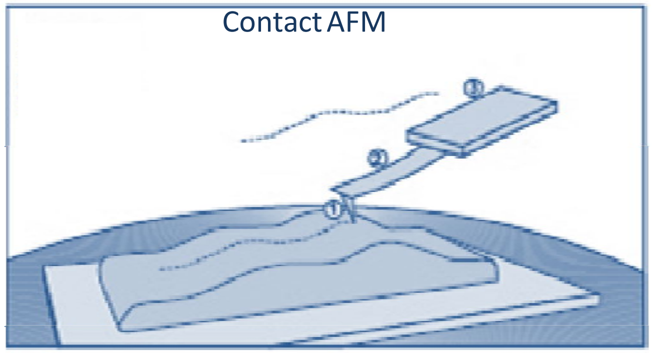


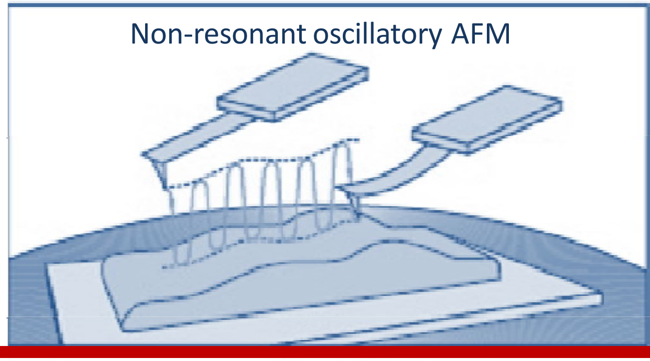
AFM is relatively slow and quite complex instrument to operate

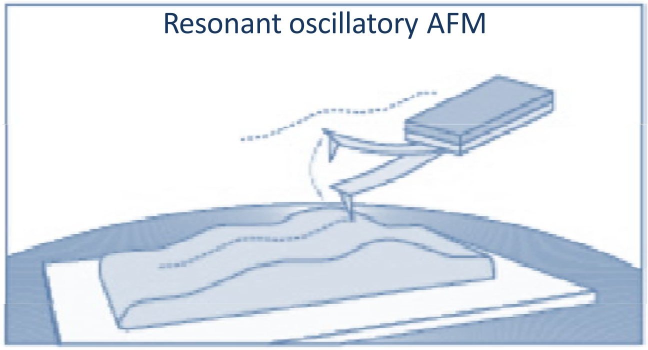
- 3D-imaging of surface topography with (sub-)nm spatial resolution
- Field of view: up to ≈100 μm in X and Y; ≈ 10 ÷ 20 μm in Z
- **Scan rate: 1 ÷ 2 Hz**
- **Imaging of nanomechanical, electrical, magnetic and other surface properties with nm-scale spatial resolution *("more than 50 AFM modes")***
- Can be combined with optical techniques in UV, visible, IR and THz ranges (AFM-Raman, nano-IR AFM, IR and THz s-SNOM)
- Can be used under different environments (vacuum, liquid, controlled atmosphere, temperature variations, etc.)

Motivation: Basic AFM Modes

AFM modes
- Contact AFM
- Non-resonant oscillatory AFM
- Resonant oscillatory AFM



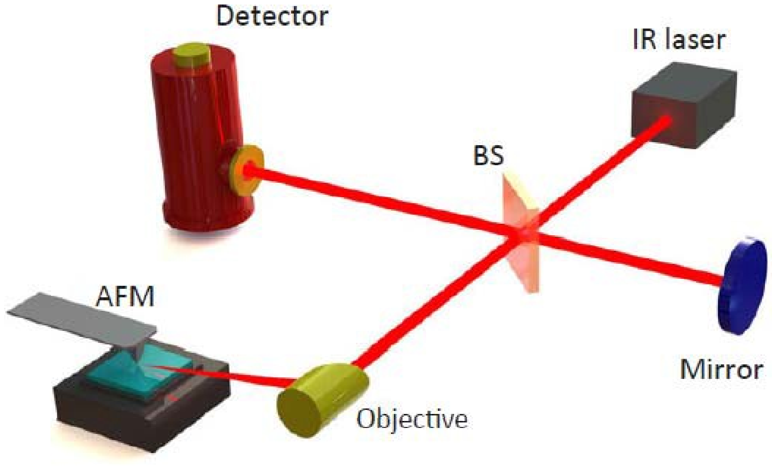




Tapping mode:
about 90% of publications where AFM is used

Motivation: VEGA AFM

Motivation: NTEGRA Nano IR – IR s-SNOM measurements



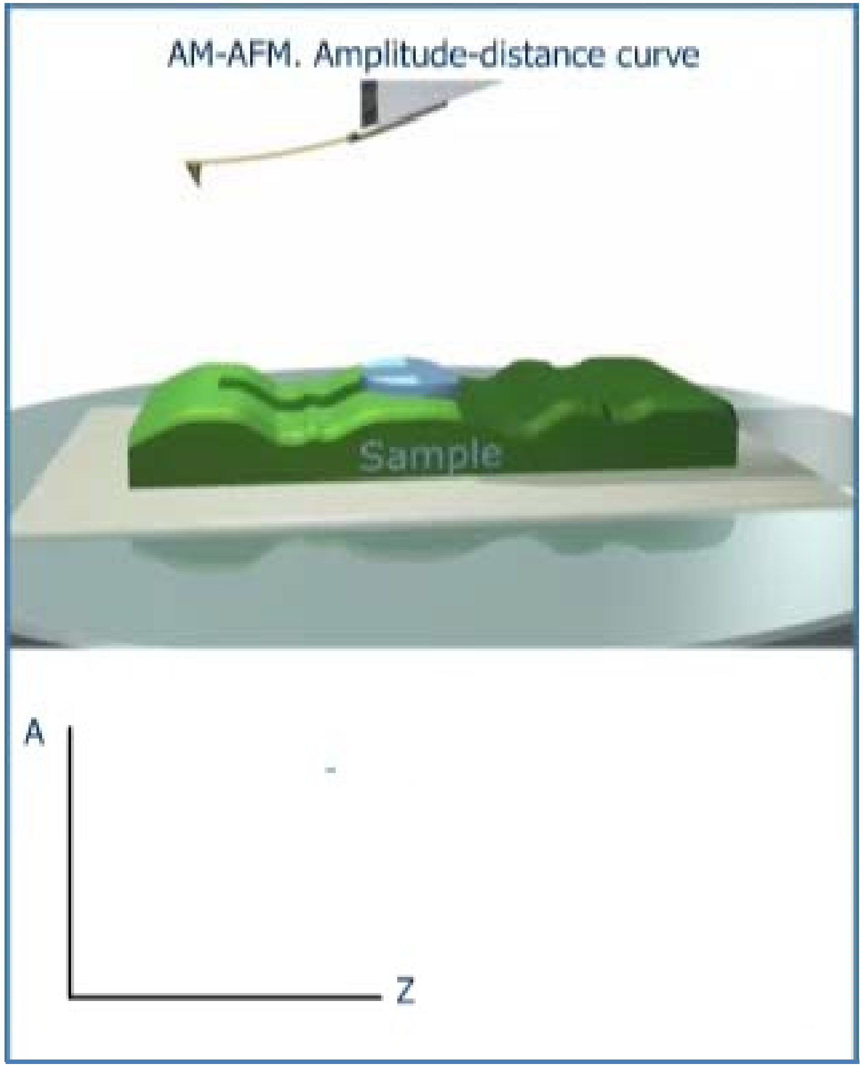
NTEGRA IR principal scheme

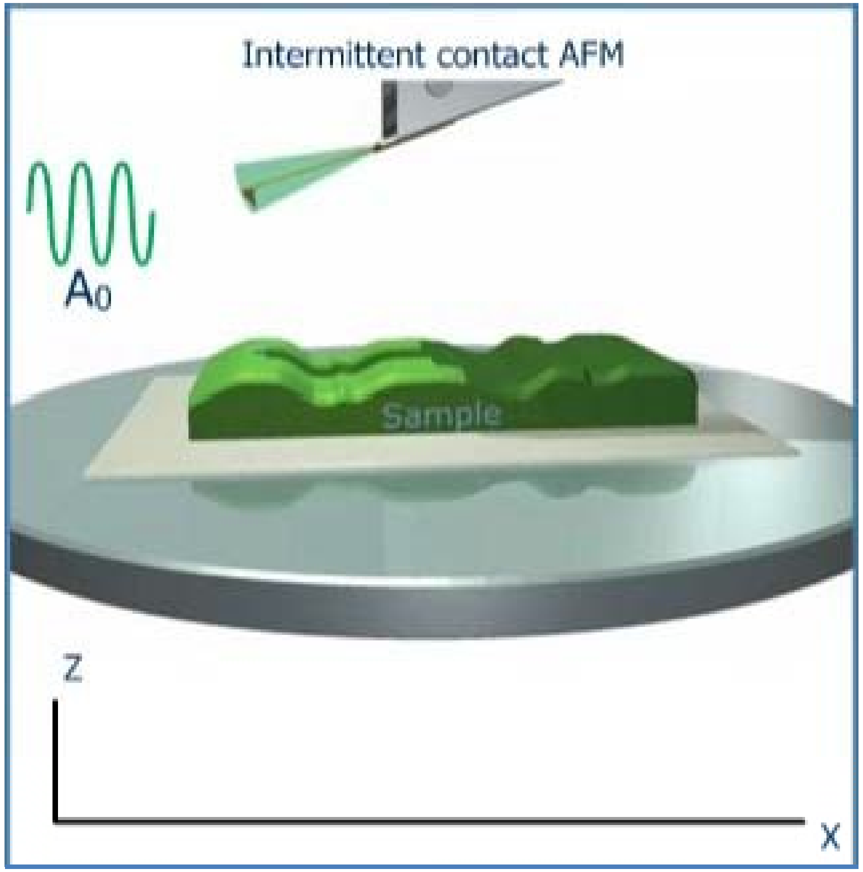
- IR s-SNOM microscopy and spectroscopy with 10 nm spatial resolution
- Wide spectral range of operation: 3-12 µm
- Incredibly low thermal drift and high signal stability
- Versatile AFM with advanced modes: SRI (conductivity), KPFM (surface potential), SCM (capacitance), MFM (magnetic properties), PFM (piezoelectric forces)
- HybriD Mode™ - quantitative nanomechanical mapping
- Integration with microRaman (optional)

Automated optimization of scanning parameters in tapping mode AFM:

physical background, basics of the algorithms and examples of application

Tapping (semicontact) AFM





Automated optimization of scanning parameters

Key scanning parameters:

- **A_0** – Amplitude of cantilever oscillations
- **SP** – Set point amplitude
- **k_i** – Integral feedback gain
- **V_x** – Scan speed

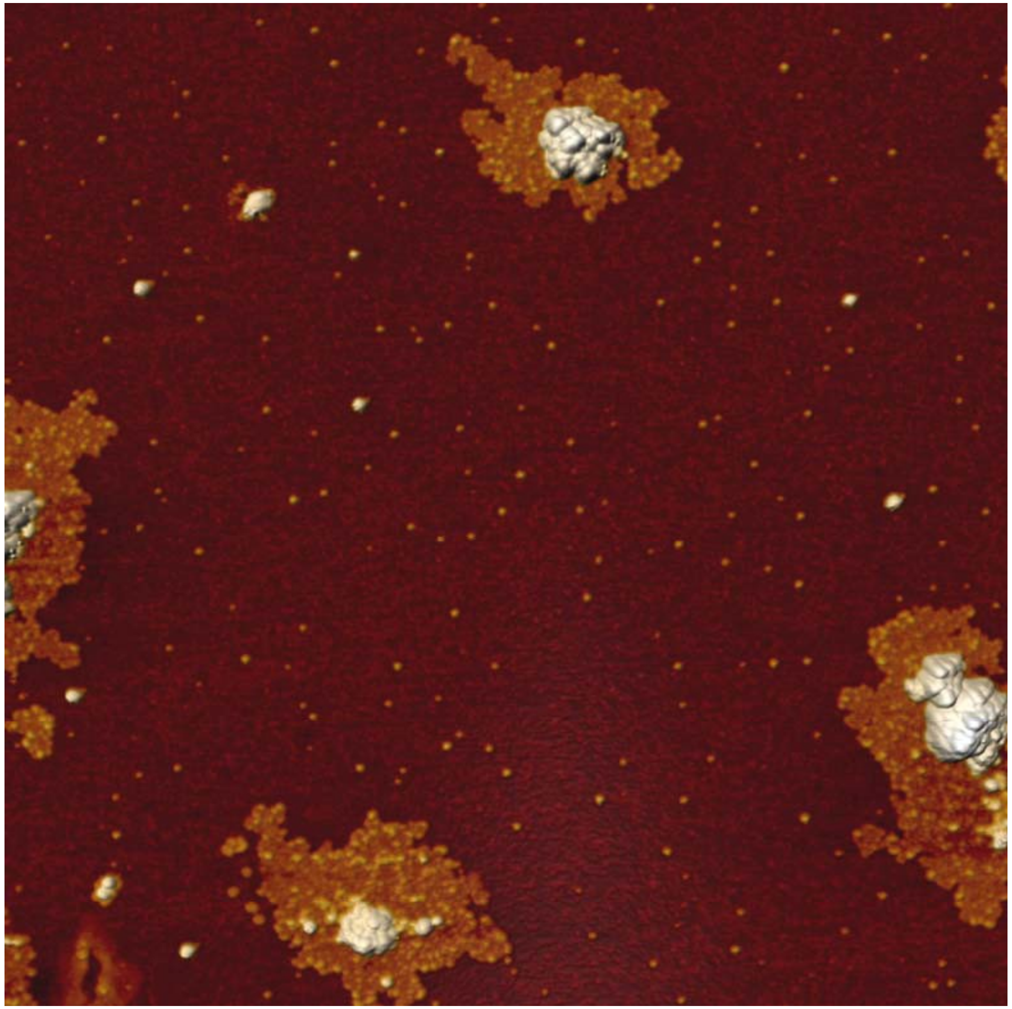
Additional scanning parameters:

- **LP** – low-pass filter band
- **k_p** - Proportional feedback gain

Known parameters:

- **Probe parameters**: resonant frequency, Q-factor
- Mag to amplitude slope conversion
- Sample roughness (?)

Automated optimization of scanning parameters



Fluoroalkanes $F_{14}H_{20}$ on Si.
Scan size 5×5 μm

Automated optimization of scanning parameters

Is it possible to **automatically optimize** scanning parameters
while operating in **tapping mode** AFM to measure the topography and phase contrast?

- How feedback system works in tapping mode AFM?
- Parachuting effects
- Mode switching in tapping mode AFM
- How to optimize integral feedback gain for tapping mode AFM?

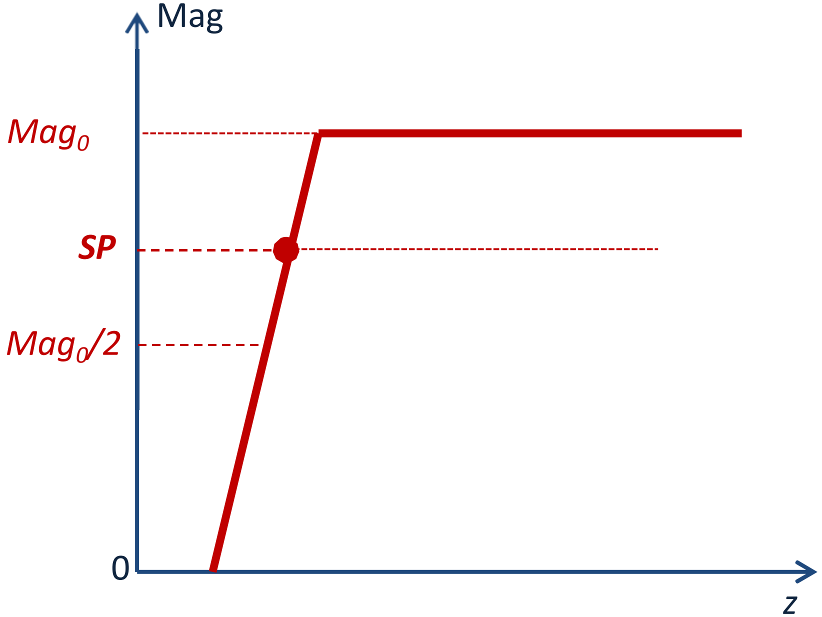
What should be objective function for optimization?

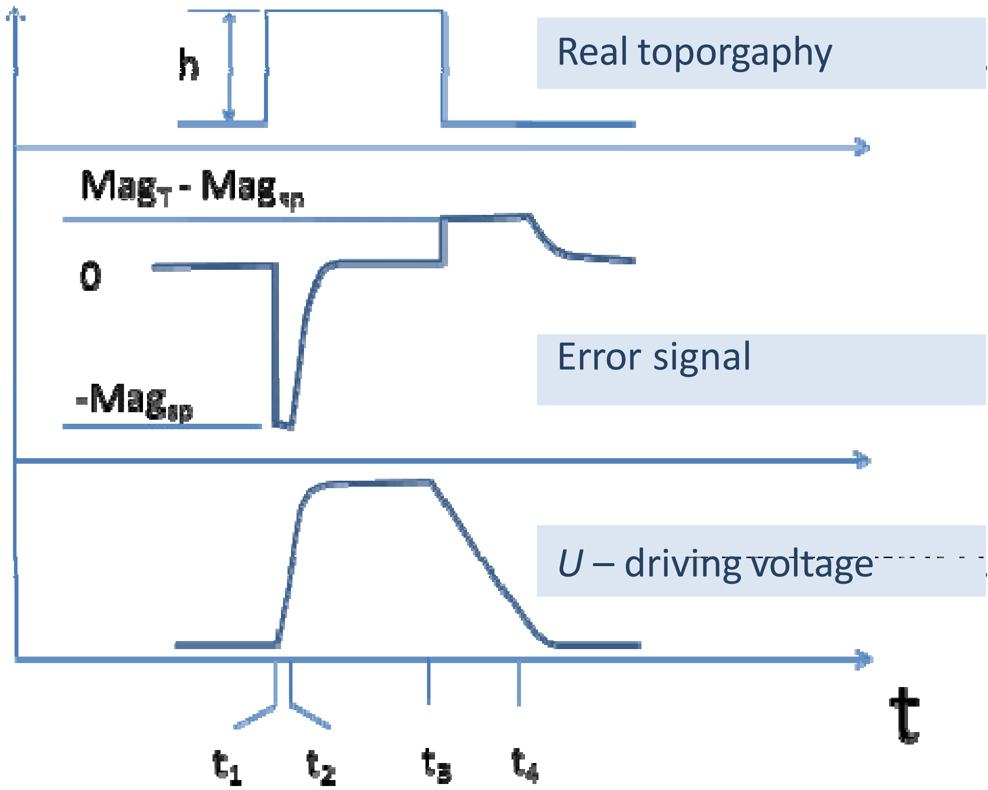
Parachuting effect in tapping mode AFM

$$-SP \leq \varepsilon \leq Mag_0 - SP$$

$$\frac{dU}{dt} = k_i \cdot \varepsilon = k_i(Mag_0 - SP)$$

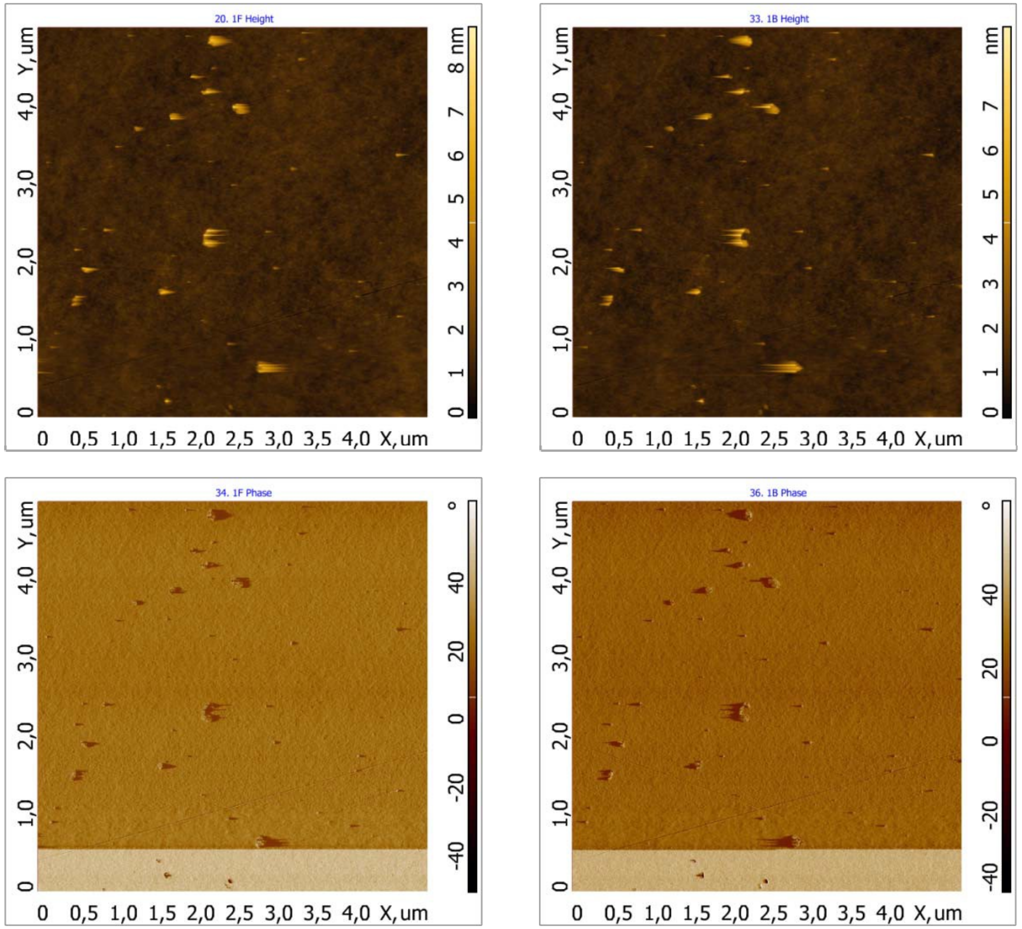
$$U = k_i(Mag_0 - SP) \cdot t + U_0 \quad t_3 < t < t_4$$

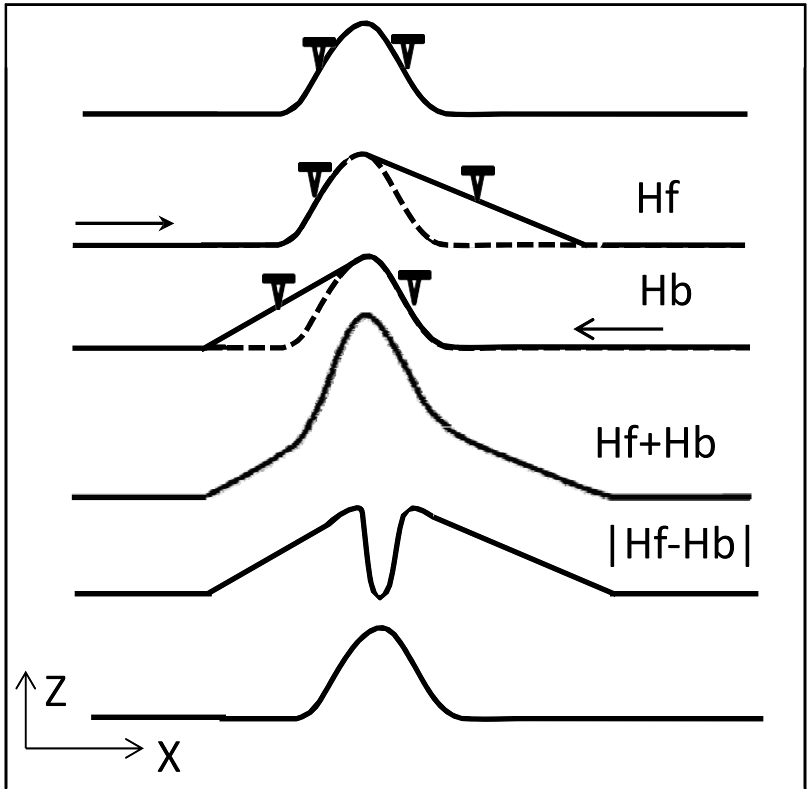




Ref.: T. Ando, "Control Techniques in High-Speed Atomic Force Microscopy," ACC: 3194-3200, 2008

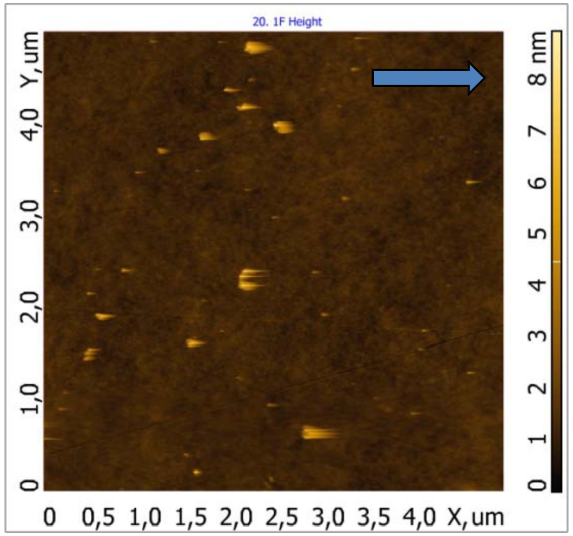
Parachuting effect in tapping mode AFM

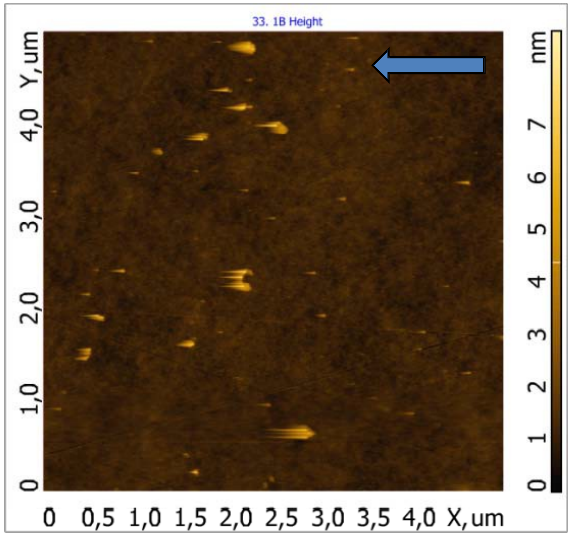


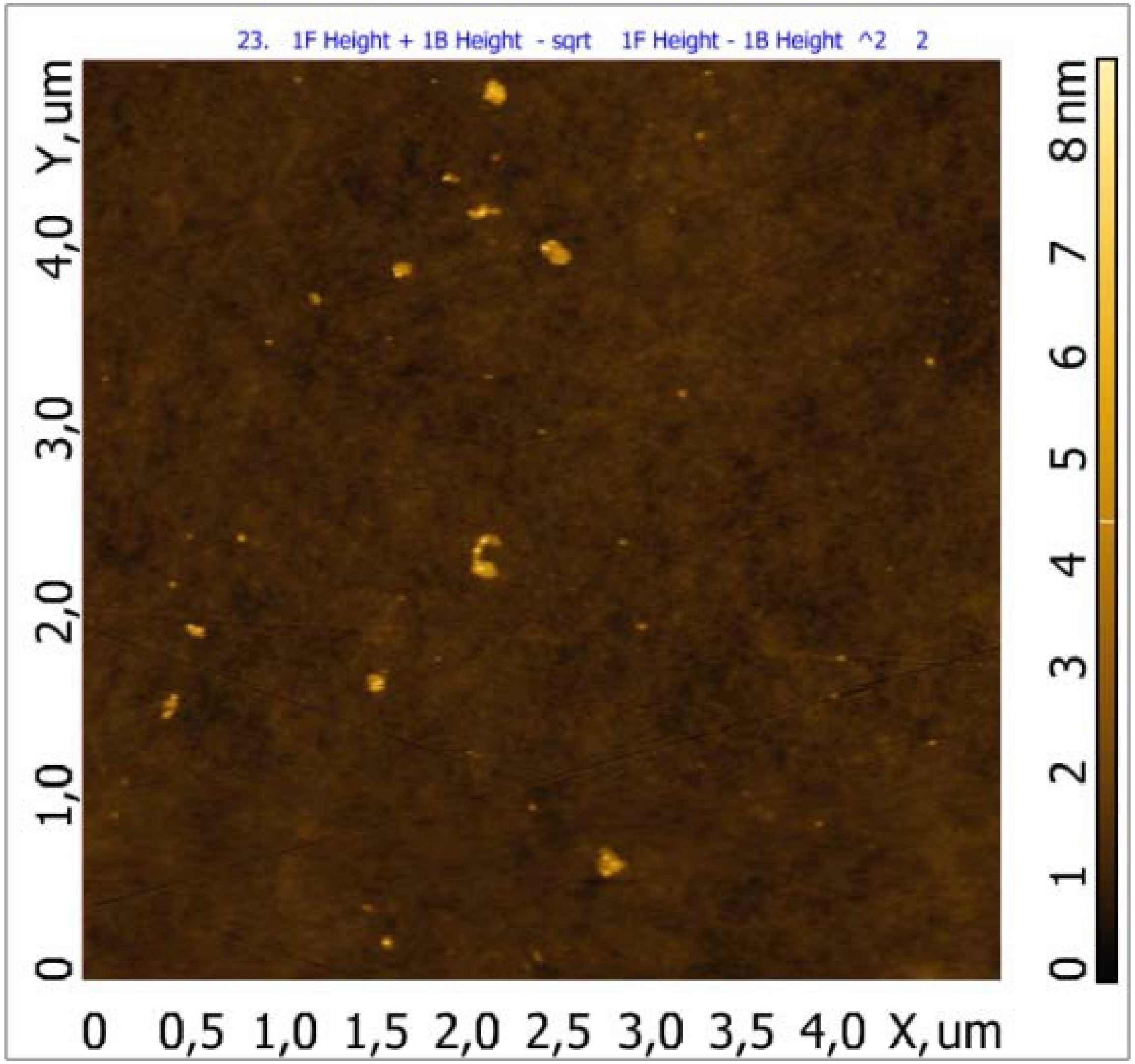


$$H = 0,5 * (H_f + H_b - |H_f - H_b|)$$

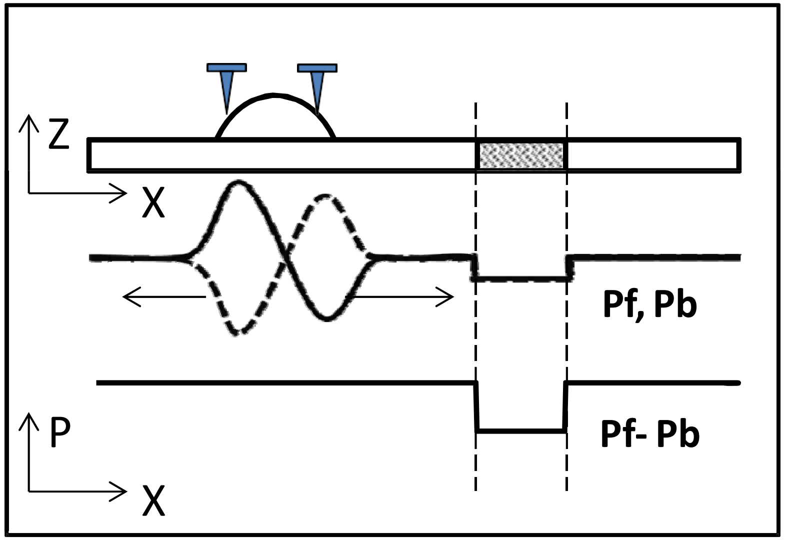
Parachuting effect in tapping mode AFM





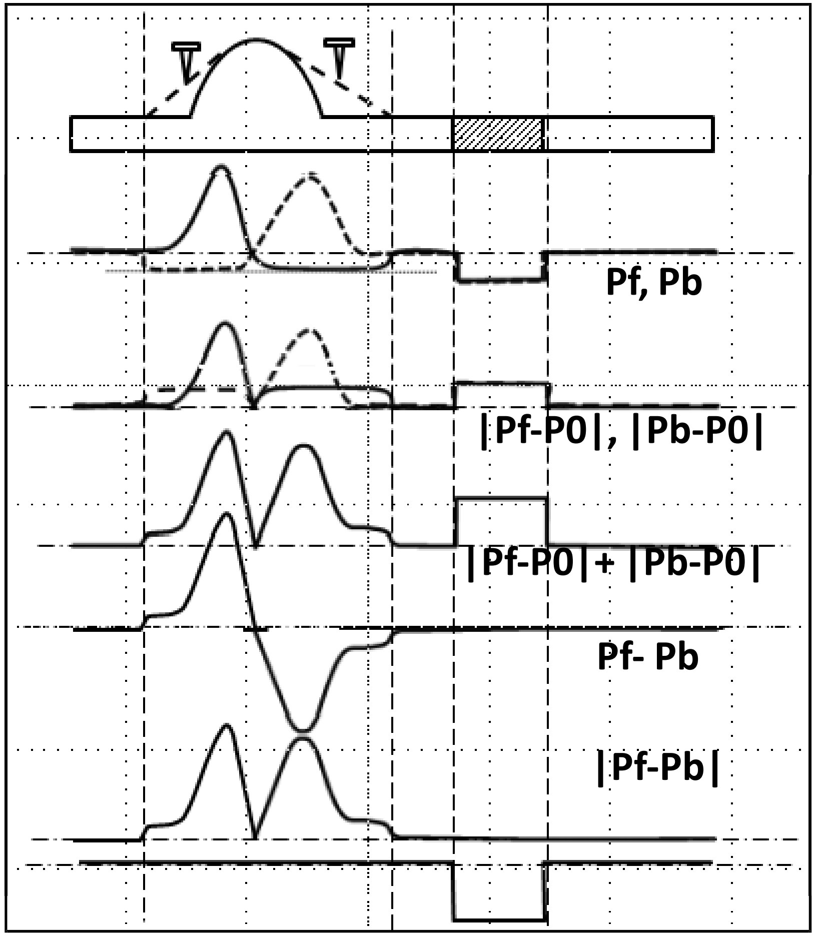


Parachuting and topography effects in phase contrast



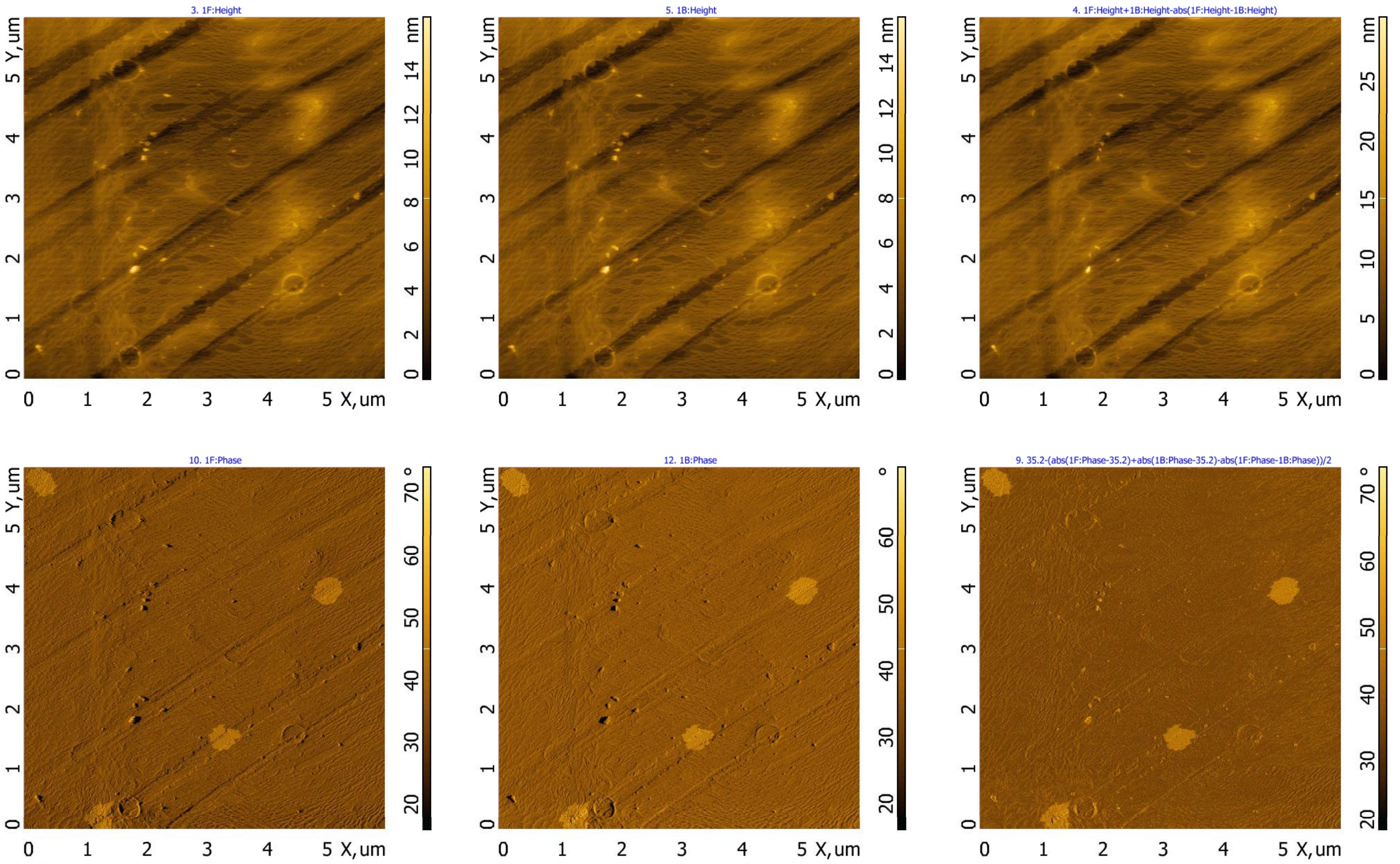
Phase signal (no parachuting) and compensation of topography influence

Po +0.5*[|Pf-Pb| - (|Pb-Po| + Pf-Po|)]

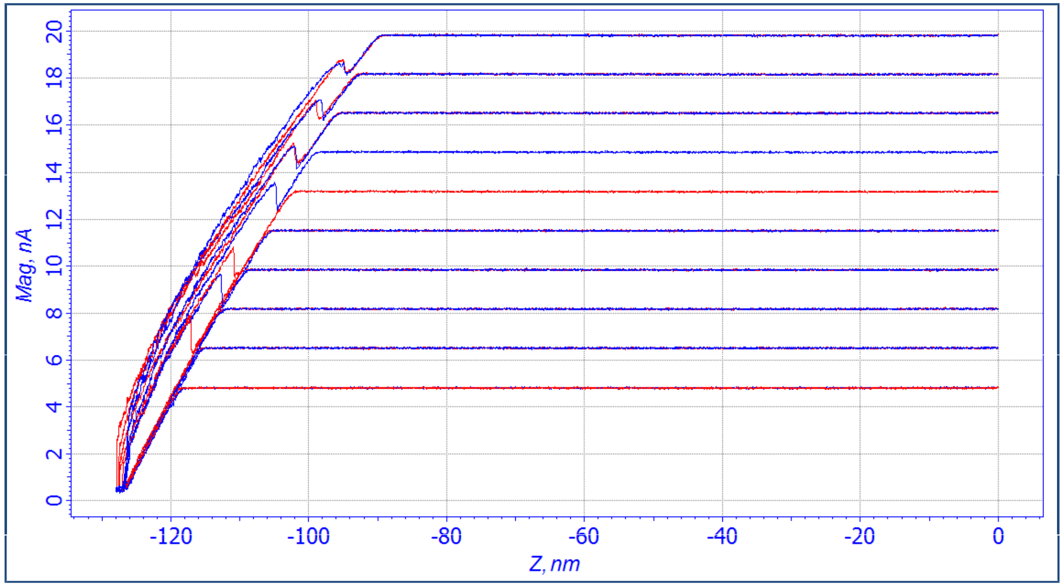


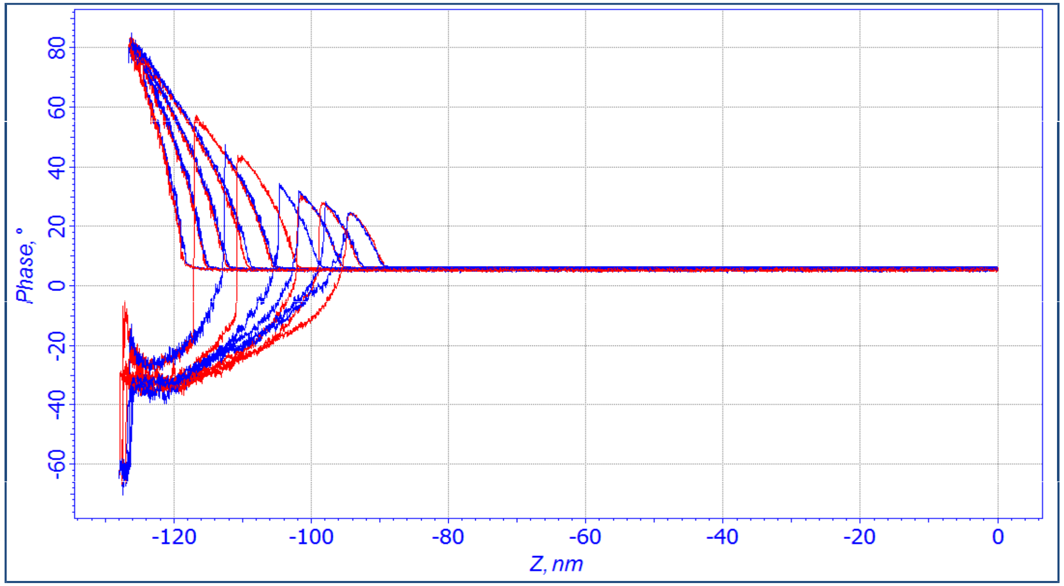
Phase signal in case of parachuting and its compensation

Parachuting and topography effects in phase contrast



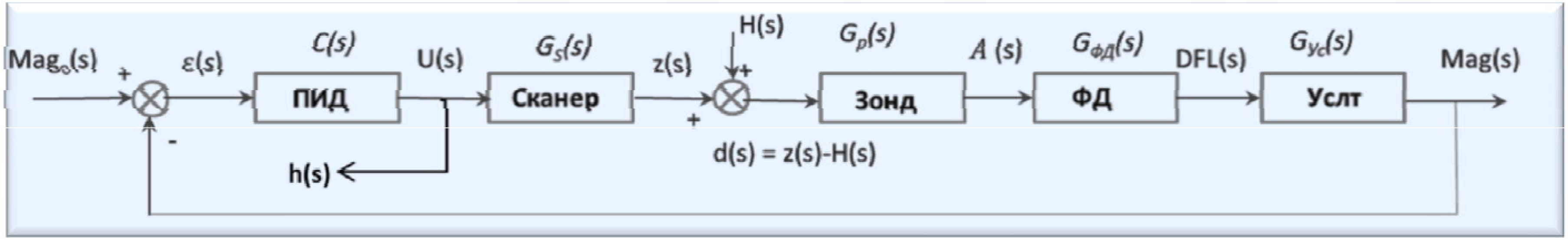
Mode switching in tapping mode AFM





Ref.: R. Garcia, R. Perez, "Dinamic atomic force microscopy methods", Surf. Science Rep. 47, 197-301, 2002

Feedback gain optimization in tapping mode AFM



$$\varepsilon = SP - Mag$$

$$\frac{dU}{dt} = k_i \cdot \varepsilon(t)$$

$$H = k_s U$$

$$Mag = k_p(H - h)$$

$$\tau_0 = \frac{1}{k_i k_p k_s}$$

$$V_x \tau_0 \cdot \frac{dH(x)}{dx} + H(x) = h(x)$$

$$\varepsilon[\mathrm{nm}] = V_x \tau_0 \cdot \frac{dH(x)}{dx}$$

Feedback gain optimization in tapping mode AFM

- Minimize **Error** signal
- Avoid mode hopping
- Avoid **Mag** signal saturation

- Integral feedback gain optimization: minimization of RMS of MAG:

$$\varepsilon = \frac{\alpha}{k_i} + \beta k_i + \gamma k_i^2 + \delta$$

- Avoid mode switching: setting amplitude so that there is no mode switching, that can be controlled using **Phase** signal

- Avoid **Mag** signal saturation: if saturation happens – decrease Set Point; if Set Point is less than 50% of free amplitude – adjust speed of scanning

$$\varepsilon[\mathrm{nm}] = V_x \tau_0 \cdot \frac{dH(x)}{dx}$$

Restart

Mode SemiContact Topo

Rate 1.40000 Hz Time: 0:06:06

Params

Direction

View

Quick

Sections

Resonance Approach

Signals	Sub
F:Height	S1
B:Height	S1
F:Mag	Off

Graphs: If:Hei

Start

Status: StandBy F =0.058 nN

Save data

Regime	Amp limit
Attr action	30

Main options

Optimization mode	Start Mode
Base	Current
N prescan	
1 1	Amp ON

Optimized parameters

	Initial	Actual
Amp	0.0010	0.0442
LockGain	20.0	38.4
zI	0.55	0.20
zP	7.00	9.80
zSP	0.900	9.800
Rate	1.4000	1.4000
lpfMag	11000.0	1000.0
Shift	0	-8

Save set

r(Height) =1.0000

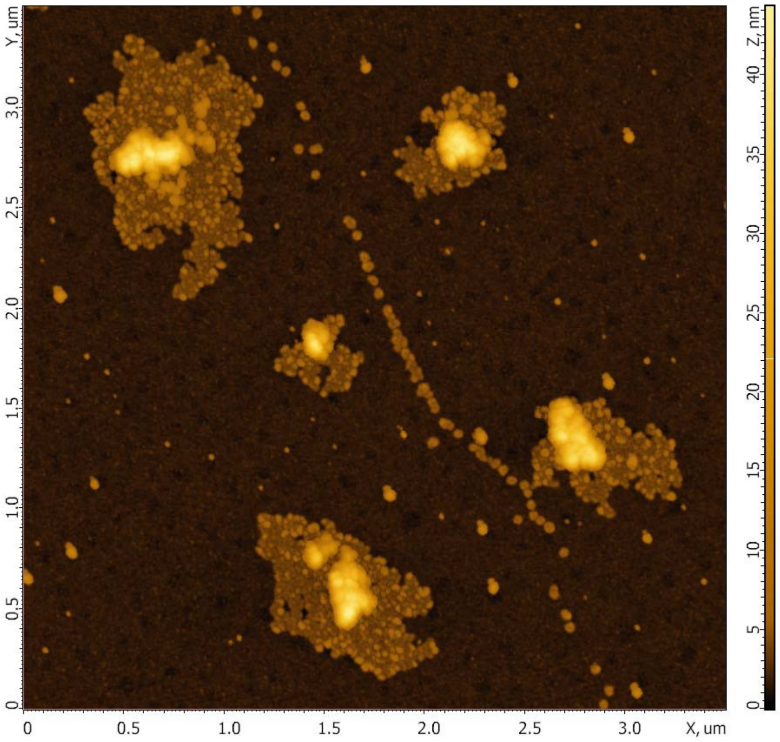
RMS(Mag) =0,0092

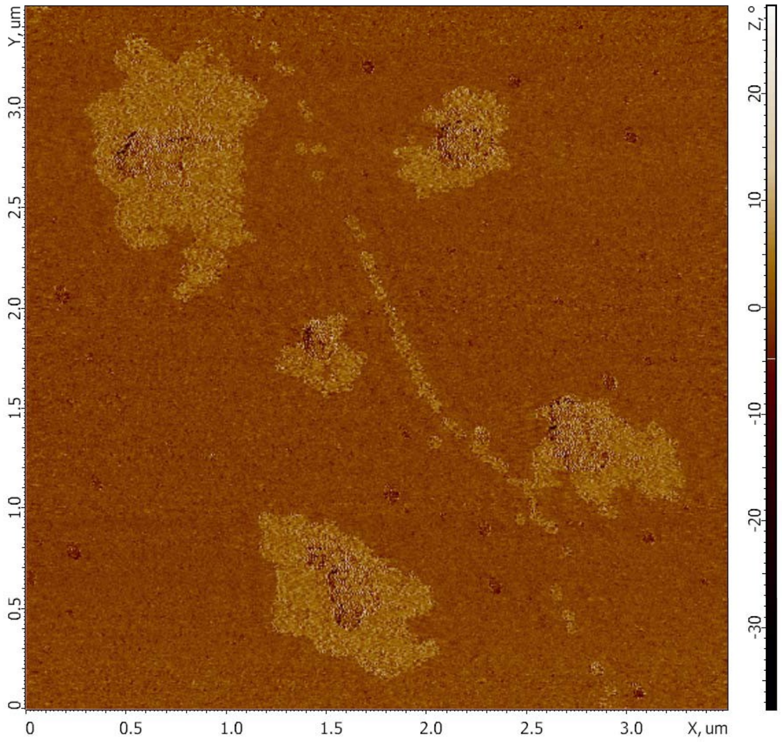
RMS(Mag) =0,0092

0 50 100 150 200 250 X, 10^-3 um

f:Height Min Order 217

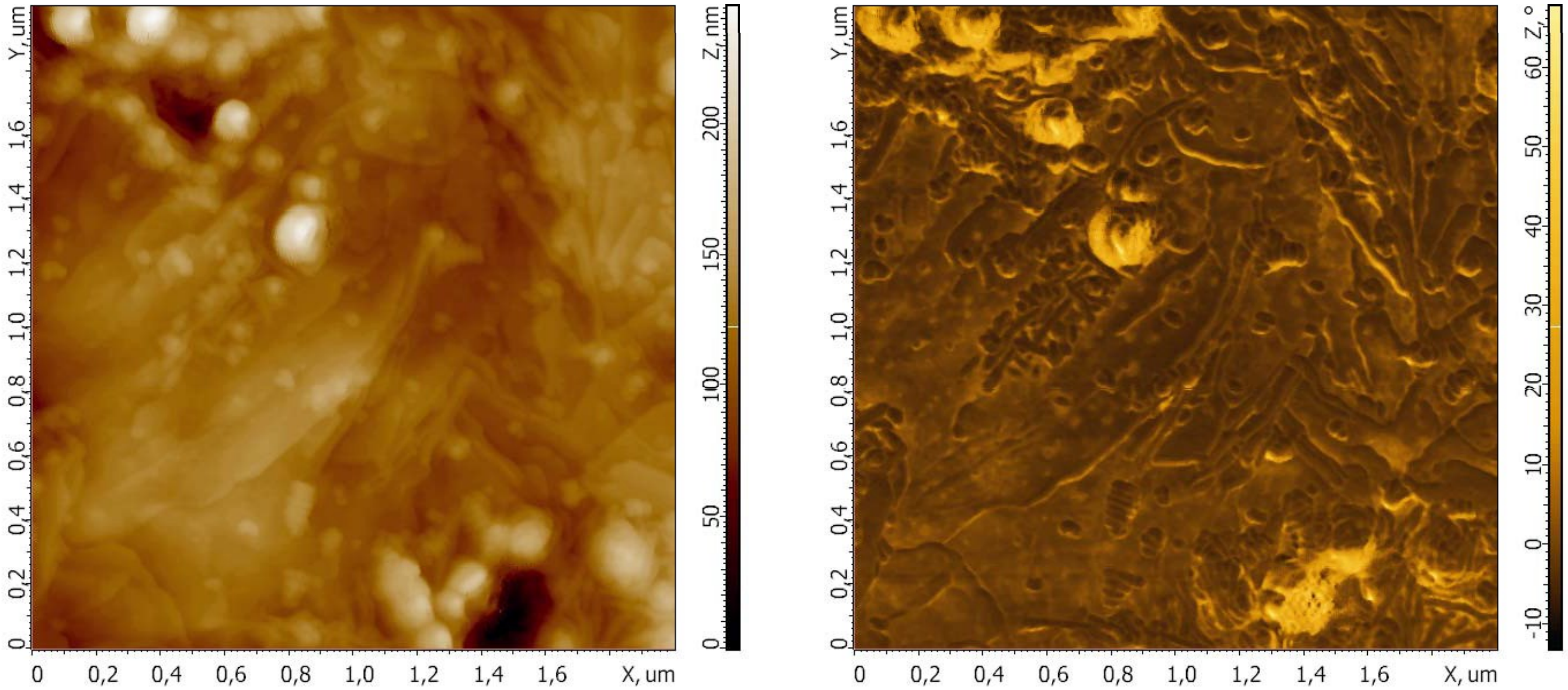
Scan Tronic: Examples of application





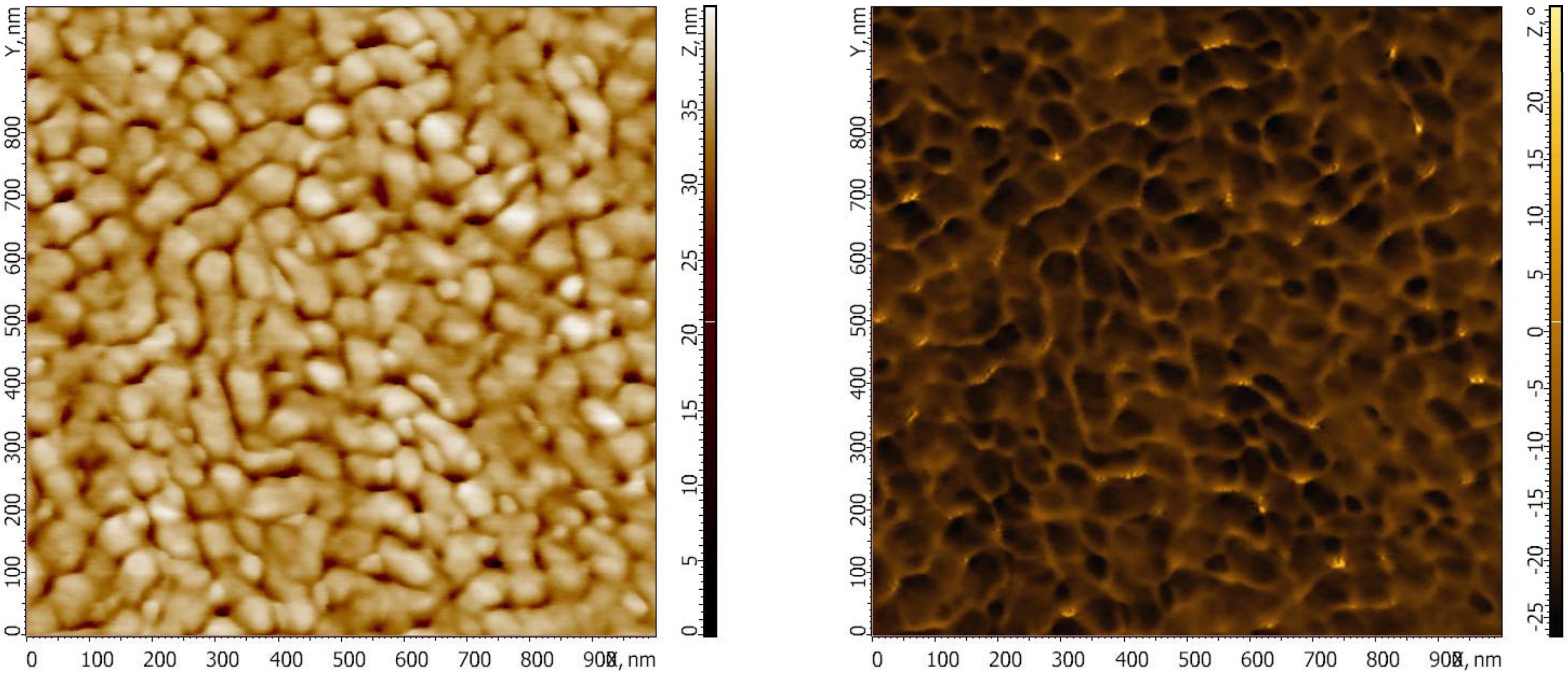
Fluoroalkanes $F_{14}H_{20}$ on Si. Left – topography, right – phase contrast
Scan size $3.5\times3.5\ \mu m$

Scan Tronic: Examples of application



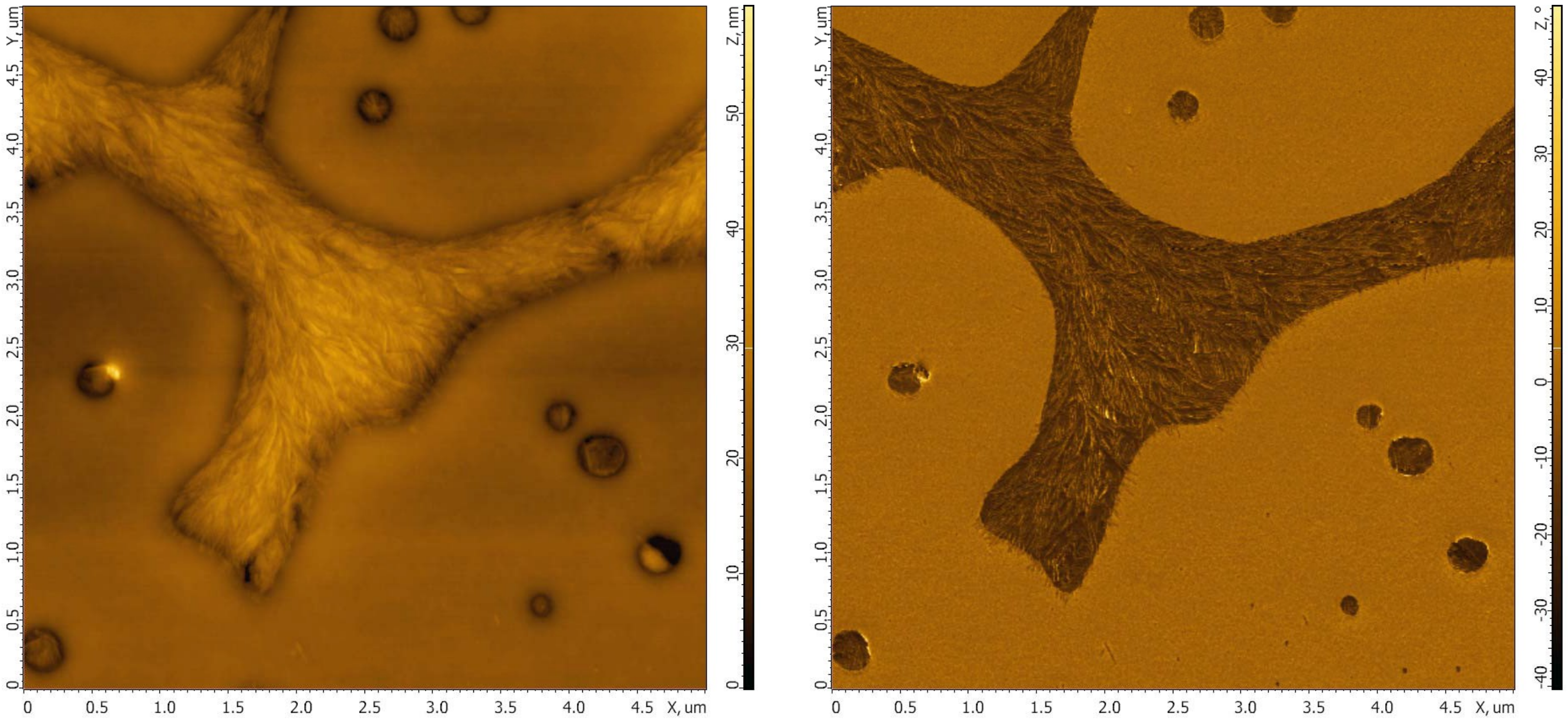
HDPE. Left – topography, right – phase contrast
Scan size 2×2 μm

Scan Tronic: Examples of application



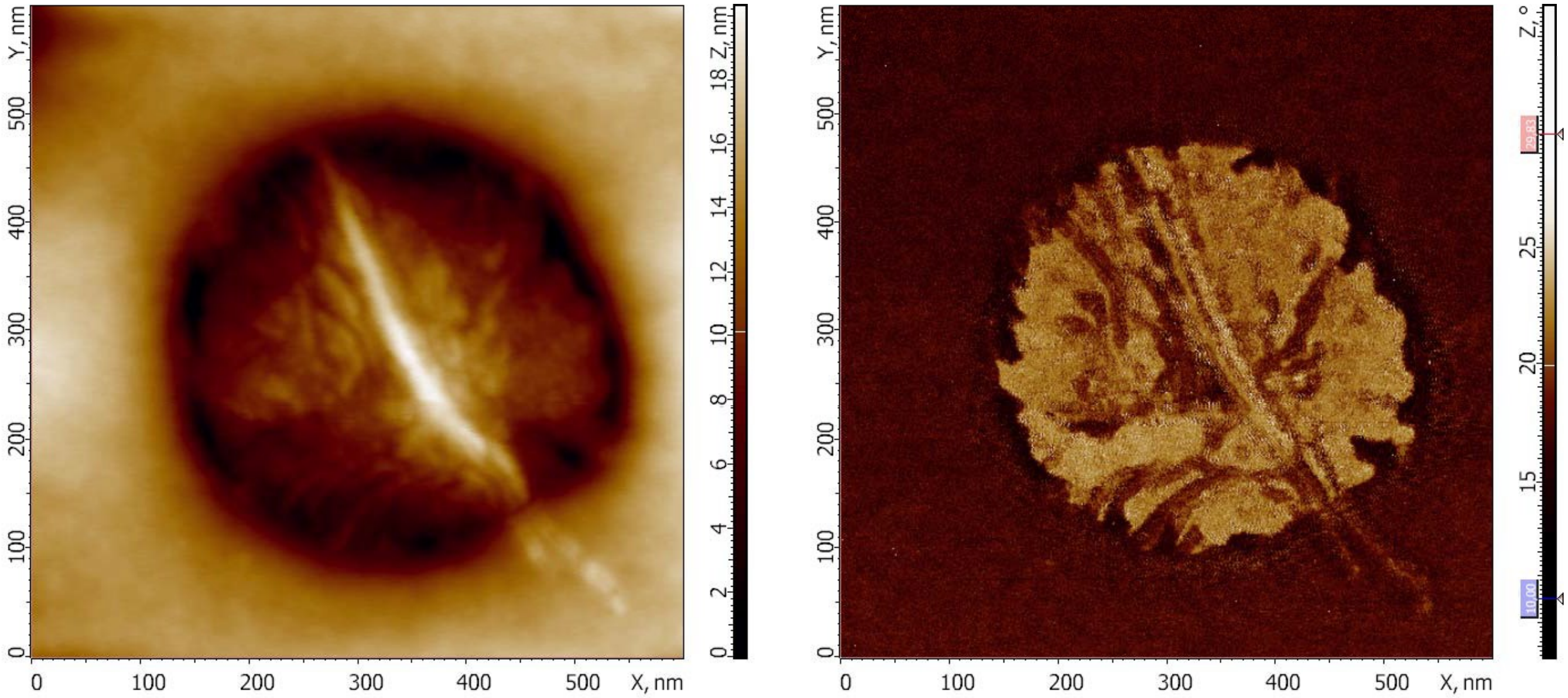
PS-b-PMMA. Left – topography, right – phase contrast
Scan size 1×1 μm

Scan Tronic: Examples of application



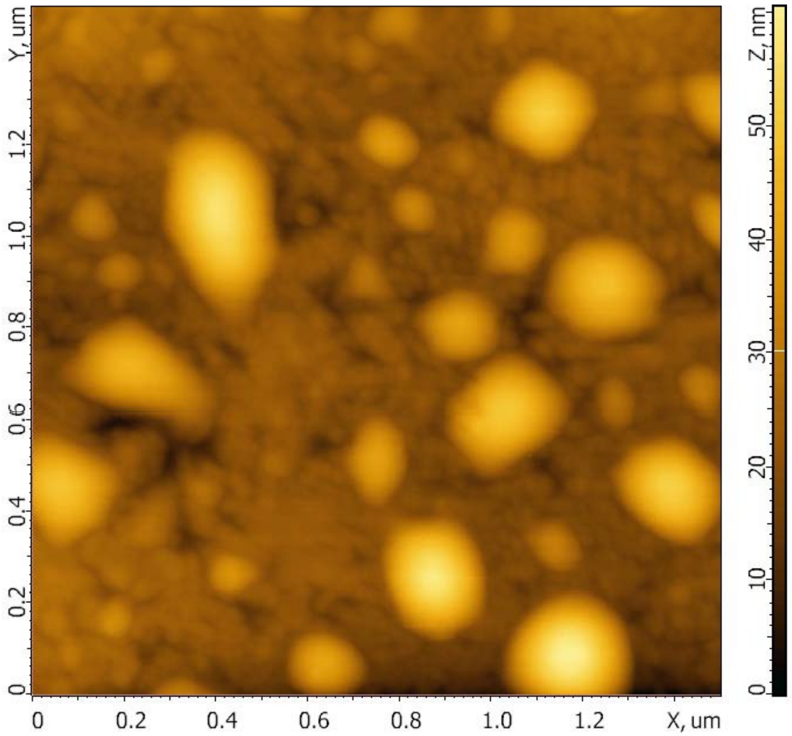
PVDF-sPS. Left – topography, right – phase contrast
Scan size 5×5 μm

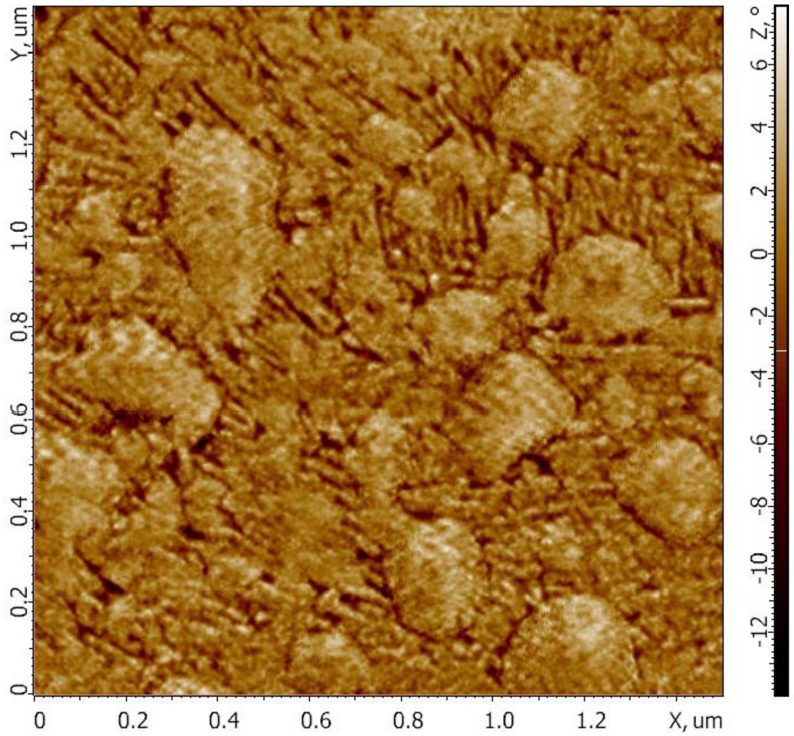
Scan Tronic: Examples of application



PVDF-sPS. Left – topography, right – phase contrast
Scan size 600×600 nm

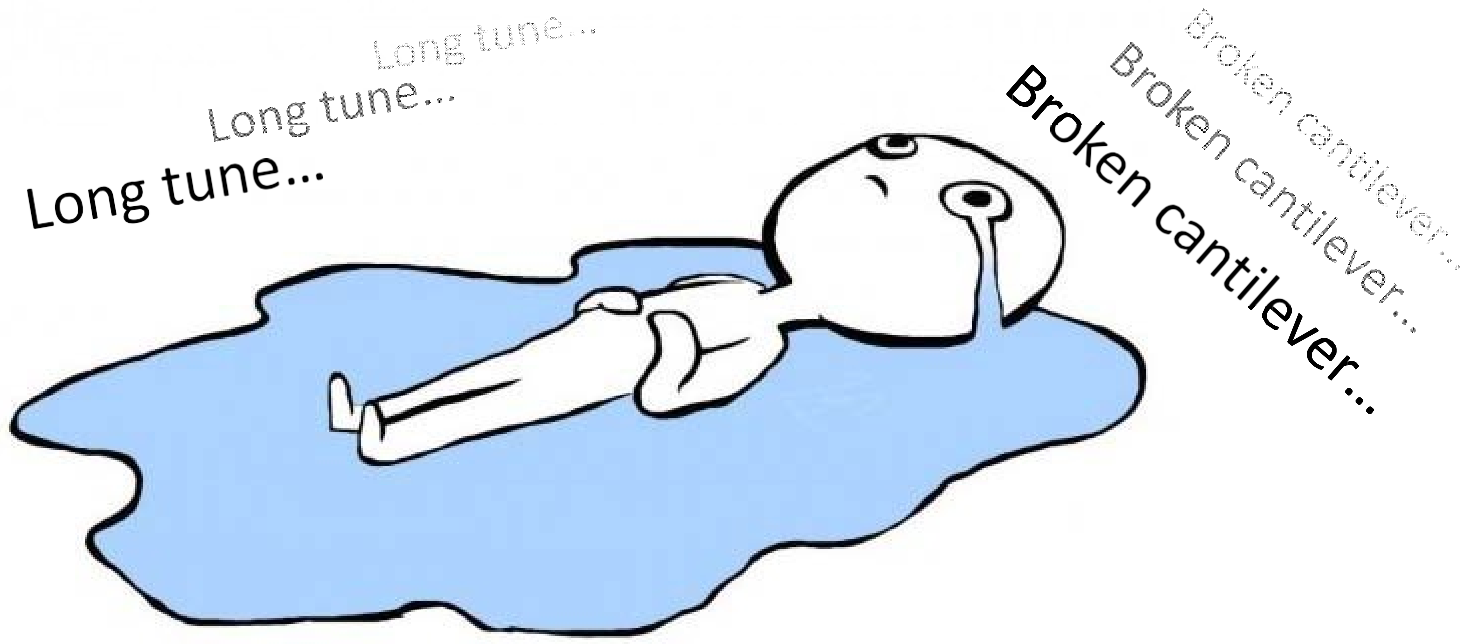
Scan Tronic: Examples of application



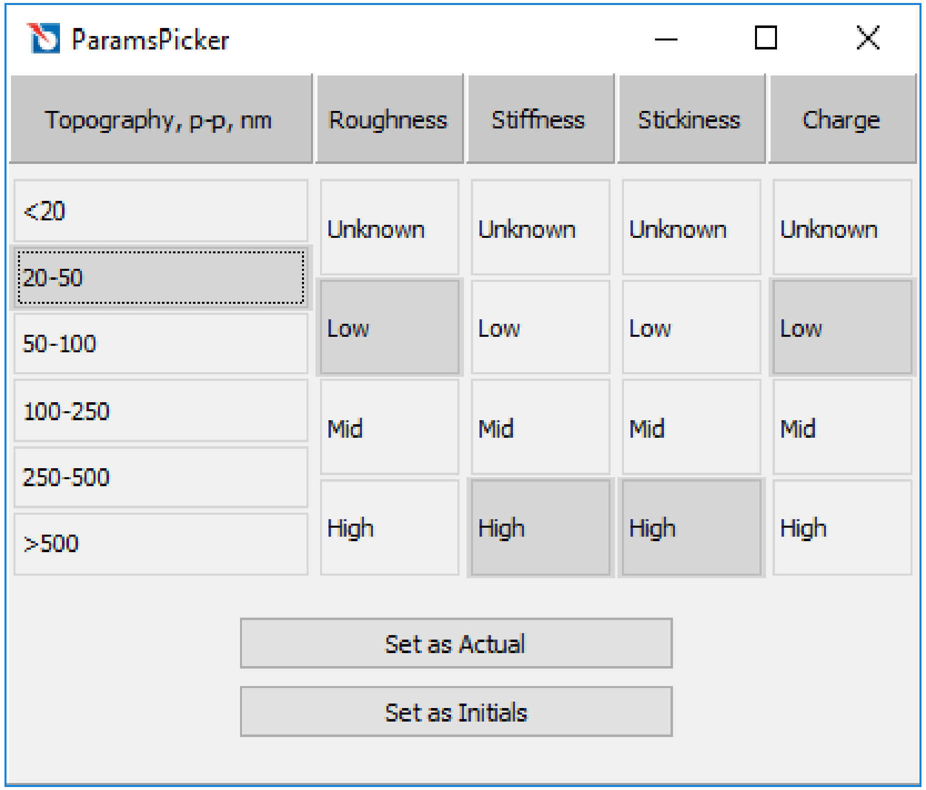


TPV. Left – topography, right – phase contrast
Scan size 1.5×1.5 μm

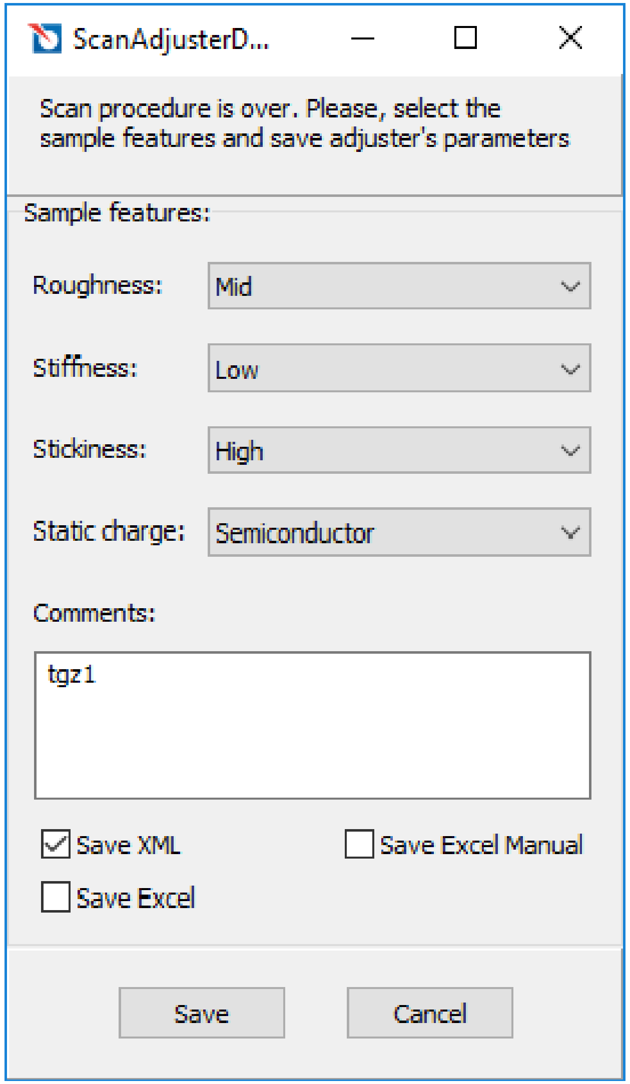
Setting initial parameters



Setting initial parameters: machine learning



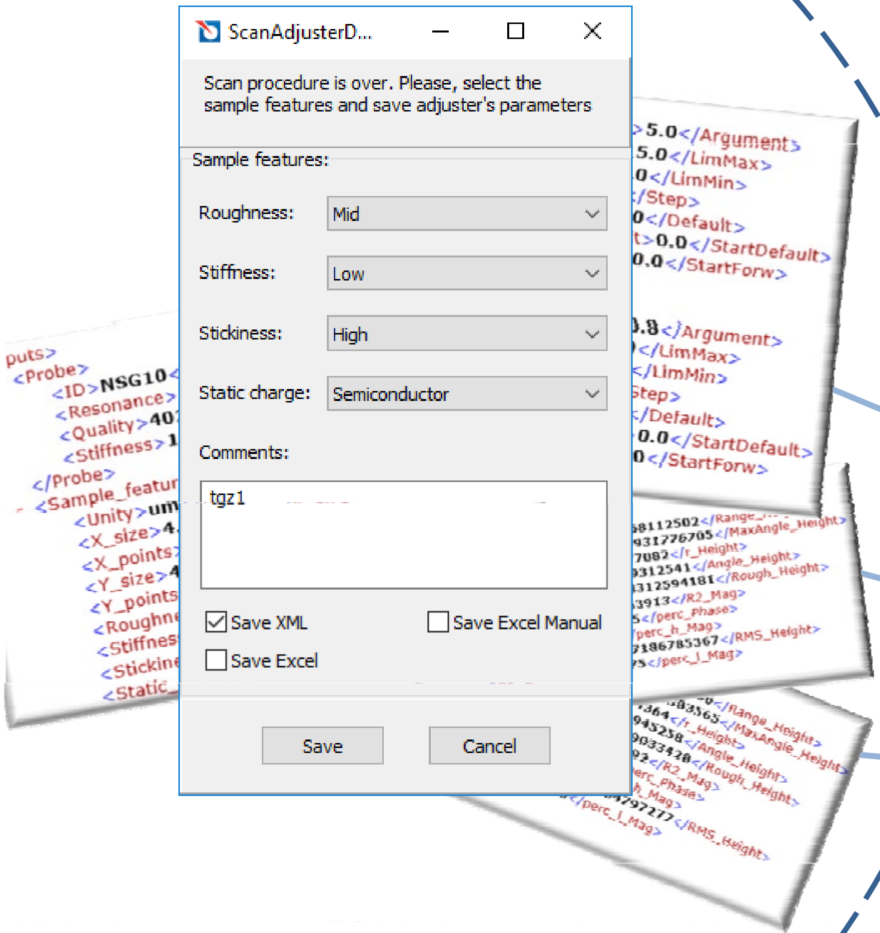
Before experiment



After experiment

Setting initial parameters: machine learning

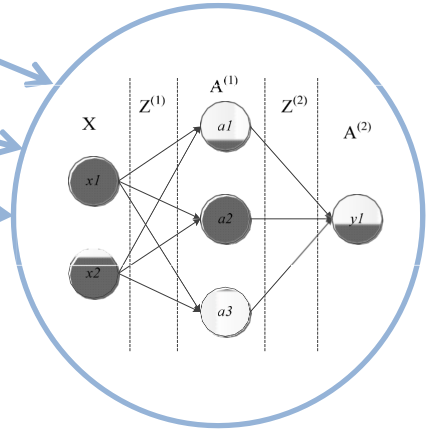
Saving



Learning

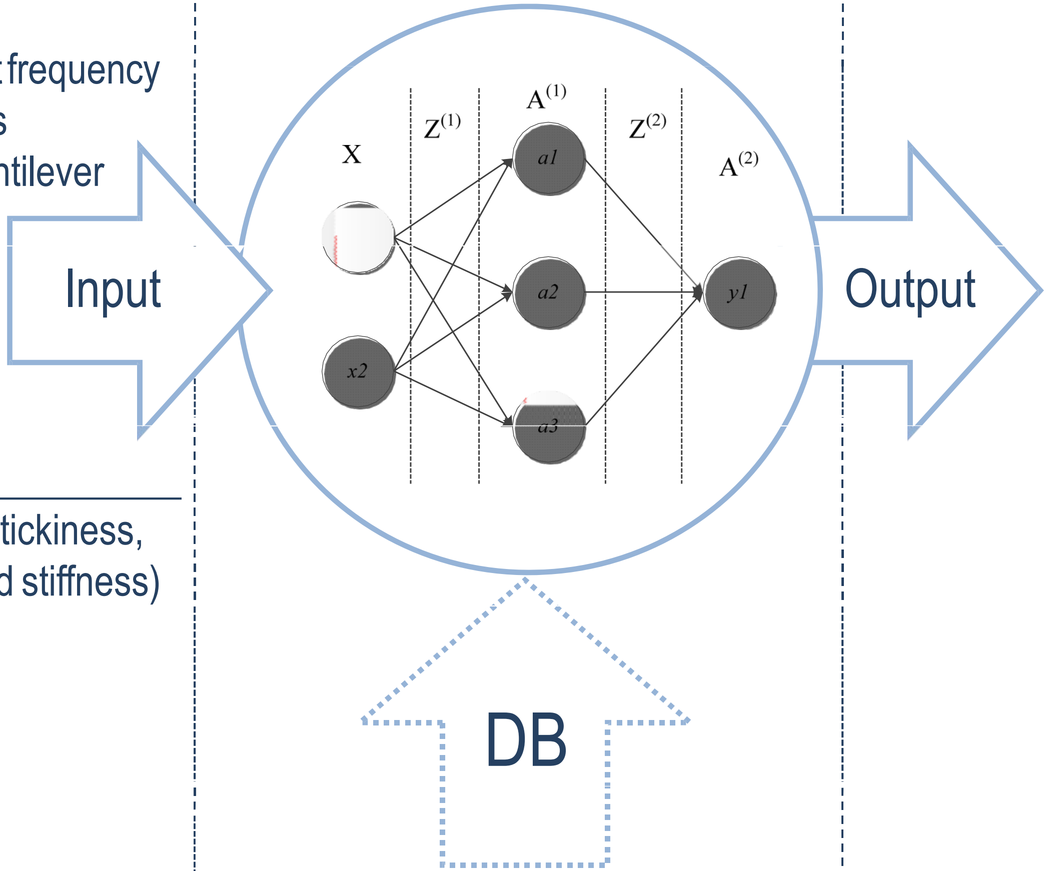
Based on the backpropagation method using the BFGS algorithm (Broyden-Fletcher-Goldfarb-Shanno)

The search for the minimum of the objective function makes it possible to find and adjust the optimal synapse weights



Setting initial parameters: machine learning

f – cantilever resonant frequency
K – cantilever stiffness
Q – Q-factor of the cantilever
Nx – number of pixels
Scan size



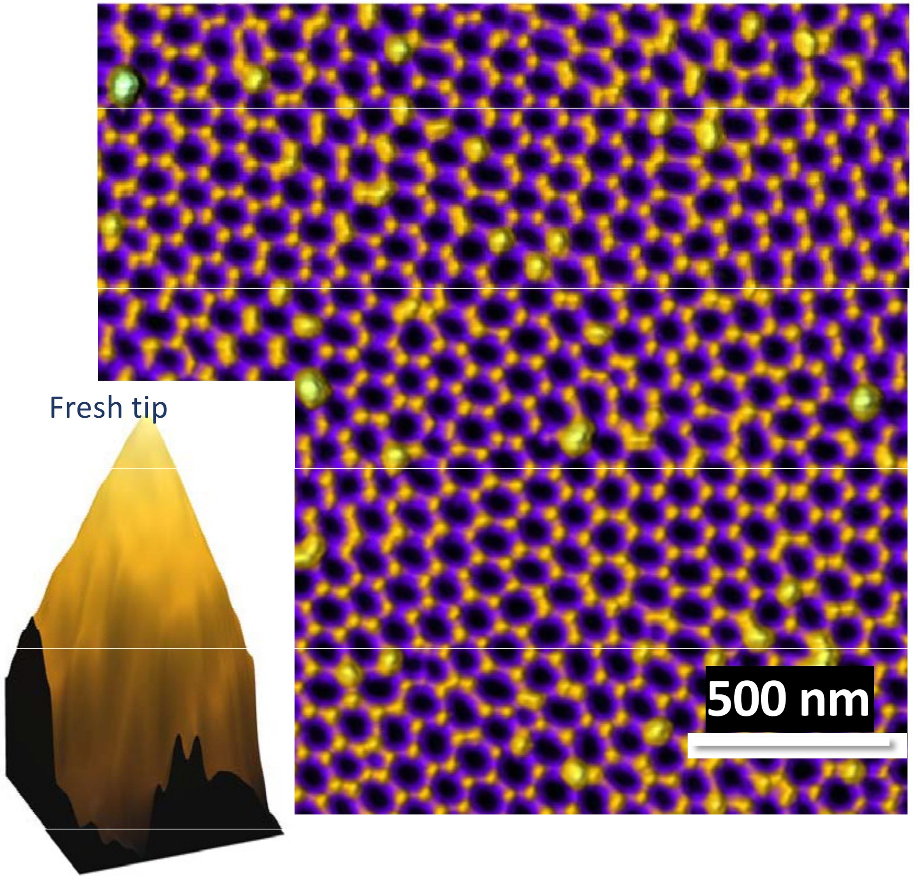
P_0 and I_0 feedback gains
SP_0 – Set Point
V_0 – Scan rate

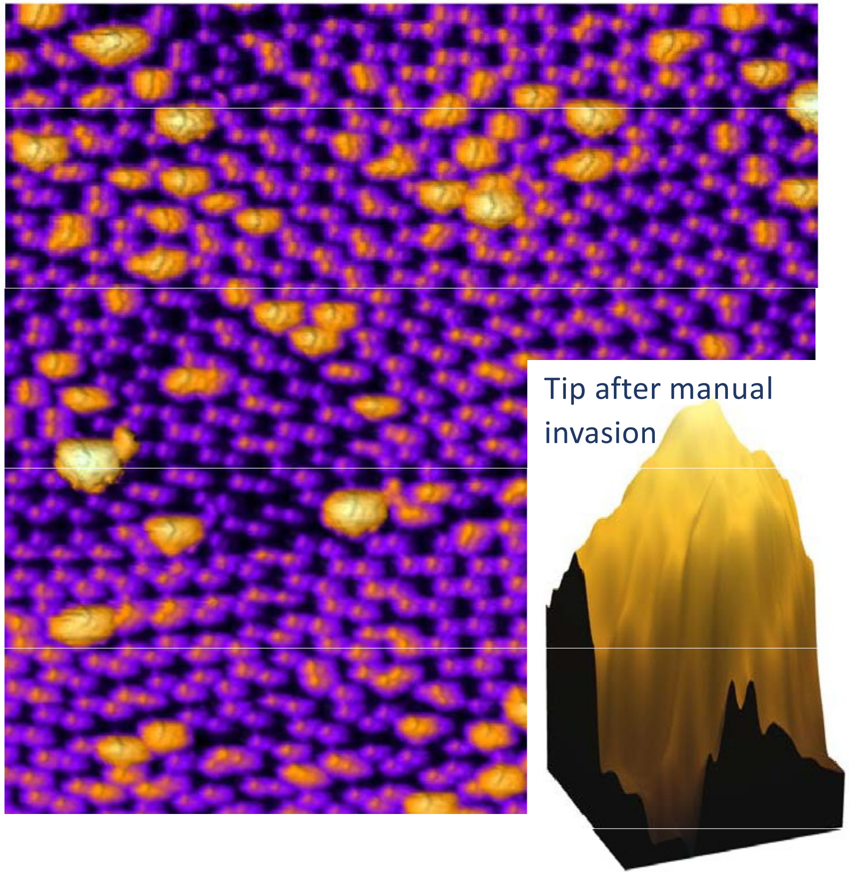
Sample parameters (stickiness, charge, roughness and stiffness)

NT-MDT
Spectrum Instruments

ScanT™

Scan Tronic: Examples of application





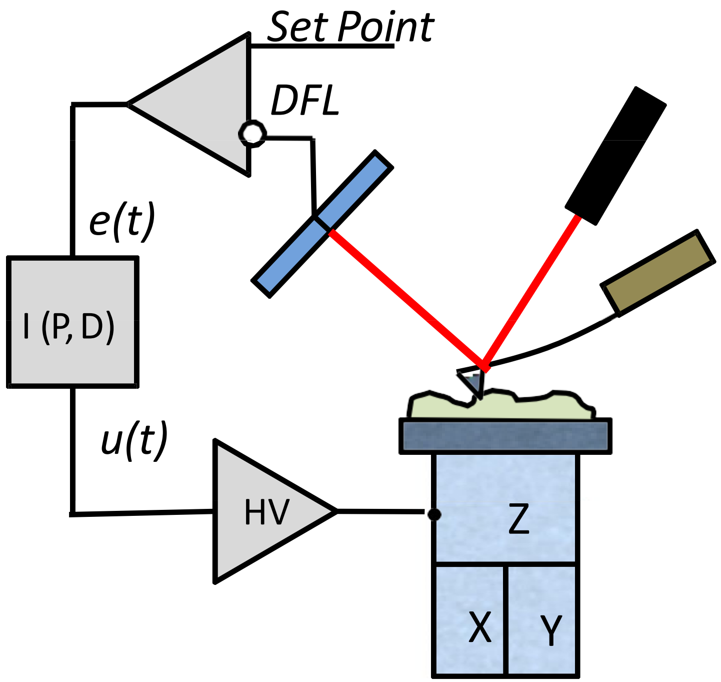
Al_2O_3 - “Grater” sample for tips.
Left – topography, ScanT used, right – manual attempt to adjust scanning parameters

Rapid Scan

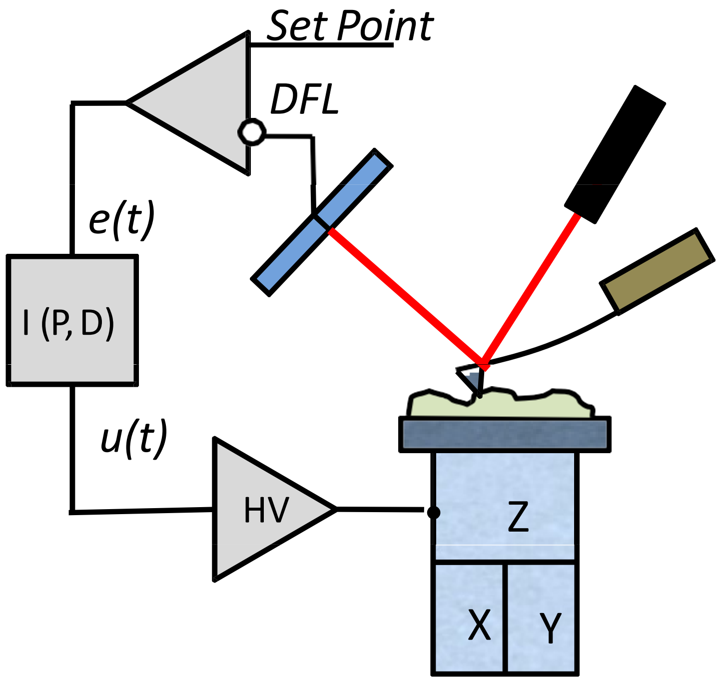
Fast scanning and High-speed scanning

- **”High-speed AFM”** is usually referred to video-rate AFM, working with the scan speed of 10 – 100 frames per second:
  - Small sample size
  - Topography imaging only
  - Very complex to operate with
  - Small FOV

- **”Fast scan AFM** is usually referred to
  - Smaller FOW (compare to regular AFM)
  - Scan rates ~ 10 lines per second

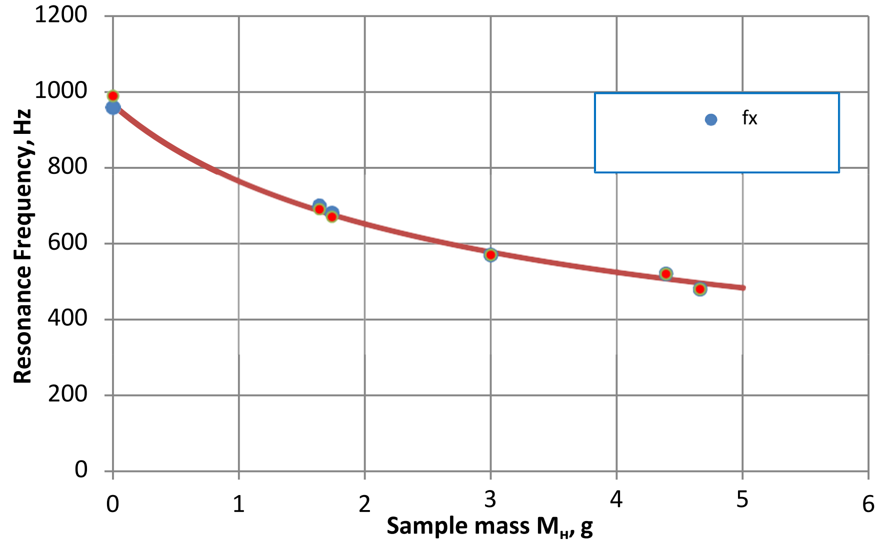


Fast scanning

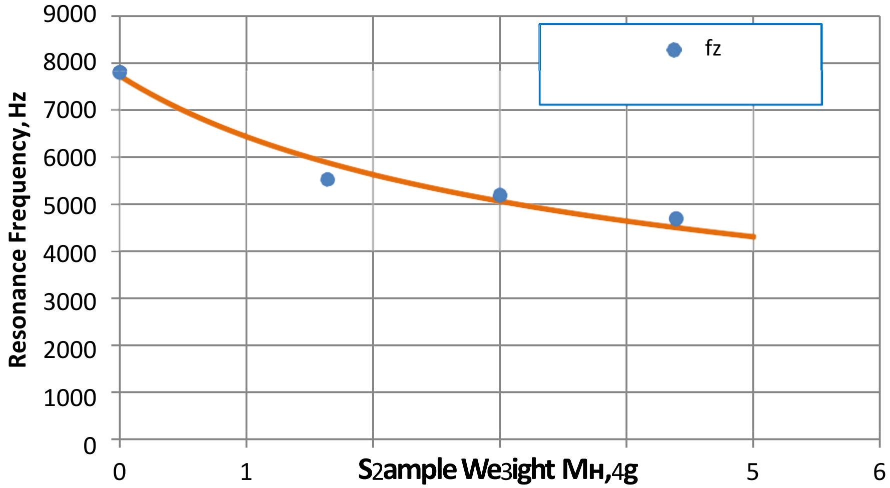


$$f_R = \frac{1}{2\pi}\sqrt{\frac{k}{(M_0 + M_\text{H})}},$$

k – effective spring constant of the scanner,
M_0 - effective mass of the scanner,
M_H - mass of load



Scanner resonant frequency (XY)

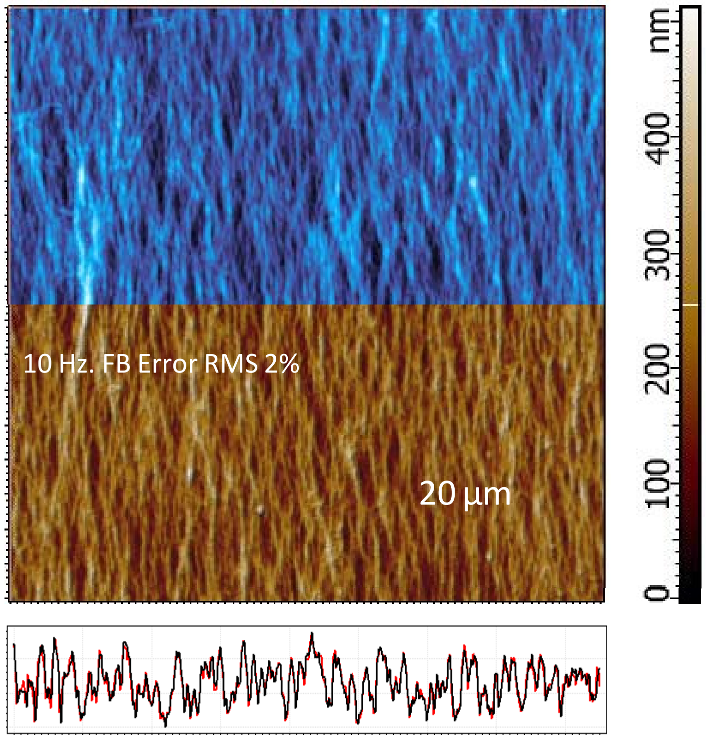


Scanner resonant frequency (Z)

Rapid Scan 100

Rapid Scan 100 technology is a combination of mechanical design and high-end digital electronical solutions which allows to speed up your AFM by an order of magnitude keeping 90 µm in-plane scaninng range.

All three access are equipped with high-precision closed-loop capacitive sensors.



90×90×0,5 µm image of collagen fibers captured @ 1 & 10Hz scanning rates

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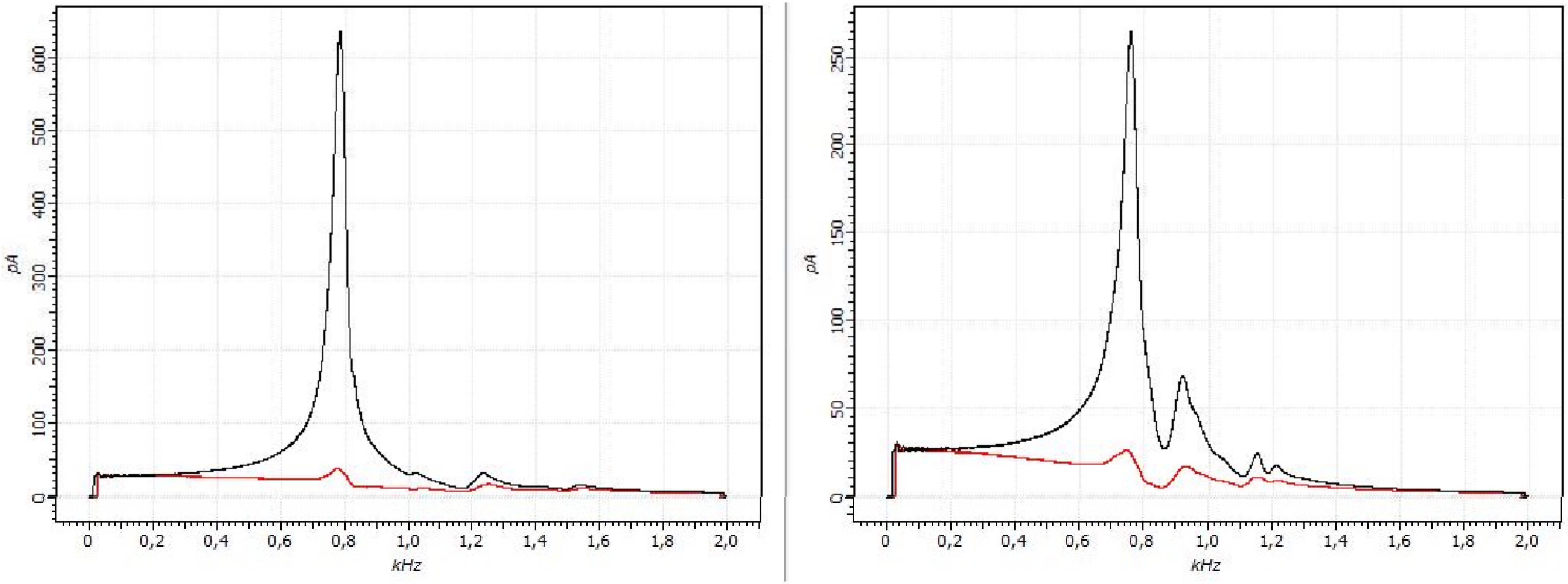
All three access are equipped with high-precision closed-loop capacitive sensors.

Parameter	Value
Travel range (XY/Z), µm	90×90×4 ±10%
Closed loop sensors (XYZ)	Capacitive
Sample size, ⌀ mm	15
Vertical noise floor, pm	30
XY position noise (Closed Loop), nm	0,1
Nonlinearity, %	0,1
Resonance Frequency (XY/Z), kHz	0,8/12
Active resonance damping	+

Rapid Scan 100: damping of scanner resonance

Low-pass filter + High-pass filter + Band-pass filter



Damping of X (left) and Y (right) scanner resonances by digital filtration

Summary

- **ScanT**™ technology allows to eliminate common AFM artefacts and drastically improve quality of AFM results

- **S anT**™ is l wering si nificantl cantilever consumption for any AFM lab

- Together with **ScanT**™, **RapidScan**™ technology increase scan rates up to ~ 10 times without compromise with maximum XY visible area

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Spring Life Shows

Thank you!