Wrocław University of Science and Technology
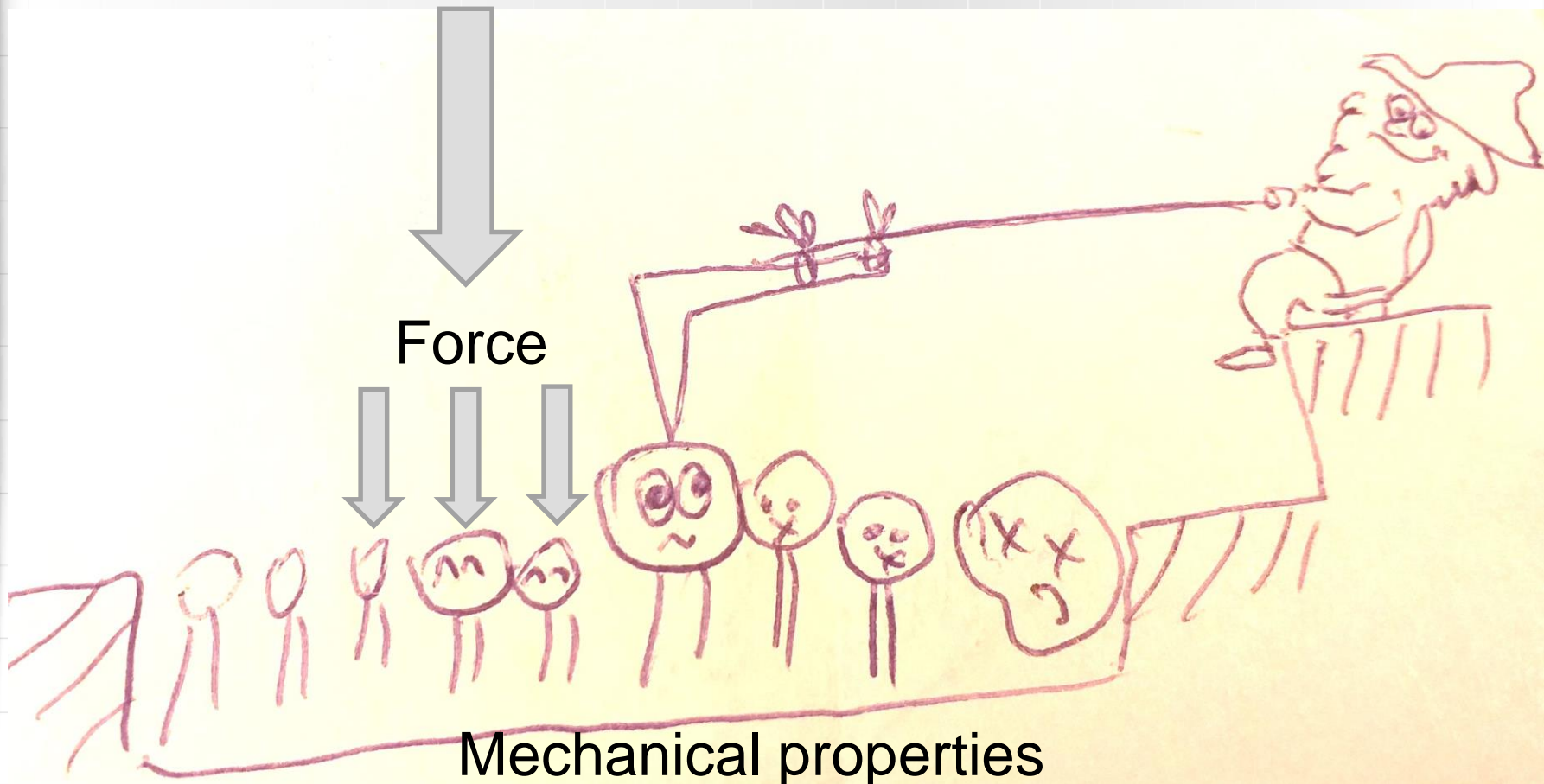
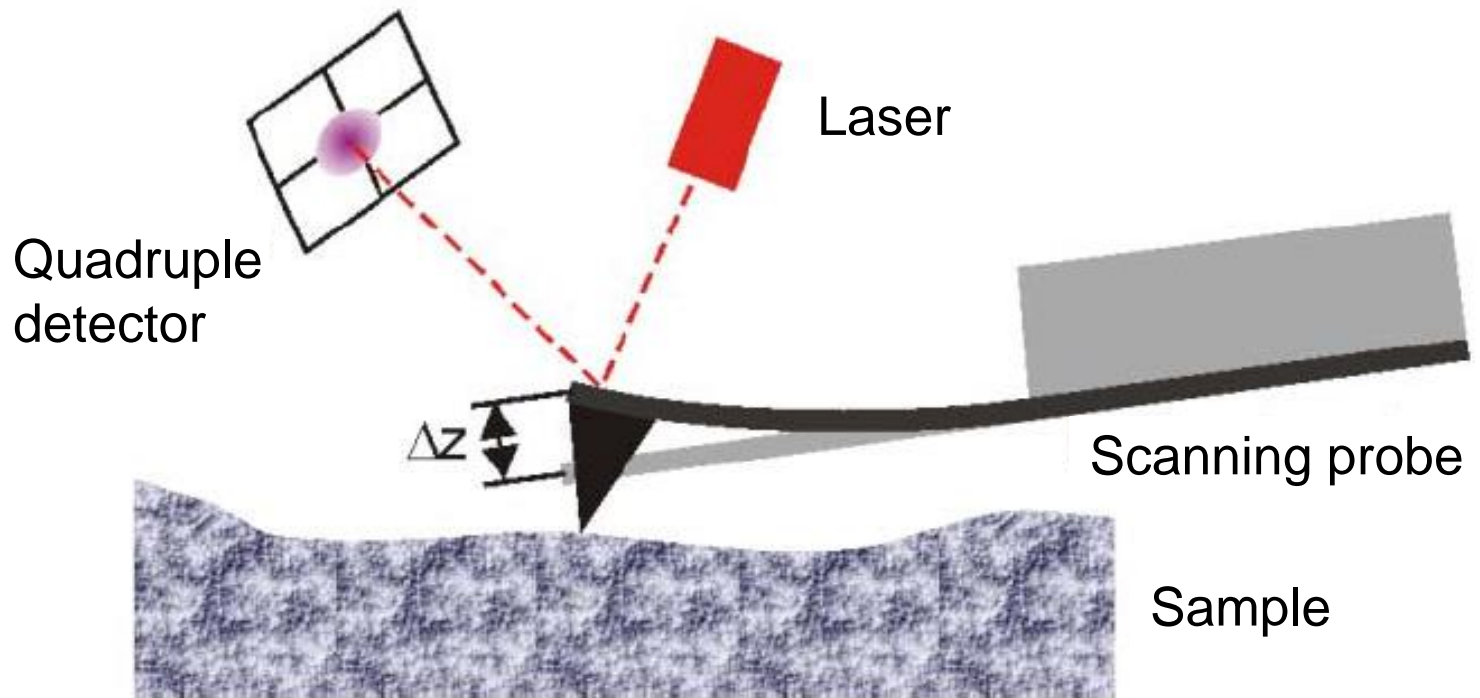# The utilization of AFM's tip-sample interaction for the surface morphology imaging and mechanical properties mapping

Andrzej Sikora
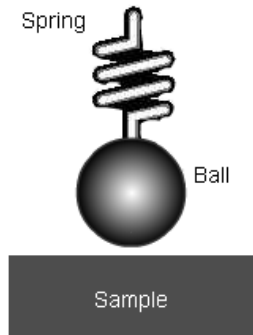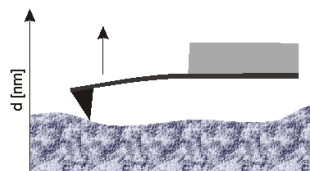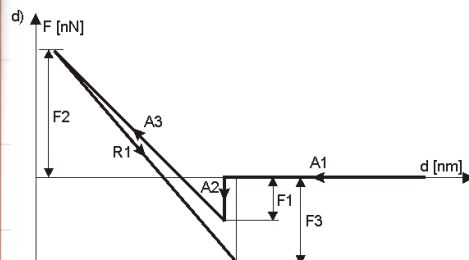
Starting from the principles…

SPM is about the tip–sample interaction observation

Force

Mechanical properties

Starting from the principles…

Laser

Quadruple detector

Δz

Scanning probe

Sample

Detecting tip-sample interaction
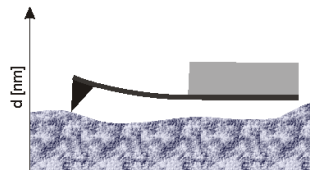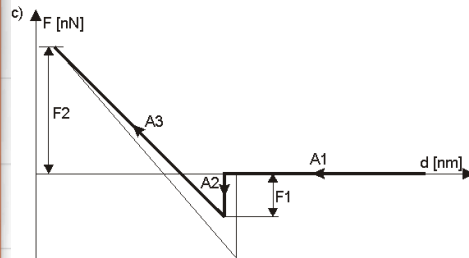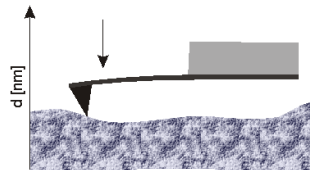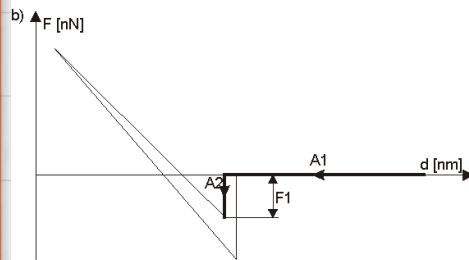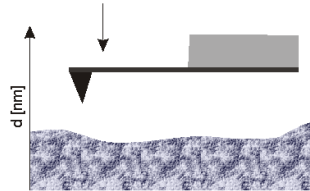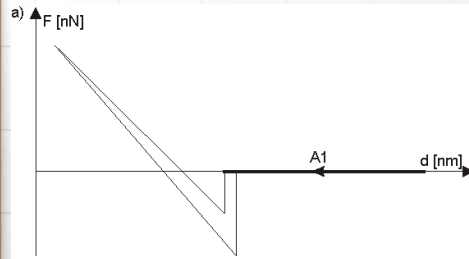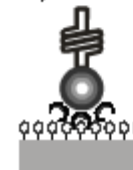
Enabling semicontact / phase imaging

Measured signal

Laser

Quadruple detector

Piezoactuator

Excitation signal

ϕ

A
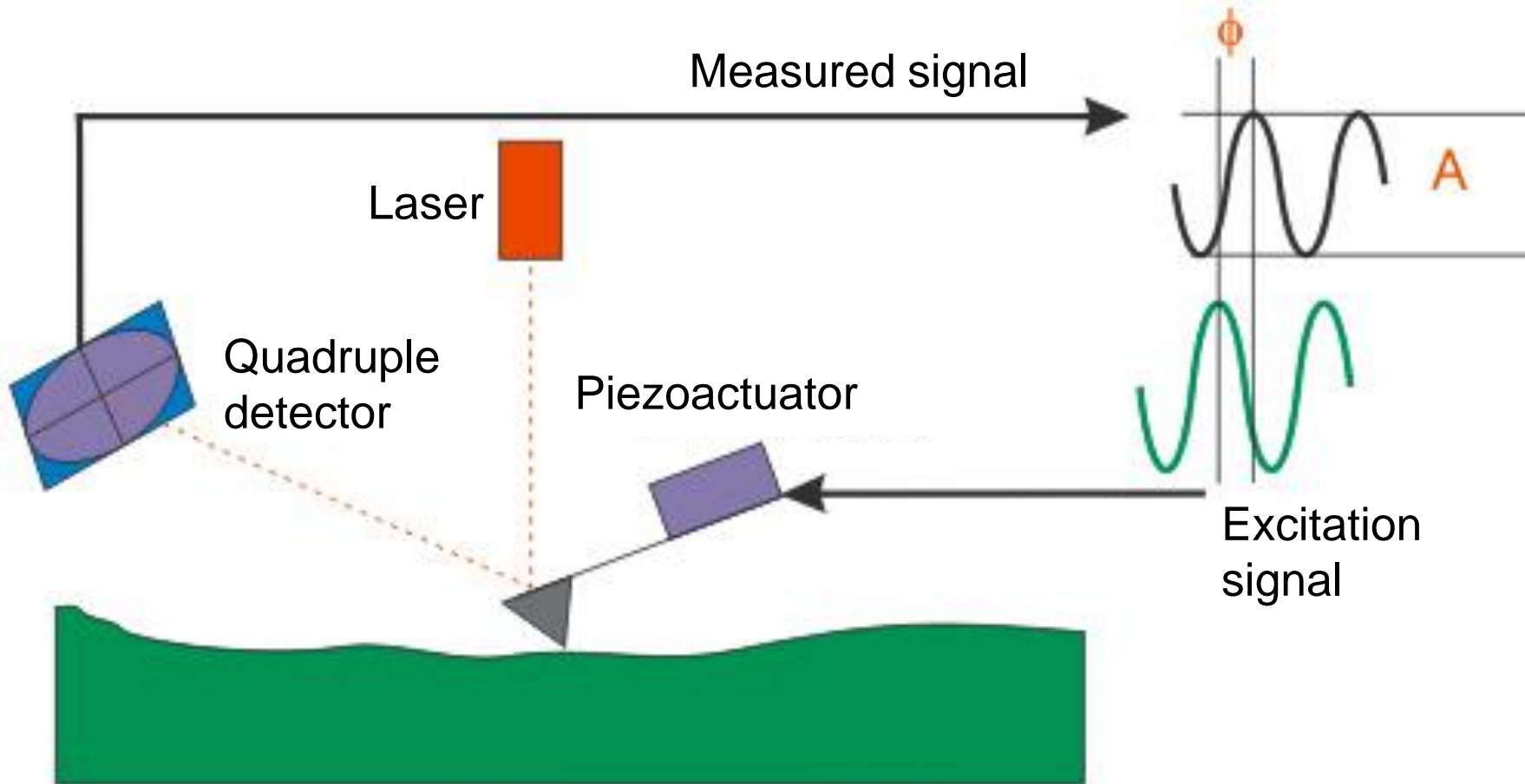
Behind phase imaging

$$\phi = \tan^{-1}\left(\frac{k}{Q\sigma}\right) \approx \frac{\pi}{2} - \frac{Q\sigma}{k} = \frac{\pi}{2} - \varepsilon a E^* \frac{Q}{k}$$

$$\sum \frac{\partial F_i}{\partial z} = \sigma$$

Tip-sample forces derrivative

Magonov S.N.S., Elings V., Whangbo M.-H.
Phase imaging and stiffness in tapping-mode atomic force microscopy
Surface Science, 375 (2)-(3), p. L385-L391, 1997

$$\phi = \tan^{-1}\left(\frac{\omega_0}{2Q^{eff}\Delta\omega}\right) \approx \frac{\pi}{2} - 2Q^{eff}\frac{\Delta\omega}{\omega_0}$$

$$Q^{eff} = 2\pi\frac{W_0}{W_d^{eff}}$$

Effective quality of the setup, connected to the energy dissipated in tip-sample setup

Whangbo M.-H, Bar G., Brandsch R.
Description of phase imaging in tapping mode atomic force microscopy by harmonic approximation
Surface Science, 411, p. L794-L801, 1998

$$\sin\phi = \frac{\omega}{\omega_0}\frac{A_{sp}(\omega)}{A_0} + \frac{Q E_{dis}}{\pi k A_0 A_{sp}(\omega)}$$

$$E_{dis} = \oint F_{ts}\frac{dz}{dt}dt$$

Energy dissipated in tip-sample setup

Bar G., Brandsch R., Whangbo M.-H.
Description of the frequency dependence of the amplitude and phase angle of a silicon cantilever tapping on a silicon substrate by the harmonic approximation
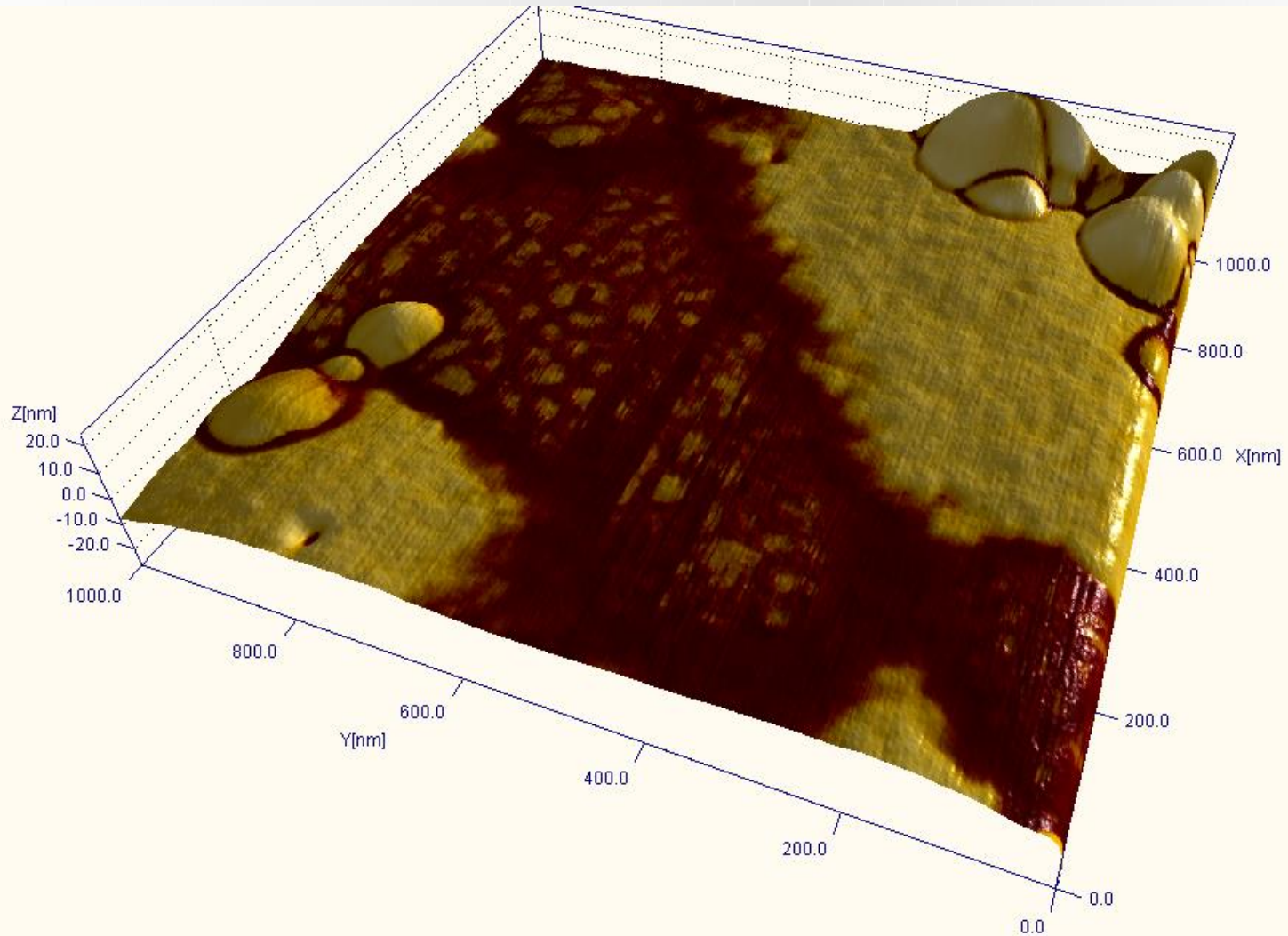Surface Science, 411 (1)-(2), p. L802-L809, 1998

6

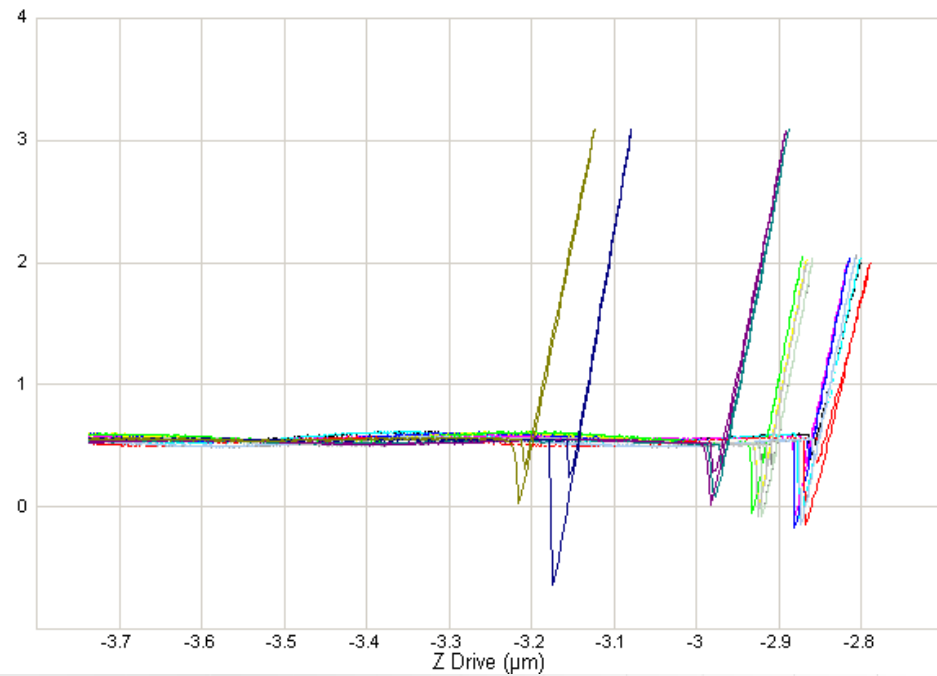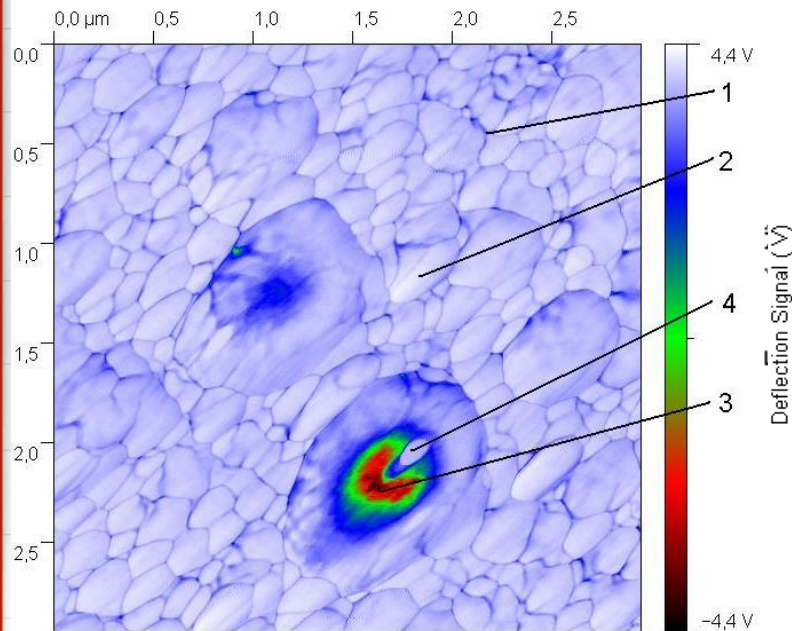# Phase imaging used for topography imaging / alcane
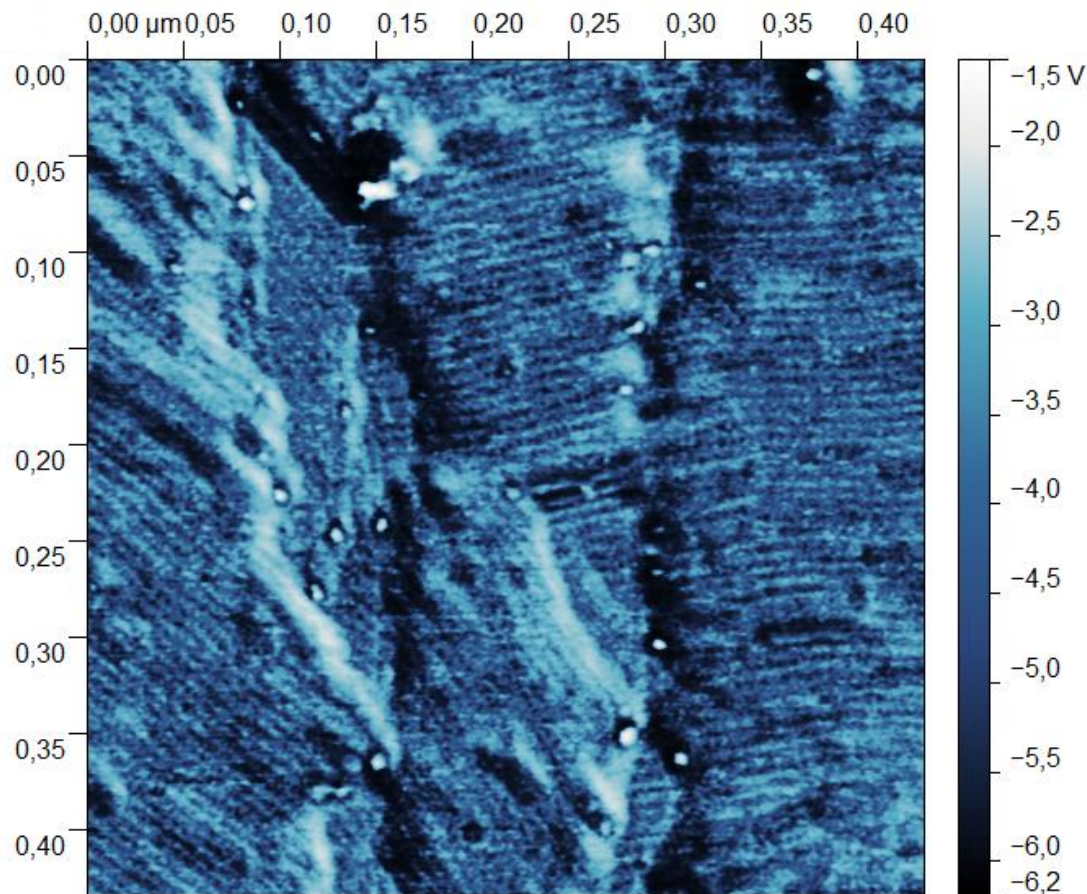
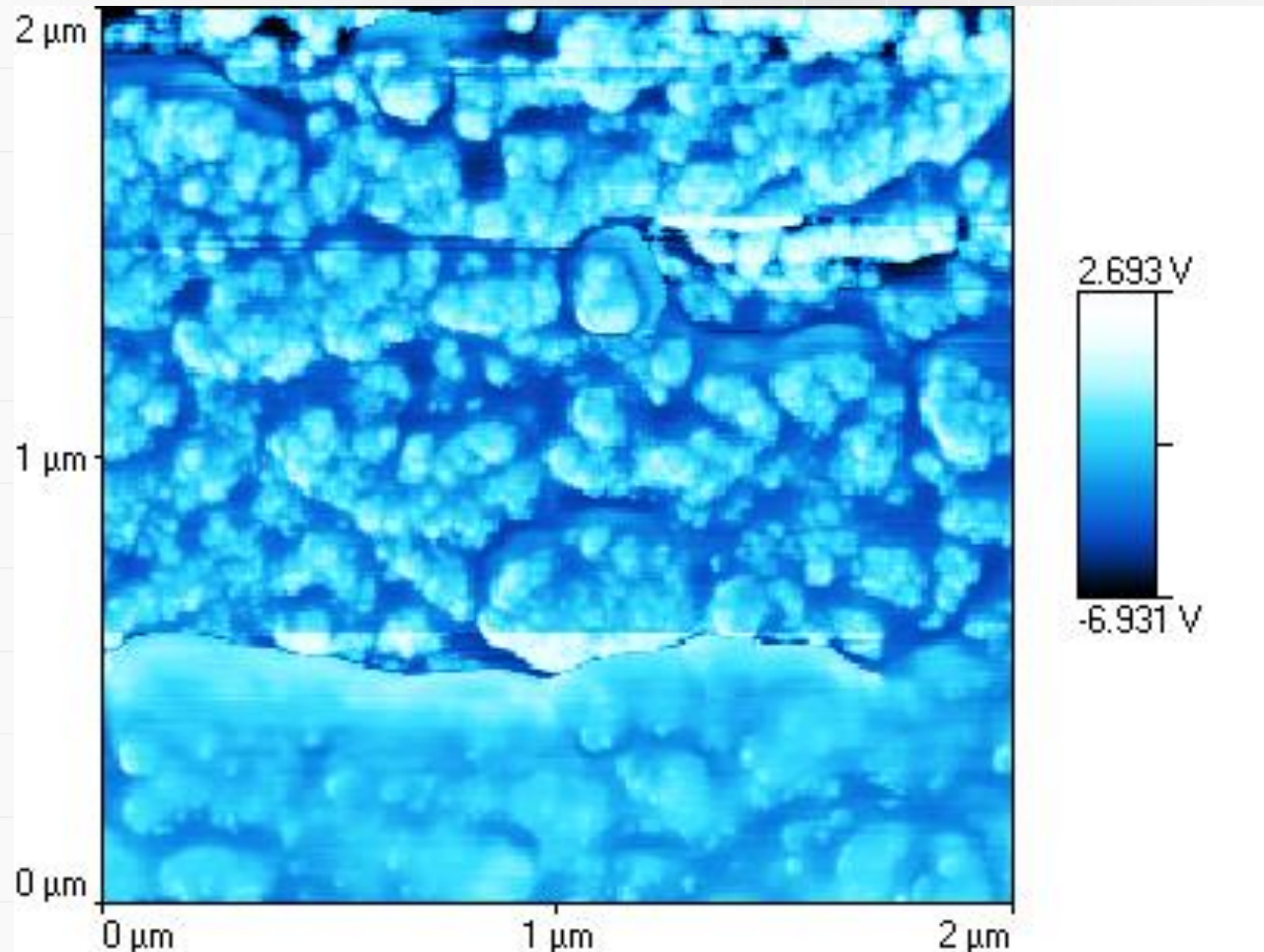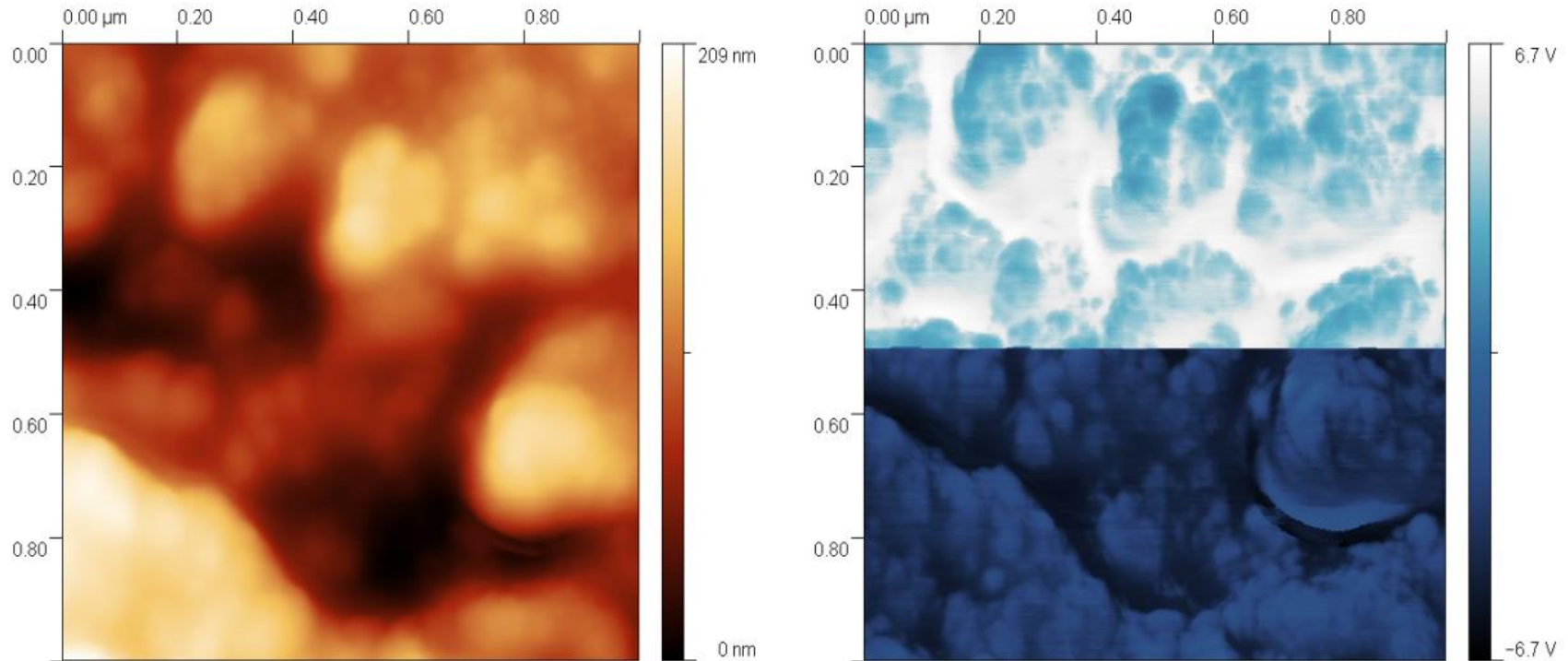# Phase imaging – polymerization map
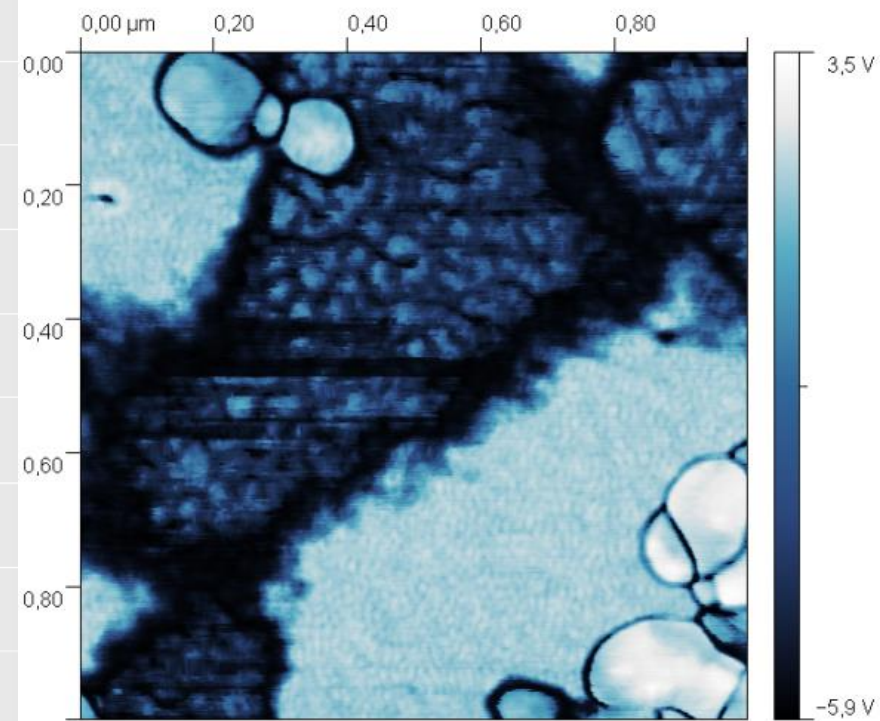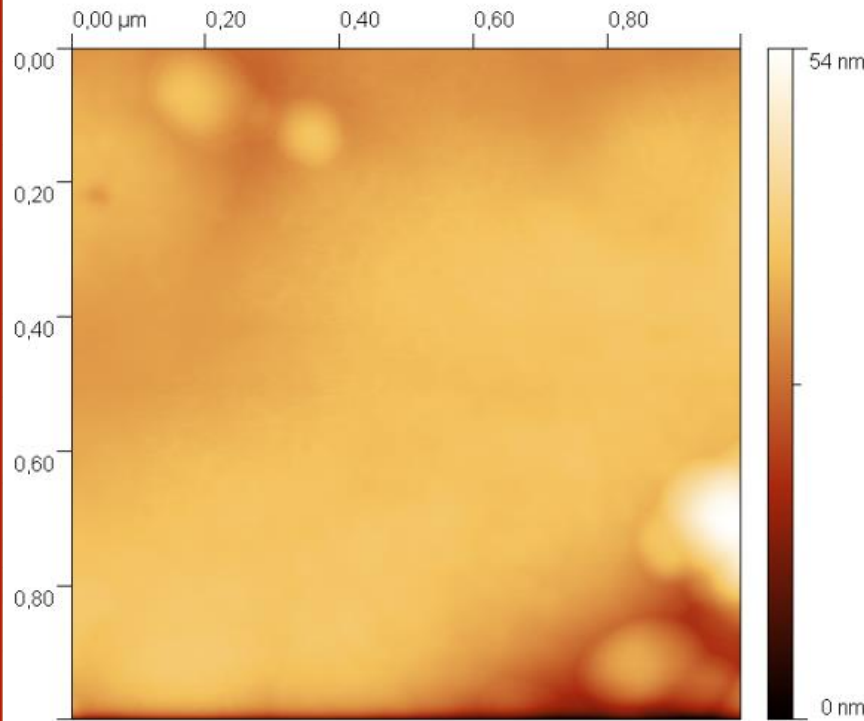
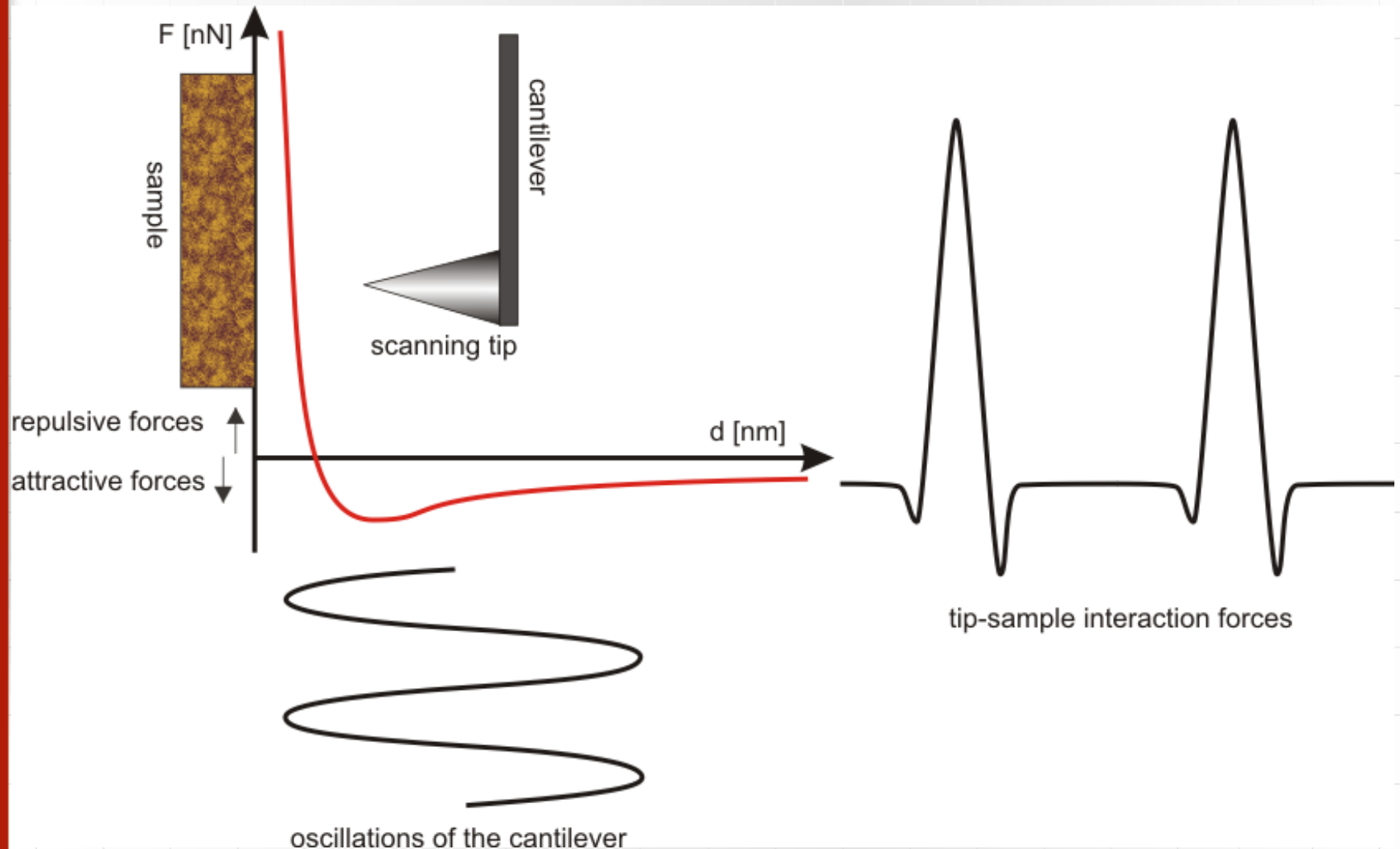# Phase imaging – polymerization map

Phase imaging vs force spectroscopy

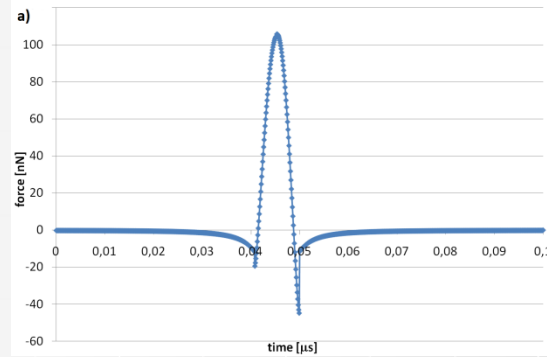# Phase imaging – there are issues…

Phase imaging – there are issues…

Every tap you make…

F [nN]

sample

cantilever

scanning tip

repulsive forces

attractive forces

d [nm]

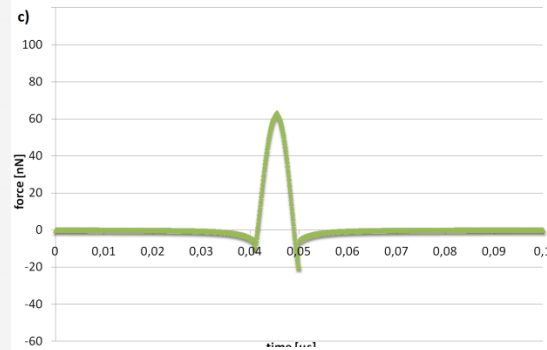tip-sample interaction forces

oscillations of the cantilever

Using DMT model to analyze signal…

time domain

FFT

10 GPa

5 GPa

1 GPa
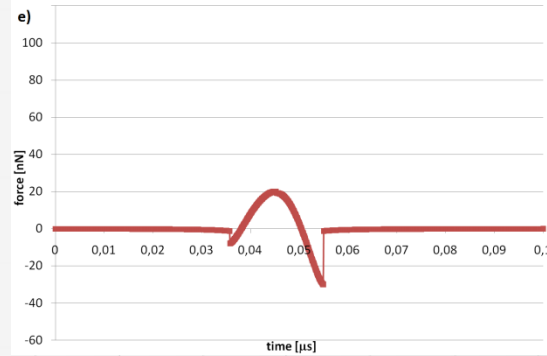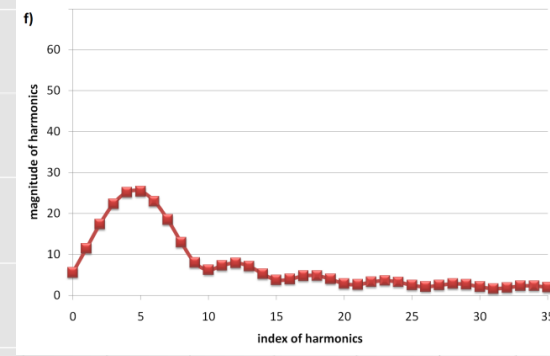
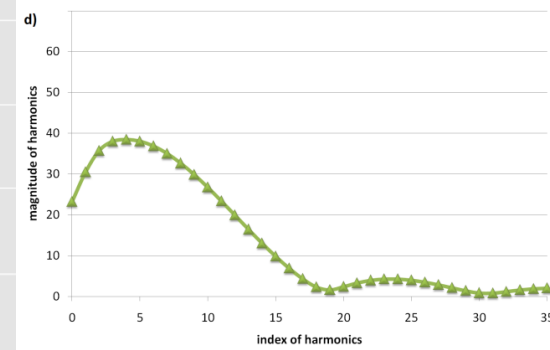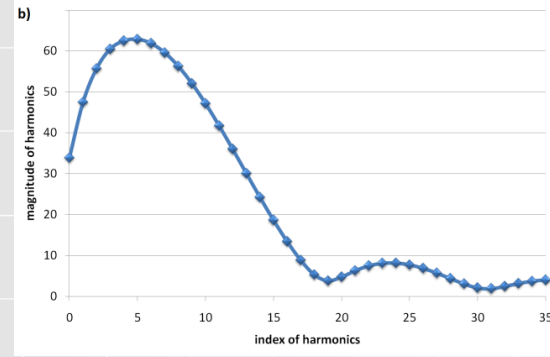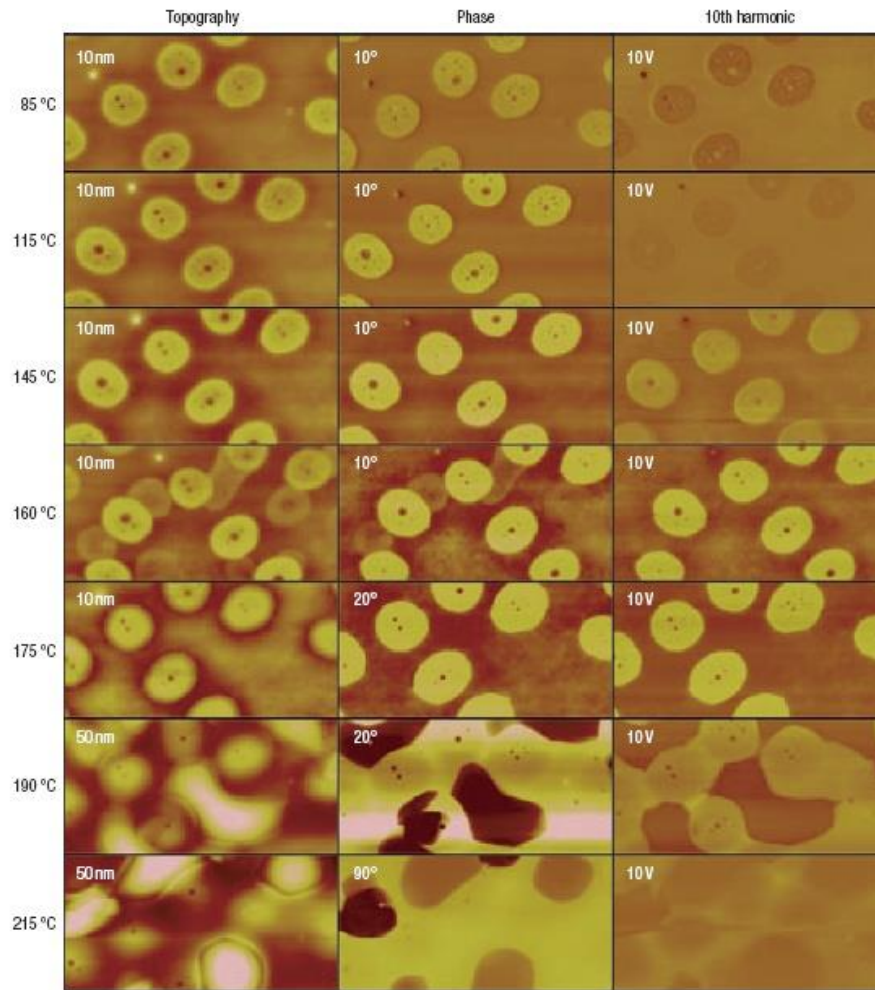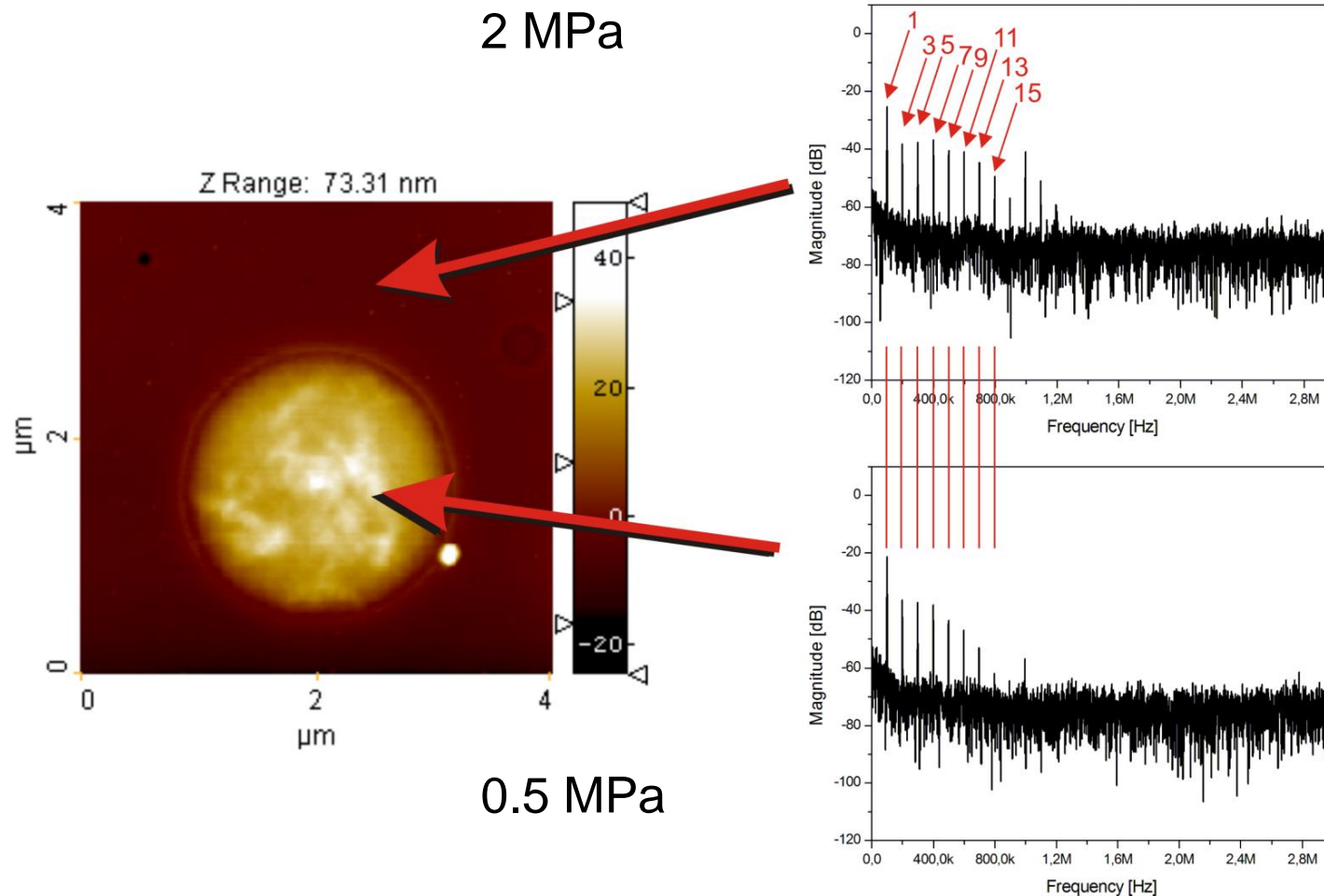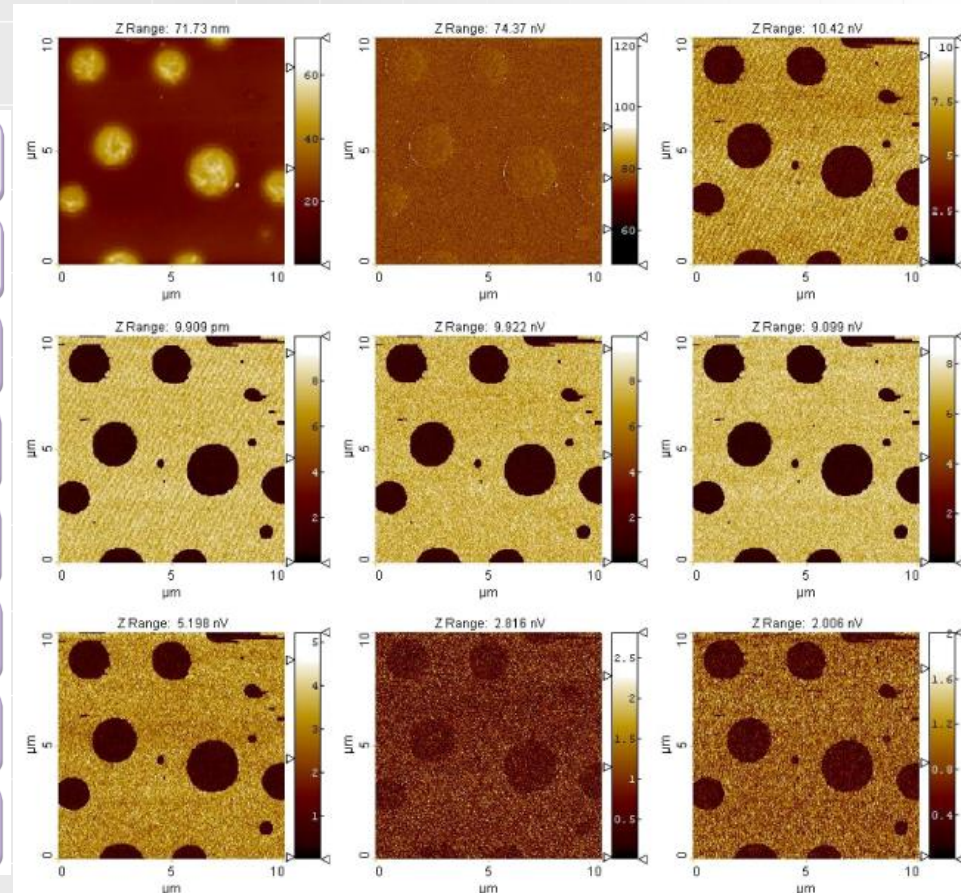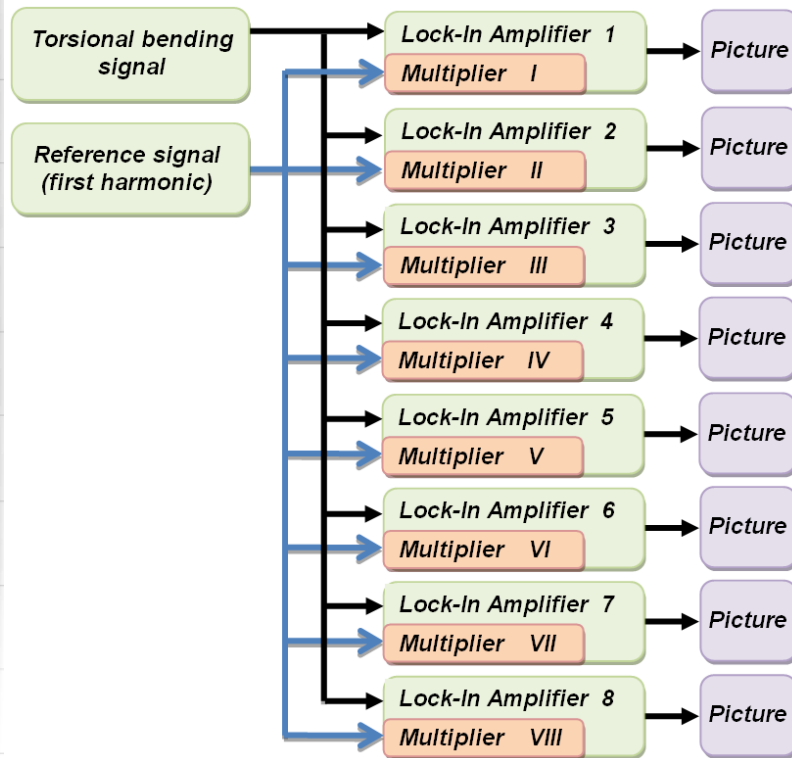# Utilizing high harmonic of the signal

Sahin O., Magonov S., Su C., Quate C.F., Solgaard O.
An atomic force microscope tip designed to measure time-varying nanomechanical forces
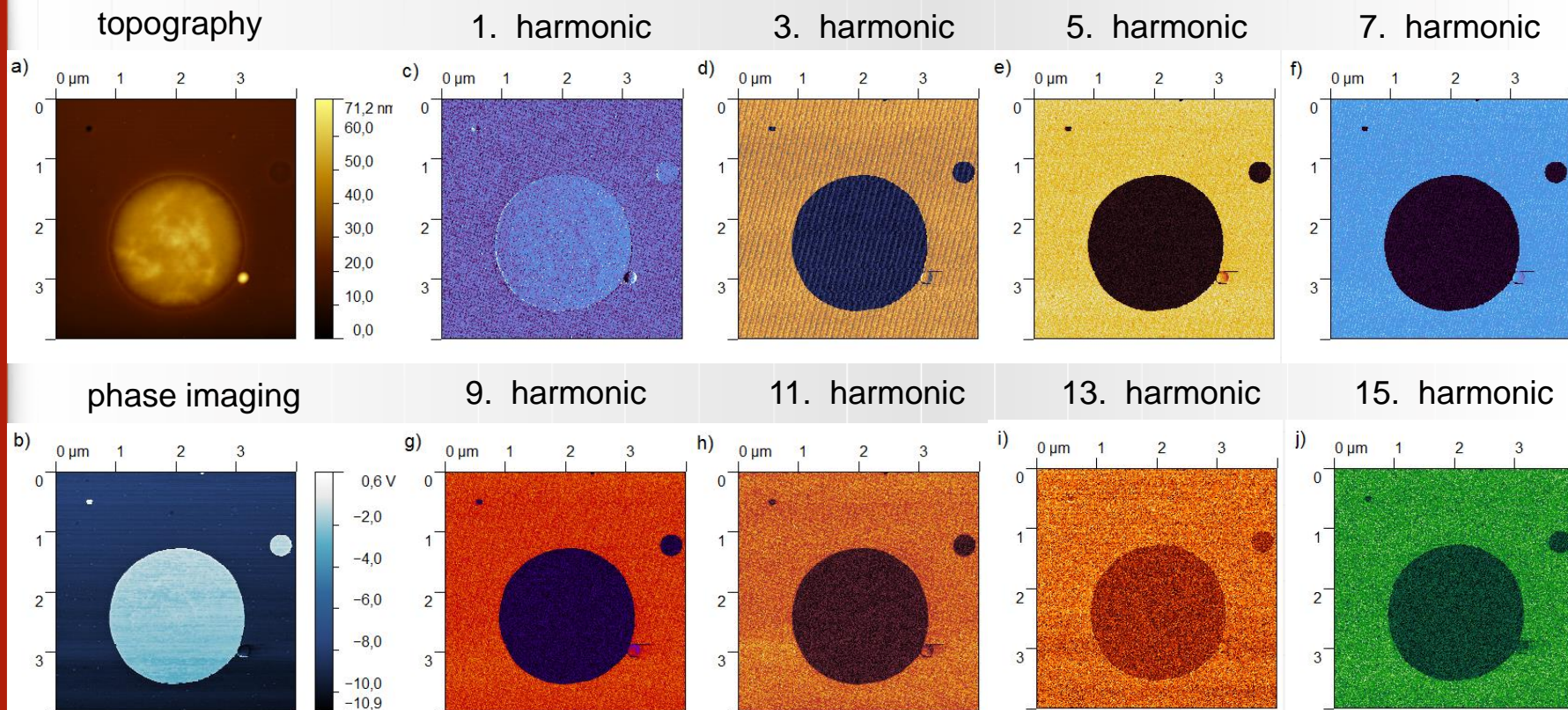Nature Nanotechnology, 2 (8), pp. 507-514, 2007

Testing the approach on PS-LDPE

…and multiplying the solution

Interesting differences can be revealed

topography · 1. harmonic · 3. harmonic · 5. harmonic · 7. harmonic

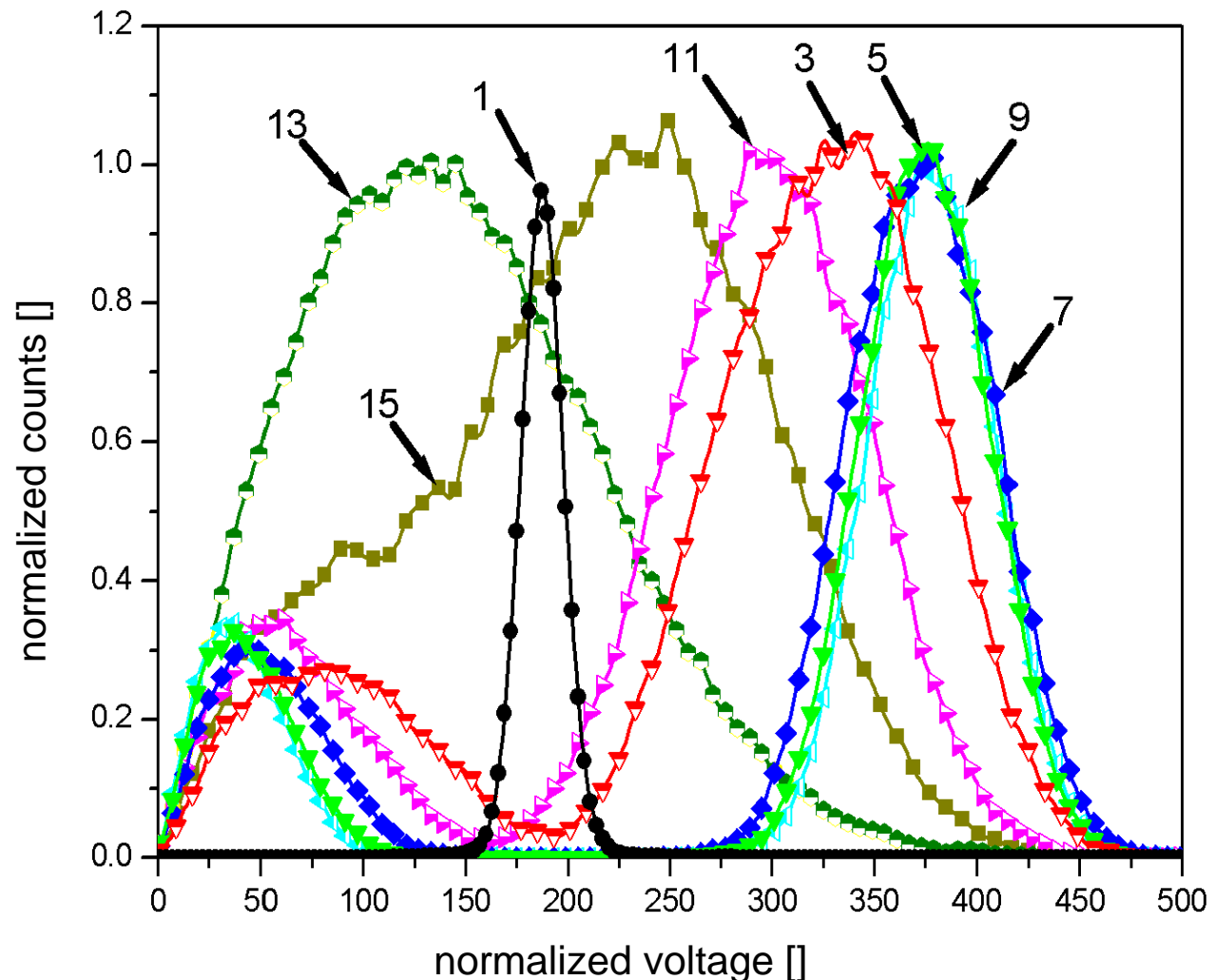phase imaging · 9. harmonic · 11. harmonic · 13. harmonic · 15. harmonic

Sikora A., Bednarz Ł.
The implementation and the performance analysis of the multi-channel software-based lock-in amplifier for the stiffness mapping with atomic force microscope (AFM)
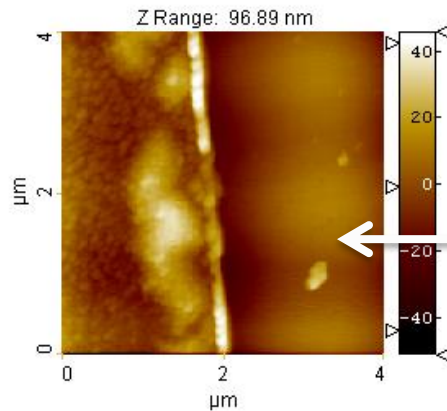Bulletin of the Polish Academy of Sciences: Technical Sciences, 60 (1), pp. 83-88, 2012

18

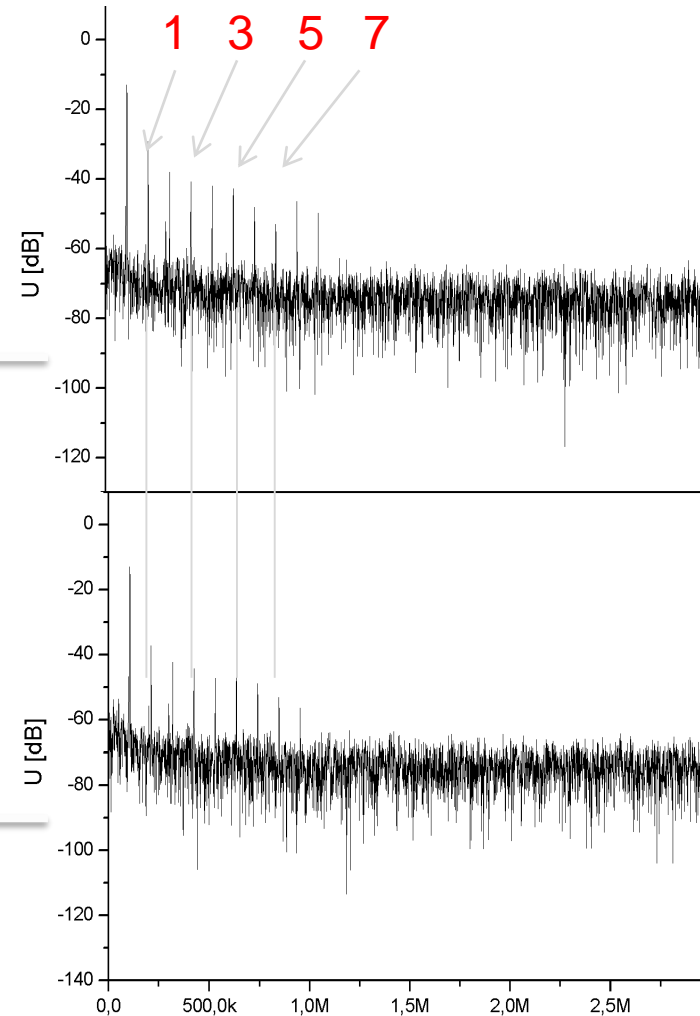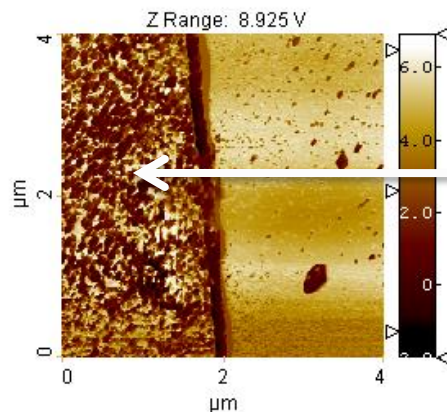# Different sensitivities can be seen for acquired harmonic maps
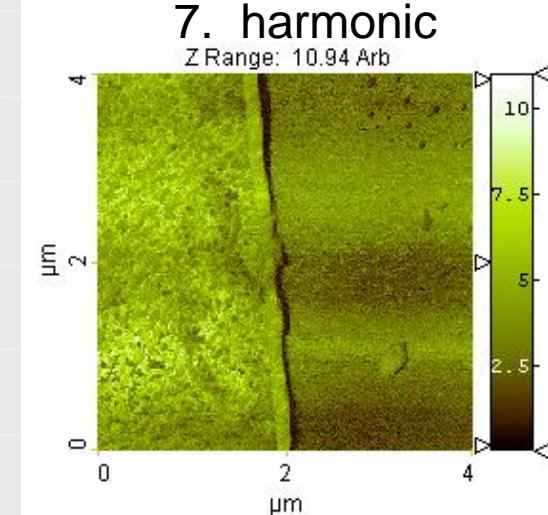
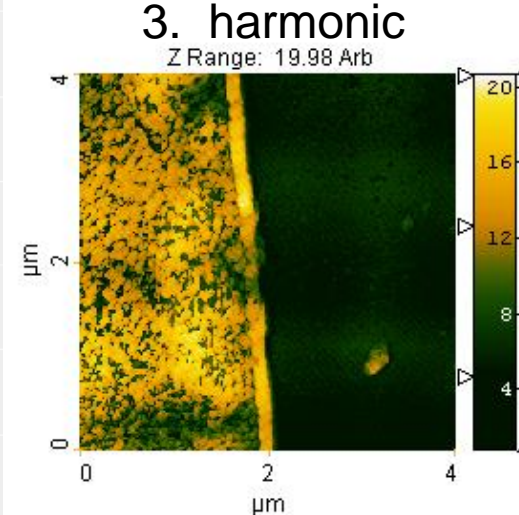# Example of stiffer surfaces…

topography

phase imaging

…looking at harmonic maps
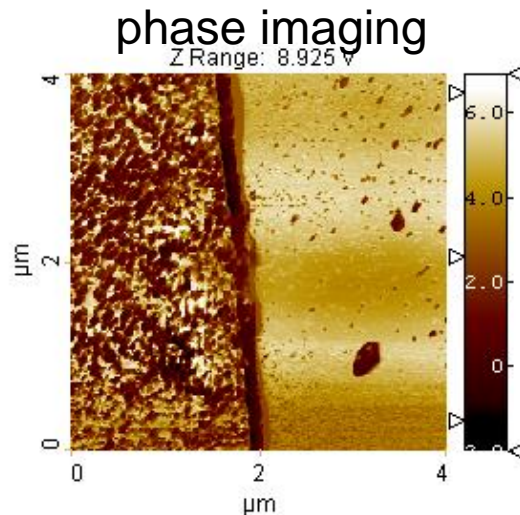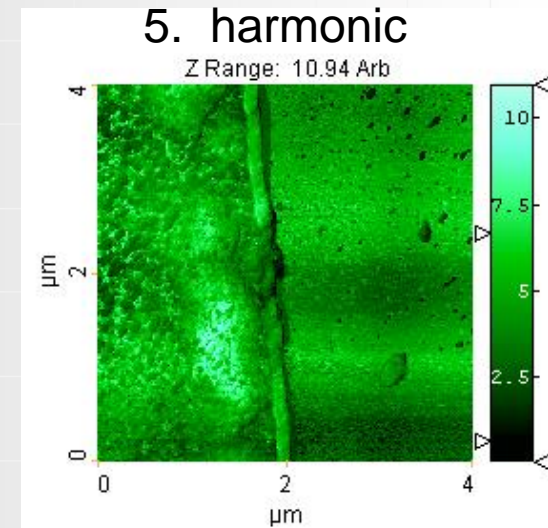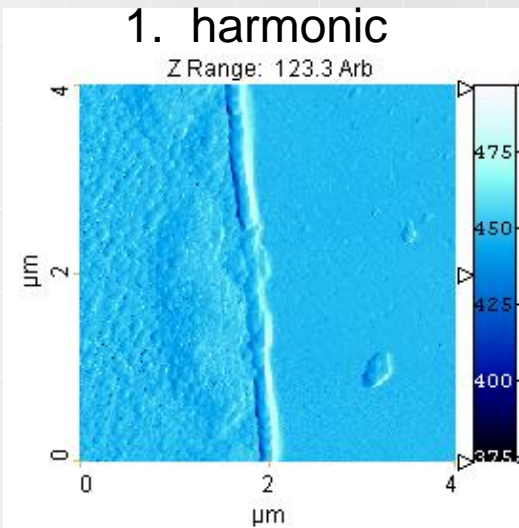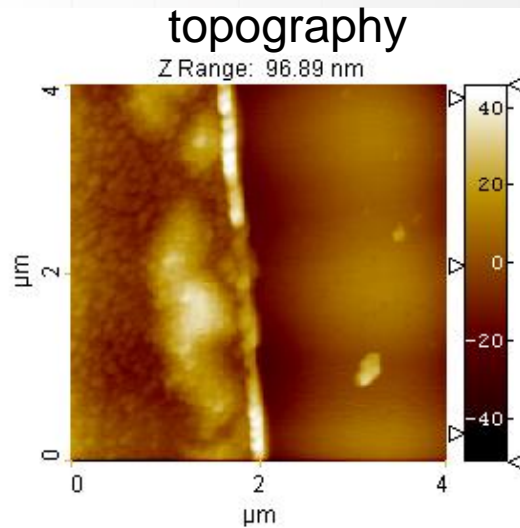
topography

1. harmonic

5. harmonic

phase imaging

3. harmonic

7. harmonic

What about acquiring force spectroscopy curve?
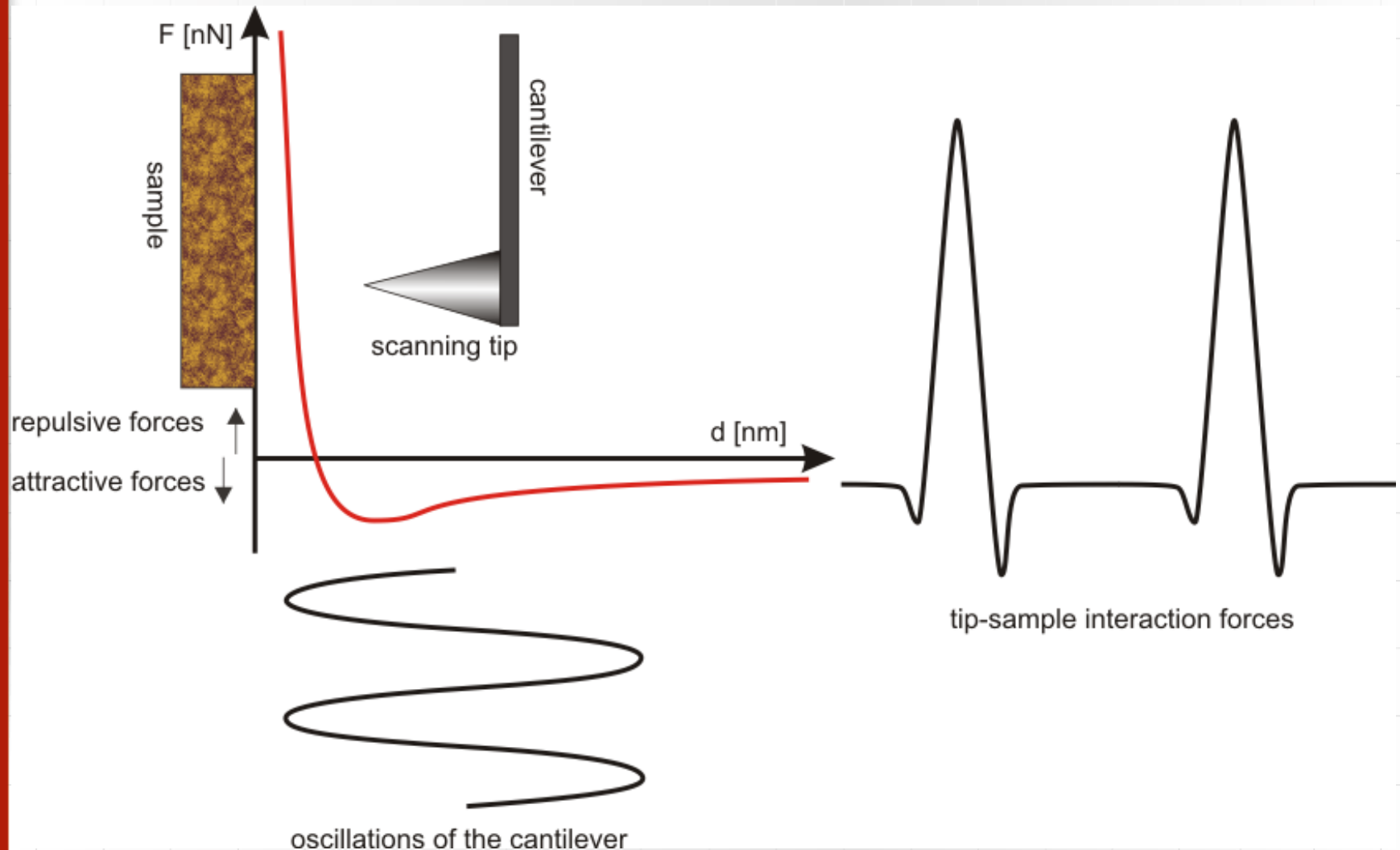
There are some obstacles on the way

Harmonic oscillator

$F_D \cos(\omega t)$

k

γ

masa

Fts

Spectrum

|H| [dB]

w/w0 [rad/s]
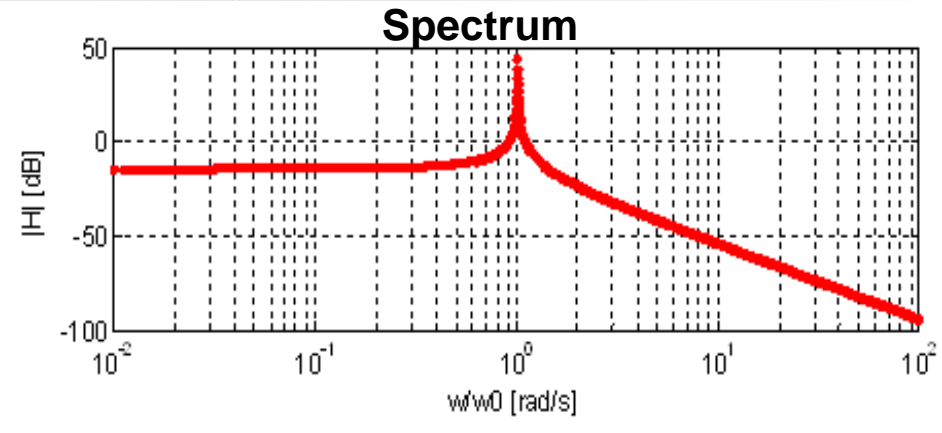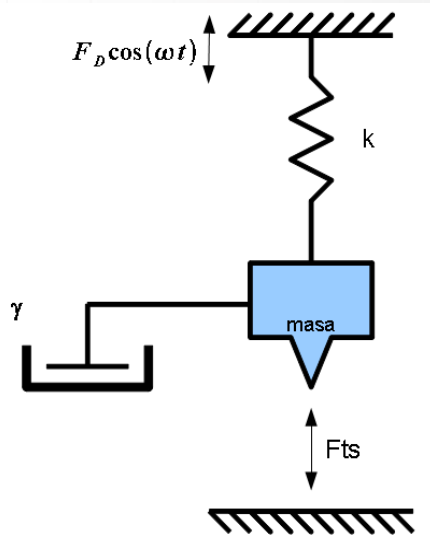
Setup transmittance

ω_0 – resonance pulsation
k – spring constant
Q – resonance quality

$$H(j\omega) = \frac{\omega_0^2 / k}{\omega_0^2 - \omega^2 + j\omega\omega_0 / Q}$$

How to remove low frequency flexural resonance
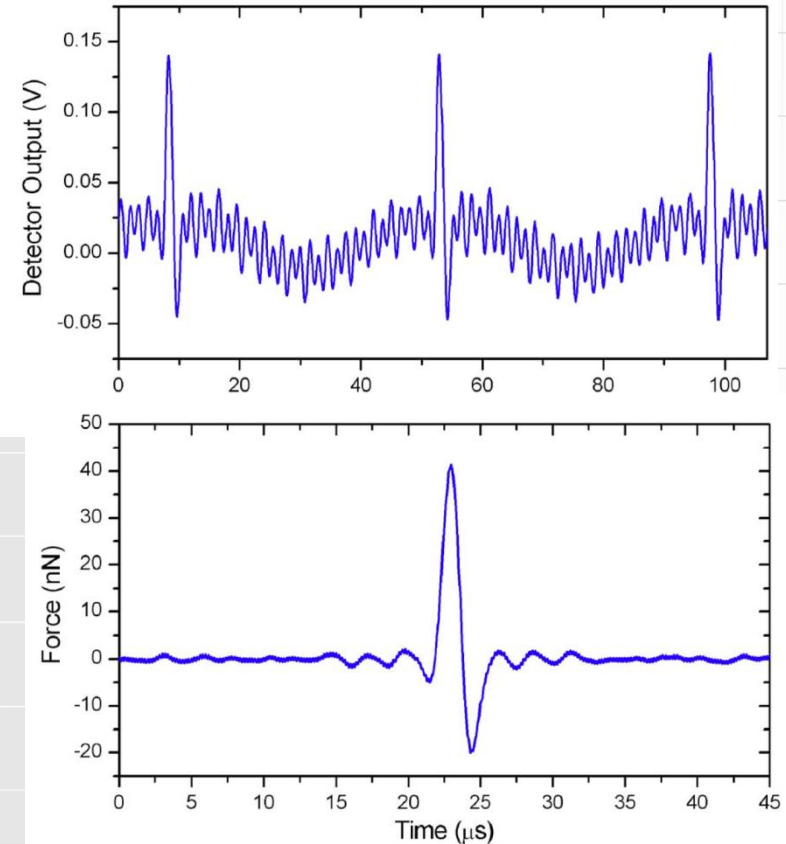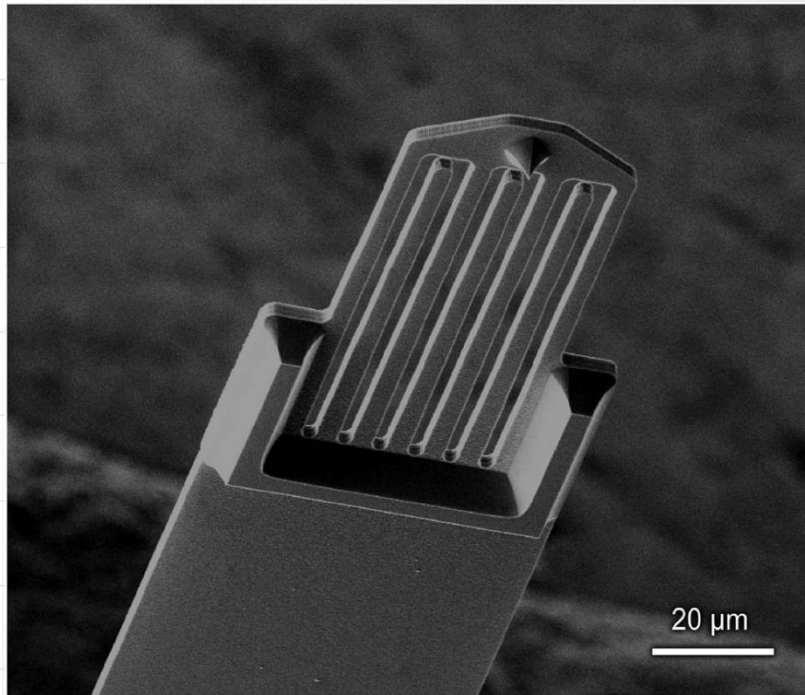
Flexural and torsional mechanical response of the cantilever
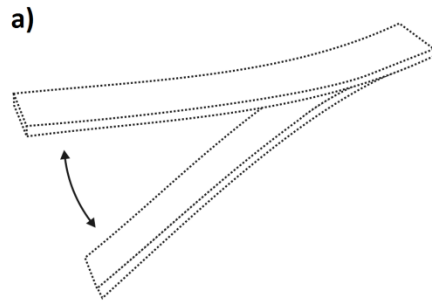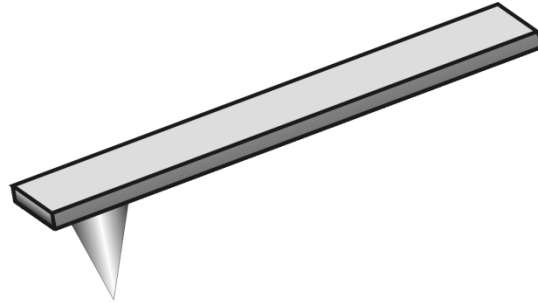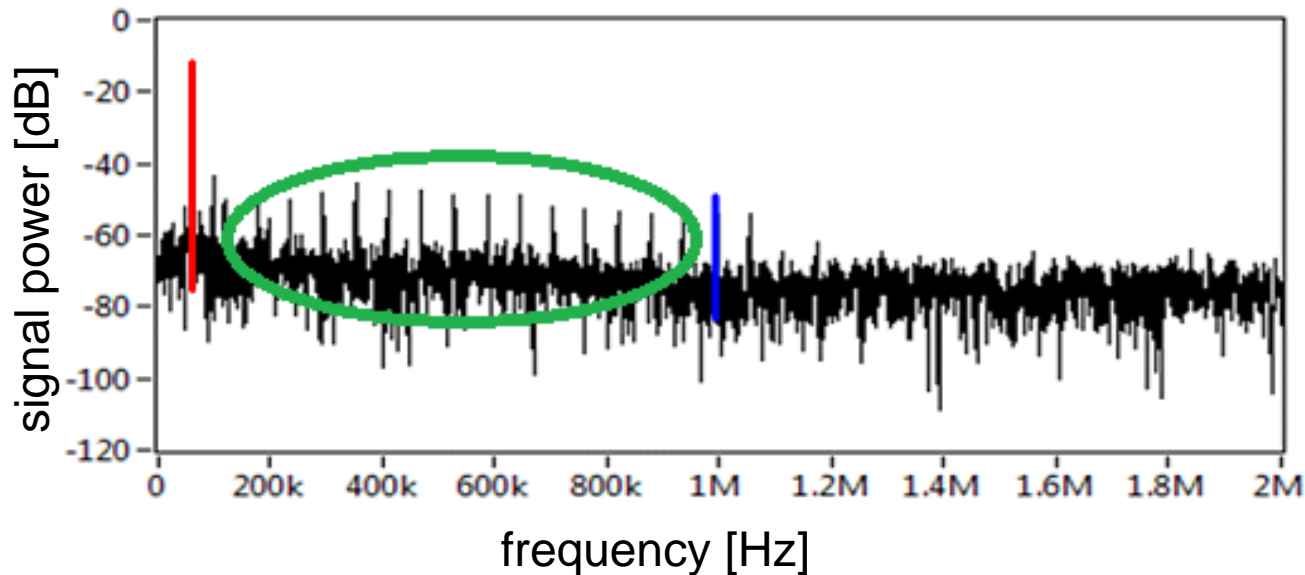
flexural oscillations
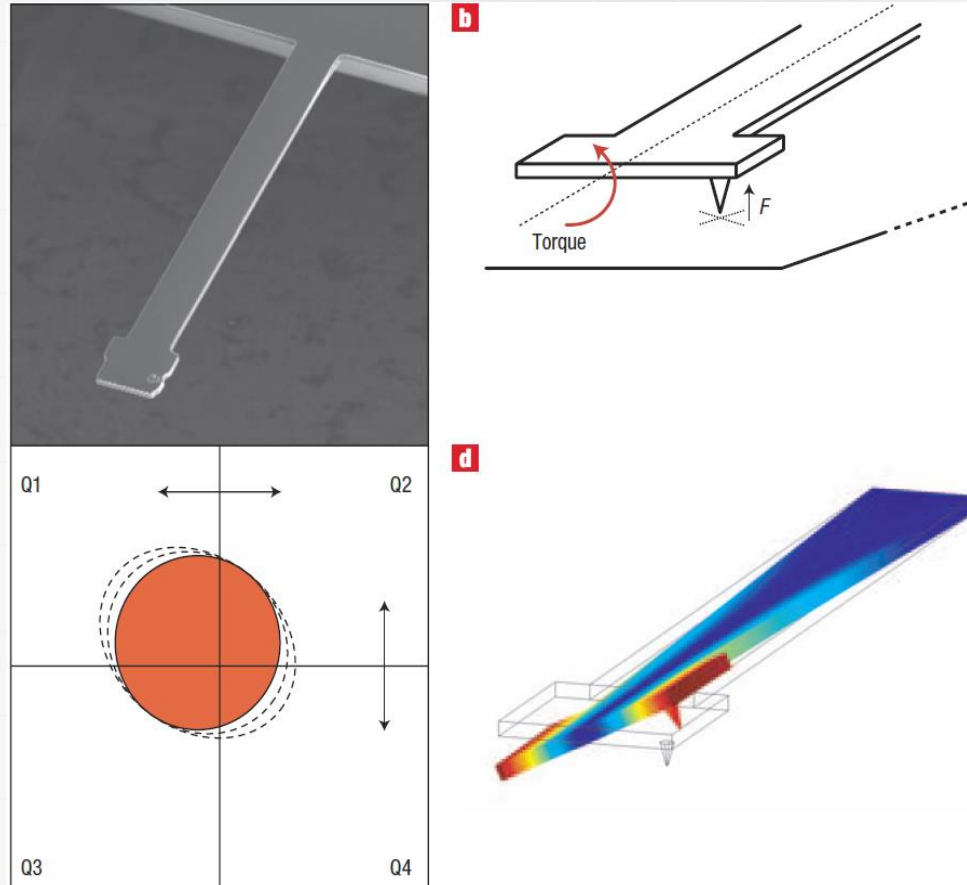
a)
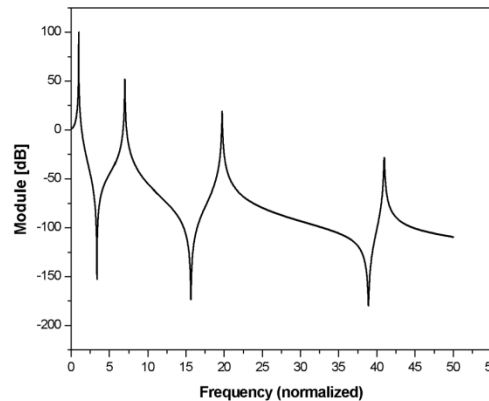
Some components of the signal are unwanted

Spectra of torsional oscillations of the cantilever

Flexural resonance frequency: 59.605 kHz
Torsional resonance frequency: 1013.285 kHz

So maybe T-shaped probe…

27

…so we could acquire interaction force

$$s = CG(f_{drive} + f_{tip})$$

$$s_X = C_X G_X (f_{drive} + f_{tip}) = H_X (f_{drive} + f_{tip})$$

$$s_Y = C_Y G_Y f_{tip} = H_Y f_{tip}$$

$$H_Y(\omega) = c_{optical} \frac{\omega_T^2 / K_T}{\omega_T^2 - \omega^2 + i\omega\omega_T / Q_T}$$

$$f_{tip}(j) = \frac{1}{N} \sum_{k=1}^{N} H_Y^{-1}(\omega_k) S_Y(\omega_k) e^{i\omega_k (j-1)}$$

Sahin O., Atalar A., Quate C.F., Solgaard O.
Harmonic cantilevers and imaging methods for atomic force microscopy
US Patent No. US6935167, 2005
Sahin O., Quate C.F., Solgaard O., Atalar A.
Resonant harmonic response in tapping-mode atomic force microscopy
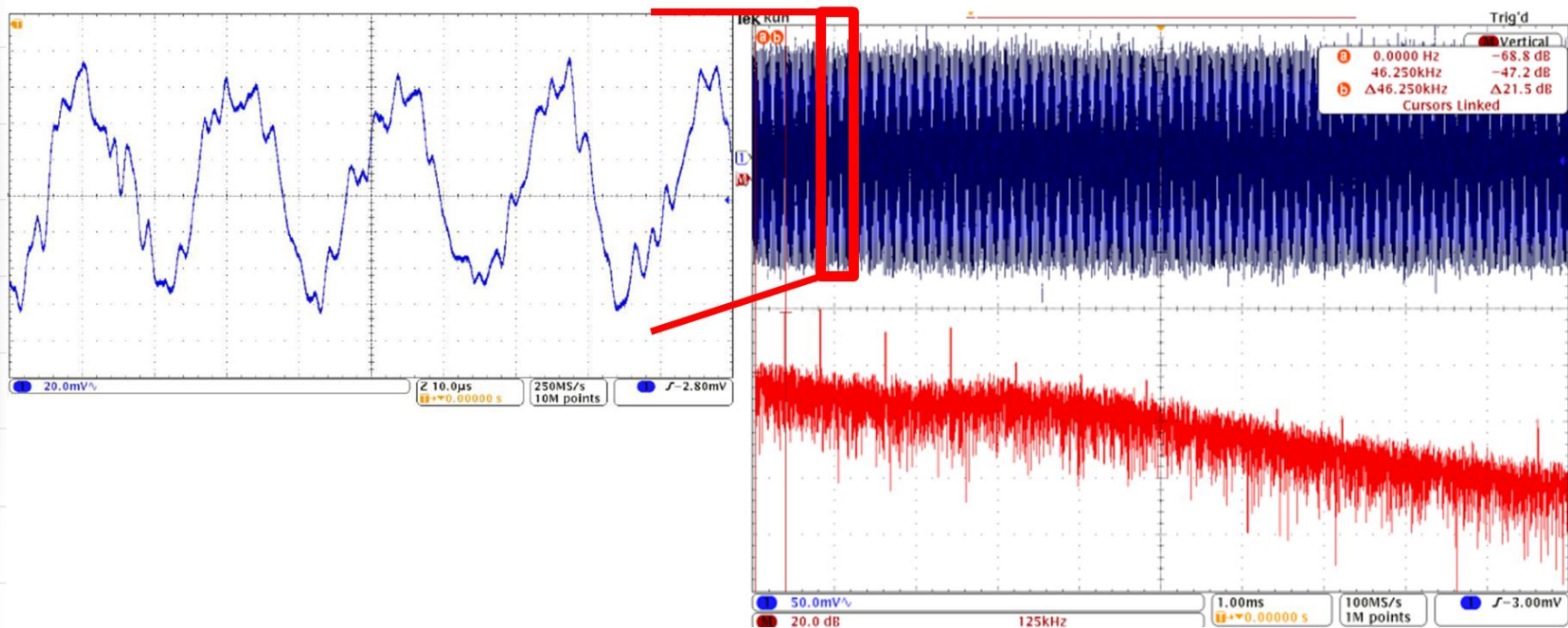Physical Review B, 69 (165416), pp. 1-9, 200
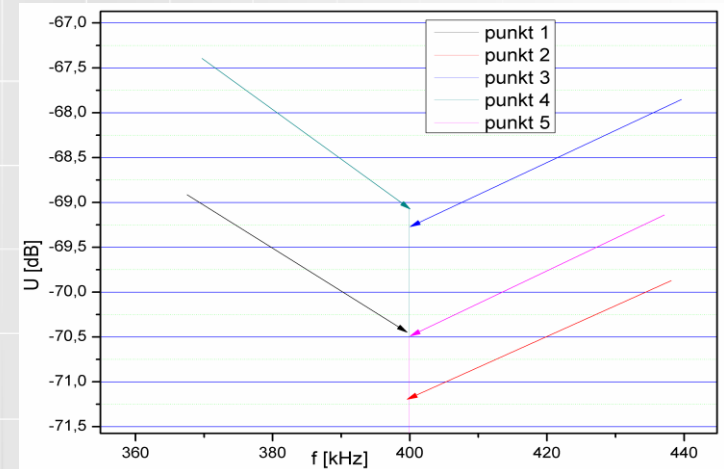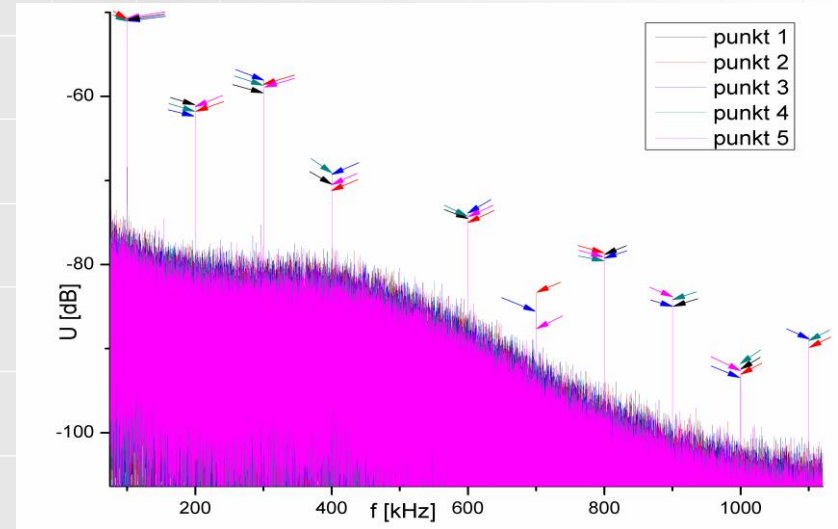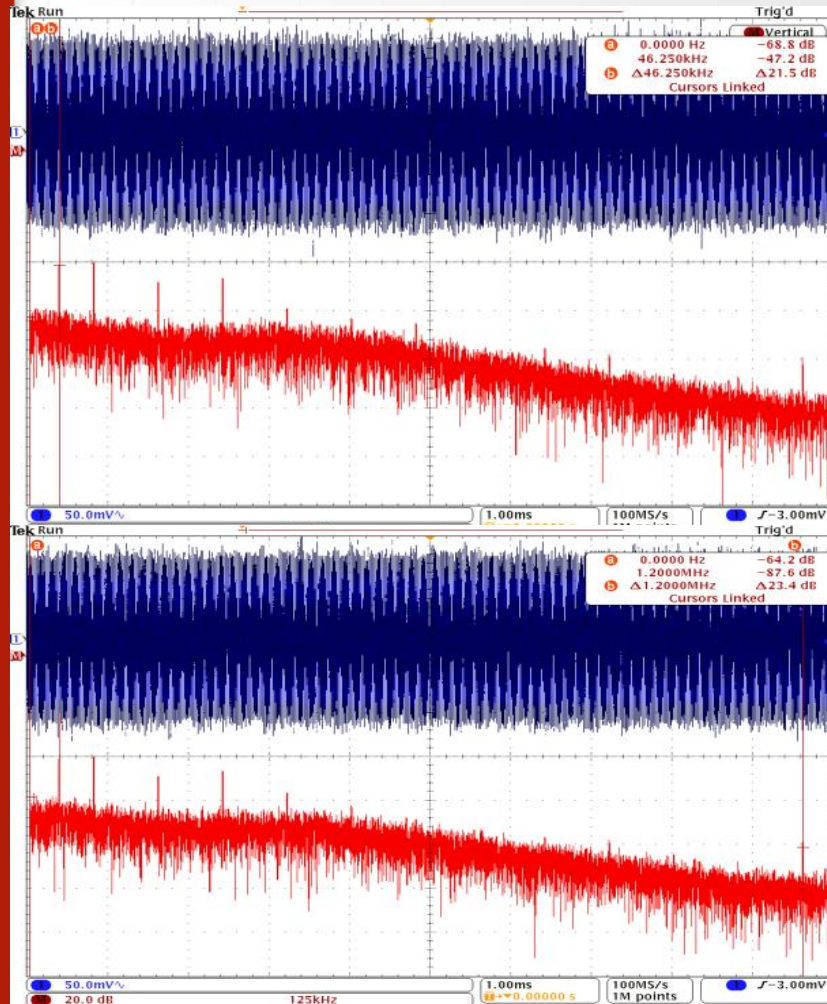
So finally it works like this
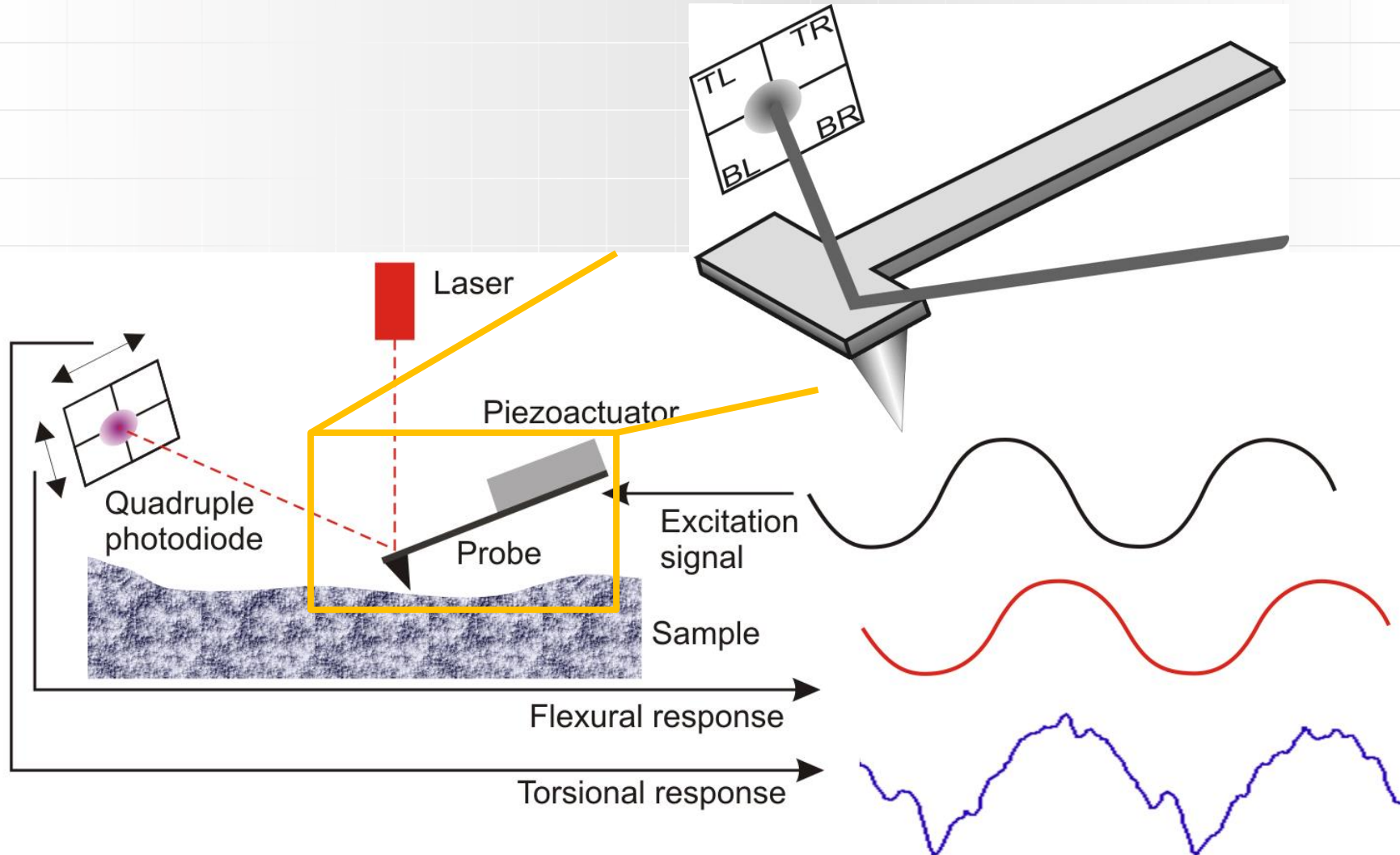
Implementation using standard AFM
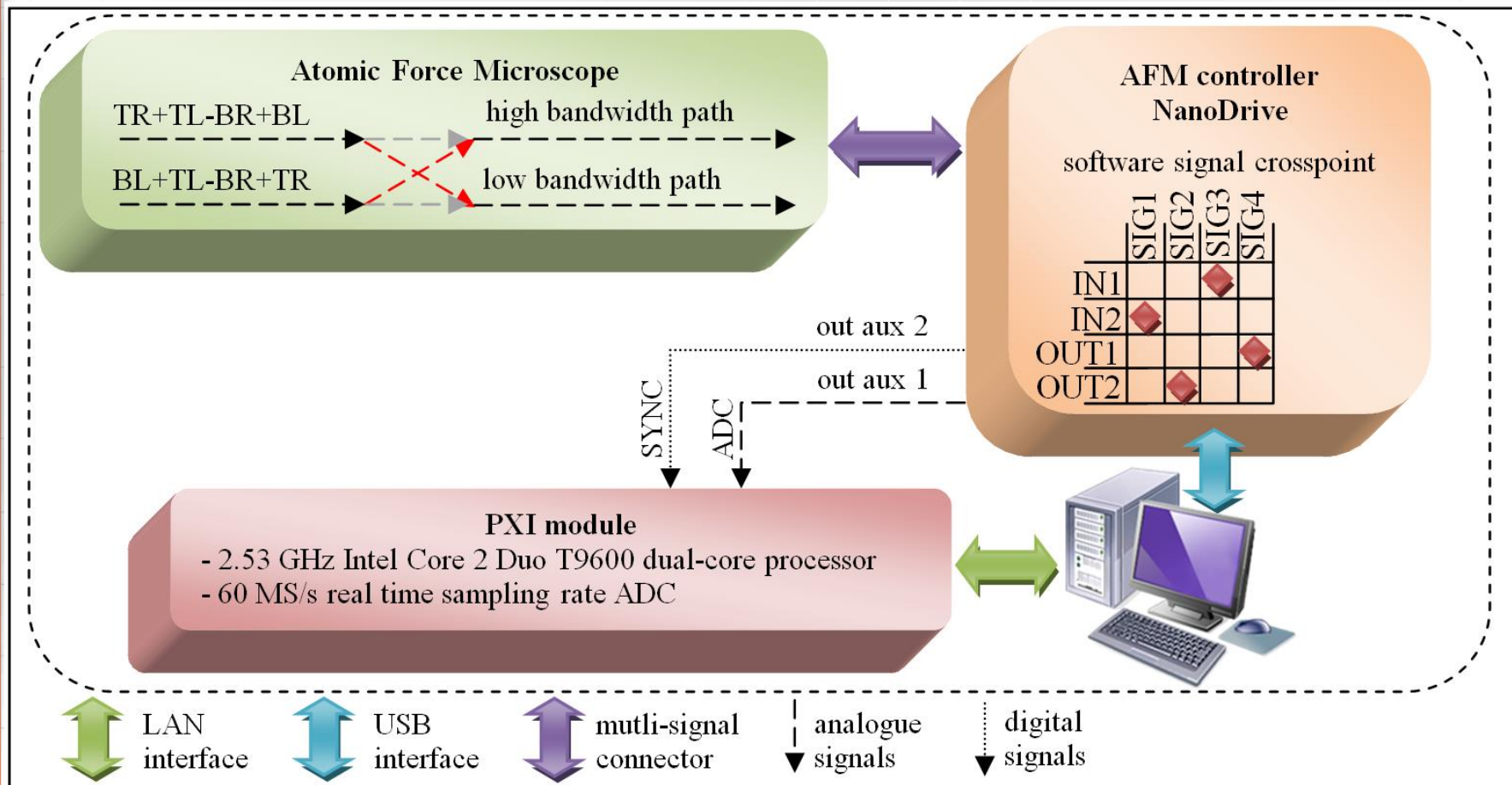
Raw torsional signal

...acquired in various spots

Reconstruction of the force spectroscopy curve
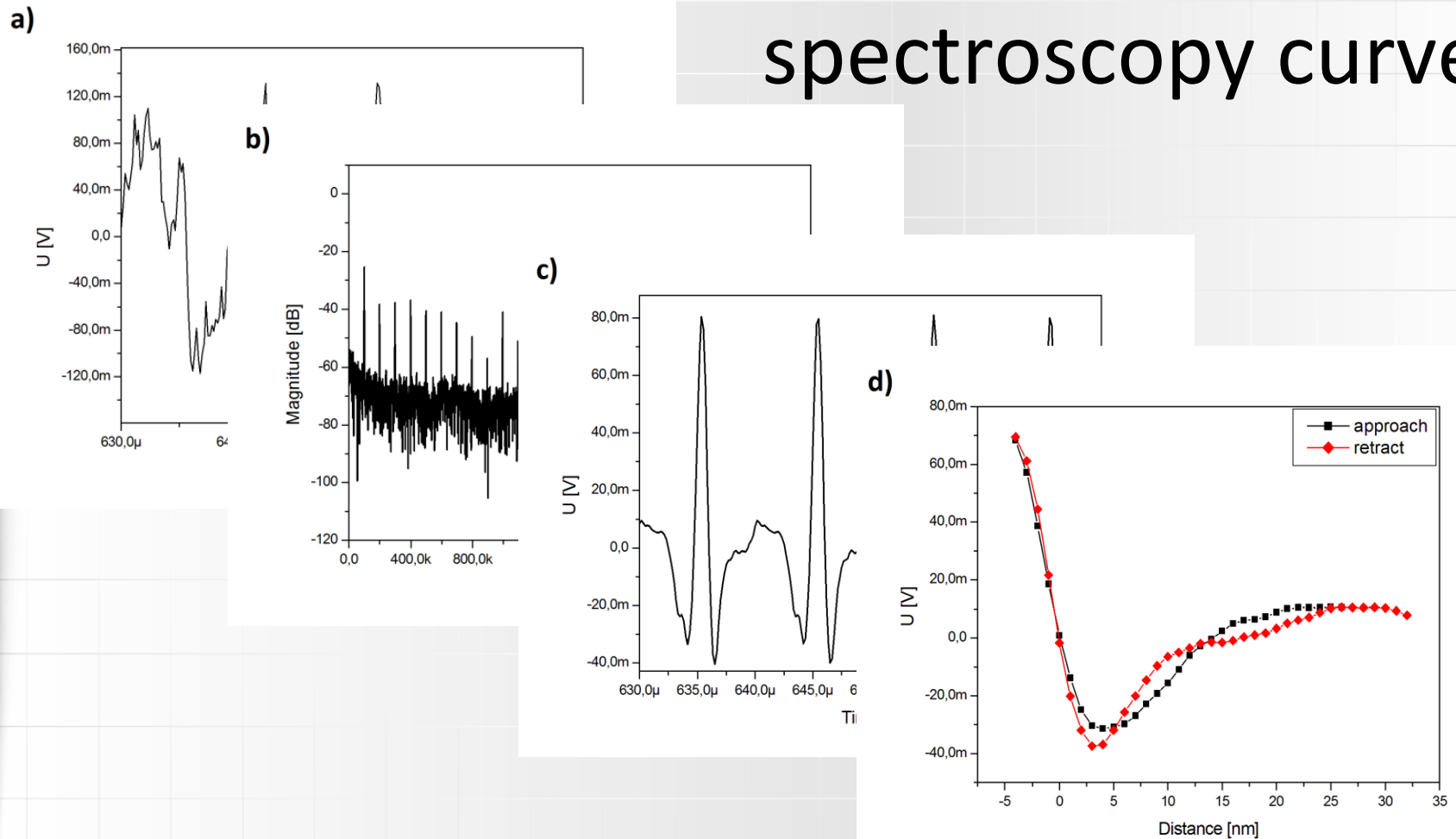
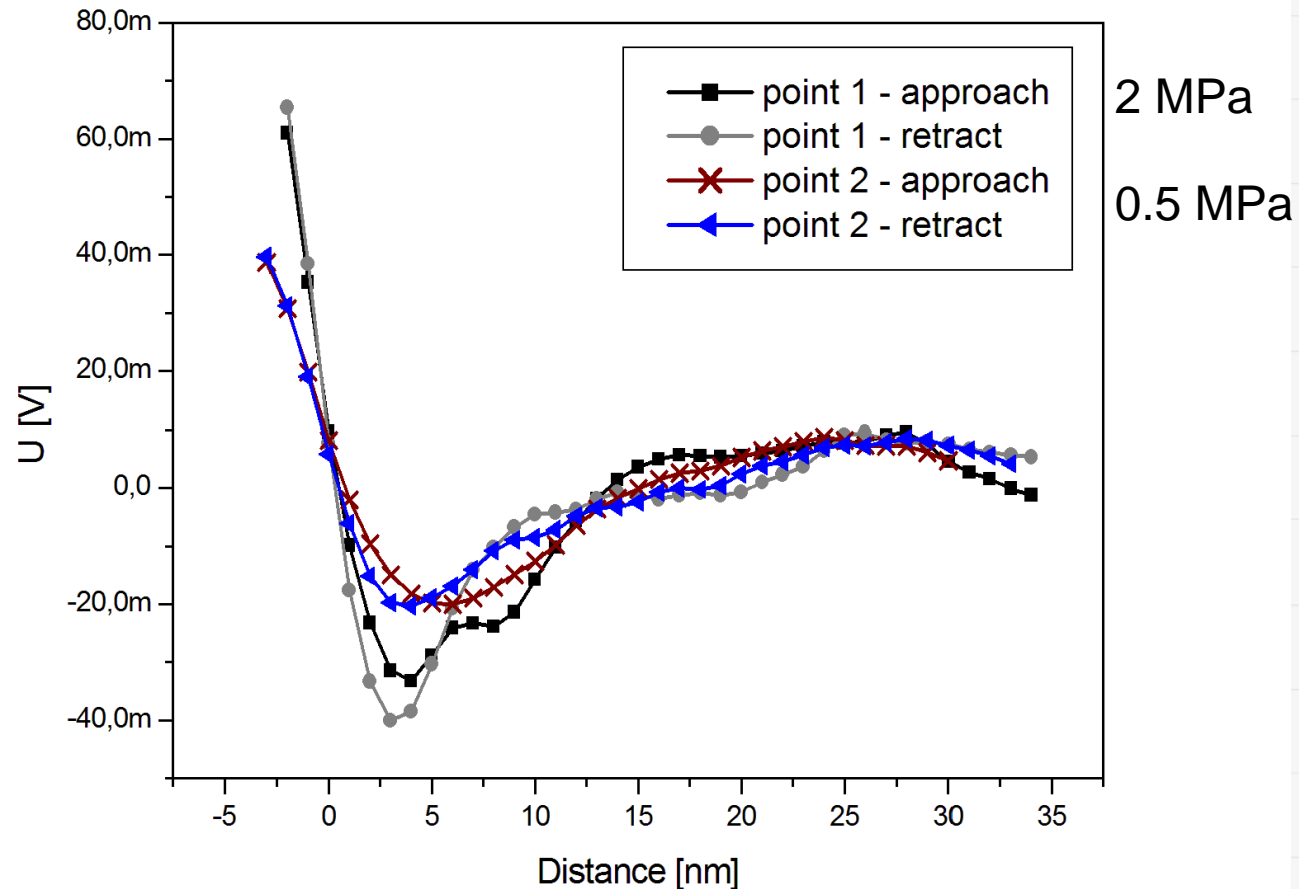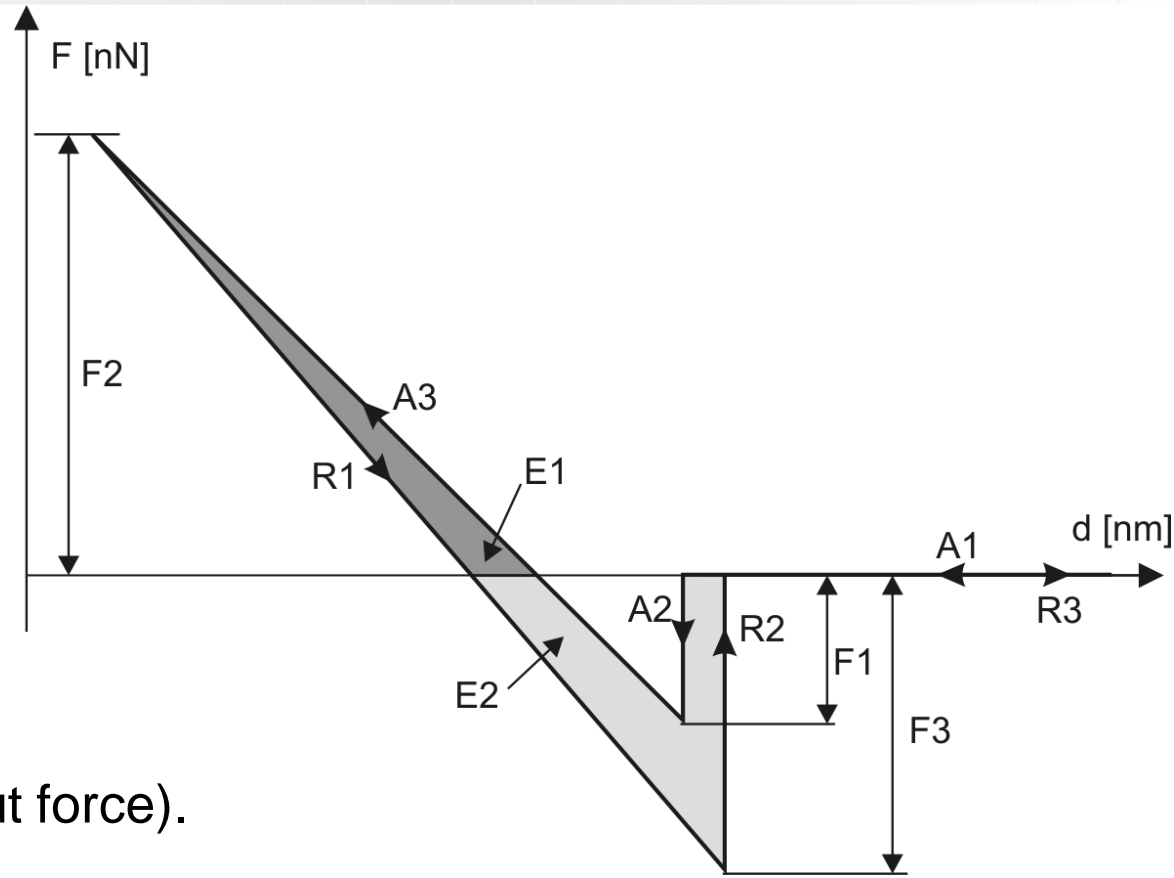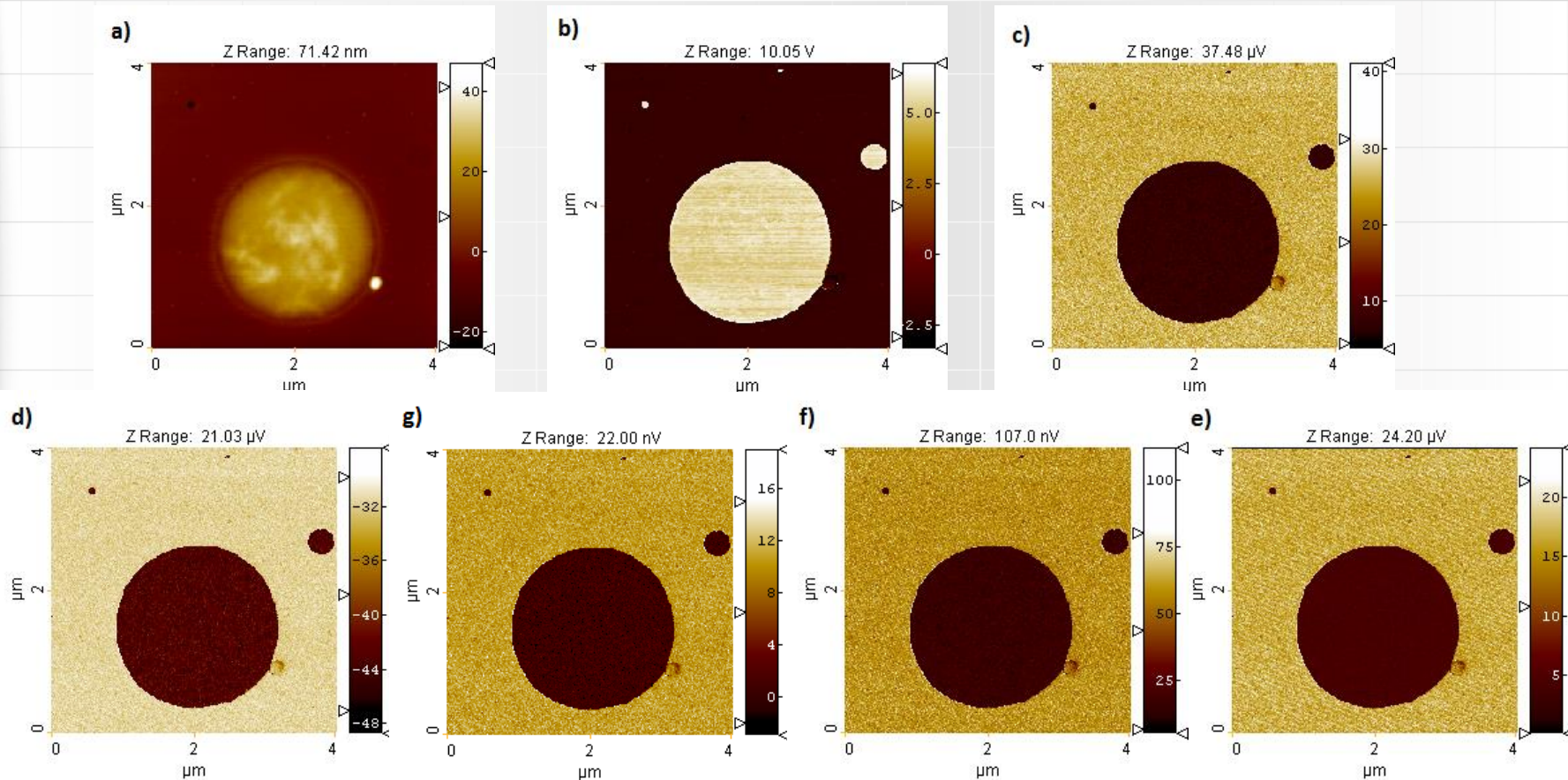# Reconstruction of the force spectroscopy curve

2 MPa

0.5 MPa

The tip-sample interaction related parameters


F1 – snap-in force,
F2 – peak force,
F3 – adhesion (snap-out force).
R1 (slope) – elasticity,
E1 – energy dissipation for deformation,
E2 – energy dissipation for tip-sample separation

Mapping PS-LDPE test sample

topography (a), phase imaging (b), adhesion (c), stiffness (d), peak force (e), energy dissipation for tip-sample separation (f), energy dissipation for deformation of the surface (g)

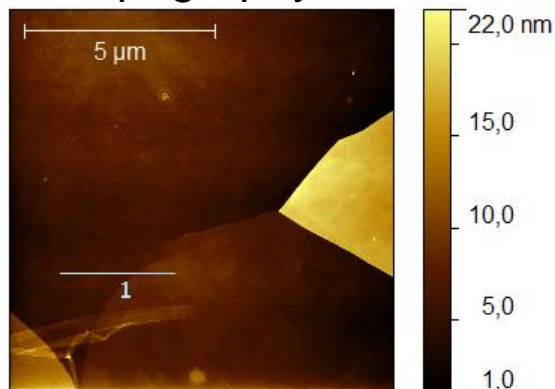# Distribution of specific parameters
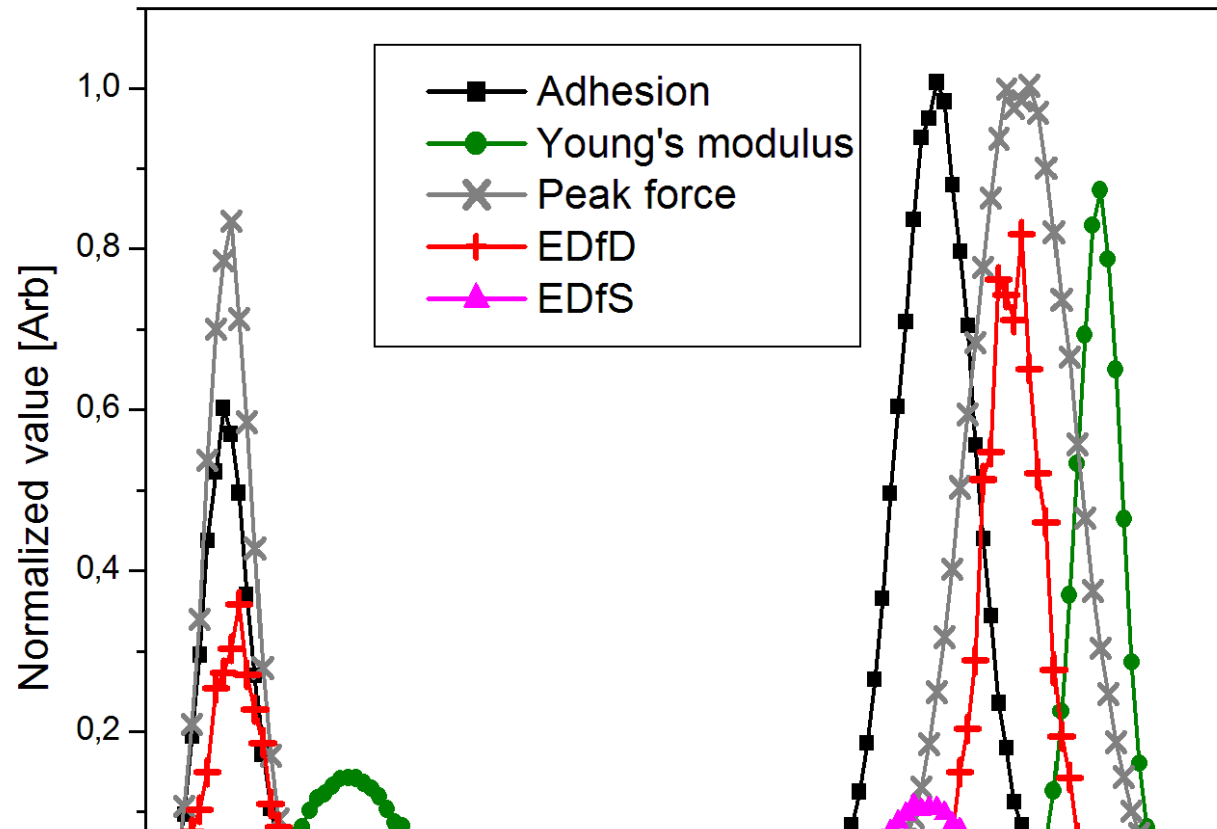
Sikora A., Bednarz L.
Mapping of mechanical properties of the surface by utilization of torsional oscillation of the cantilever in atomic force microscopy
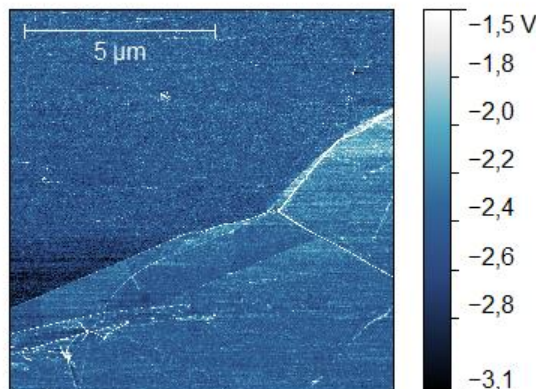Central European Journal of Physics, 9 (2), pp. 372-379, 2011
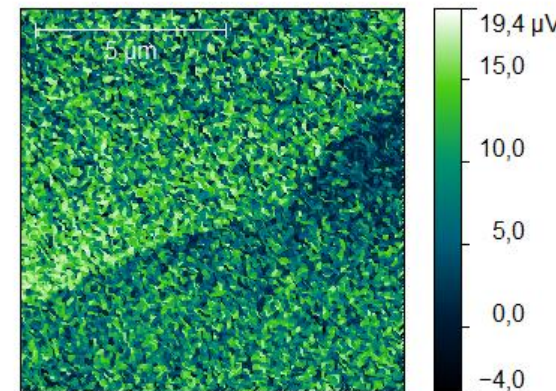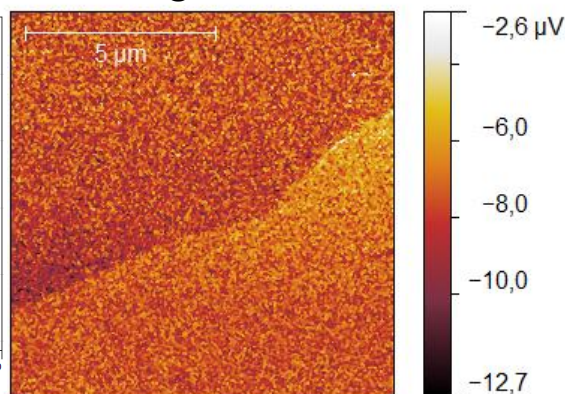
Graphene flakes on SiO$_2$
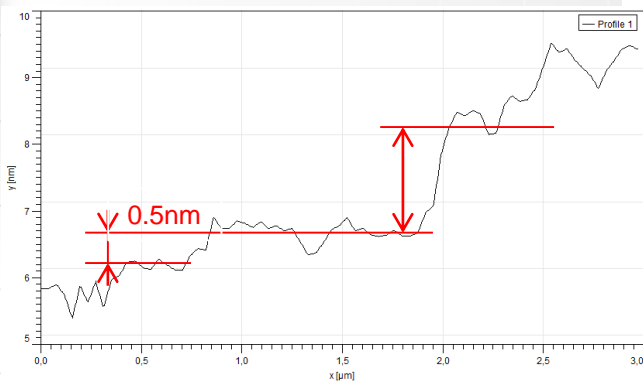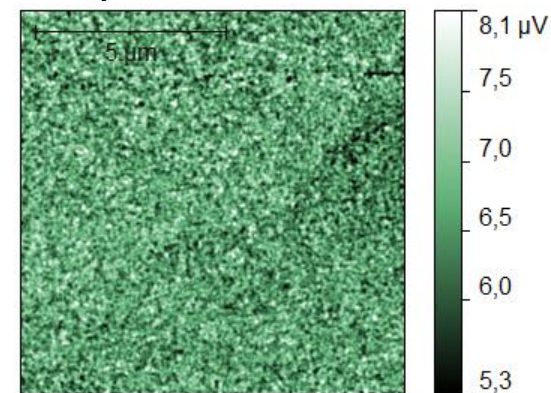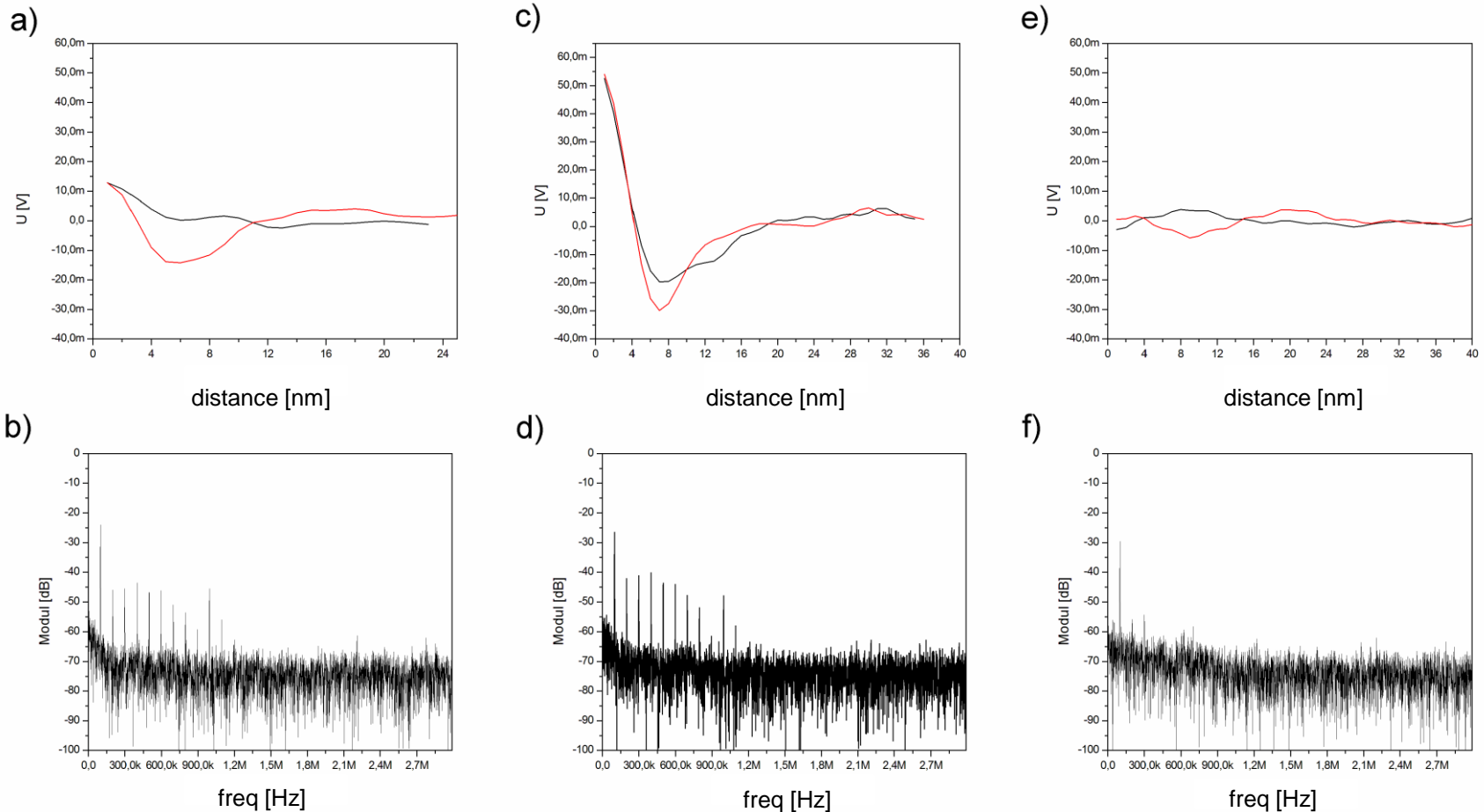
topography

phase imaging

adhesion

Young modulus

peak force

Sikora A., Woszczyna M., Friedemann M., Ahlers F.J., Kalbac M.
AFM diagnostics of graphene-based quantum Hall devices
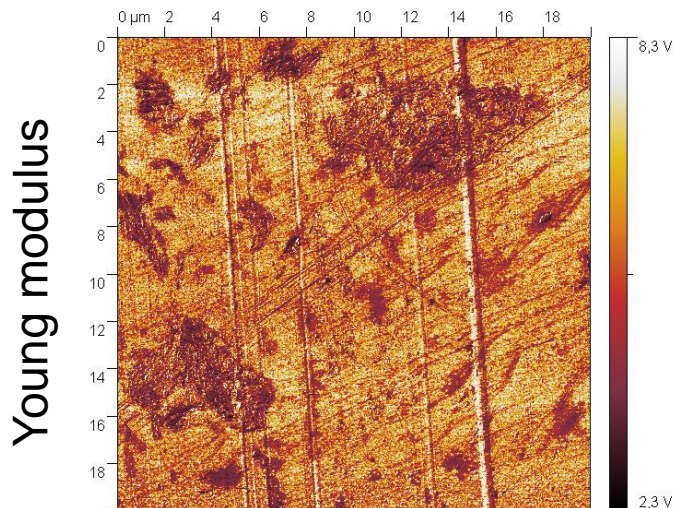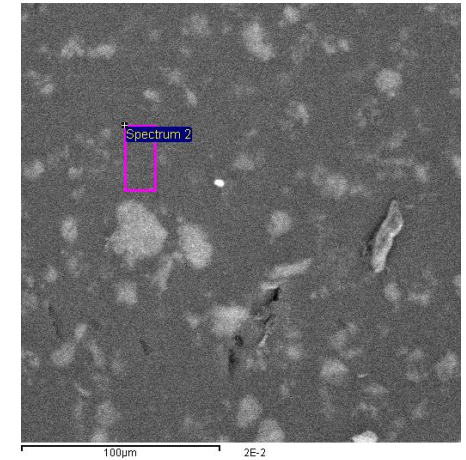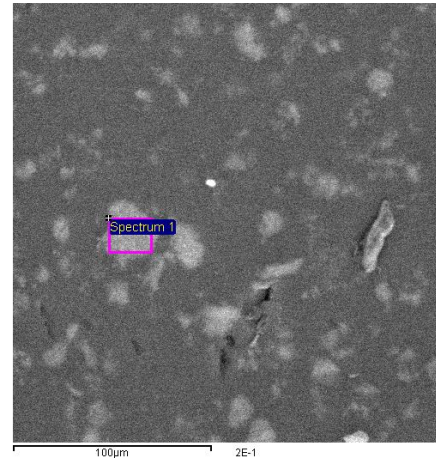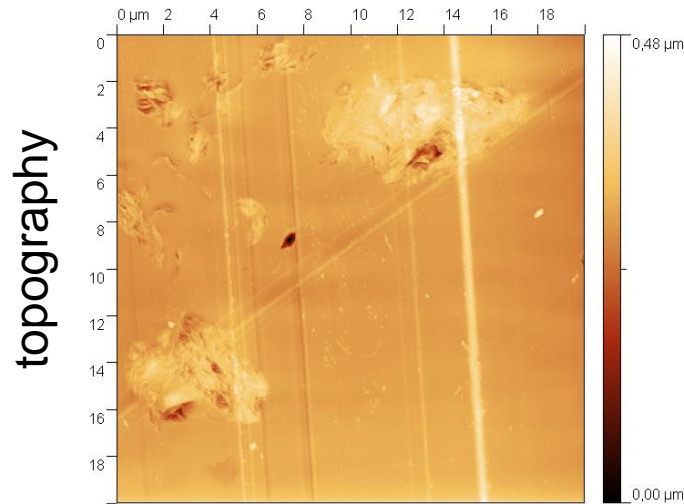Micron, 43, pp. 479-486, 2012

I can't get no interaction…

too close   OK   too far

Epoxy resin with silica nanofiller

topography

Young modulus

Element	Weight %	Atomic %		Weight%	Atomic%
C K	54.30	65.82		72.60	78.15
O K	26.49	24.11		26.68	21.56
Mg K	0.43	0.26		0	0
Al K	4.78	2.58		0	0
Si K	13.74	7.12		0.24	0.11
Cl K	0.26	0.11		0.48	0.18
Totals	100.00			100.00	

Calcium deposits from human vains
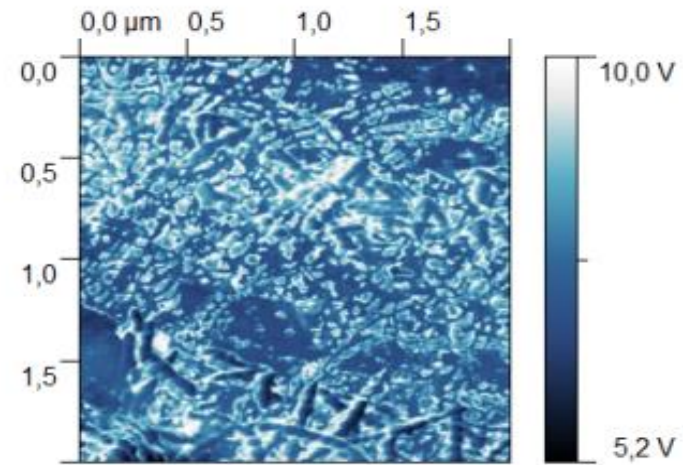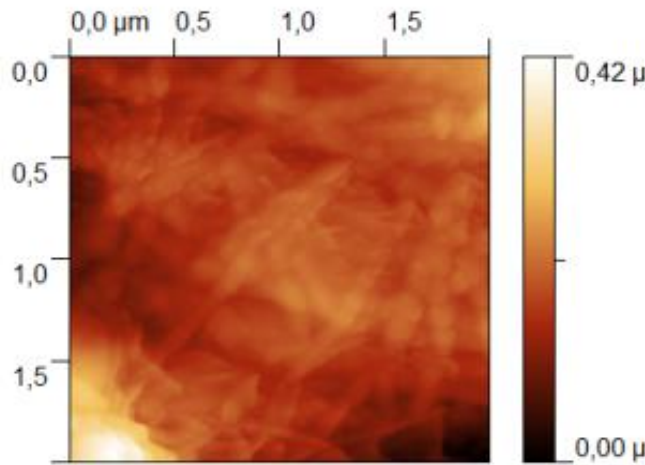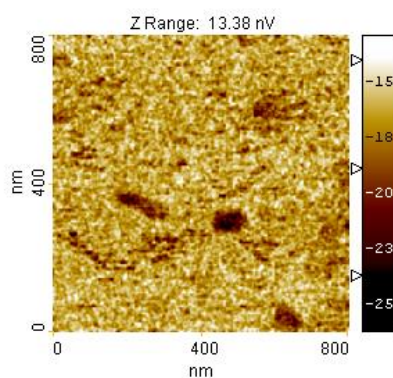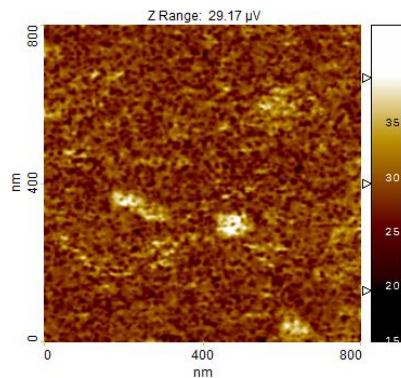
Manganese cathode materials for lithium ion batteries
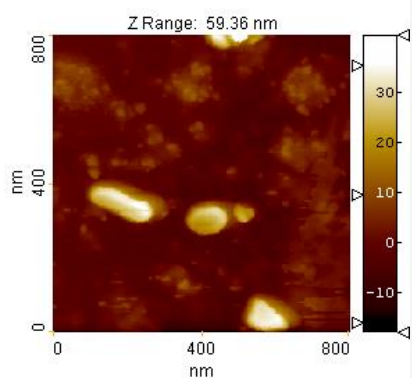
topography   stiffness   adhesion
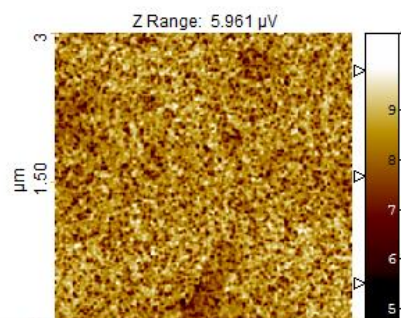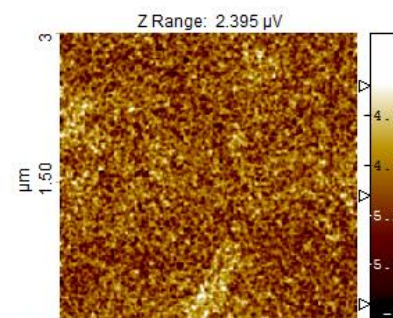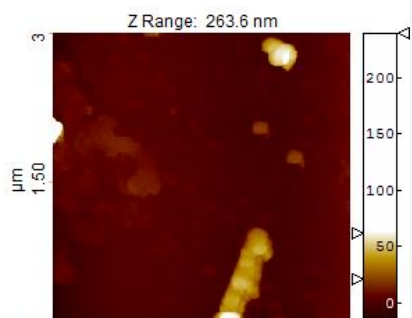
LiMn$_2$O$_4$ (L47A)

Indication of the different indentation of the surface
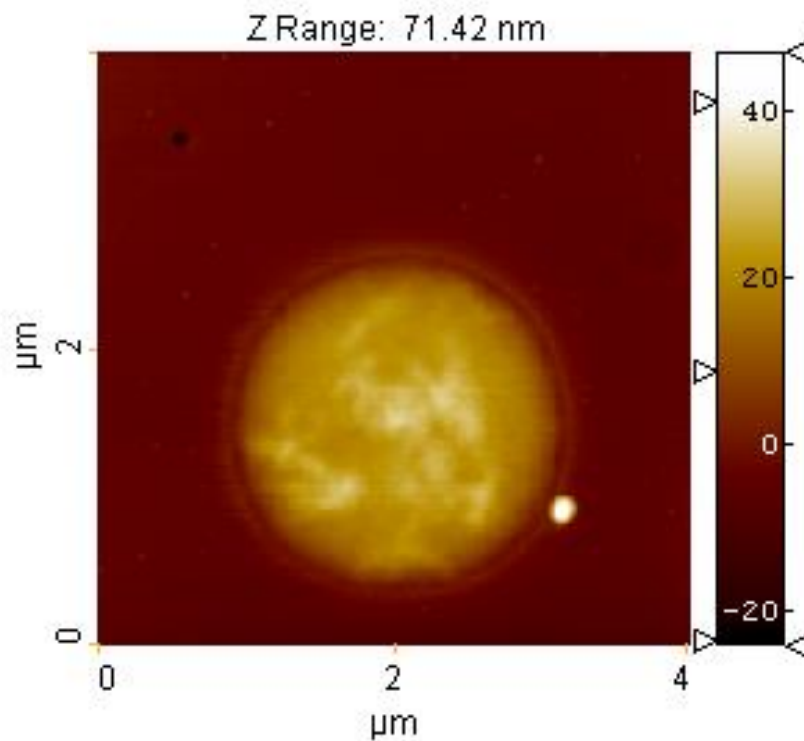
topography
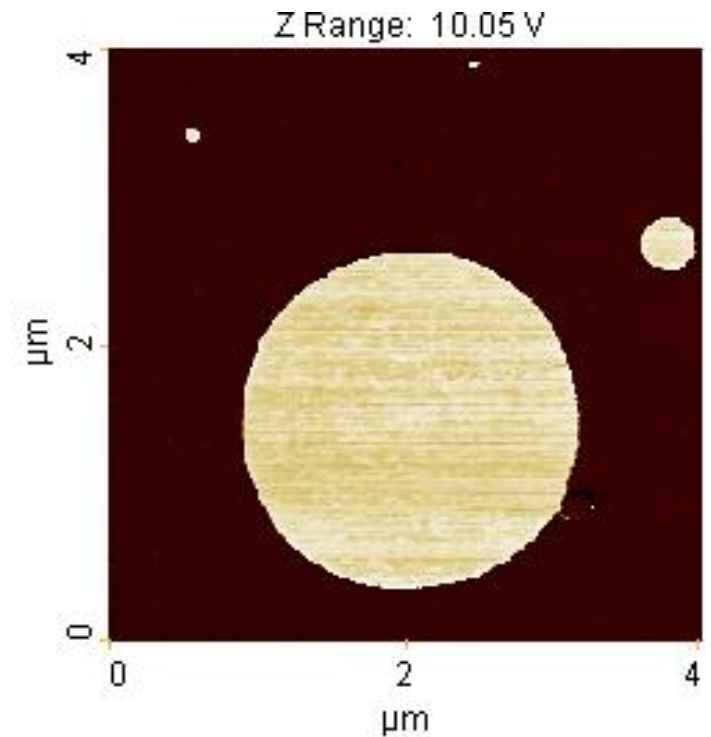
topography correction map

Tip-sample interaction information may have metrological application

2 MPa

0.5 MPa

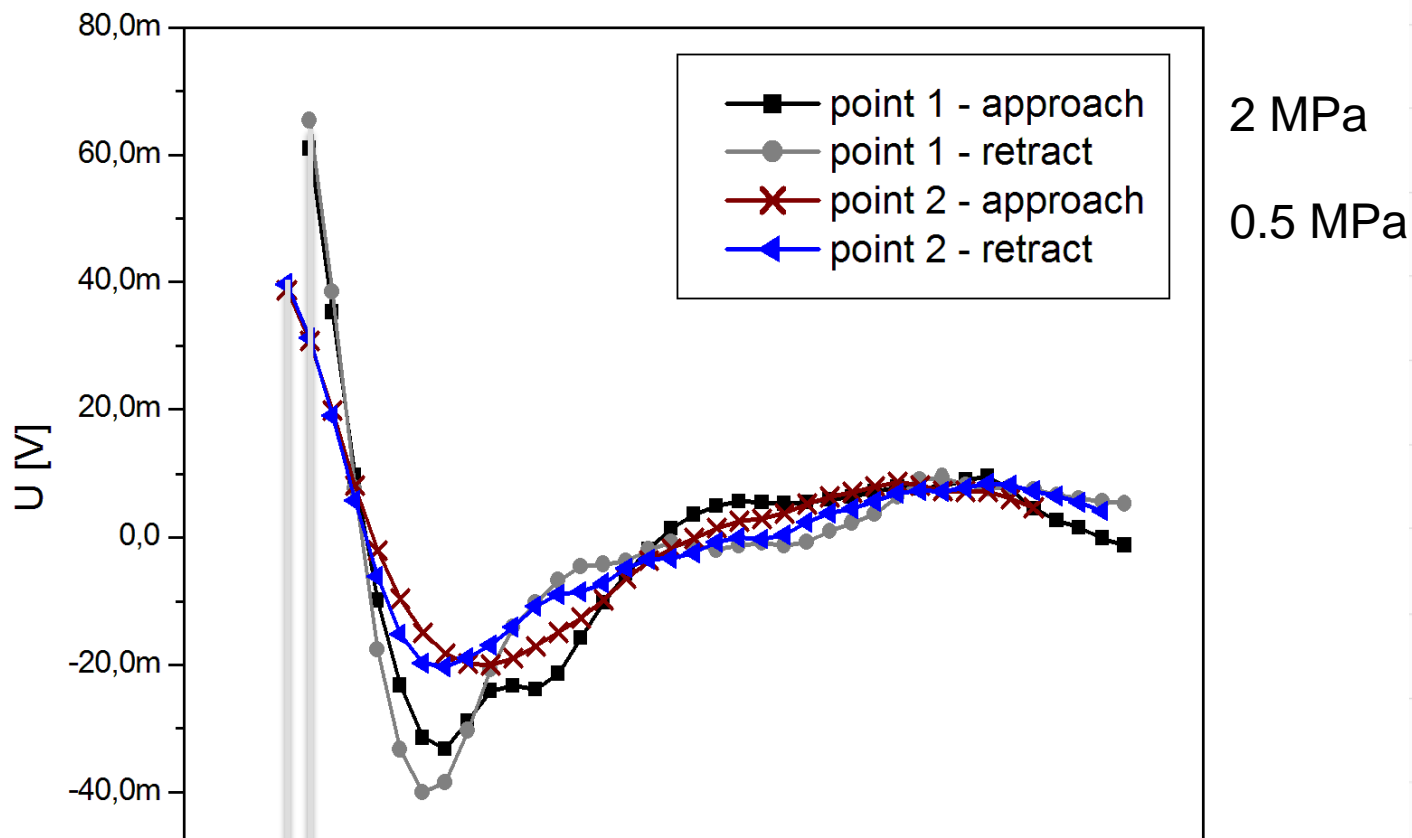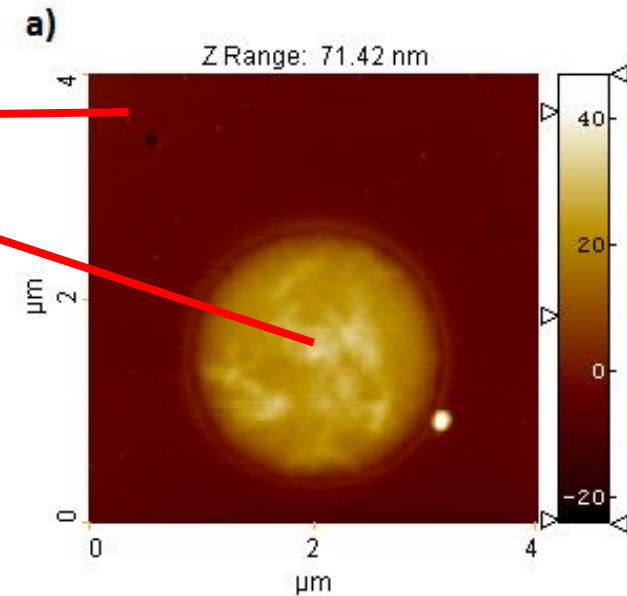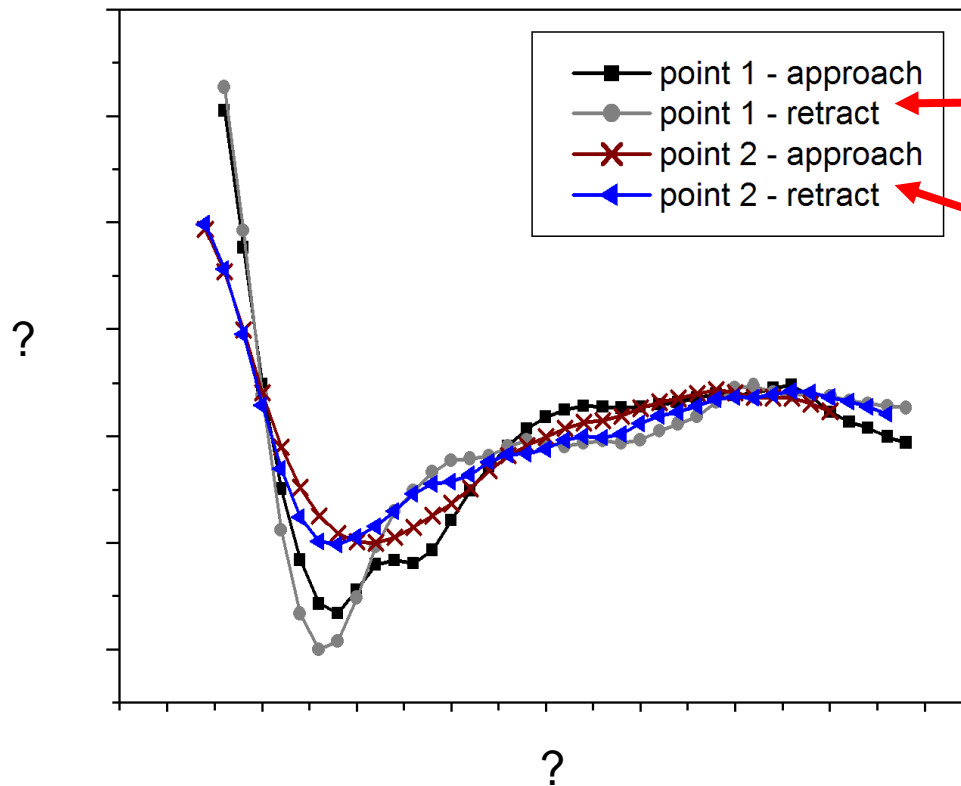**Sikora A.**, Bednarz L.:
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Measurement Science and Technology, 22 (9), p. 94005, 2011

System calibration

A. Sikora
Quantitative normal force measurements by means of atomic force microscopy. Towards the accurate and easy spring constant determination
Nanoscience and Nanometrology 2016; 2(1): 8-29

Acknowledgements

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Thank you for your attention