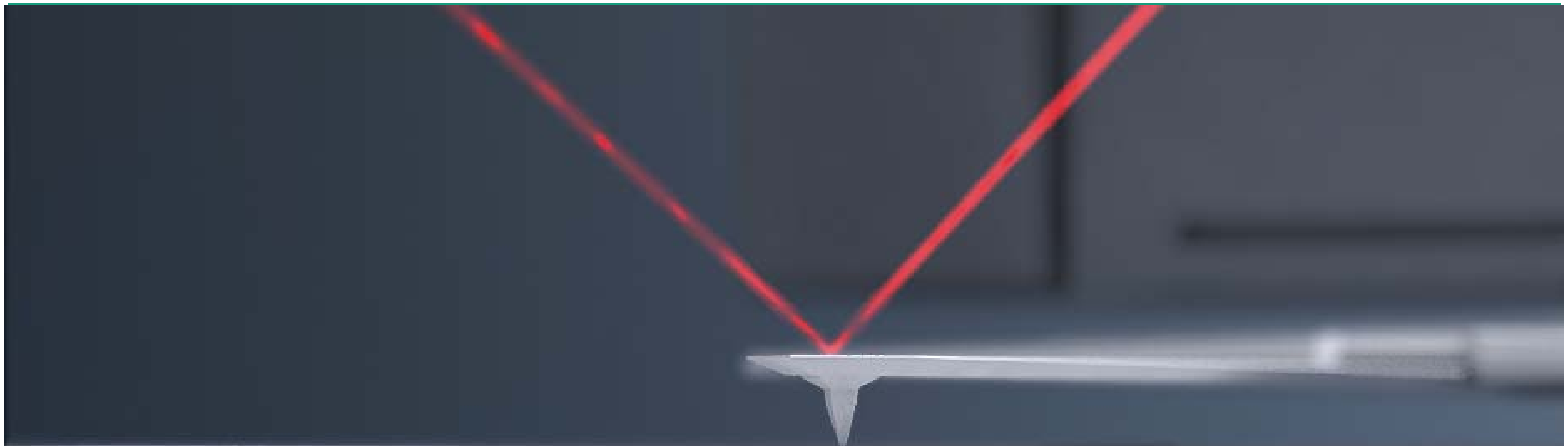
Mapping and evaluation of visco-elastic properties on micro- and nanoscale by use of modified atomic force microscopy



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Fraunhofer IZFP

Overview

Elastic and viscoelastic interactions

Kelvin-Voigt model

Elastic and viscoelastic characterization of materials on macro- and micro-scale

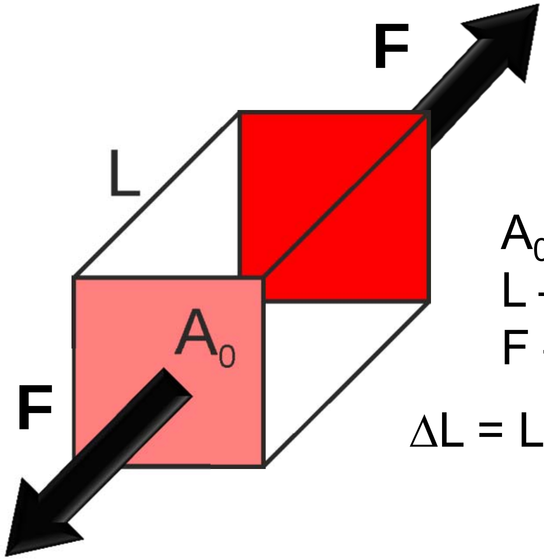
How can we use AFM high resolution methods?

Principle of atomic force acoustic microscopy (AFAM)

Tip-sample interactions, or where is the elastic and viscoelastic component?

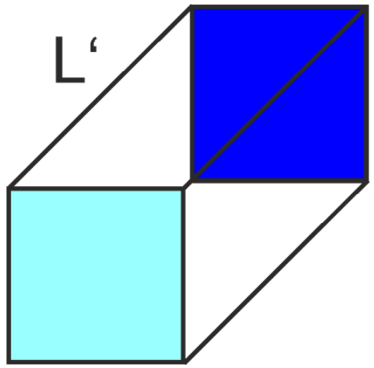
What is also possible…

Elastic interaction



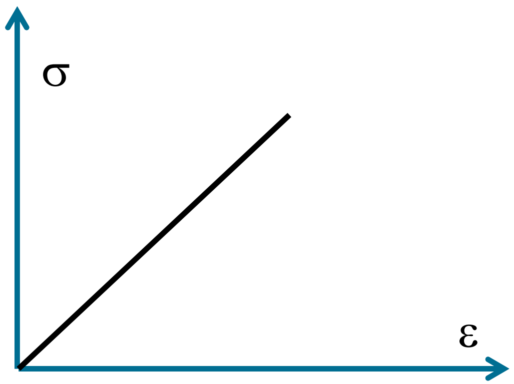
A_0 – surface area
L – length of the element
F – applied load (force)

ΔL = L'-L – change in the length



$$\sigma = \frac{F}{A_0}$$ - applied stress;

$$\varepsilon = \frac{\Delta L}{L}$$ - resulting strain



$$E = \frac{\sigma}{\varepsilon}$$ - Young's modulus

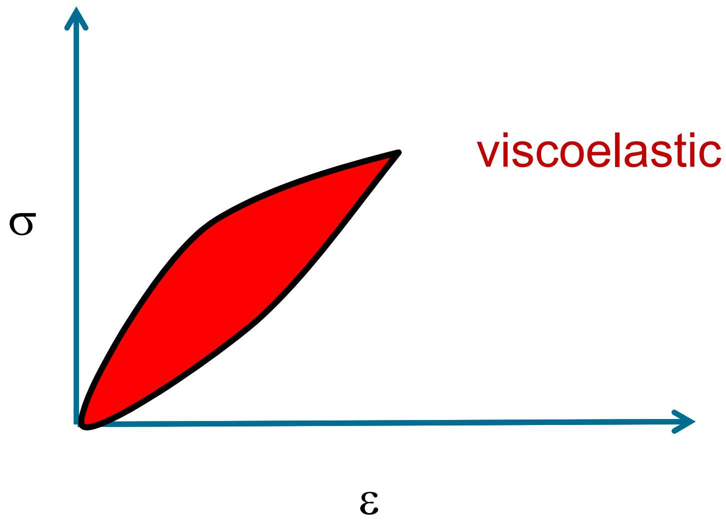
Elastic deformation is reversible

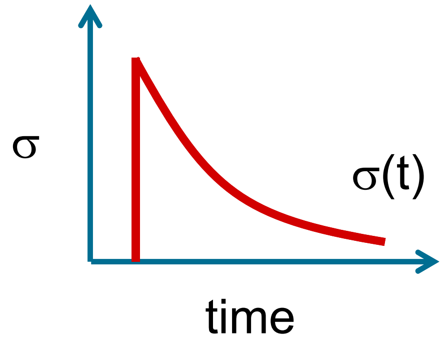
Viscoelasticity

Trademarks of viscoelasticity:

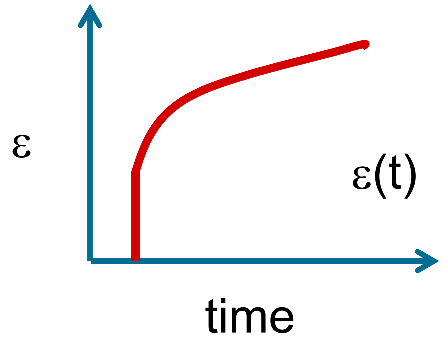
time dependence of material properties

energy dissipation



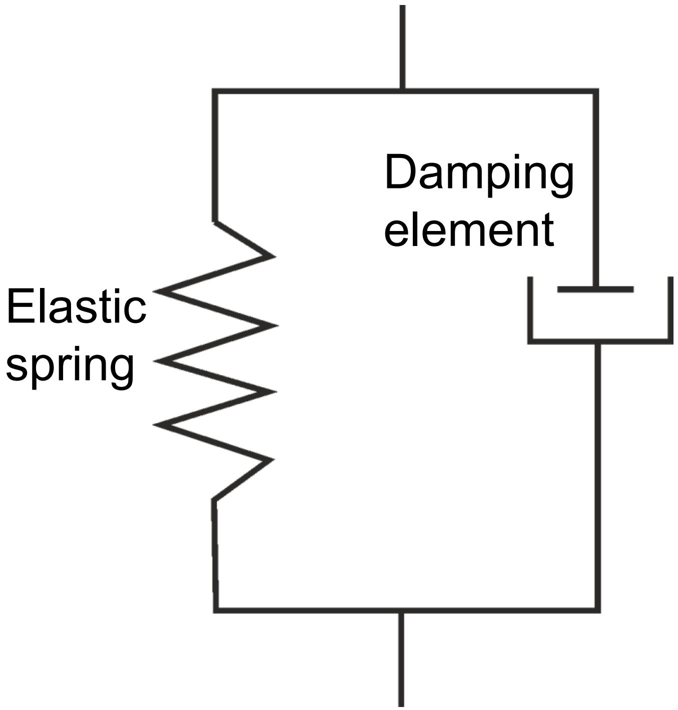


ε = const.
stress relaxation



σ = const.
retardation (creep)

Kelvin-Voigt model



Kelvin-Voigt model describes the time dependence in the stress-strain change rate:

$$\sigma(t) = E\varepsilon(t) + \eta \frac{d\varepsilon(t)}{dt},$$

η is the viscosity

Static methods: $\varepsilon(t) = \frac{\sigma_0}{E}\left(1 - e^{-\lambda t}\right)$,

where $\lambda = \frac{E}{\eta}$ is rate of relaxation

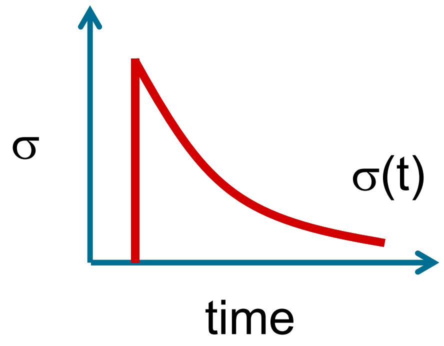
Dynamic methods: $E^{dyn}(\omega) = E' + iE''$,
where E' and E'' are the storage and loss moduli

Methodology for bulk materials

<u>Static testing methods</u>:
measurements of relaxation and retardation

time dependent modulus of elasticity

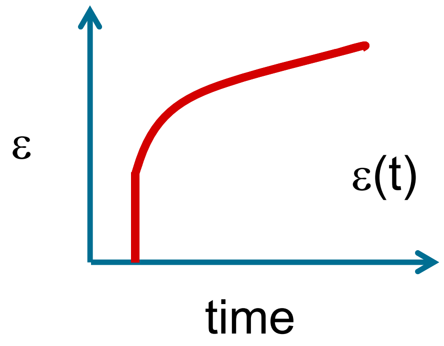
$$E(t) = \frac{\sigma(t)}{\varepsilon_0}$$



stress relaxation

time dependent compliance

$$C(t) = \frac{\varepsilon(t)}{\sigma_0}$$



retardation (creep)

Methodology for bulk materials

<u>Dynamic-Mechanical analysis (DMA):</u>

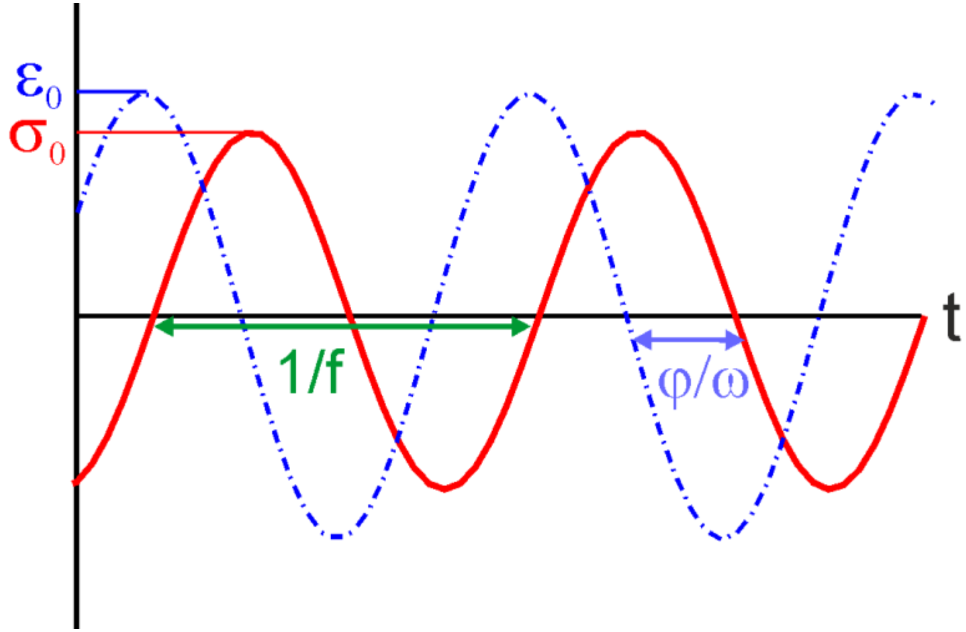
forced vibrations:

$$\varepsilon(t) = \varepsilon_0 sin\omega t,$$

$$\sigma(t) = \sigma_0 \sin(\omega t + \varphi)$$

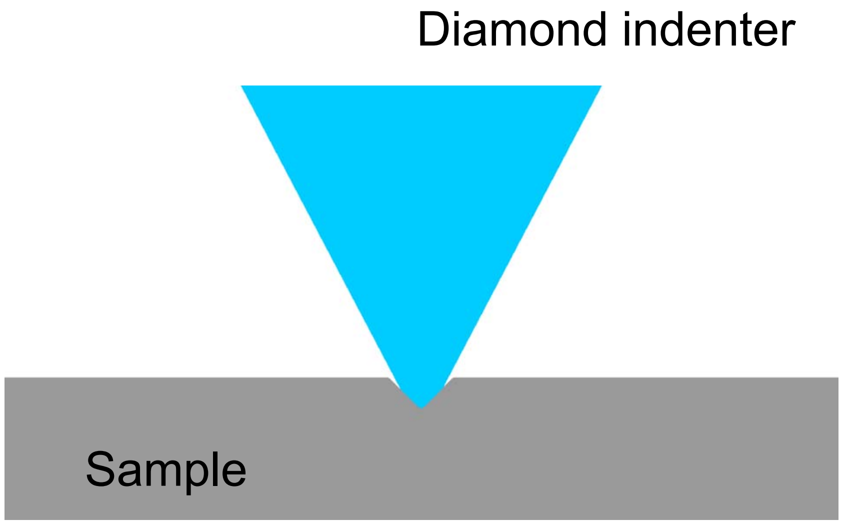
storage modulus $E' = \frac{\sigma_0}{\varepsilon_0} cos\varphi$

loss modulus $E'' = \frac{\sigma_0}{\varepsilon_0} sin\varphi$



What about thin film systems?

Nanoindentation



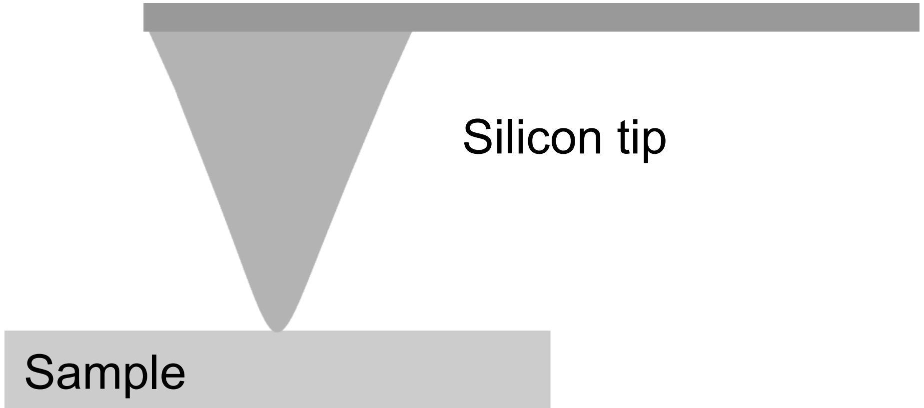
Force range: 100 nN – several mN

Tip radius: 100 nm – 500 nm - 20 µm

Film thickness limit : ca. 200 nm

Indentation depth: 5 nm – 20 µm

AFM based methods

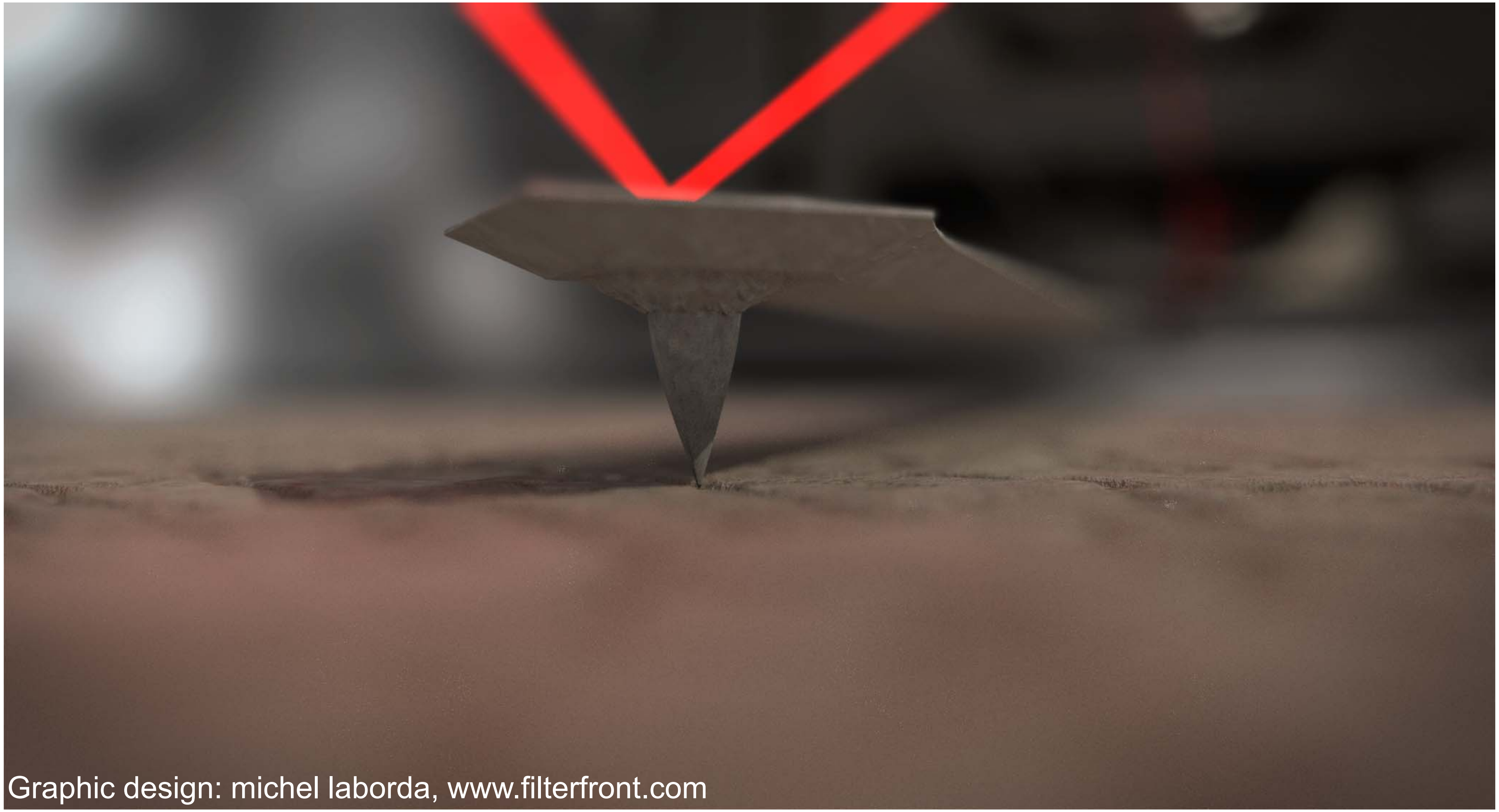


Force range: 1 nN – several µN

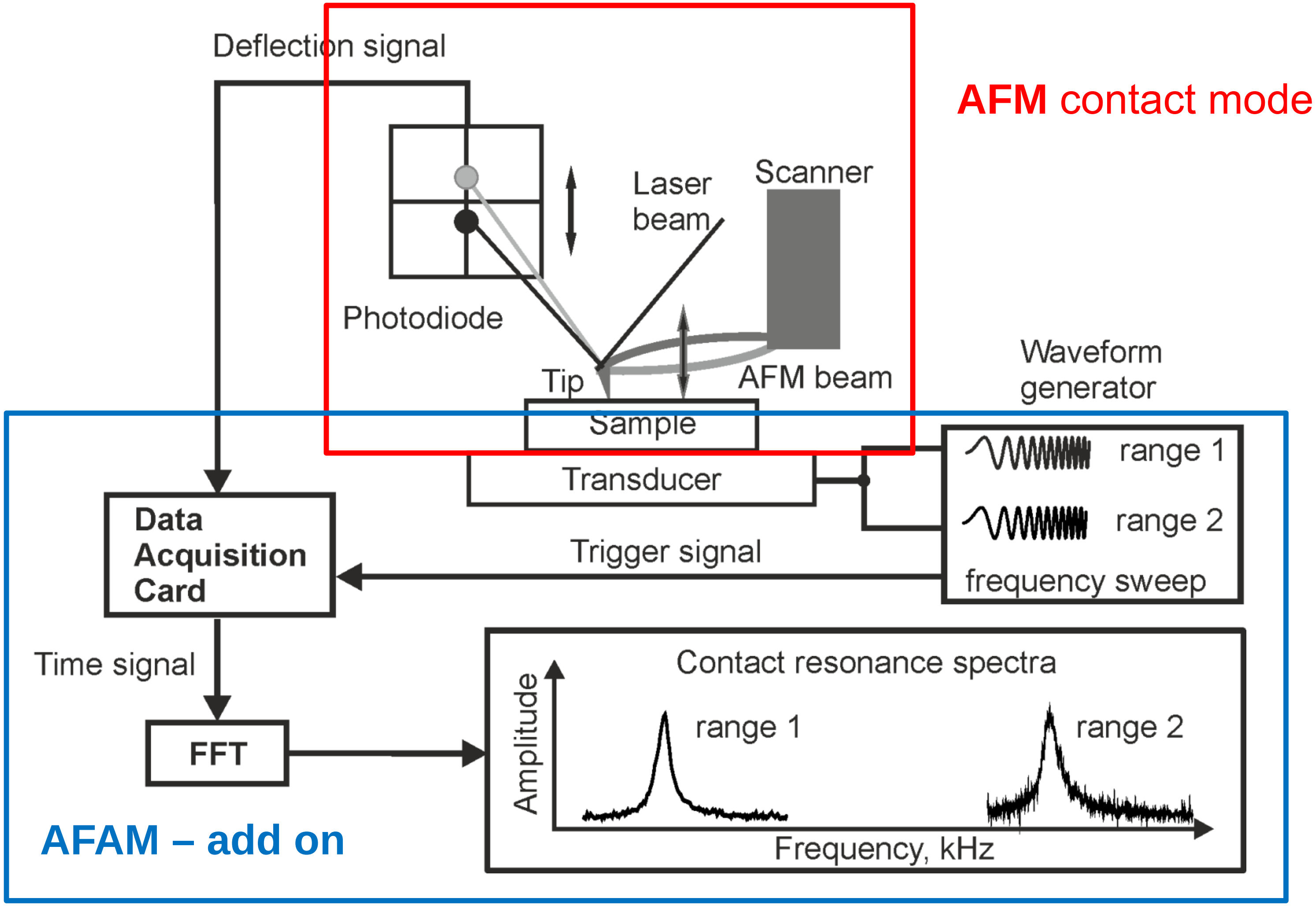
Tip radius: 10 nm – 150 nm – 500 nm

Film thickness limit : ca. 50 nm

AFM methods

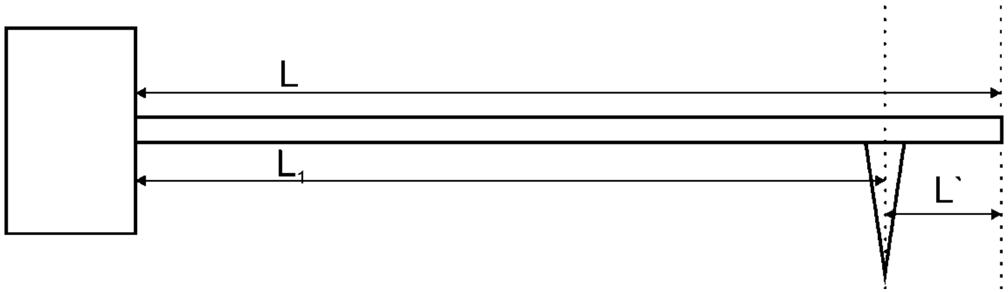


What is AFAM?



Deflection signal
AFM contact mode
Laser beam
Scanner
Photodiode
Tip
AFM beam
Waveform generator
Sample
Transducer
range 1
range 2
frequency sweep
Data Acquisition Card
Trigger signal
Time signal
FFT
Contact resonance spectra
Amplitude
range 1
range 2
Frequency, kHz
AFAM – add on

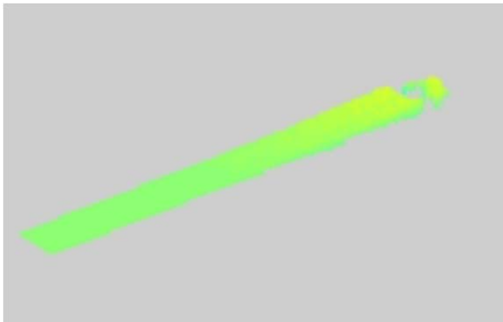
Free resonance frequencies of an AFM cantilever



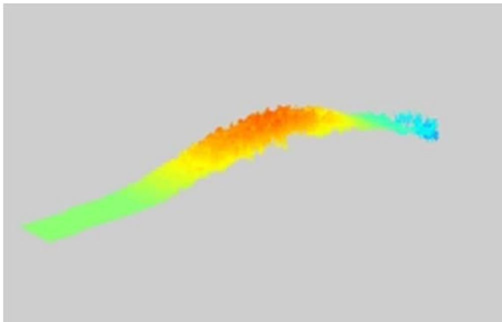
$$f^{1}_{free} = 0.162\sqrt{\frac{E}{\rho}}\frac{T}{L^2}$$

L – length
T – thickness
E – Young's modulus
ρ – density

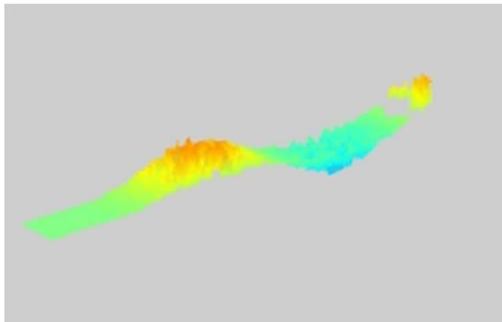
1st Mode f = 85.84 kHz



2nd Mode f = 532.84 kHz

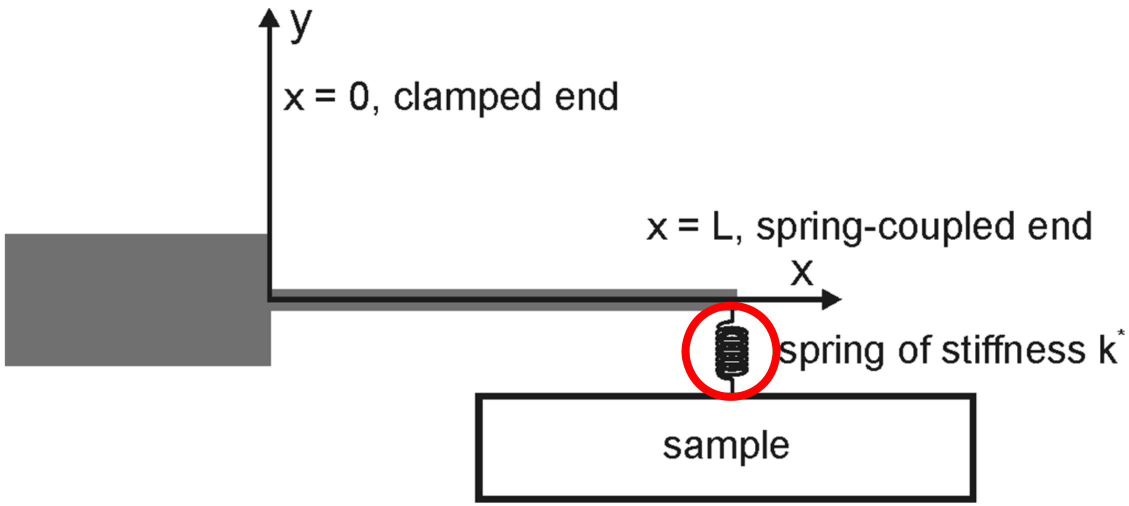


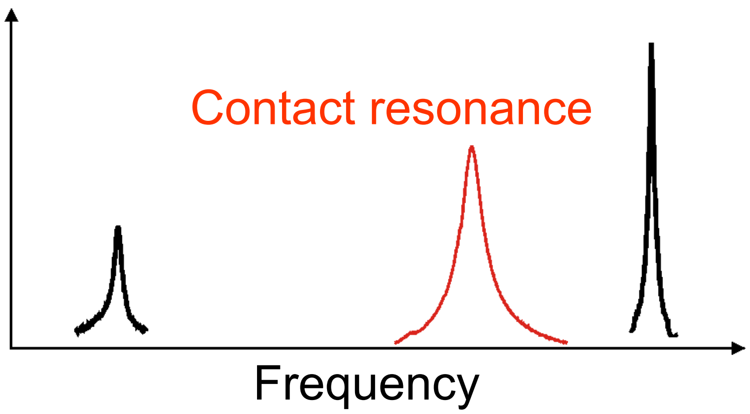
3rd Mode f = 1486.1 kHz



Cantilever vibrations measured at IZFP-Saarbrücken

Contact resonance frequencies of an AFM cantilever





contact mechanics

dynamic behavior of the cantilever

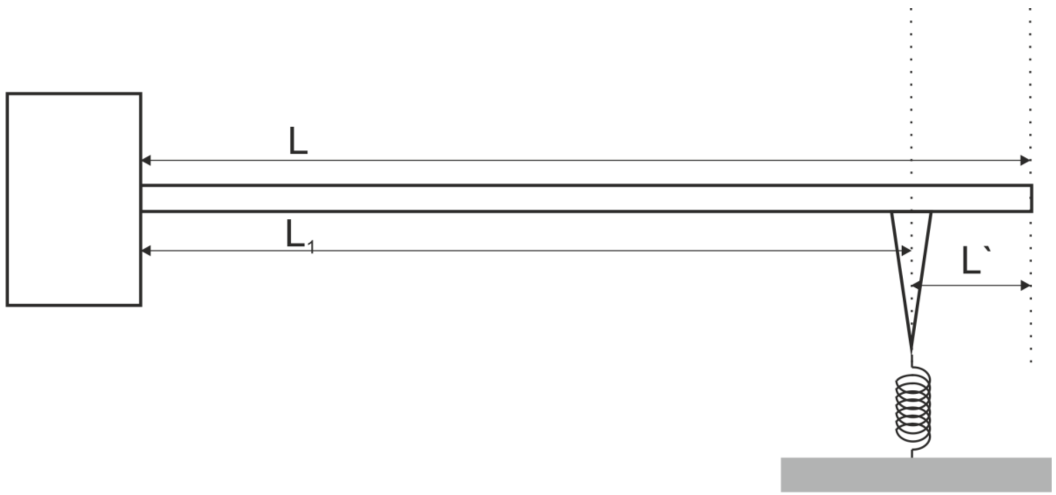
tip-sample contact stiffness

$$k^* = 2aE^*$$

Value of the contact resonance frequency f_n ⟵ k^* ⟶

$$\frac{1}{E^*} = \frac{1}{M_{tip}} + \frac{1}{M_{sam}}$$

Equations for k^* – simple model for elastic interactions



$L = L_1 + L'$

L_1/L – tip position parameter

Contact resonance frequency f_n is a function of:

geometry and mechanical properties of the beam – parameter α

Free resonance frequencies – $f_{n(free)}$

Tip-sample contact stiffness – k^*

Tip-position parameter - $L_1/L = r$

Spring constant - k_c

Equations for k* – the simple model

$$\frac{k^*}{k_c}[-(\cosh\alpha Lr\sin\alpha Lr-\sinh\alpha Lr\cos\alpha Lr)$$

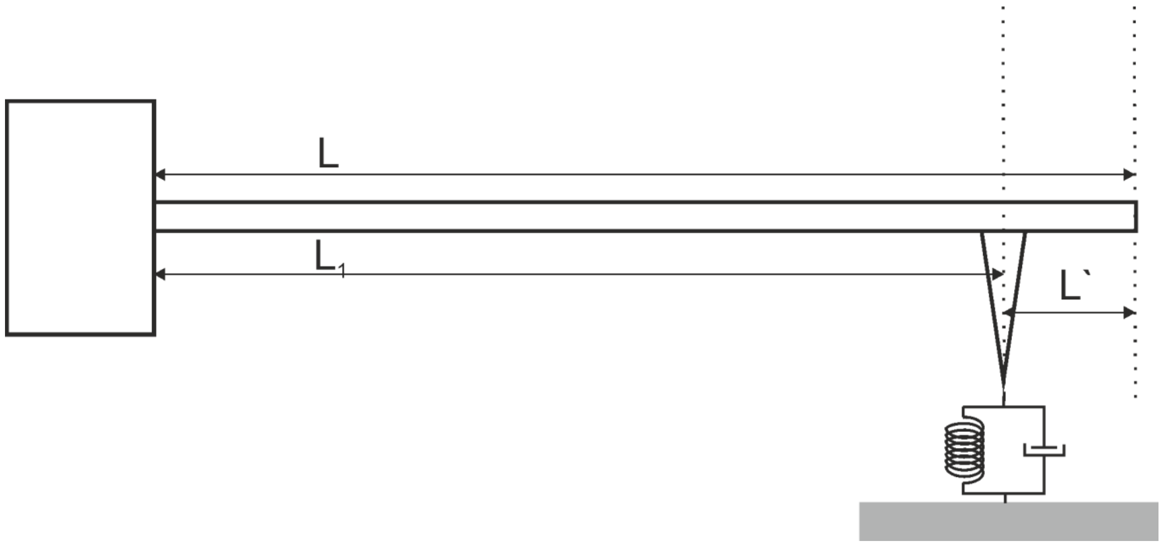
$$\times\begin{pmatrix}1+\cos\alpha L(1-r)\cosh\alpha L(1-r)\\+(\cosh\alpha L(1-r)\sin\alpha L(1-r))-\sinh\alpha L(1-r)\cos\alpha L(1-r)\end{pmatrix}\times$$

$$\times(1-\cos\alpha Lr\cosh\alpha Lr)]=2\frac{(\alpha Lr)^3}{3}(1+\cos\alpha L\cosh\alpha L)$$

$$\alpha L=(\alpha_n L)_{free}\sqrt{\frac{f_{n(cont)}}{f_{n(free)}}}$$

n	$(\alpha_n L)$free
1	1.87
2	4.69
3	7.88

Equations for k* – simple model for viscoelastic interactions



Contact resonance frequency f_n is additionally a function of:

$\chi = \frac{2\pi f_{n0}}{Q_{n0}}$ – damping of the cantilever in air

Q_{n0} – Q factor of the free resonance frequency

Q_n – Q factor of the contact resonance frequency

Equations for k* – simple model for viscoelastic interactions

$$\left(k'+i\beta(\alpha Lr)^2\right)\left[-\left(\cosh\alpha Lr\sin\alpha Lr-\sinh\alpha Lr\cos\alpha Lr\right)\right.$$

$$\times\begin{pmatrix}1+\cos\alpha L(1-r)\cosh\alpha L(1-r)\\ +(\cosh\alpha L(1-r)\sin\alpha L(1-r))-\sinh\alpha L(1-r)\cos\alpha L(1-r)\end{pmatrix}\times$$

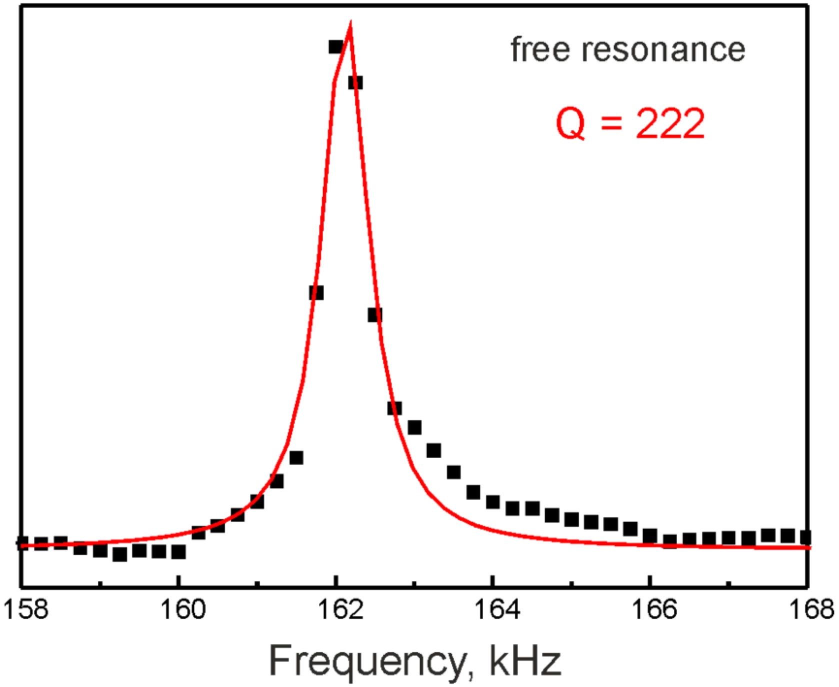
$$\left.\times\left(1-\cos\alpha Lr\cosh\alpha Lr\right)\right]=2\frac{(\alpha Lr)^3}{3}\left(1+\cos\alpha L\cosh\alpha L\right)$$

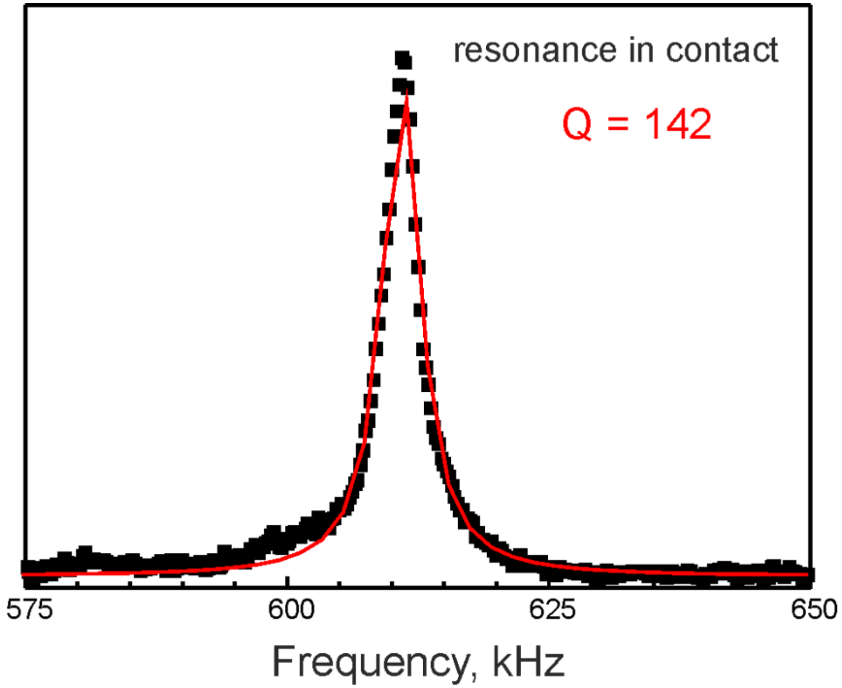
$$\alpha L=(\alpha_n L)_{free}\sqrt{\frac{f_{n(cont)}}{f_{n(free)}}}\left(1+i\left(2\pi f_{n(cont)}-\chi Q_{n(free)}/8\pi f_{n(cont)}Q_{n(con)}\right)\right)$$

k' is the normalized contact stiffness
β is the damping constant

Source: D.C. Hurley, JAP, 2004, J. Killgore, Langumuir, 2011

Q-factor





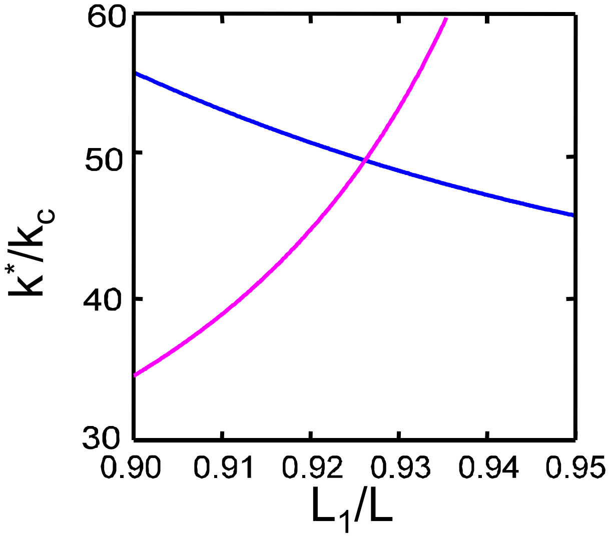
From the analysis of the resonance spectrum we can determine values of the contact resonance frequencies, Q factor, and damping of the cantilever in air

Determination of k^* from the contact resonance frequencies

k^* - tip-sample contact stiffness = ?
r – tip position parameter = ?

We can calculate the values of k* as a function of tip position parameter for a given value of the contact resonance frequency measured with the AFAM system

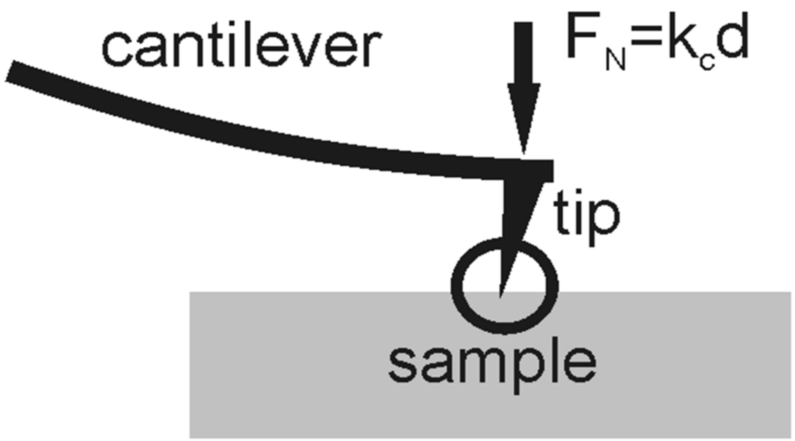
For f_1

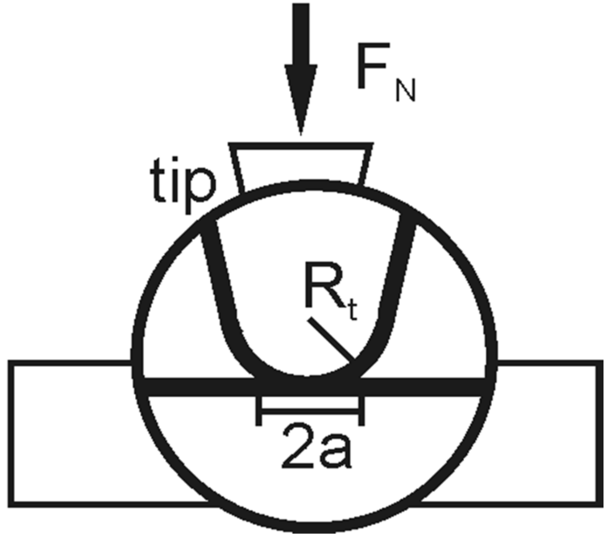


For f_2

We have to measure the contact resonance frequencies for at least two modes!

Contact mechanics





k^* - contact stiffness

a – contact radius

E^*- reduced Young's modulus

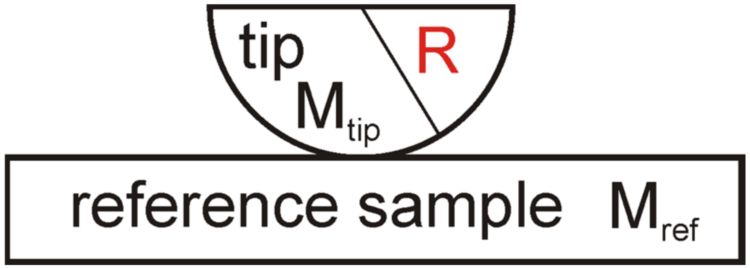
R – tip radius

F – static load

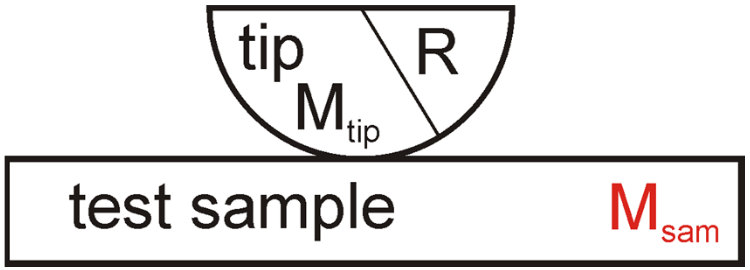
$$a = \sqrt[3]{\frac{3RF}{4E^*}}$$

$$k^* = \sqrt[3]{6RFE^{*2}}$$

How to calculate the indentation modulus M?



$$k^*_{ref} = \sqrt[3]{6RFE^{*2}_{ref}}$$



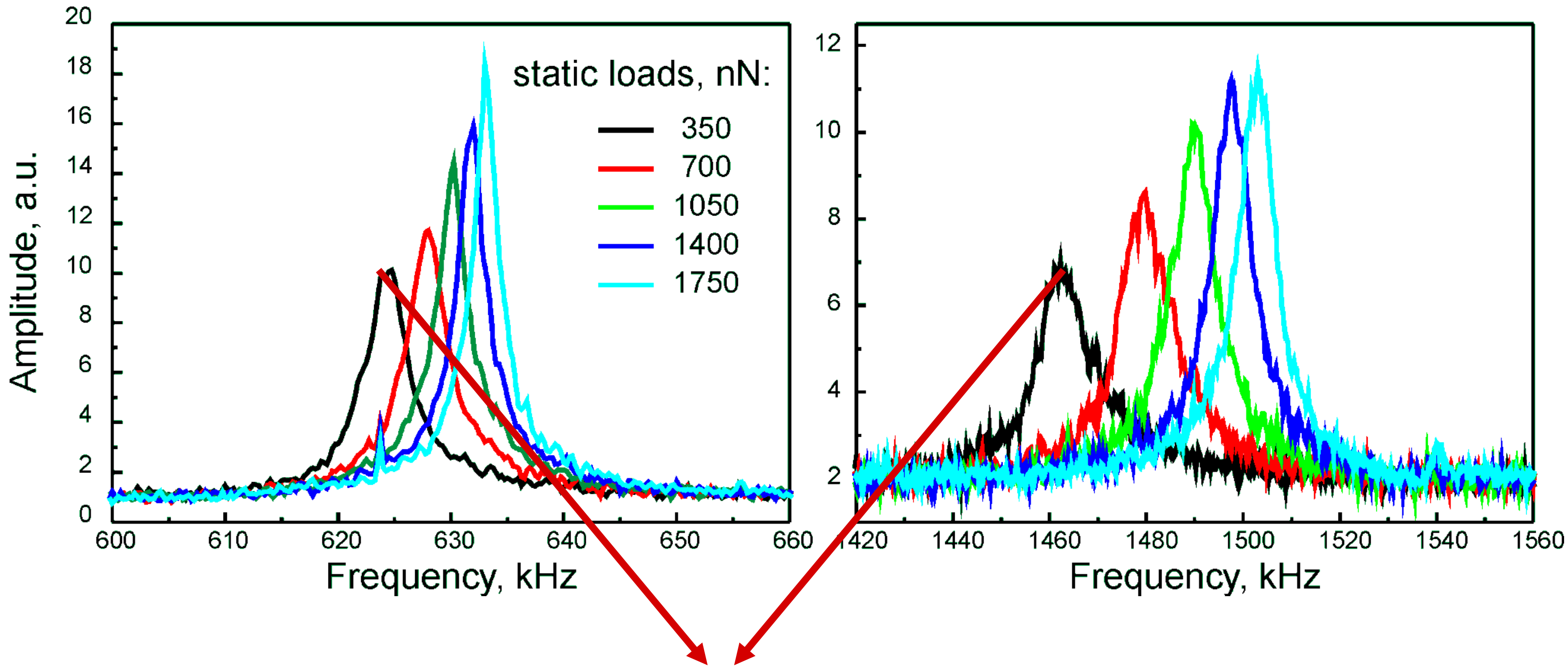
$$k^*_{sam} = \sqrt[3]{6RFE^{*2}_{sam}}$$

$$E^*_{sam} = E^*_{ref}\left(\frac{k^*_{sam}}{k^*_{ref}}\right)^{3/2}$$

$$M_{sam} = \left(\frac{1}{E^*_{sam}} - \frac{1}{M_{tip}}\right)^{-1}$$

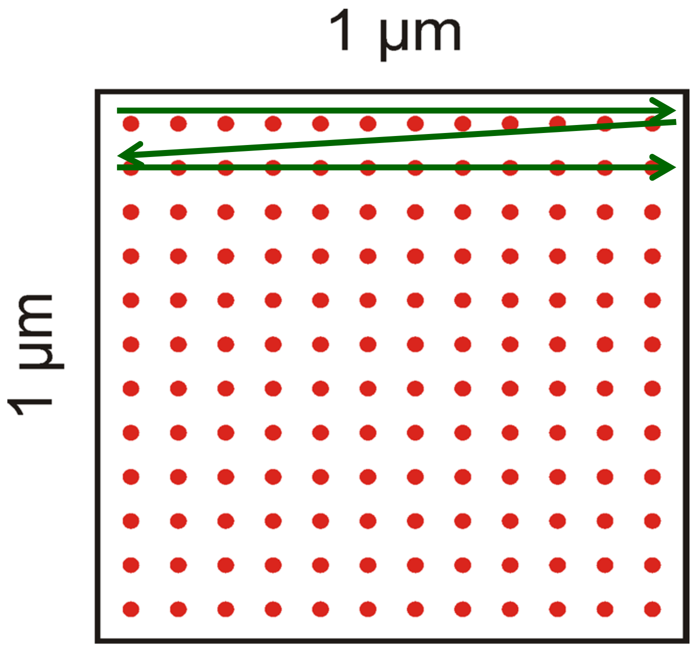
Spectroscopy mode: single point measurements

Contact resonance spectra acquired on Si (100) sample

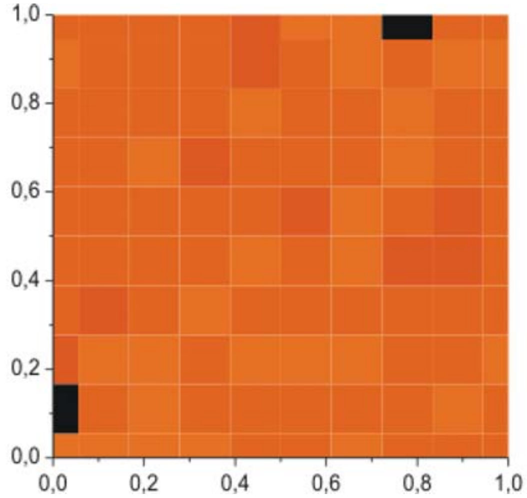


We use the values of the contact resonance frequencies to calculate k*

Principle of the grid measurement



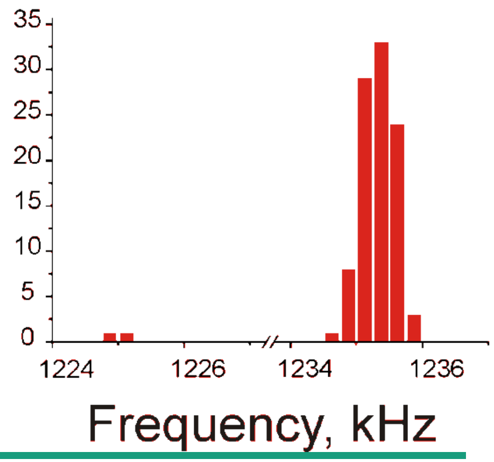
The results can be presented …



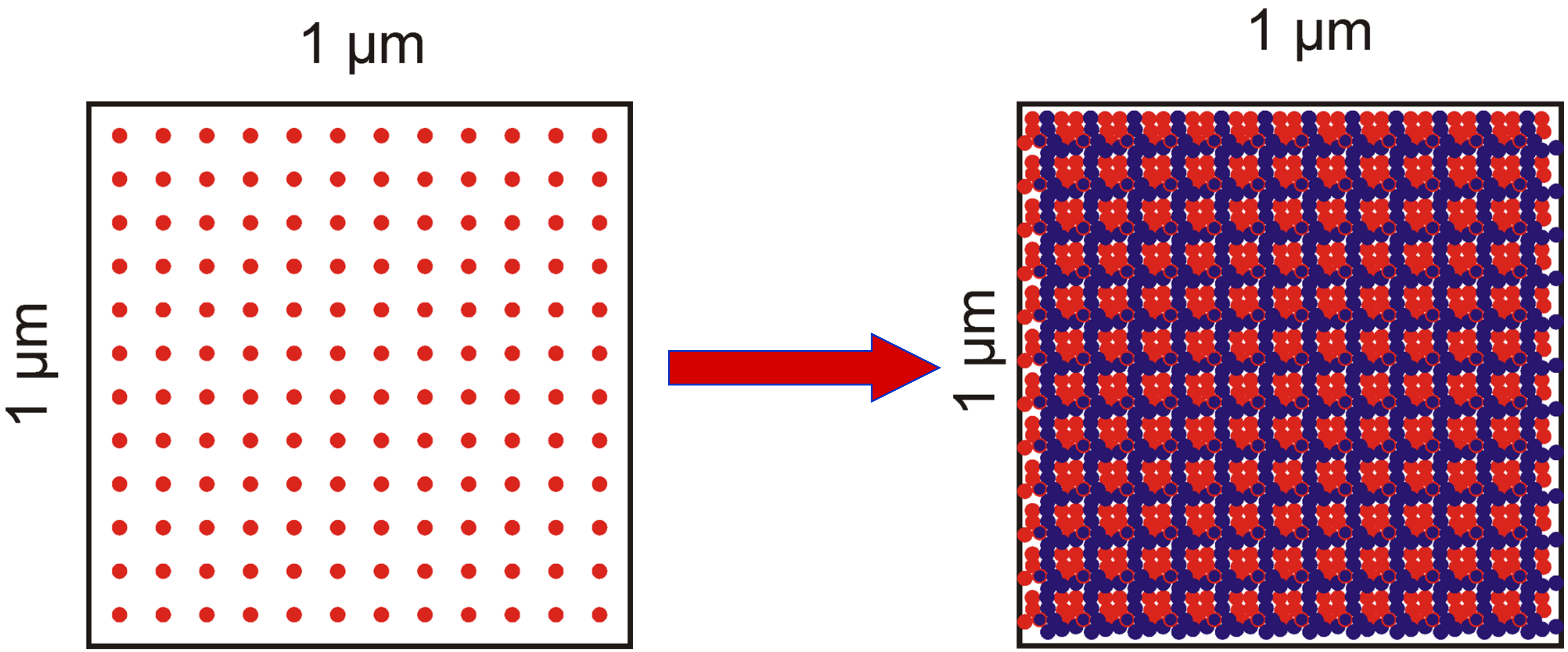
…as grids….

The values of contact resonance frequencies are determined as a function of a position on the sample surface at a fixed value of the applied static load.

…or histograms.



And for inhomogeneous samples…



10 × 10 points

128 × 128 points and more

In the near future

AFAM Dynamic-Mechanical analysis (DMA):

forced vibrations:

$$F(t) = F_0 sin\omega t$$

Determine k*, ε, σ and calculate

storage modulus $E' = \frac{\sigma_0}{\varepsilon_0} cos\varphi$

loss modulus $E'' = \frac{\sigma_0}{\varepsilon_0} sin\varphi$