Centrale de Technologie
en
Micro et nanoélectronique

CR-AFM
(Contact Resonance Atomic Force Microscopy)

Atelier DFRT
Nanomécanique

6 octobre 2016

Richard Arinero

SUMMARY

1. Contact-resonance AFM : Introduction
2. Indirect vs direct modulation methods
3. Modelling of the cantilever dynamics
   - Analytical models
   - Finite elements modelling
4. Contact mechanics
5. Calibration of the cantilever's force constant
6. Calibration of contact resonance frequency
7. Imaging techniques (from fixed frequency to DFRT)
8. Some recent advances

AFM nanomechanics : 2 different approaches

1. <u>« Vibrating » contact mode</u>

At low frequencies Force modulation (Maivald 1991)

At high frequencies

Ultrasonic force microscopy (UFM) (Kolosov 1993)

Contact-resonance (Yamanaka, Arnold 1994)

→ DFRT (Dual Frequency resonance tracking) (Rodriguez 2007)
DART (Dual AC resonance tracking) (Asylum 2011)

2. <u>Force distance spectroscopy approaches</u>

Force – distance curves (1986)

Force – volume (mid 1990's)

Pulsed Force microscopy (van der Werf 1994) (Witec early 2000's)

→ Harmonix (Sahin 2007), Peak-Force (Bruker 2008), QI (JPK 2012)

THE ANCESTOR OF CR-AFM

Force modulation microscopy

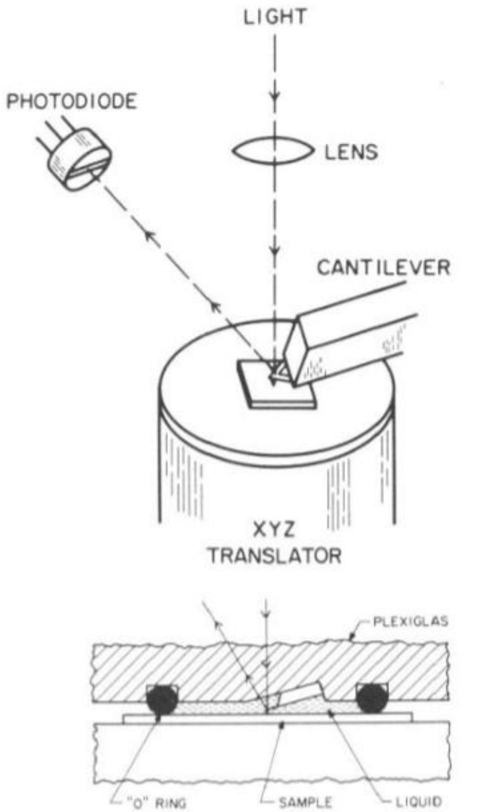


Figure 1. Schematic diagram of our AFM. Laser light is focused on a cantilever that reflects it onto a segmented photodiode. The photodiode senses the deflection of the reflected beam and thus the deflection of the cantilever. In operation a feedback loop controls the vertical position of the sample and hence the force exerted on it by the cantilever. This is accomplished by moving the sample up and down as it is scanned.

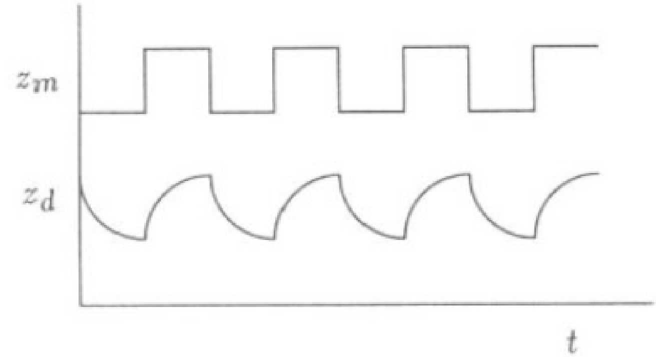


Figure 4. Diagram showing the modulation applied to the *xyz* translator, ΔV_m, and the corresponding photodetector response, ΔV_d.

The quantity $\Delta z_d/\Delta z_m$ is then used to create a force modulation image.

$$k_s = \Delta F/(\Delta z_m - \Delta z_d) = k_c/(\Delta z_m/\Delta z_d - 1)$$

$$\Delta z_m/\Delta z_d = k_c/k_s + 1.$$

For $k_c/k_s \ll 1$, variations in k_s are not picked up since the response in Δz_d is too small.

Maivald Nanotechnology 1991

THE ANCESTOR OF CR-AFM

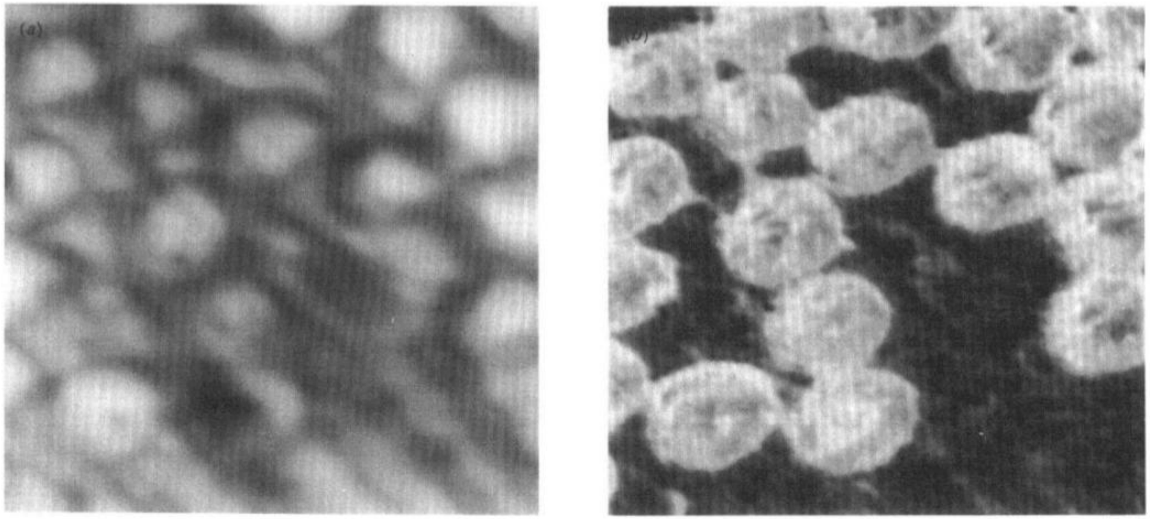


Figure 2. Image of the carbon fibre and epoxy composite in air. Intensity corresponds to (*a*) height in the topographic image, (*b*) stiffness in the force modulation image. Image width is 32 μm.

k_c=3000 N/m

r=10 μm

Δz_m=25nm

Hertz model

$$\Delta F=(M_s^2 r d^3)^{1/2}$$

$$M_s=(\sqrt{2}k_c/a_s)[R(\Delta V_m/\Delta V_d)-1]^{-1}.$$

M_s: indentation modulus

On infinitely hard sample

$$\Delta z_d/\Delta z_m=(1/R)\Delta V_d/\Delta V_m$$

$$\Delta z_d/\Delta z_m=1, \text{ and } R=\Delta V_d/\Delta V_m.$$

R : calibration factor

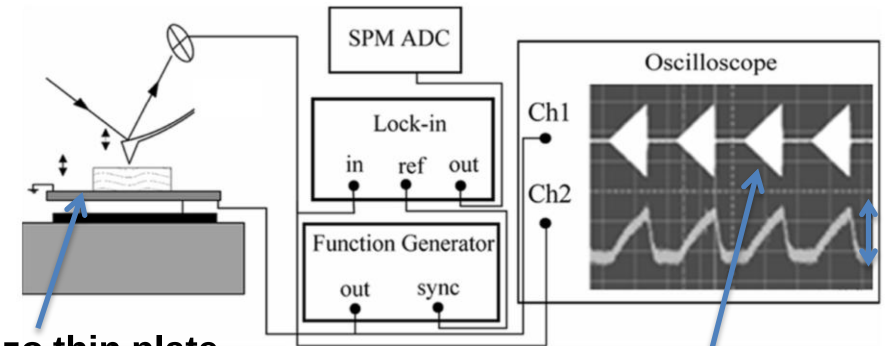
For the epoxy, we measure $\Delta V_d/\Delta V_m=0.10$, resulting in a measured modulus $M_s=7.0\times10^{10}$ Pa.

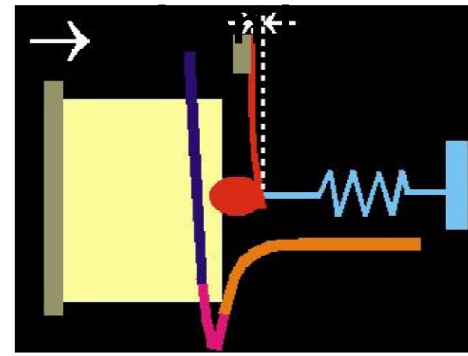
Calculation very sensitive to the value chosen for a_s (contact radius)

ULTRASONIC FORCE MICROSCOPY (UFM)

Kolosov 1993
Dinelli 2000
Cuberes 2001

Non linear detection of f(d) separation → Mechanical diode effect





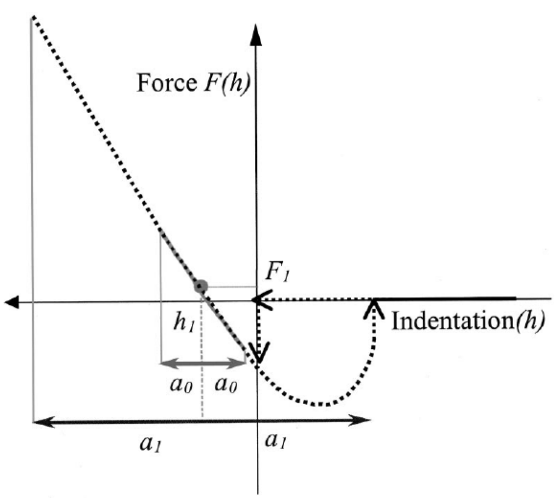
Amplitude used To create elastic image

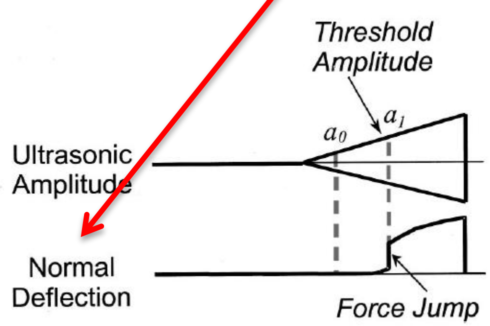
Piezo thin plate
~ f_{ult} few MHz

Sawtooth modulation (few volts)
~ few kHz

Averaged force

$$F_m(h_1, a) = \frac{1}{T_{ult}} \int_{T_{ult}} F[h_1 - a\cos(2\pi f_{ult} t)]\,dt$$





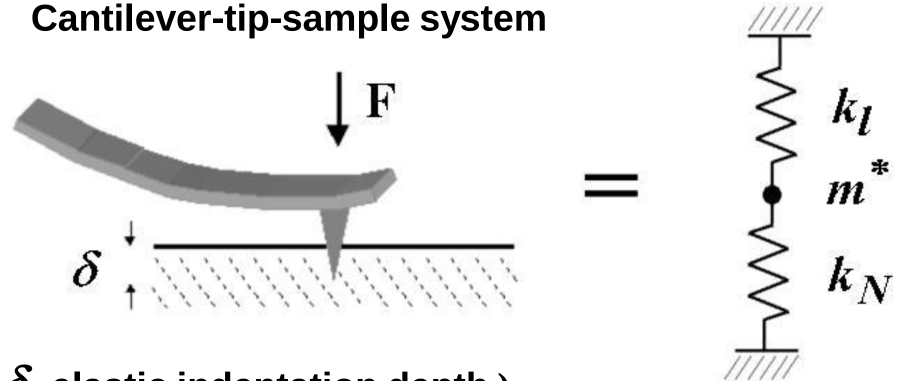
Advantage:
k_c ~10^5 times higher at 3 MHz

Disadvantage:
Quantification not easy

CR-AFM

IDEA: To probe the local elastic deformations of the tip-sample system by means of cantilever's resonance frequency in contact mode

Cantilever-tip-sample system



(δ elastic indentation depth)

First approximation :

contact = spring = normal contact stiffness

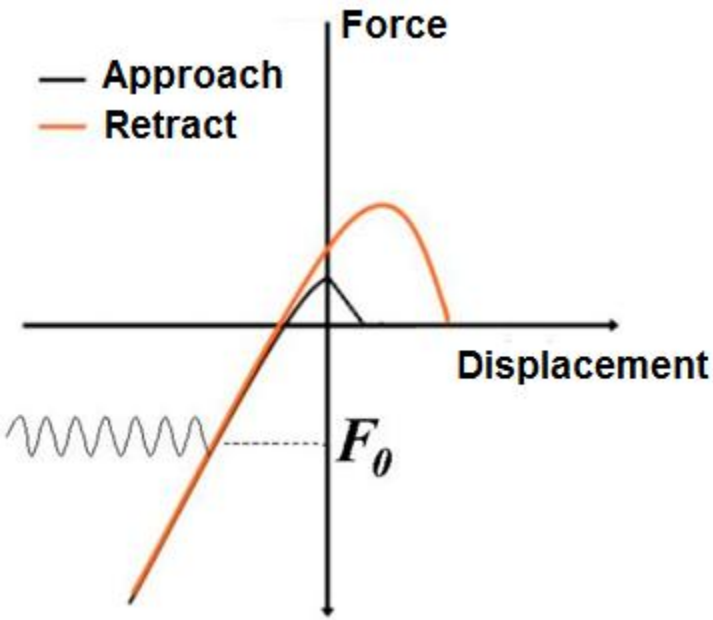
$$k_N = \frac{\partial F}{\partial \delta} = 2aE^*$$

k_N **depends on :**

- **the contact area**
- **Elastic properties of the surface (E,ν)**

The variation of k_N according to F is predicted by Contact Mechanics Theory

CR-AFM



Applied force is modulated

$$F = F_0 + F_{excitation} \times \sin \omega t$$

if $F_{excitation}$ is small

<u>LINEAR REGIME</u>

Amplitude of vibration and resonance frequency depends on the contact stiffness k_n

<u>Problem to solve</u> : **1. Relation between resonance frequency and k_N**

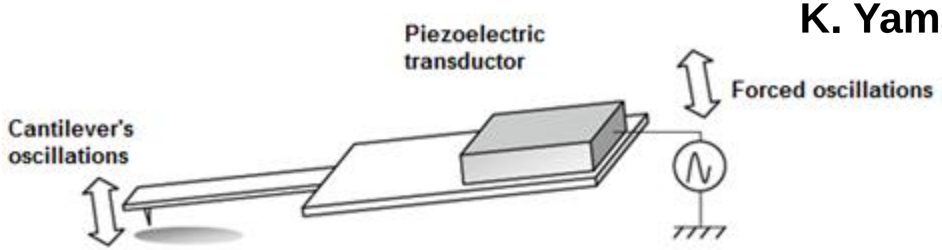
2. Relation between k_N and E

INDIRECT MODULATION METHODS

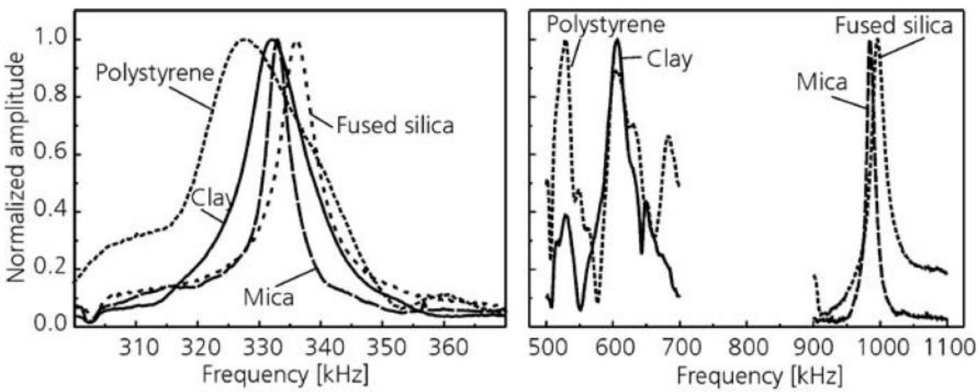
The cantilever is <u>not directly</u> excited

Modulation via the cantilever holder

U. Rabe et al *Surf. Interf. Anal.*, 27, 1999
K. Yamanaka et al, *Jpn. J. Appl. Phys.*, 35,1996



CR-AFM excited by means of piezoelectric actuation of the cantilever's holder

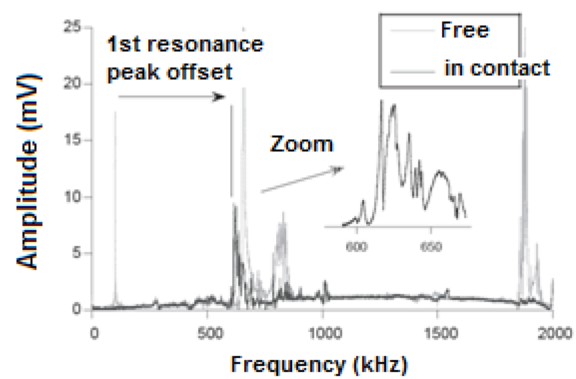


Contact-resonance spectra on polystyrene, clay, mica and fused silica . The spectra of the first and the second contact-resonance frequency of a cantilever with a spring constant of 1.5 N/m are shown

Prasad et al (2002) Geophys Research Lett 29:13-1

The sensitivity to Hard samples is Better using higher harmonics

F. Mege, PhD thesis 2011



Silicon sample

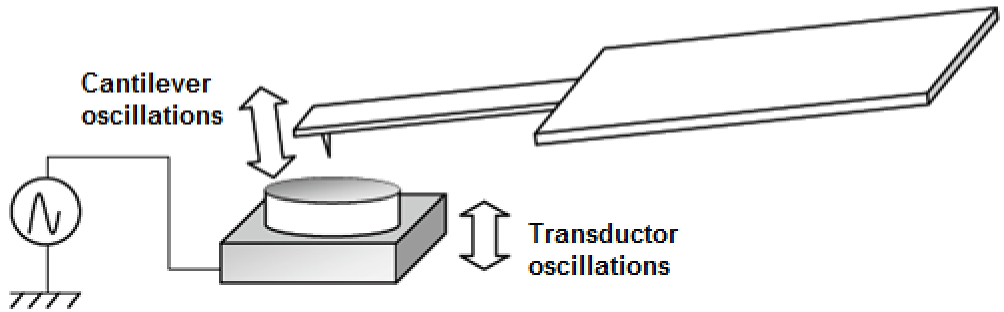
INDIRECT MODULATION METHODS

The cantilever is <u>not directly</u> excited

Modulation via the sample holder

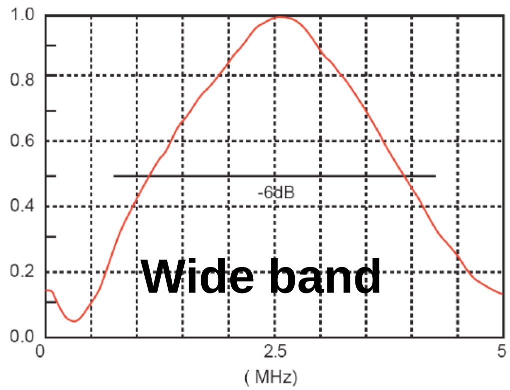
U. Rabe and W. Arnold., *Applied Physics Letters*, 64, 1994
U. Rabe et al, *Review of Scientific Instruments*, 67, 1996

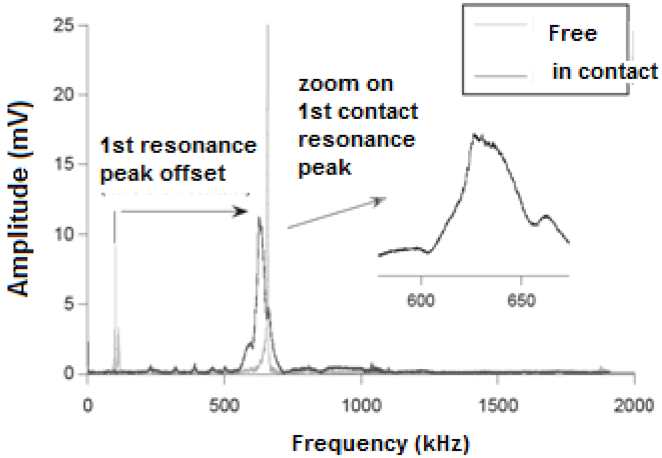
AFAM (Atomic force Acoustic microscopy)



CR-AFM excited via a piezo transductor located under the sample

Panametrics-NDT V106-RM





Silicon sample

F. Mege, PhD thesis 2011

Possible subsurface observation of subsurface defects

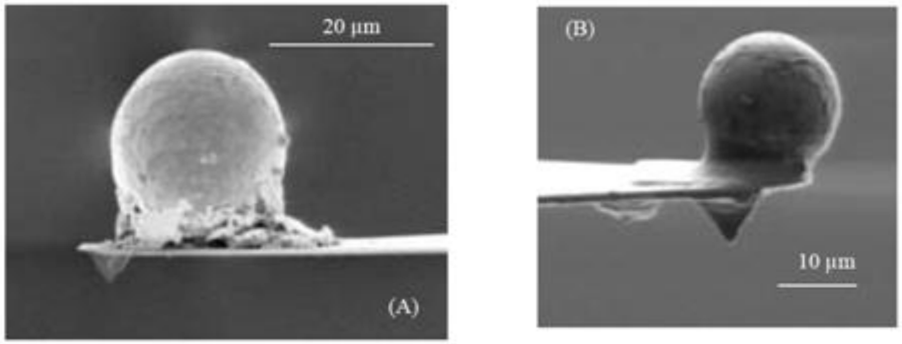
Possible mechanical coupling effects

DIRECT MODULATION METHODS

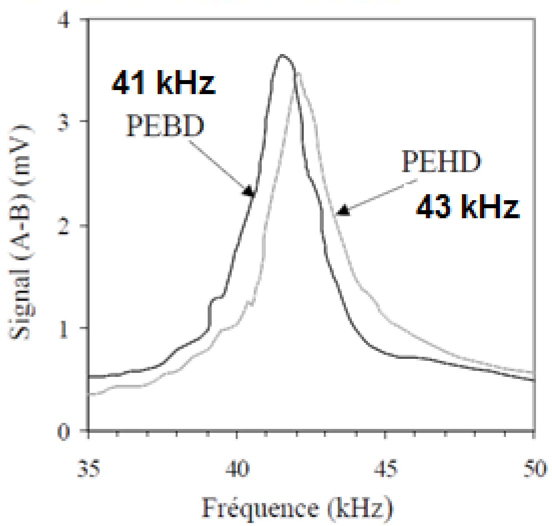
The cantilever is <u>directly</u> excited

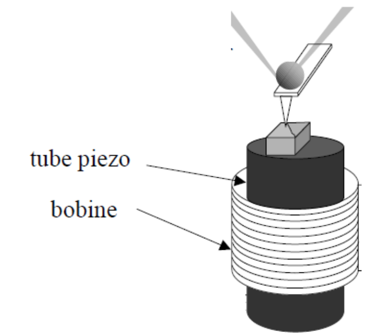
Magnetic excitation

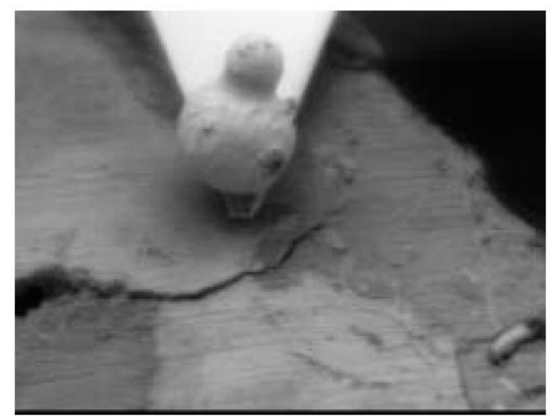
O. Piètrement, PHD Thesis (2000)



Cantilevers with magnetic particles glued at their extremity







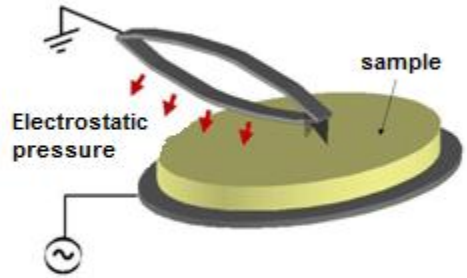
DIRECT MODULATION METHODS

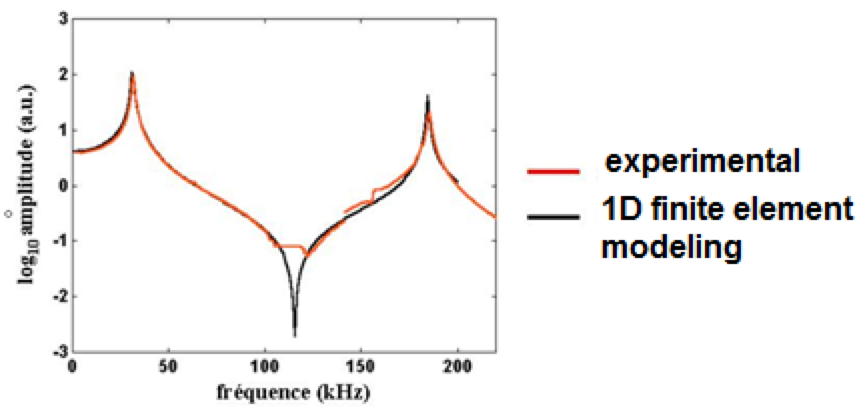
The cantilever is <u>directly</u> excited

Electrostatic excitation

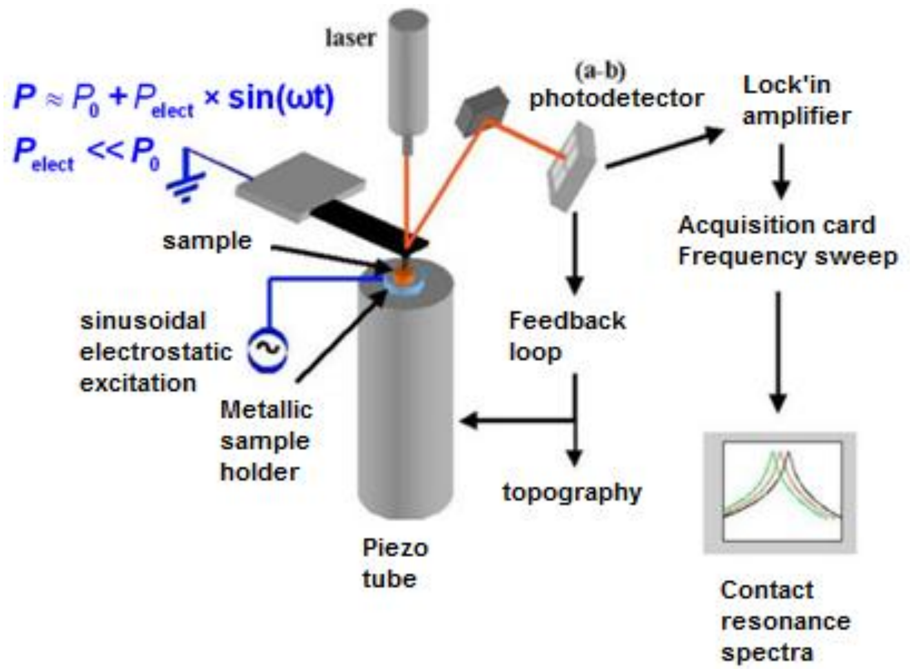
$$F_{élec} = \frac{1}{2} V^2 \frac{\partial C}{\partial z} = F_0 + F_\omega + F_{2\omega}$$

S. Cuenot, PhD thesis 2002
R. Arinero, PhD Thesis 2003





Example of free cantilever vibrating in the air

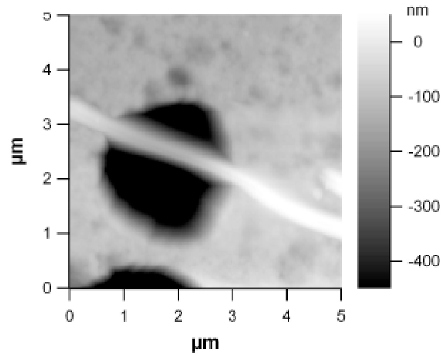


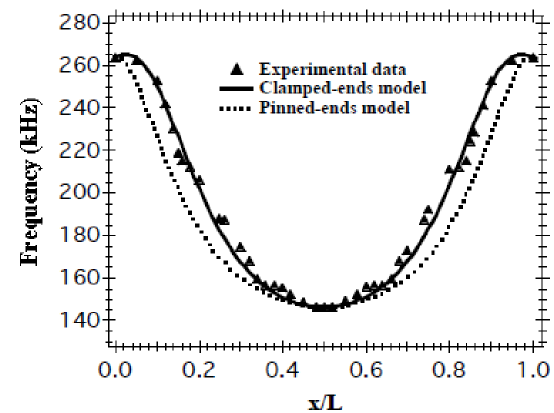
DIRECT MODULATION METHODS

Elastic Modulus of suspended nanotubes

<u>The cantilever is directly</u> excited

Polypyrrole nanotubes





Cuenot et al, JAP, 93, 2003

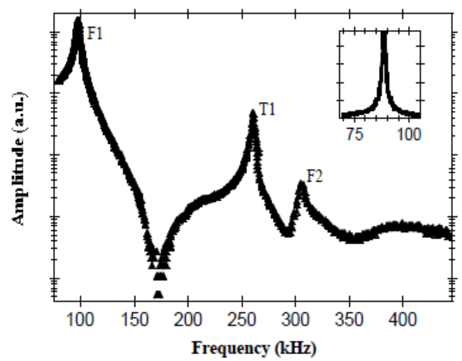
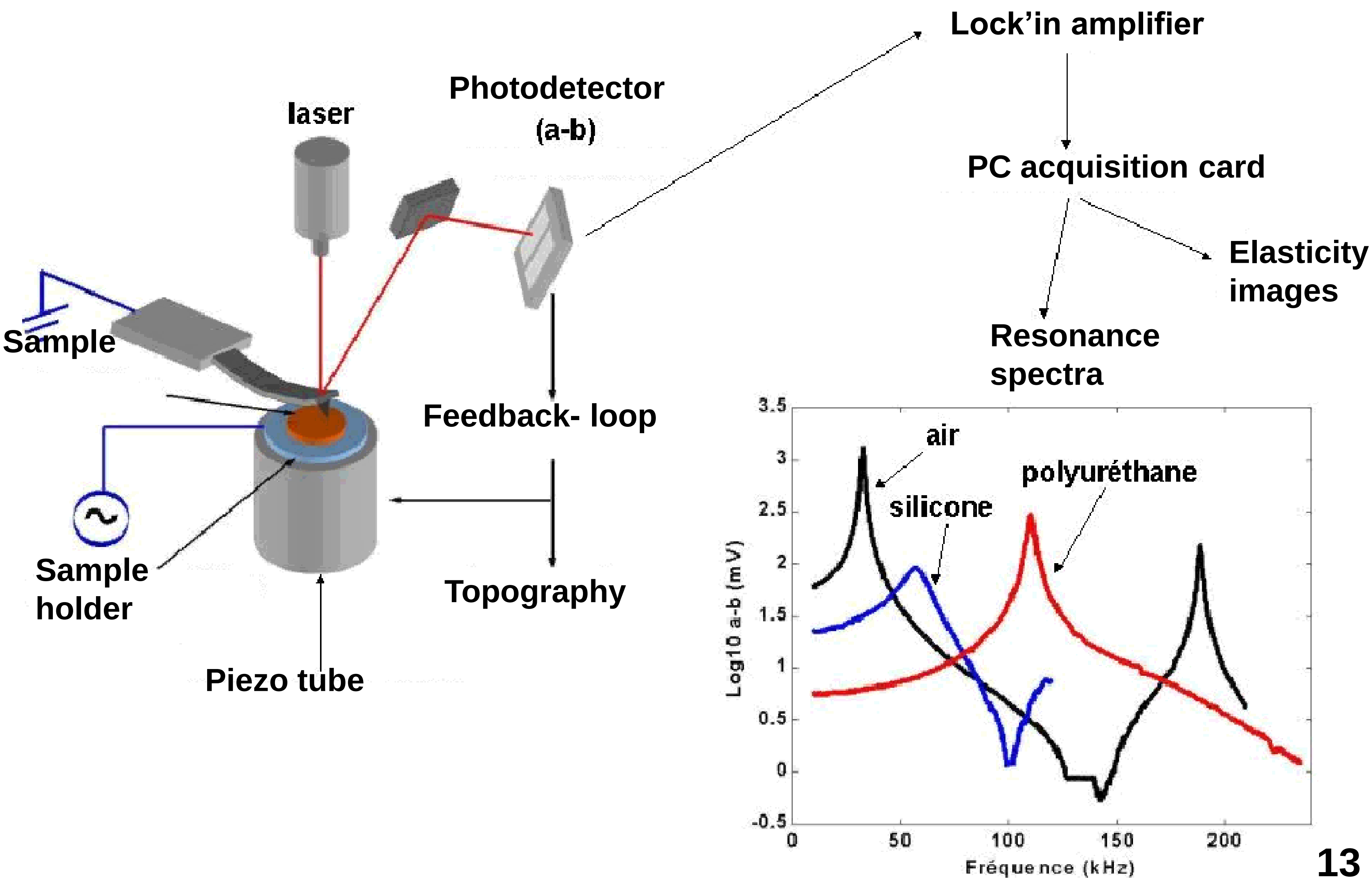


Fig. 2: Typical resonance spectrum measured for a cantilever in contact with a nanotube (logarithmic ordinate axis) (see text for peak labeling). In the insert, the resonance peak of the free cantilever is given for comparison purposes.

Uncertainty between 15 and 30%
For diameter larger than 100 nm

Uncertainty between 25 and 45%
For diameter lower than 50 nm

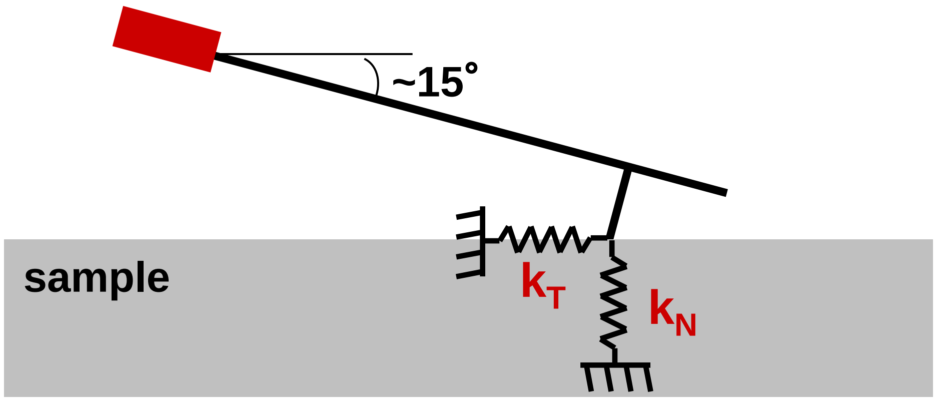
CR-AFM


Lock’in amplifier
PC acquisition card
Elasticity images
Resonance spectra
Photodetector (a-b)
laser
Sample
Feedback- loop
Topography
Sample holder
Piezo tube
air
silicone
polyuréthane
Log10 a-b (mV)
Fréquence (kHz)
3.5
3
2.5
2
1.5
1
0.5
0
-0.5
0
50
100
150
200

MODELLING OF THE CANTILEVER DYNAMICS

Objective: to find the link between resonance frequency and contact stiffness

Geometry of the problem



Normal and horizontal components of the tip displacement

CONTACT = A normal spring k_N + An horizontal spring k_T

2 situations

- $k_T = cte \neq 0$ → **« Glued contact »**
- $k_T = 0$ → **« Sliding contact »**

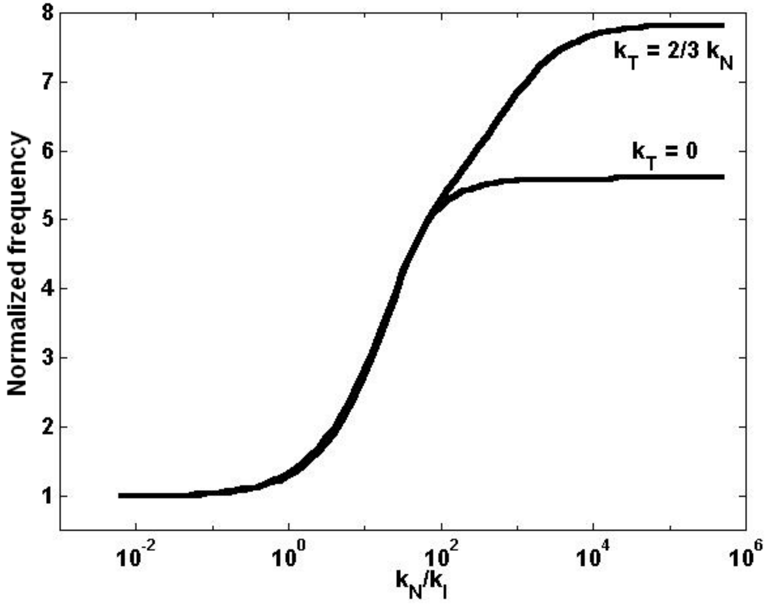
ANALYTICAL MODELS

✓ The most frequently applied model

✓ Resolution of the Euler-Bernouilli beam equation with adapted limit conditons

✓ Analytical model allows to understand the influence of several parameters: k_T, heigth and position of the tip, excitation mode (direct or indirect excitation)…

k_T modifies the curve $f_{rés} = f(k_N)$

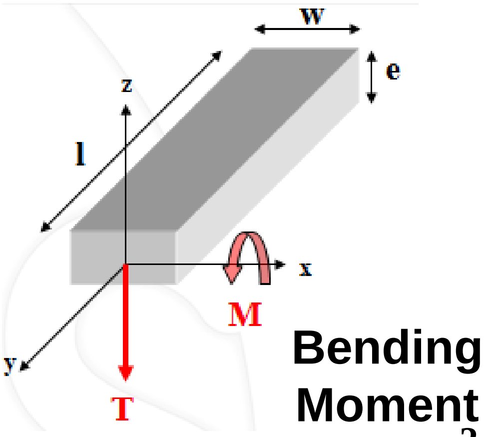


Problem: Analytical model do not allow to take into account the exact geometry Of the cantilever (V-shaped…) and the distributed electrostatic excitation.

MODELLING OF THE CANTILEVER DYNAMICS

$$-EI\frac{\partial^4 z}{\partial y^4}dy = (\rho S dy)\frac{\partial^2 z}{\partial t^2}$$

Clamped-free oscillations



Bending Moment

$$M = EI\frac{d^2 z}{dy^2}$$

Shear Force

$$T = -\frac{dM}{dy} = -EI\frac{d^3 z}{dy^3}$$

Harmonic solution $z(y,t) = z(y)\sin\omega t$

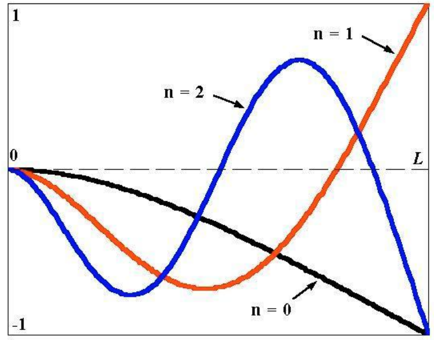
$$z(y) = Ae^{\beta y} + Be^{-\beta y} + Ce^{i\beta y} + De^{-i\beta y}$$

A, B, C, D determined from boundary conditions

$$z = 0 \quad and \quad \frac{\partial z}{\partial y} = 0 \quad for \quad y = 0$$

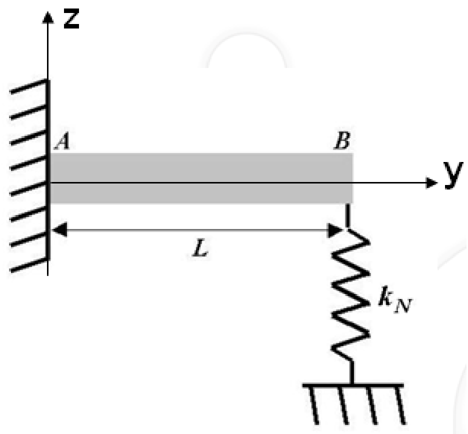
$$\frac{\partial^2 z}{\partial y^2} = 0 \quad and \quad \frac{\partial^3 z}{\partial y^3} = 0 \quad for \quad y = l$$

System of 4 equations
With 4 unknowns A, B, C, D



Beam deformation of modes n

MODELLING OF THE CANTILEVER DYNAMICS



Simple case

Yamanaka et al

Clamped beam coupled with a normal spring

✓**point A :** $z(0) = 0$ **(null deflection)**

$\partial z(0) / \partial y = 0$ **(null slope at the clamped end)**

$$z(y) = A_1 \sin c_n y + A_2 \cos c_n y + A_3 sh c_n y + A_4 ch c_n y$$

✓**point B :** $\partial^2 z(L) / \partial y^2 = 0$ **(null moment)**

Condition for a non trivial solution :

(*) $$1 + \cos c_n L ch c_n L = \frac{3k_N}{k_l c_n^3}\left(\cos c_n L sh c_n L - \sin c_n L ch c_n L\right)$$

$EI \, \partial^3 z(L) / \partial y^3 = k_N z(L)$

(shear force in equilibrium with spring reaction)

with $k_l = 3EI / L^3$

$$c_n = \left(48\pi^2 \rho / Ee^2\right)^{1/4} f_n^{1/2}$$

f_n is obtained experimentally, k_n is deduced from (*)

MODELLING OF THE CANTILEVER DYNAMICS

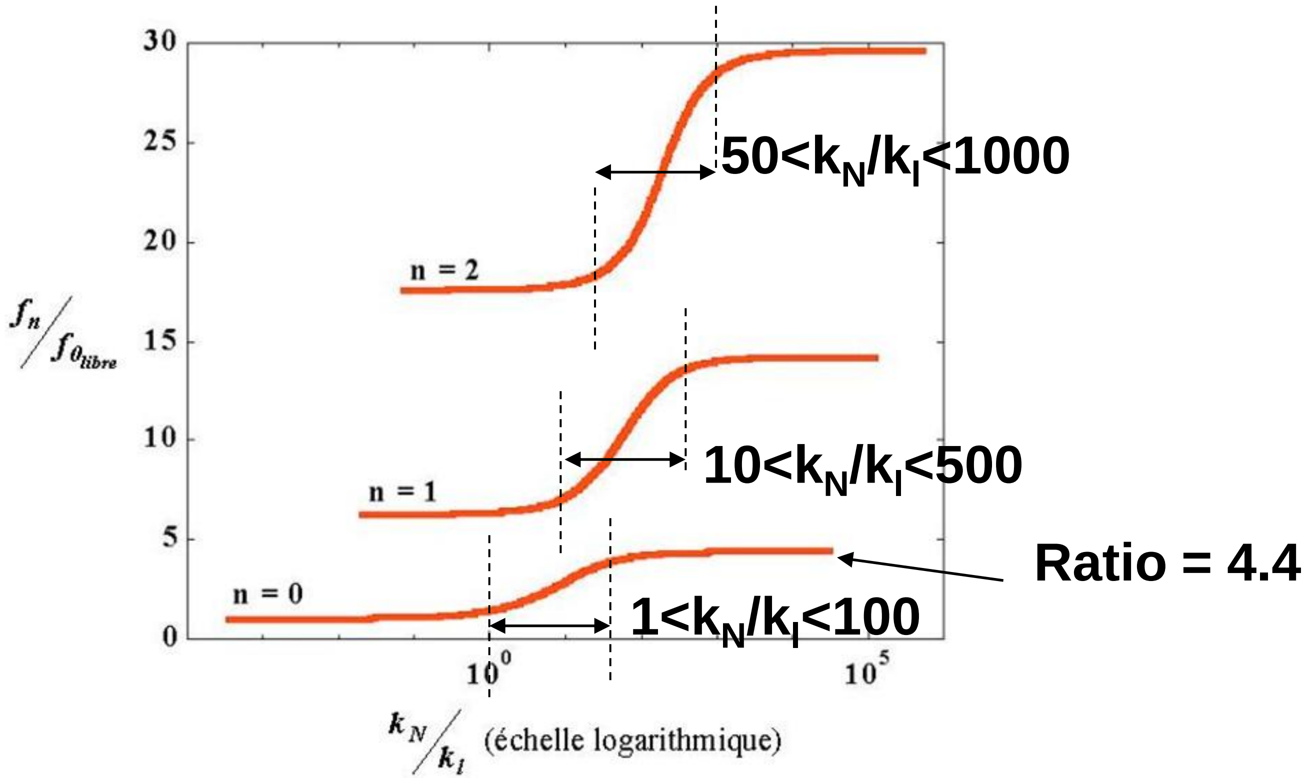
Example:

Probe : $k_L \approx 3$ N/m, $R \approx 50$ nm
$F_0 \approx 200$ nN

Soft sample (Low T_g polymer)
$k_N \approx 10$ N/m
OK using n=0

Hard sample (Silicon)
$k_N \approx 1000$ N/m
Not OK using n=0
OK using n>0

Sensitivity of the cantilever with respect to the sample rigidity

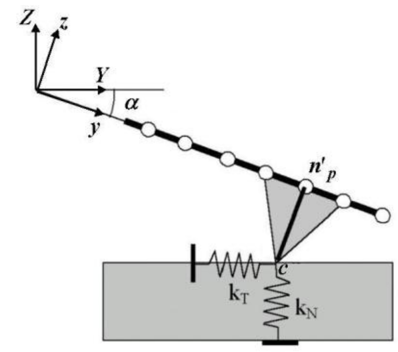


fréquence de résonance des trois premiers modes, normalisée à la fréquence de résonance libre du mode fondamental, en fonction du rapport raideur du ressort sur raideur

FINITE ELEMENT MODELLING (FEM)

1D Model

R. Arinero, G. Lévêque
Review of Scientific Instruments, **74** (1), 104-111, 2003



✓ cantilever = N elements of 1 µm length

✓ for each element : 4 degrees of liberty (slope θ and transversal displacement z at extremities)

• Kinetic energy → <u>Mass matrix</u> $[M]$

• Elastic potential energy → <u>Rigidity matrix</u> $[K]$

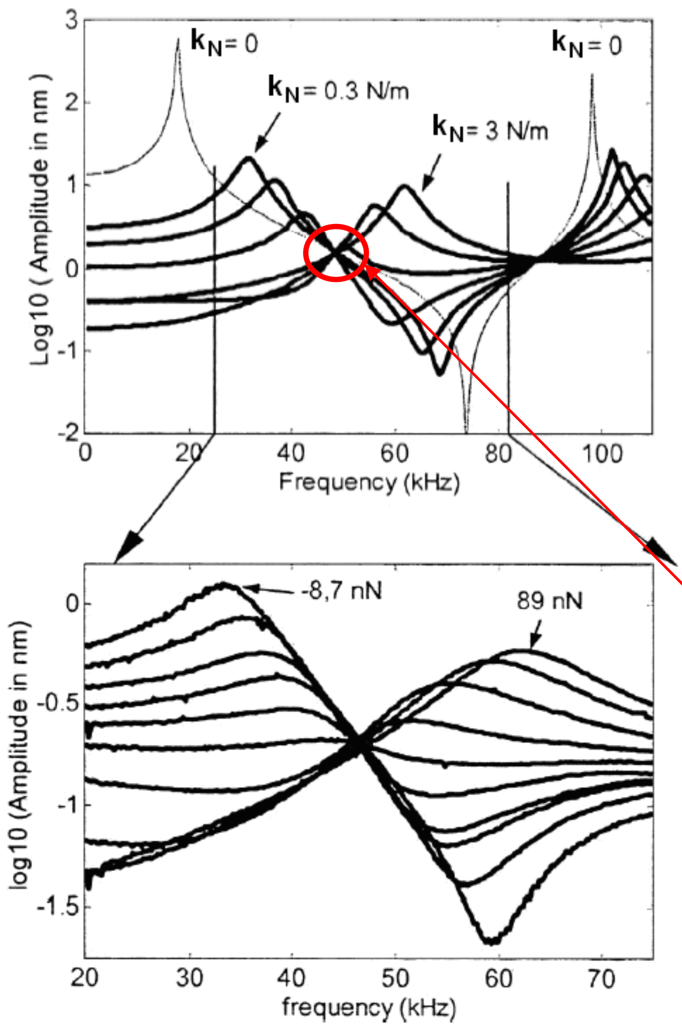
✓Tip (kinetic nrj) and contact (potential nrj) = Additional elements in the matrix

Natural frequencies

Forced vibrations

$$([K]-\omega^2[M])\{q\}=\{F\}$$

FINITE ELEMENT MODELLING (FEM)



Experimental verification of FEM model

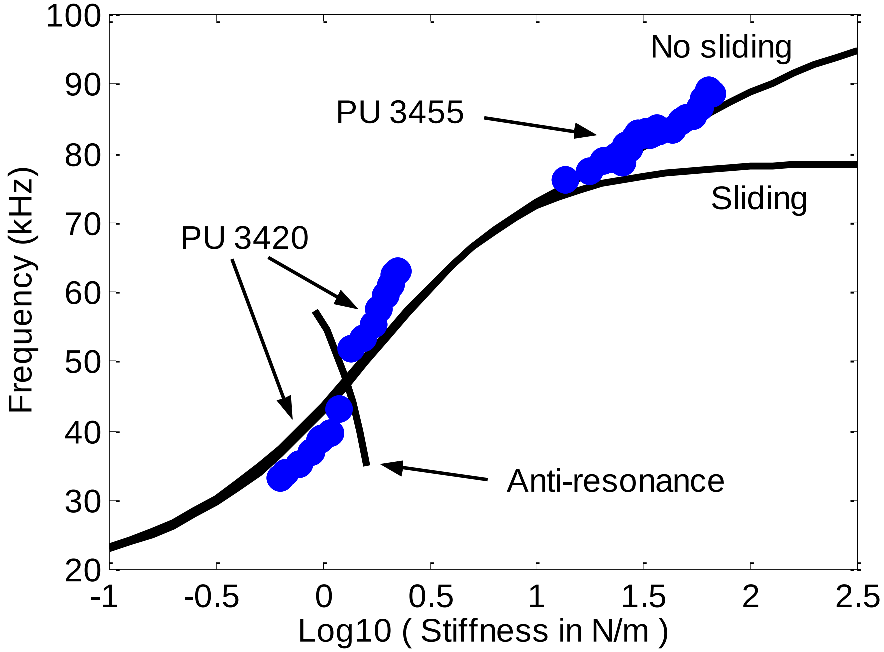
Top: *apparent amplitude versus excitation frequency calculated for a different contact stiffness. The tangential stiffness is taken as* $k_T = 2/3\,k_N$. *All curves include a damping effect, obtained by multiplying the stiffness real values by the arbitrary complex number: 1+i/5, in order to be more similar to the experimental curves below.* ***Bottom:*** *apparent amplitude measured on a polyurethane sample (PU 3420) for an applied force increasing from -8.7 nN (closed to the pull-off force) to 89 nN.*

Increasing F_{res} with $F_{Applied}$ (or k_N)

Node of detection
(disparition of the resonance peak for specific values of k_N !)

Arinero, Levêque,
Rev. of Sc. Inst., 74, 2003

FINITE ELEMENT MODELLING (FEM)



Arinero, Levêque,
Rev. of Sc. Inst., 74, 2003

Experimental verification of FEM model

Resonance frequency of the AFM cantilever, in two cases :

The sliding contact case ($k_T = 0$) and the no-sliding contact ($k_T = 2/3\ k_N$).

Dots : experimental points corresponding to two polyurethane samples (PU3455 and PU3420).

CONTACT MECHANICS

Objectives : Find the relationship between k_N and E

Existing models:
- sphere-plane models for homogeneous et isotropric samples
- Indentation depth << R

✓ **Hertz (1885):**
- The oldest
- Without adhesion

$$k_{N_{Hertz}} = \left(6 E^{*2} R F\right)^{1/3}$$

E^*: Reduced elastic modulus $$\frac{1}{E^*} = \frac{1-\nu_{Tip}^2}{E_{Tip}} + \frac{1-\nu_{Sample}^2}{E_{Sample}}$$

→ Not frequently used in AFM,
Adapted for high applied charges

CONTACT MECHANICS

✓ **DMT Model :**
- **Adhesive model**
- **« Offset » of Hertz model**

$$k_{N_{DMT}} = \left(6E^{*2}R\left(F + F_{ad_{DMT}}\right)\right)^{1/3}$$

Adhesion force $F_{ad_{DMT}} = 2\pi R w_{ad}$

Adapted to hard samples, low adhesion force and low tip radius R

✓ **JKR Method :**
- **Adhesive model**
- **Evaluation of the energy of adhesion between two solids**

$$k_{N_{JKR}} = 2E^{*}a_{JKR}\frac{1-\frac{1}{2}\left(\frac{a_{0_{JKR}}}{a_{JKR}}\right)^{3/2}}{1-\frac{1}{6}\left(\frac{a_{0_{JKR}}}{a_{JKR}}\right)^{3/2}}$$

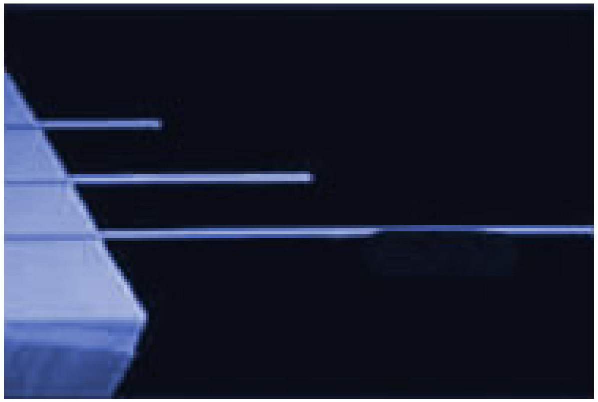
avec

$$\begin{cases} a_{JKR} = \left[\frac{3R}{4E^{*}}\left(\sqrt{F_{ad_{JKR}}} + \sqrt{F_{ad_{JKR}} + F}\right)^{2}\right]^{1/3} \\ a_{0_{JKR}} = \left[\frac{3RF_{ad_{JKR}}}{E^{*}}\right]^{1/3} \\ F_{ad_{JKR}} = \frac{3}{2}\pi R w_{ad} \end{cases}$$

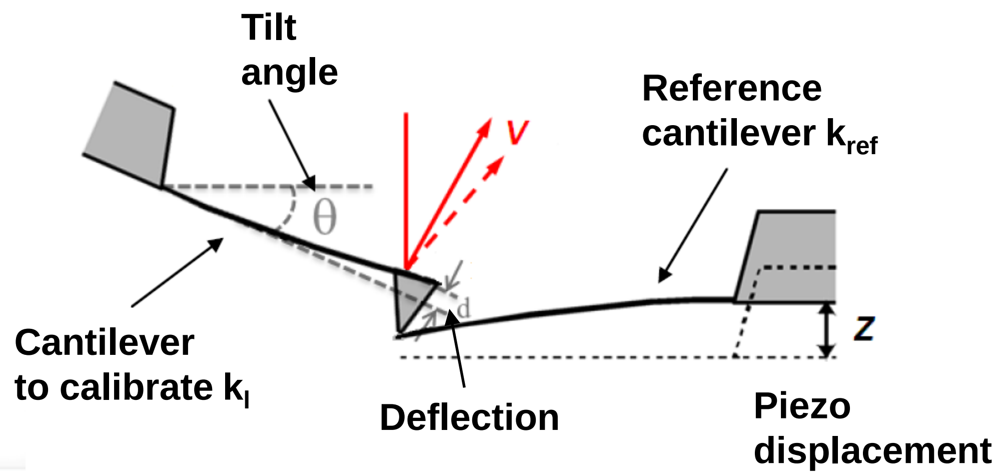
Adapted to soft samples, high adhesion force and high tip radius R

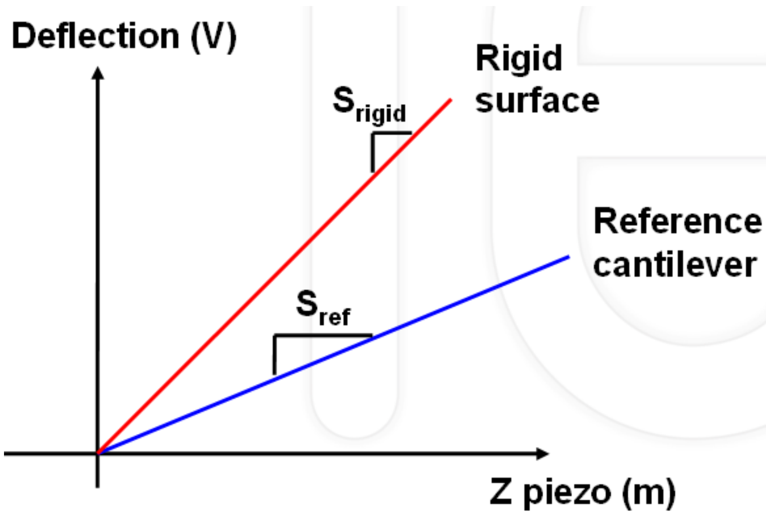
CALIBRATION OF THE CANTILEVER'S FORCE CONSTANT

Reference cantilevers



Butt *et al.*, 2005; Gibson *et al.*,1996





$$k_l = k_{ref}\left(\frac{S_{rigid}}{S_{ref}} - 1\right)\cos^2\theta$$

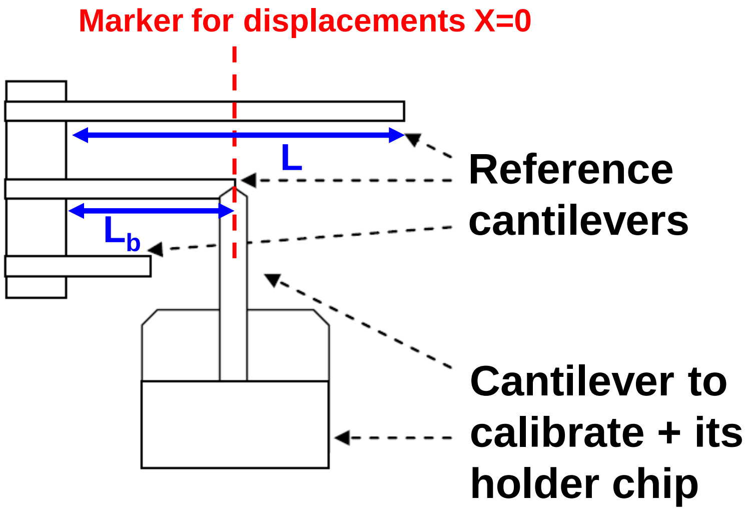
At the extremity of the cantilever

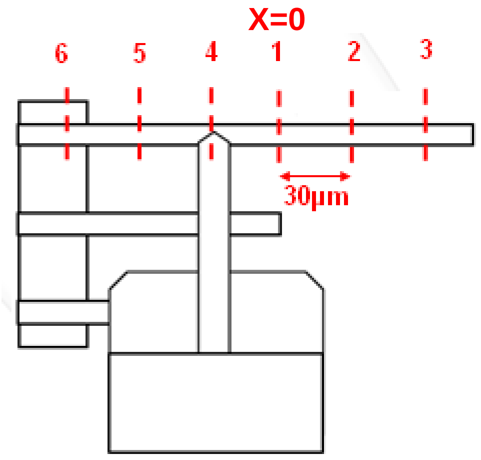
Tilt angle correction

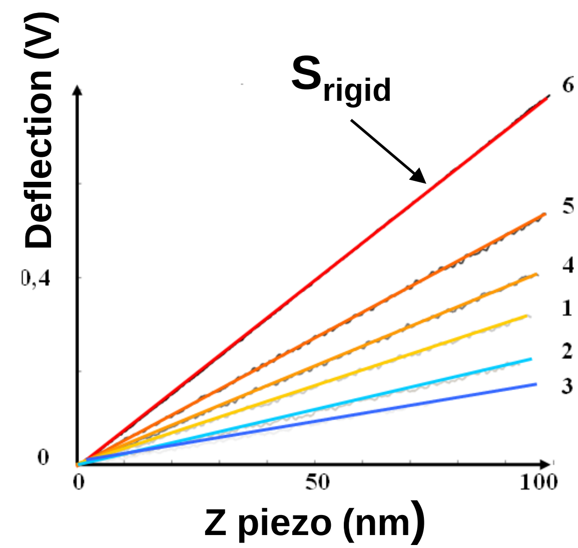
CALIBRATION OF THE CANTILEVER'S FORCE CONSTANT

PROTOCOL

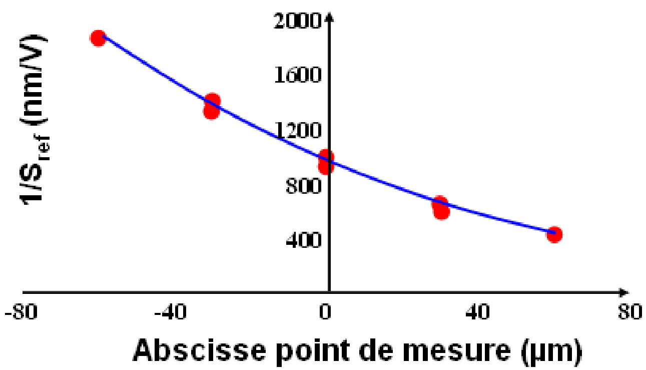
K. Bytebier PhD thesis (2009)







$$\frac{S_{ref}}{S_{rigid}} = \left[1 + \frac{k_l}{k_{ref} \left(\frac{L}{L - \Delta L} \right)^3 \cos^2 \theta} \right]^{-1}$$



$$\Delta L = L - L_b - \Delta X$$

Calibrated k_l=3.4±0.5 N/m

CALIBRATION OF THE CANTILEVER'S FORCE CONSTANT

Thermal noise

Hutter 1993
Butt 1995

Acquisition of the thermal noise during a finite time interval + Fourier transform

Mean square amplitude of thermally driven cantilever can be used to calibrate k_l

Boltzmann constant
$k_B = 1.38.10^{-23}$ J/K

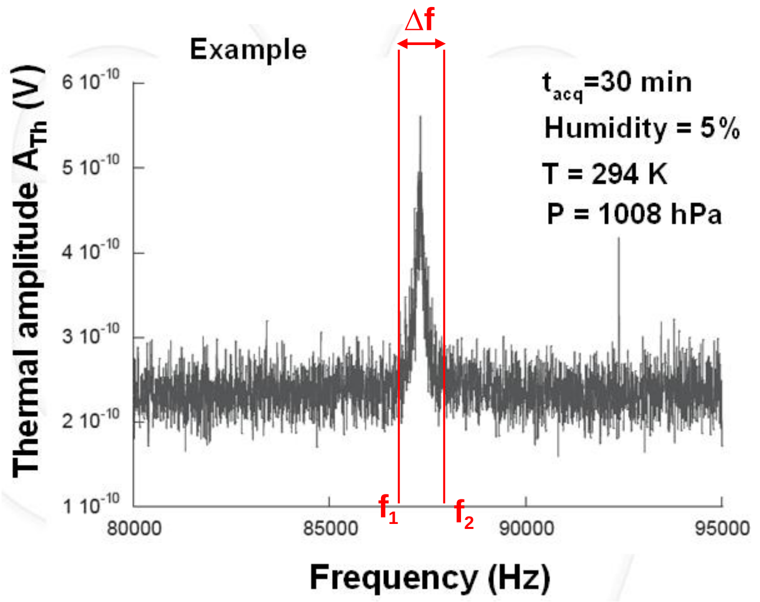
$$k_l = \frac{12 k_B T}{\alpha_i^4 \left\langle A_{Th}^2 \right\rangle}$$

Constant relative to mode *i*
α_1=1.875 (first mode)

$$\left\langle A_{Th}^2 \right\rangle = \frac{1}{\Delta f} \int_{f_1}^{f_2} A_{Th}^2(f)\,df$$

$$\left\langle A_{Th}^2 \right\rangle \cong 3.16 \times 10^{-14} V_{rms}^2$$

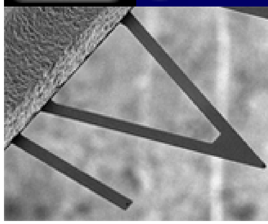
To convert in m^2 using



CALIBRATION OF THE CANTILEVER'S FORCE CONSTANT

http://www.ampc.ms.unimelb.edu.au/afm/calibration.html

Search ? Department of Mathematics & Statistics



Atomic Force Microscope Cantilevers (Calibration method of Sader)

THE UNIVERSITY OF MELBOURNE

Back to Introduction

NEW!

Web App | Download on the App Store

Sader Method - iPhone and Web Apps

Bibliography on AFM Cantilevers and Force Measurements

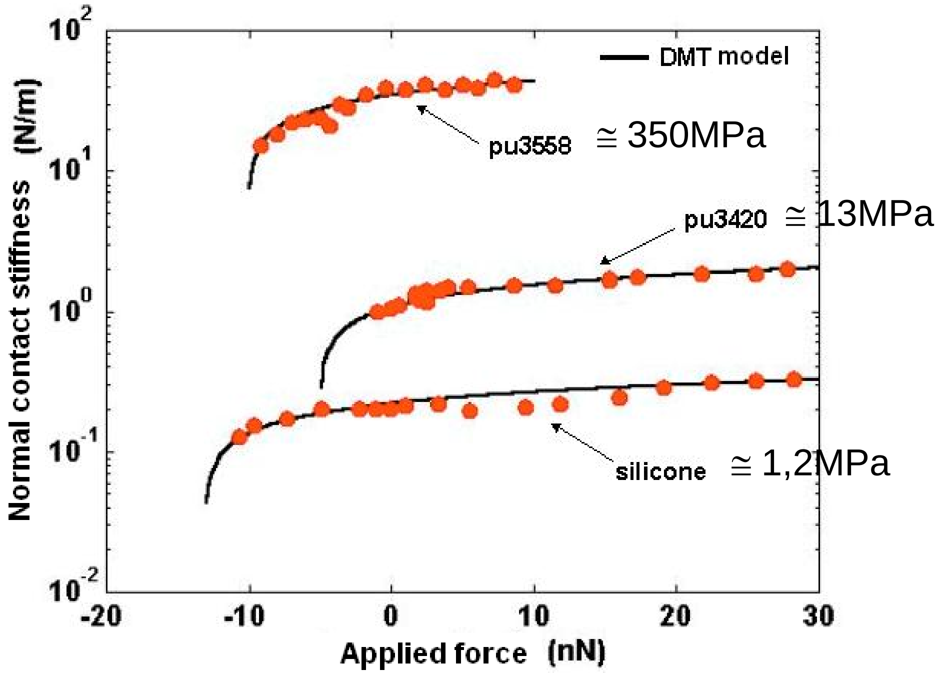
Online Calibration

- Normal spring constant
- Torsional spring constant

To use these Java applets to perform an online calibration of the normal and torsional spring constants of rectangular AFM cantilevers, just enter the length and width (in microns), the appropriate fundamental resonant frequency (in kHz) and the corresponding quality factor, and press the calculate button.

COMPLETE PROTOCOL EXAMPLE

Polymer samples



Experimental part : **Measurement of f_{res} versus $F_{applied}$**

Theoretical part : **1. Calculation of k_N using FEM**

2. k_N versus $F_{applied}$ curves are fitted by a wall adapted contact mechanics model

CALIBRATION OF CONTACT RESONANCE FREQUENCY

MOTIVATIONS : **Problems related to previous method**

- **The data involved in the different models (R, k_l…) are not reliable enough**

  (The apex of the tip is not exactly a sphere)

- **In the case of very hard materials δ is indetectable**
- **Contact models are isotropic**

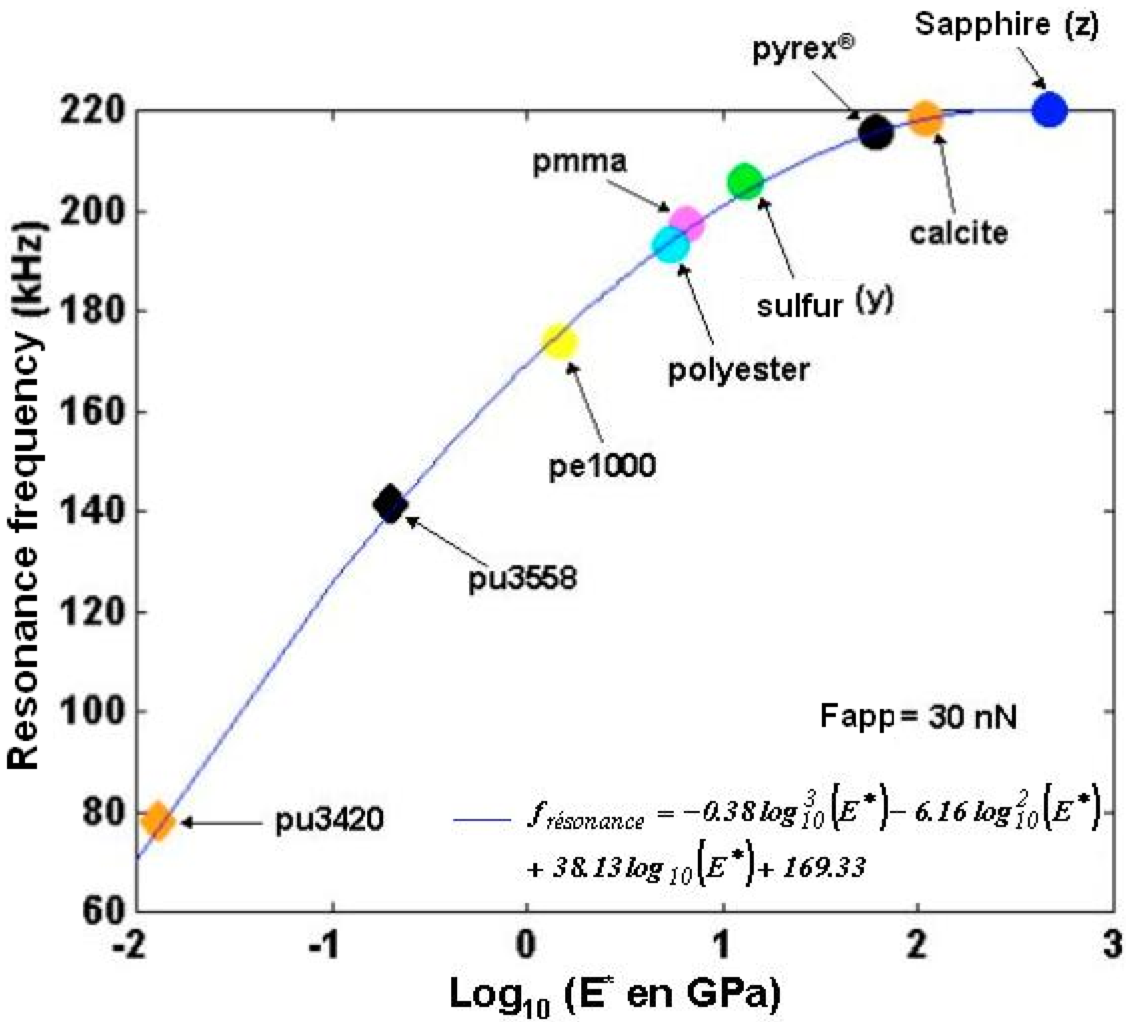
IDEA : **We want to directly link resonance frequency and elastic modulus Based on the master curve obtained with reference samples**

REFERENCE SAMPLES:

1. **Must cover a broad range of modulus (polymers, metals, cristals, ceramics)**
2. **Must be homogeneous**
3. **Must be stable in the time**

CALIBRATION OF CONTACT RESONANCE FREQUENCY

MASTER CURVE FOR A PARTICULAR CANTILEVER

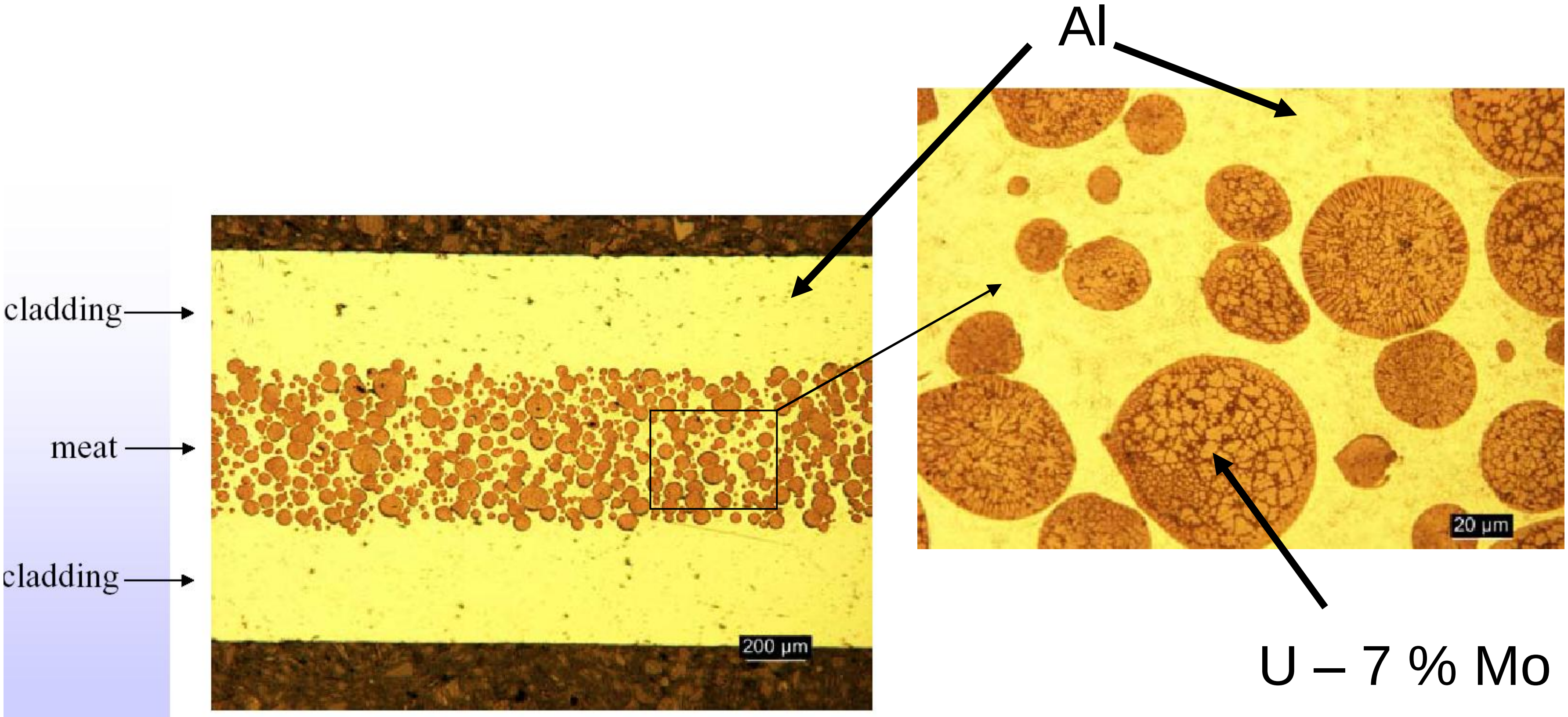


EXAMPLE CASE STUDY

Mechanical properties of heterogeneous nuclear fuels at the submicrometer scale

Laux, Arinero, CFM (2005)

UMo Nuclear Fuel



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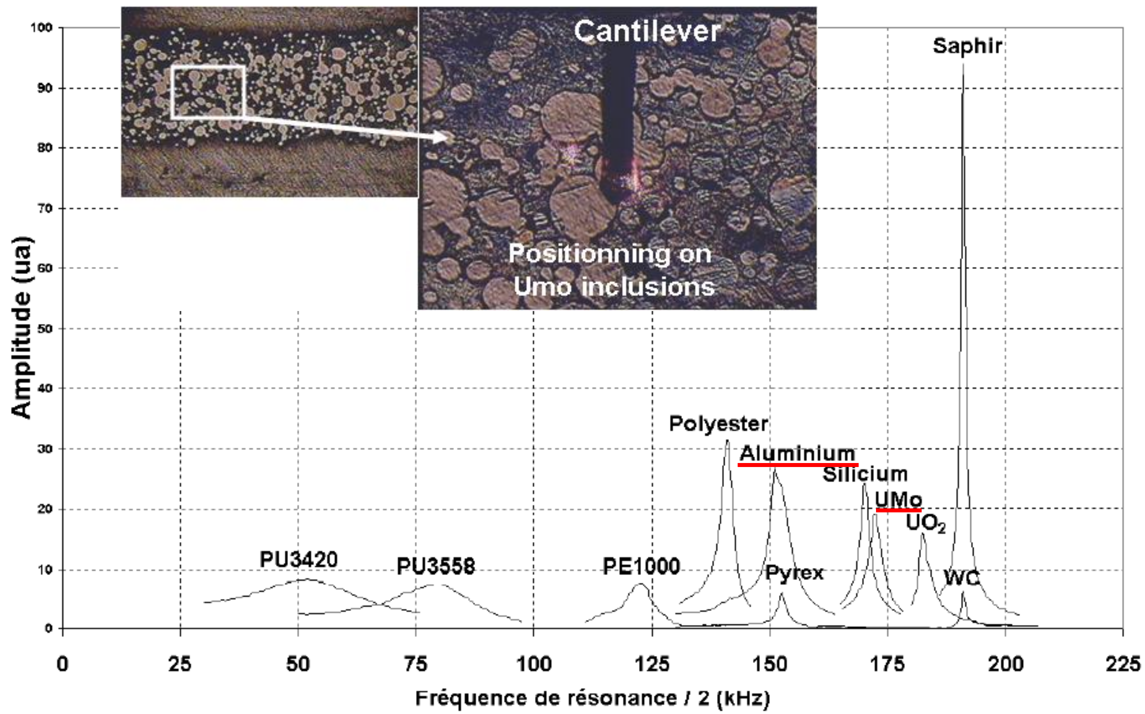

EXAMPLE CASE STUDY

Mechanical properties of heterogeneous nuclear fuels at the submicrometer scale

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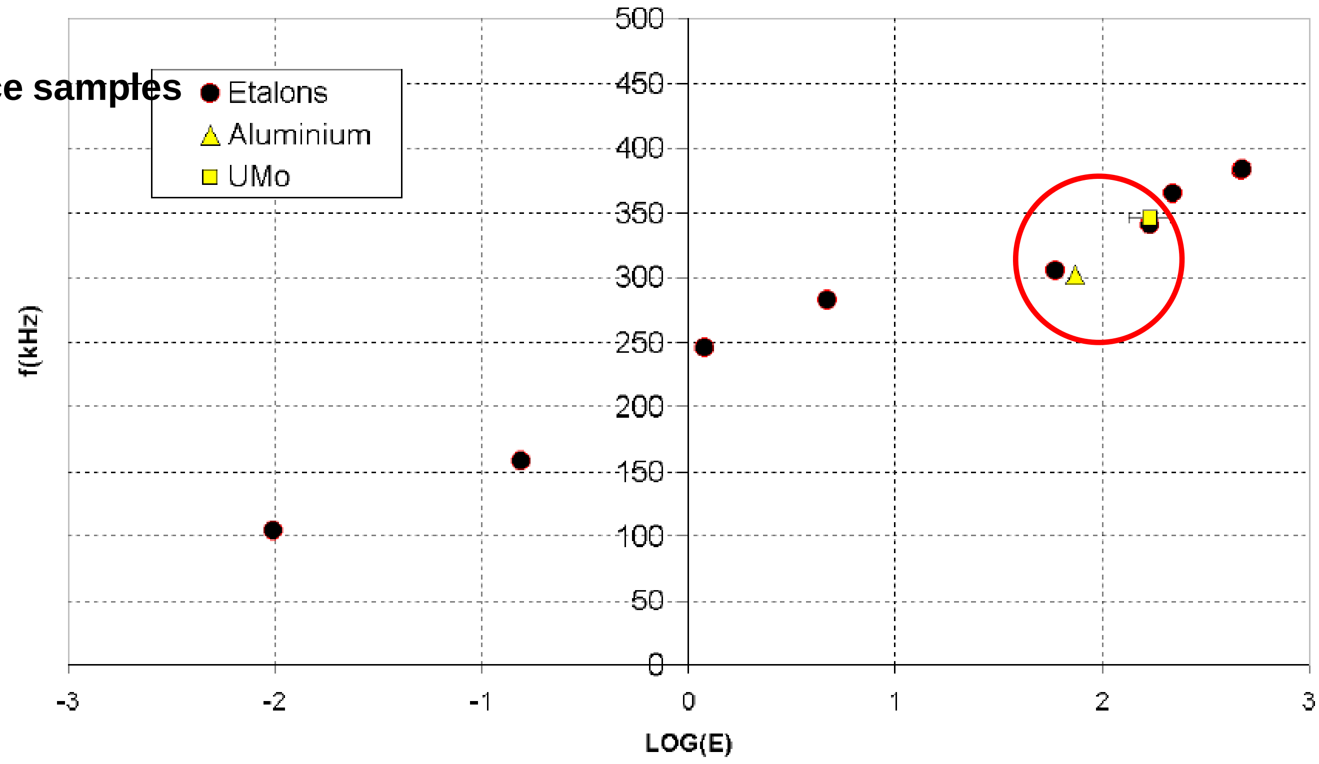
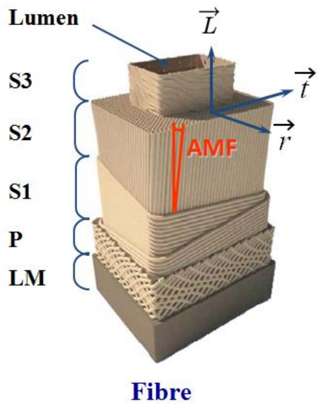
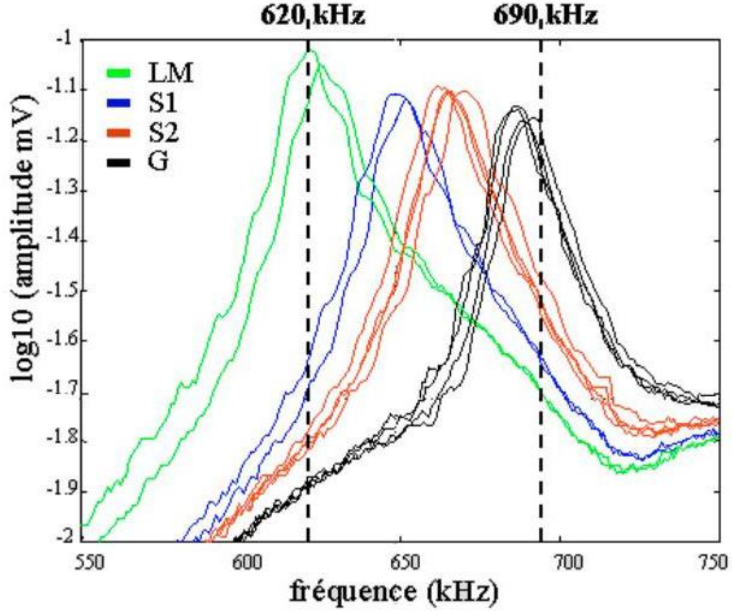
Reference samples

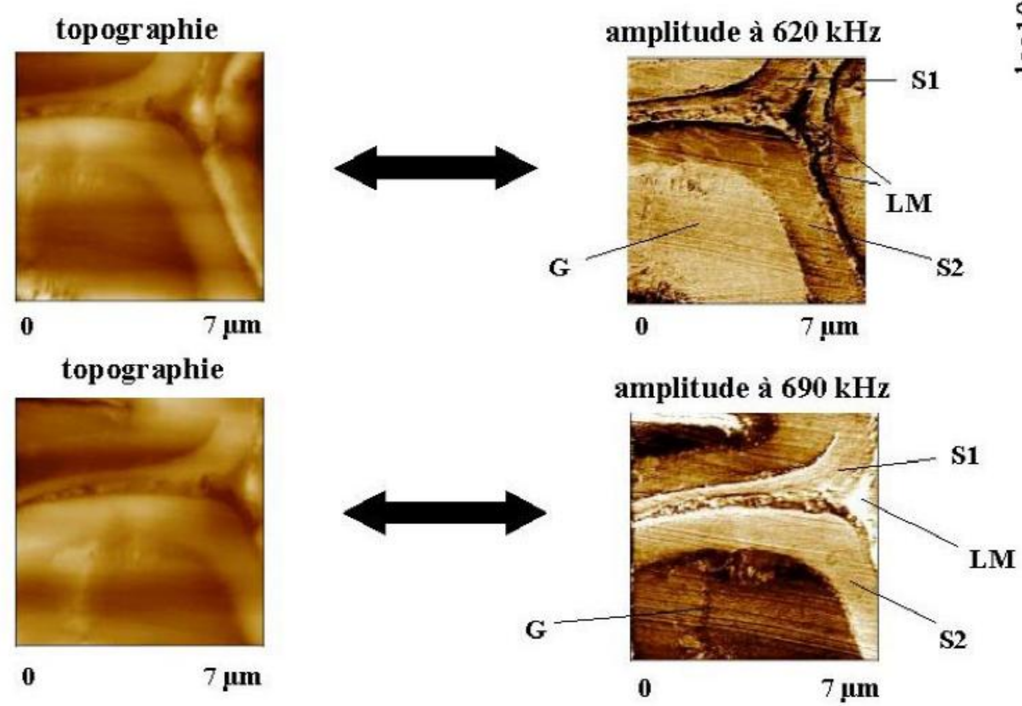


ELASTIC CONTRAST AT A FIXED IMAGING FREQUENCY



Wood cell-wall Mechanical Properties (Arinero, Arnould, Clair)





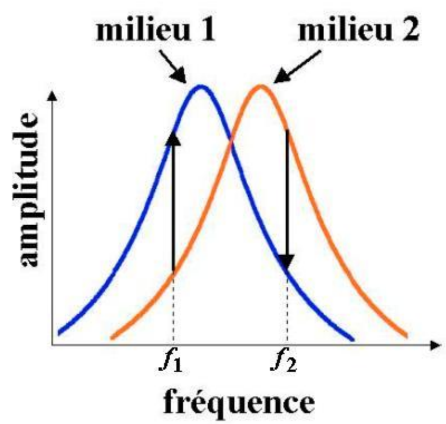


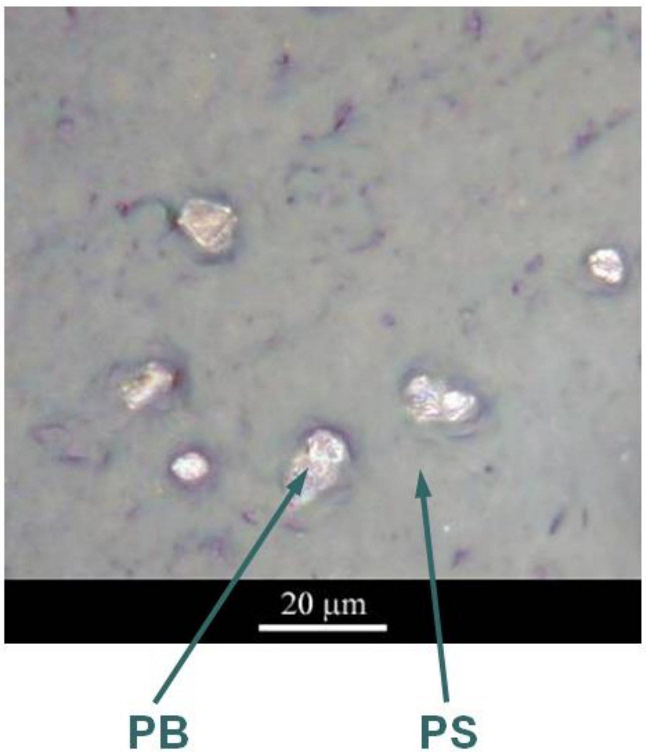
figure VI.13 : *Couches de la paroi cellulaire du bois de chêne vert. A gauche, images topographiques, les niveaux de couleur s'étendent de 0 à 300 nm. A droite, images « élastiques » à 620 kHz et 690 kHz. A 620 kHz, les zones foncées correspondent aux couches les plus souples. A 690 kHz, les zones foncées correspondent aux couches les plus rigides.*

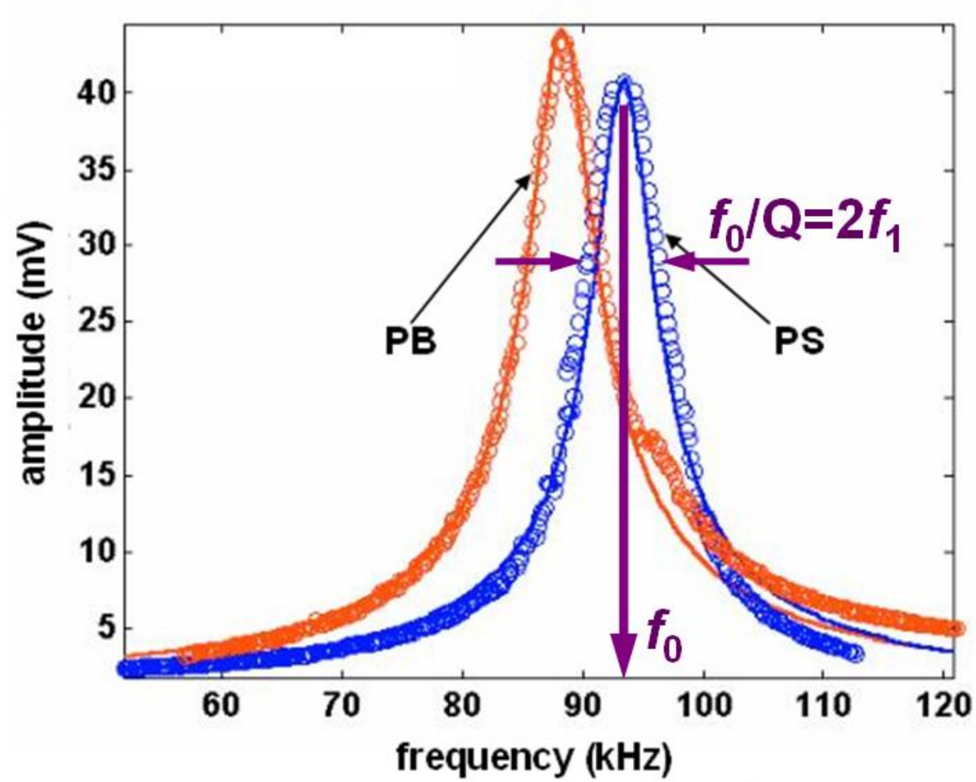
IMAGE PROCESSING FOR RESONANCE FREQUENCY MAPPING

Maps obtained on HIPS (high-impact polystyrene)

Arinero *et al.*, Rev. of Sc. Inst., 78, 2007

[University of Cambridge]





$f_0 \rightarrow k_N$ $Q \sim \dfrac{1}{\tan\delta}$

IMAGE PROCESSING FOR RESONANCE FREQUENCY MAPPING

Imaging frequency conditions

f_{im} close to f_0

$$C^* = Ce^{j\psi}$$

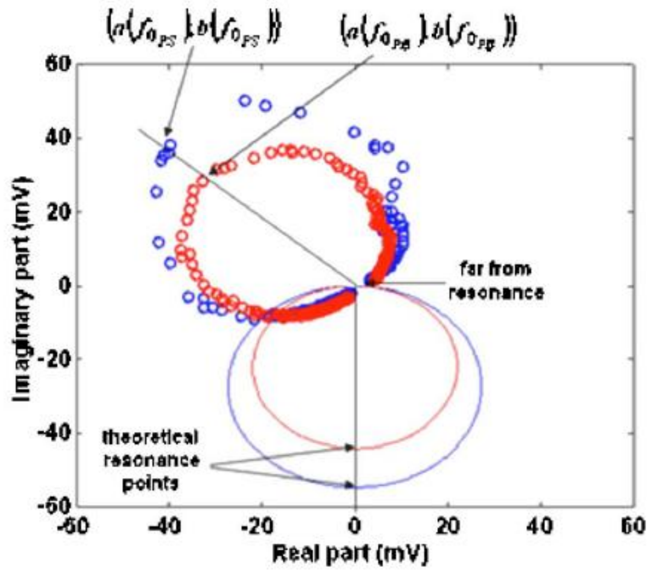
To determine

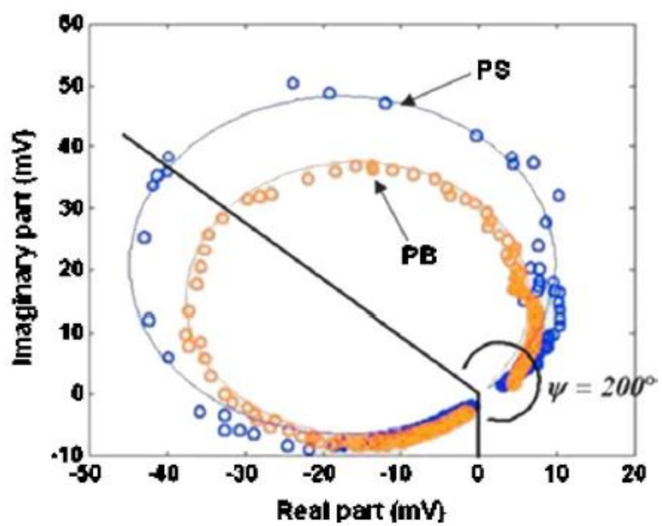
$$a + jb = X_0 e^{j\varphi}$$

$$a + jb = \frac{C^*}{f_0 - f + jf_1}$$

$$f_0 = f_{im} + \frac{C(a\cos\psi + b\sin\psi)}{a^2 + b^2}$$

$$f_1 = \frac{C(a\sin\psi - b\cos\psi)}{a^2 + b^2}$$





Lorentzian curve

$$X_0(f) = \left| \frac{C}{f_0 - f + jf_1} \right|$$

$$C \approx 9.5 \times 10^4$$

Ψ=200°

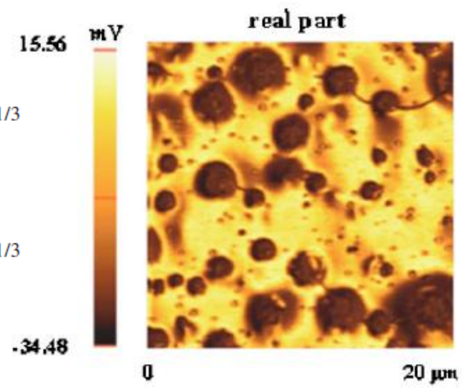
Arinero *et al.*, Rev. of Sc. Inst., 78, 2007

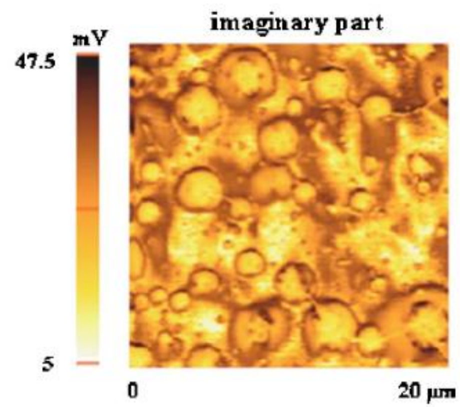
IMAGE PROCESSING FOR RESONANCE FREQUENCY MAPPING

Arinero *et al.*,
Rev. of Sc. Inst., 78, 2007

$$k'_{N_{DMT}} = E'^{*}\left[\frac{6R(F_0+F_{ad})}{E_0^{*}}\right]^{1/3}$$

$$k''_{N_{DMT}} = E''^{*}\left[\frac{6R(F_0+F_{ad})}{E_0^{*}}\right]^{1/3}$$





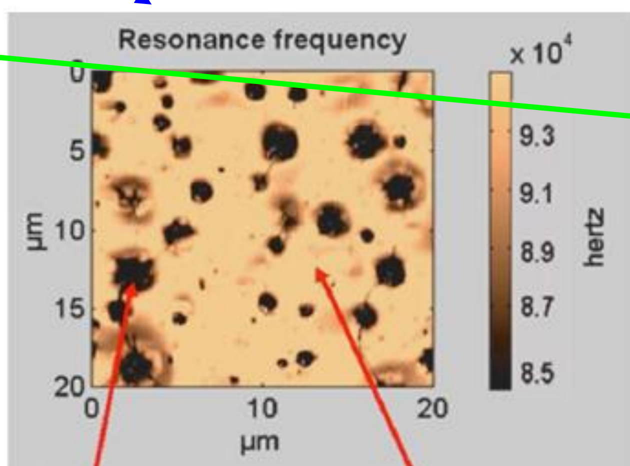
	f_0 (kHz)	f_1 (kHz)	$k'_{N_{DMT}}$ (N/m)	$k''_{N_{DMT}}$ (N/m)	E'^{*} (GPa)	E''^{*} (GPa)
PS	94±3	1.75±0.2	2.74[a]	0.1[a]	0.29[a]	0.025[a]
PB	86±3	2.75±0.2	2.22[a]	0.15[a]	0.01[a]	0.0016[a]

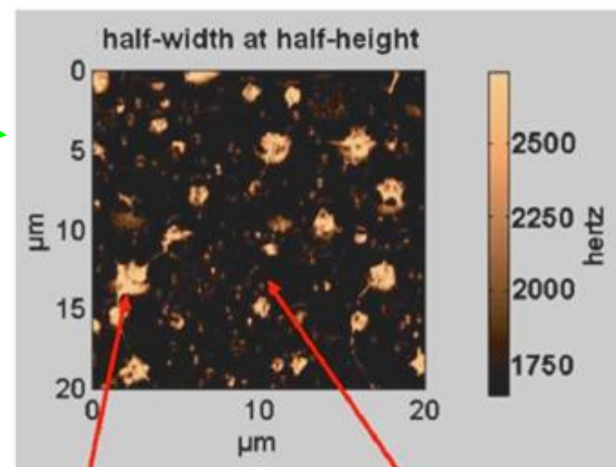
[a] $\Delta k_N / k_N \approx 0.5$ and $\Delta E^{*}/E^{*} \approx 1$.

$$f_0 = \frac{1}{2\pi}\sqrt{\frac{k'_{N_{DMT}} + k_l}{m_{eff}}}$$

$$f_1 = \frac{k''_{N_{DMT}}}{8\pi^2 m_{eff} f_0}$$

Image processing





~ E'

Storage modulus

PB $\mathbf{f_0}$ PS

~ E''

Loss modulus

PB $\mathbf{f_1}$ PS

RESONANCE TRACKING WITH PLL

Pll (phase locked loop)

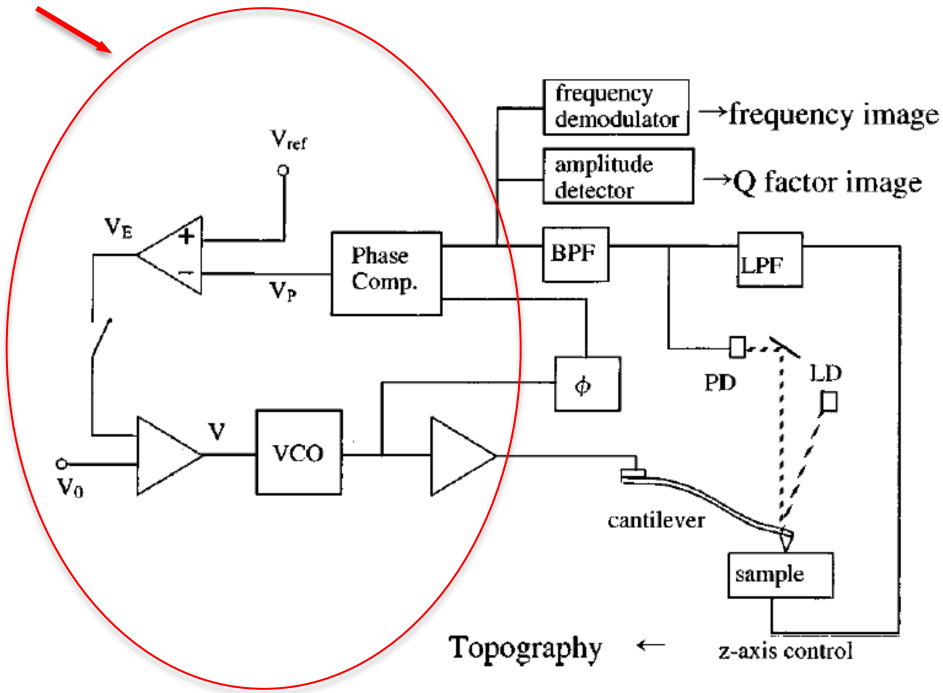
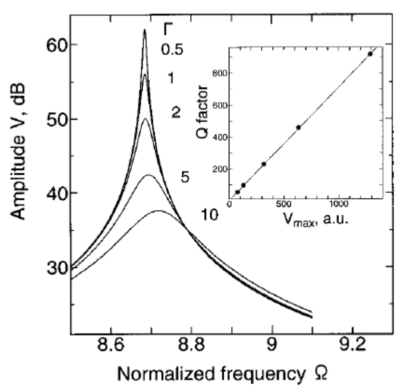


FIG. 2. Block diagram of the UAFM for resonance frequency and Q factor mapping.



$$\Omega = \omega / \sqrt{k/M}$$

$$\Gamma = \gamma / \sqrt{Mk}$$

$Q \sim V_{max}$

s/k=200 and Γ=0.5, 1, 2, 5, 10

Yamanaka et al.

Appl. Phys. Lett., Vol. 78, No. 13, 26 March 2001

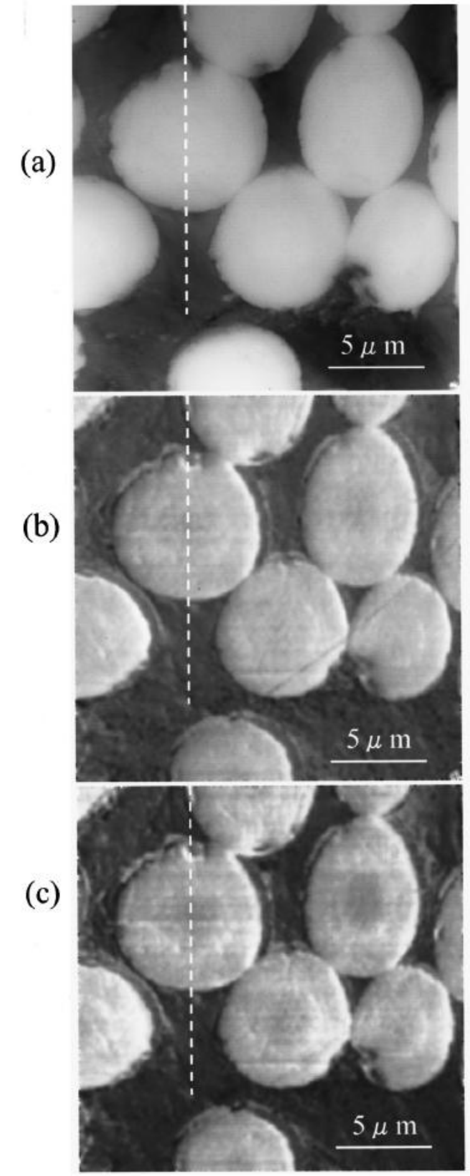
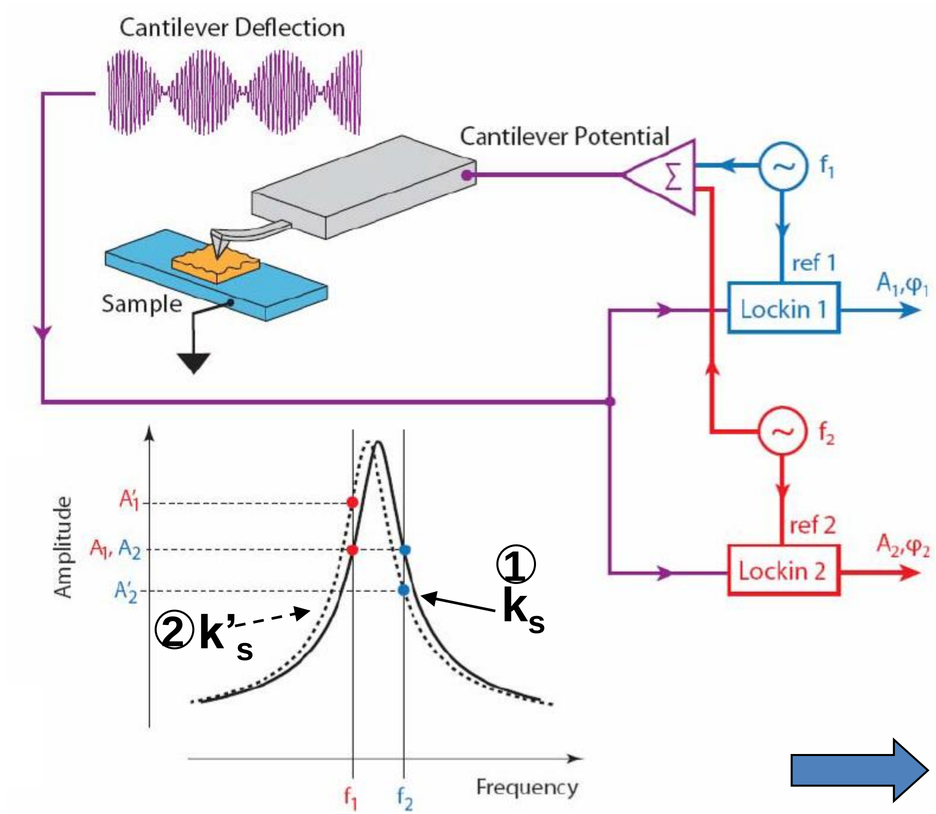


FIG. 4. Images of CFRP. (a) Topography (maximum height difference of 500 nm). (b) Resonance frequency image with a gray scale from 170 to 180 kHz. (c) Q factor image with a gray scale from 70 to 250.

DRFT OR DART

Dual ac resonance tracking (DART)

B J Rodriguez *et al* 2007 *Nanotechnology* 18



Use of 2 lock-in amplifiers

Oscillation = Sum of two frequencies f_1 and f_2 near to resonance

① Stiffness k_s (before starting feedback)

$A_1=A_2=A_R/2$

$$f_c = \frac{f_1 + f_2}{2}$$

② Stiffness k'_s

$A'_1 - A'_2 > 0$: $k'_s < k_s$

$A'_1 - A'_2 < 0$: $k'_s > k_s$

Resonance tracking:

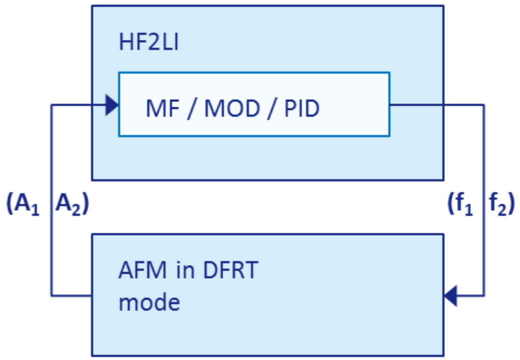
$\Delta f = f_1 - f_2 = \text{constant}$

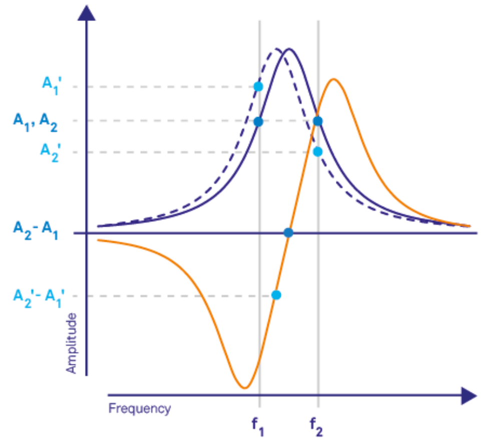
Feedback loop to maintain $A'_1 = A'_2$

$$f_c' = \frac{f_1' + f_2'}{2}$$

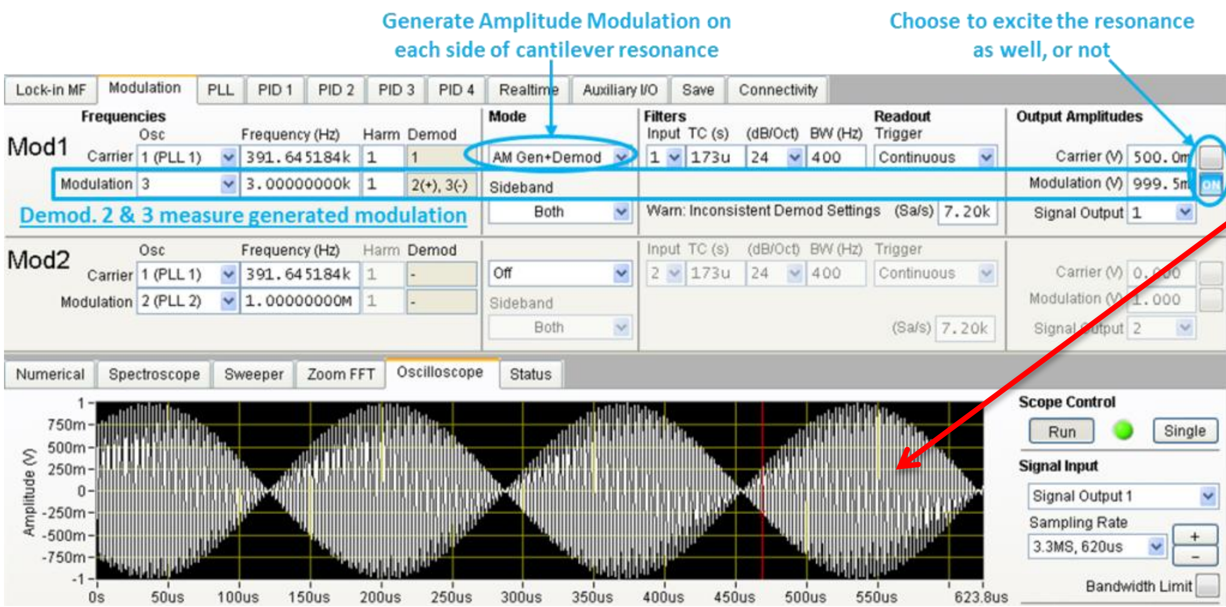
Q calculated by damped Spring harmonic oscillator model

DFRT BY ZURICH INSTRUMENTS

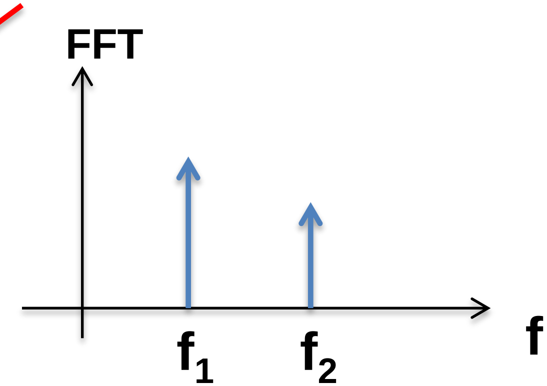




Typically,
Δf≥ 2 × imaging (feedback) bandwith



Double-sideband suppressed-carrier modulation



$$f_{1,2} = f_0 \pm f_{mod}$$

SUMMARY OF IMAGING METHODS FOR THE CR -AFM

Methods	What it does	Benefits	Disadvantages
Fixed frequency[2]	The cantilever response is measured at a fixed frequency, which varies as the contact resonance frequency shifts.	Simple to implement and produces elastic contrast images.	Produces only qualitative results since the frequency shift itself is not measured. Contrast is lost if the peak shifts too far from the selected frequency.
PLL frequency tracking[1]	A phase-locked loop (PLL) uses the phase of the cantilever response to track the contact resonance frequency.	The actual contact resonance frequency is tracked.	Difficult to tune the PLL to achieve stable frequency tracking due to spurious phase shifts in the response. Does not measure the Q of the resonance.
Frequency sweep (chirp)[3,4,5]	A frequency sweep (chirp) is done at each point. The cantilever response is Fourier analyzed to recover the full frequency response.	Measures the entire frequency response, so both the frequency and Q are obtained. Additional analysis is possible based on more complex models.	Mapping is quite slow when collecting large numbers of pixels. Each sweep must be done slowly enough for the cantilever to respond (rate limited by Q).
DART[6,7,8]	The amplitude and phase response at two frequencies (bracketing the contact resonance) is measured, which enables the contact resonance to be tracked.	Provides both the contact resonance frequency and Q. The tracking is extremely fast, so DART imaging can be done at normal imaging rates.	The full response is not measured, so analysis is more limited than frequency sweep or band excitation methods.
Band Excitation[8,9]	A continuous band of frequencies is excited. The cantilever response is Fourier analyzed to recover the full frequency response.	The entire frequency response is measured. By exciting the entire band at once, it is much faster than other full spectrum techniques (e.g. sweep).	Data transfer bandwidth limitations make the current implementation significantly slower than DART. Future speed improvements are possible.

From Asylum (CR-AFM application note)

DRFT OR DART: SOME RECENT ADVANCES

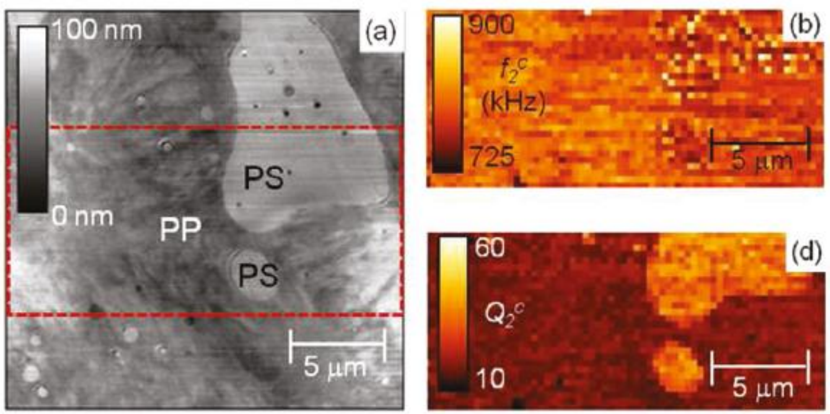
Polystyrene polypropylene blend

Killgore et al, langmuir, 27, 13983, 2011

PS regions: f_2=792.1 ±31.7 kHz, Q_2=37.3 ±5

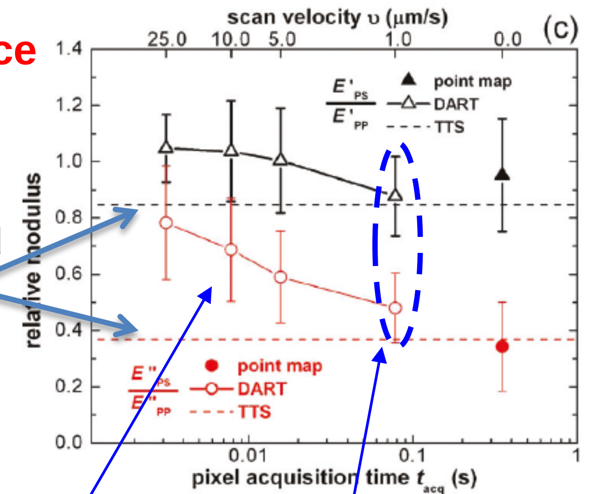
PP regions: f_2=801.7 ±17.4 kHz, Q_2=18.4 ±2.7

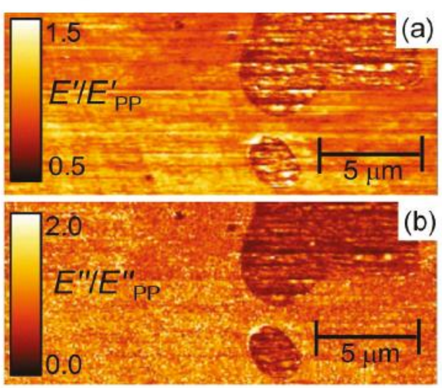
Scan velocity effects



Dual ac resonance tracking images

Expected at 1MHz (DMA low freq and low T + Time Temp superposition)





Cantilever modelling +contact mechanics model

Higher scan speeds NO ! (contact mechanics or instruments effects)

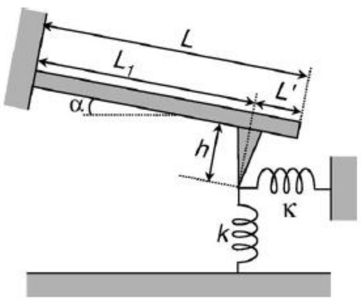
V=1 µm/S OK !

Image duration ~1h
15 µm*15 µm
256*256 pixels

DRFT OR DART: SOME RECENT ADVANCES

Measurement of Poisson's ratio with Contact-resonance atomic force microscopy

Hurley and Turner, JAP, 102 (2007)



k and K provided by Flexural and torsional Resonance respectively

Young's modulus

$$k = 2aE^*$$

$$M = E/(1-\nu^2)$$

$$E_s^* = E_{\text{ref}}^* \left(\frac{k_s}{k_{\text{ref}}}\right)^m$$

$$\frac{1}{E_s^*} = \frac{1}{M_{\text{tip}}} + \frac{1}{M_s}$$

Shear modulus

$$\kappa = 8G^*a$$

$$N = G/(2-\nu)$$

$$G_s^* = G_{\text{ref}}^* \left(\frac{\kappa_s}{\kappa_{\text{ref}}}\right)\left(\frac{k_s}{k_{\text{ref}}}\right)^{m-1}$$

$$\frac{1}{G_s^*} = \frac{1}{N_{\text{tip}}} + \frac{1}{N_s}$$

Contact area

Hertzian Contact m=3/2

Flat punch m=1

Reference sample

Material	Source	M	N	$\nu=\frac{M-4N}{M-2N}$	$G=N(2-\nu)$	$E=M(1-\nu^2)$
SiO_2	Literature	74.9	17.0	0.171	31.1	72.7
Glass	Literature	84.7	18.7	0.206	33.6	81.1
	Expt. $m=1$	81±5	18±2	0.21±0.11	32±5	76±6
	Expt. $m=3/2$	85±8	19±3	0.17±0.16	35±8	79±10

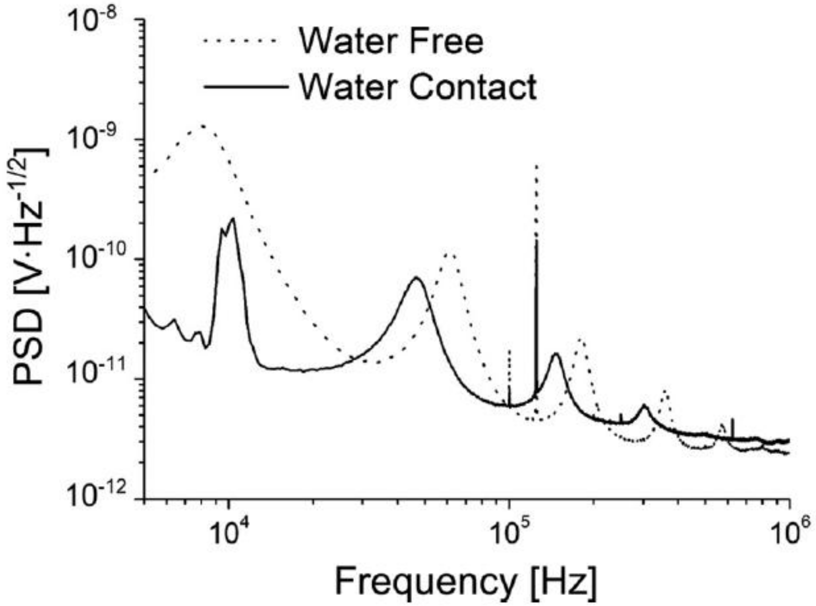
«Unknown» material

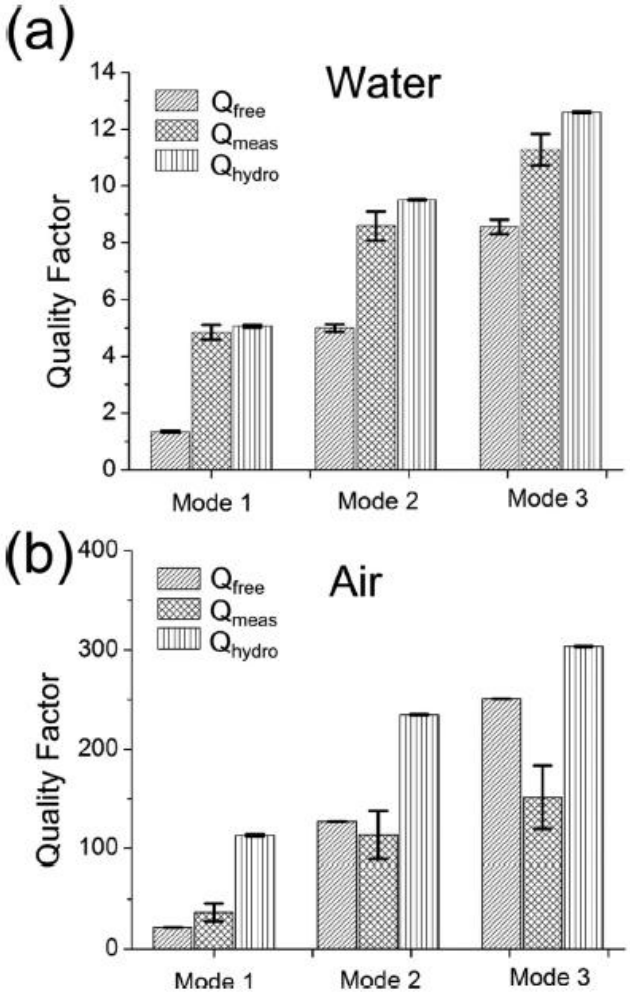
$$\nu = \frac{M-4N}{M-2N}$$

DRFT OR DART: SOME RECENT ADVANCES

LIQUID CR-AFM

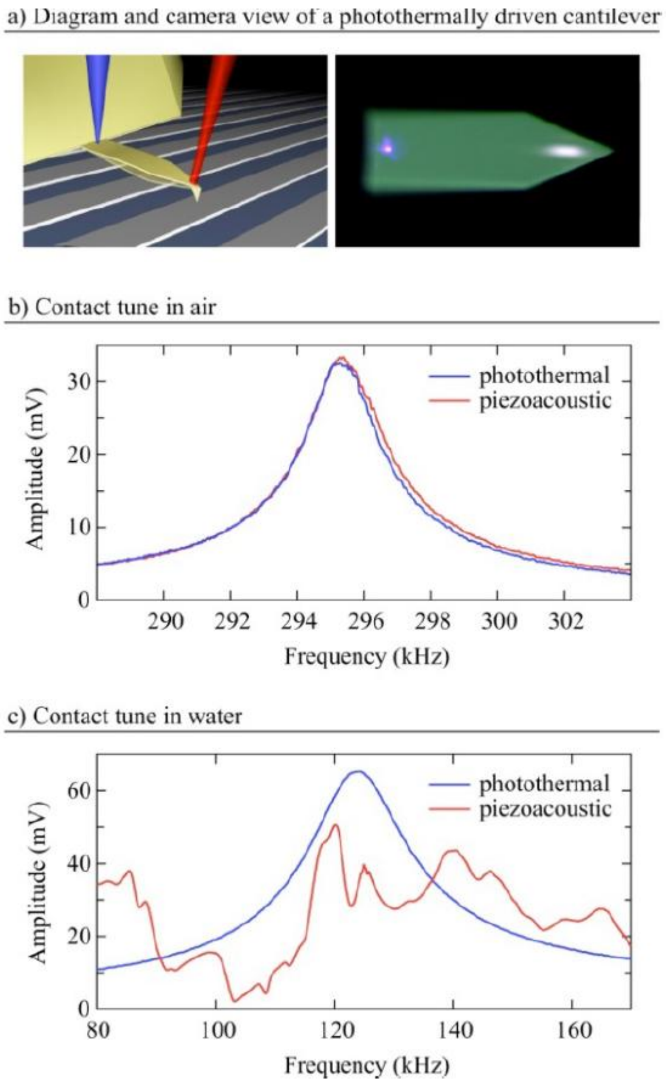
Sample: glass blade





Tung JAP 2014

DRFT OR DART: SOME RECENT ADVANCES



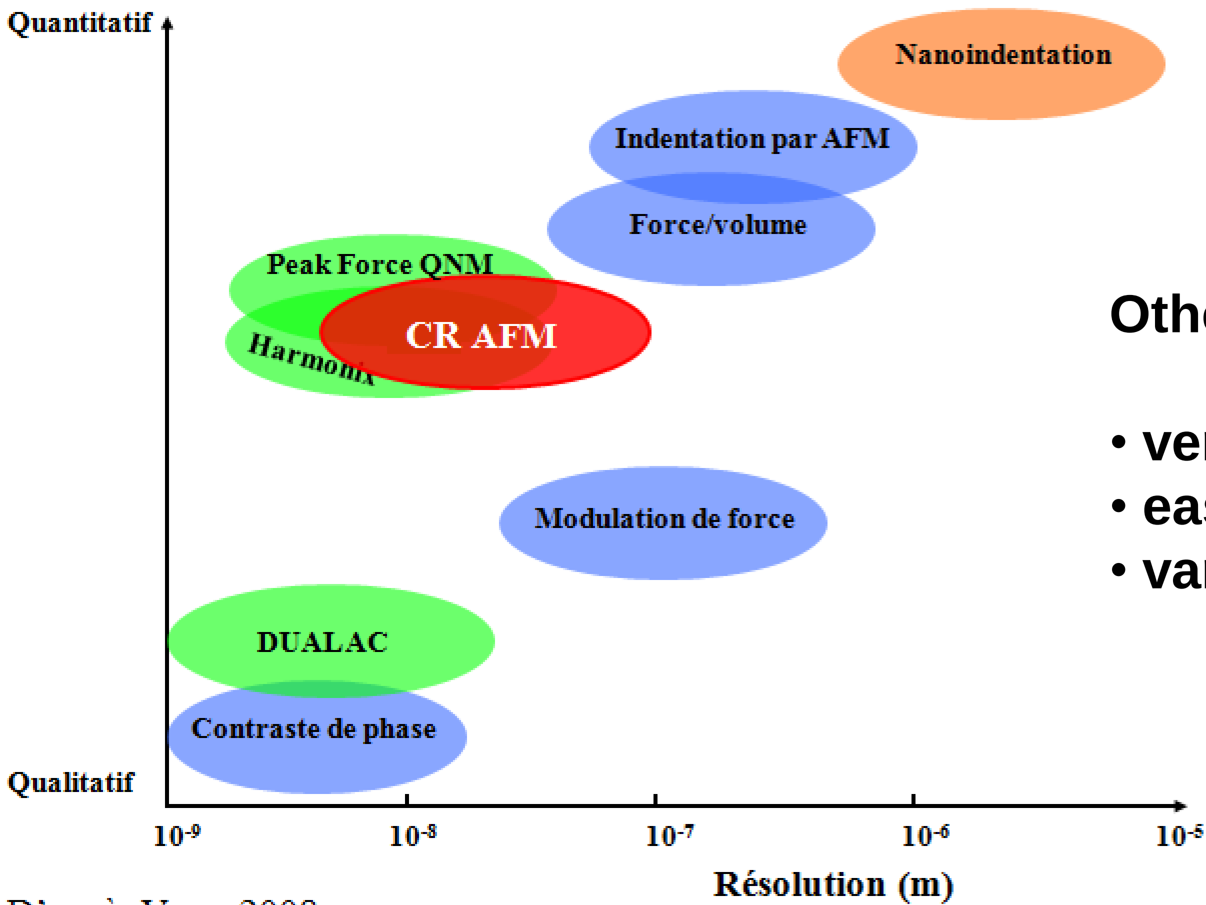
Photothermally Excited Contact Resonance Imaging in Air and Water

amplitude cantilever oscillation is induced by modulating the blue laser power that is focused at the base of the cantilever

Advantage of using photothermal actuation becomes clear when the contact resonance tunes are performed in water

Kocun, Proksch, Asylum

CONCLUSION



D'après Veeco 2008

Other aspects to consider

- **versatility**
- **ease of implementation**
- **variety of materials**

THANK YOU FOR YOUR ATTENTION