The Force Feedback Microscope: an AFM for soft condensed matter

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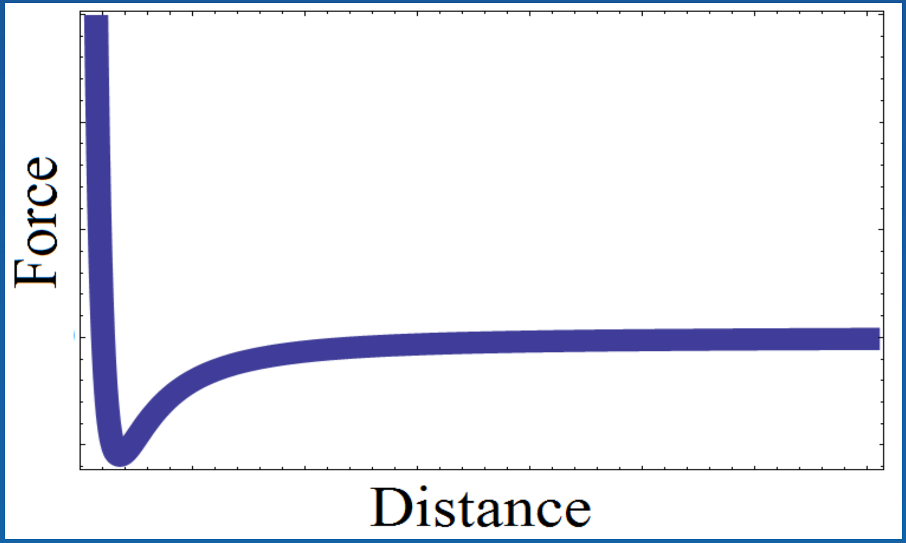
Outline

- *Motivation*
- *The Force Feedback Microscope*
- *Results*
- *Theory*
- *Conclusions*

The interaction Force

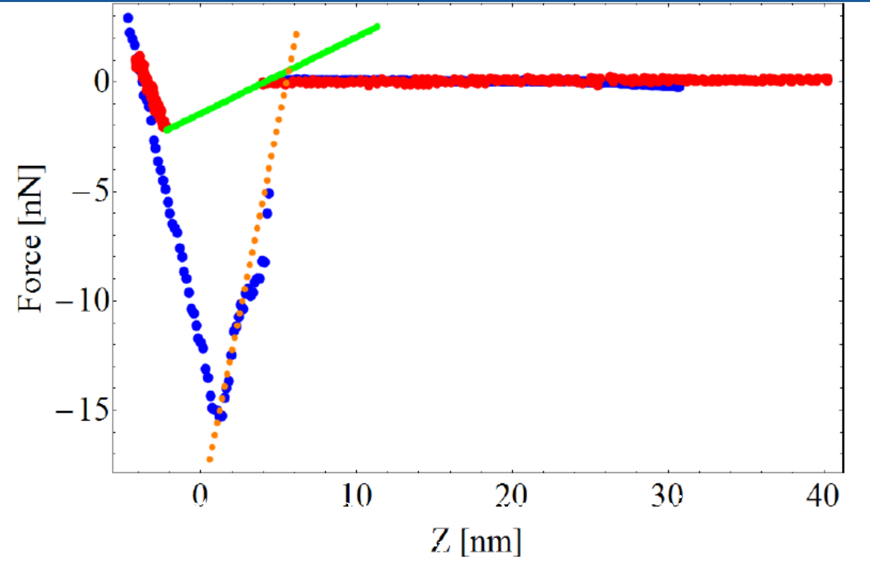


...all things are made of atoms - little particles that move around in perpetual motion, attracting each other when they are a little distance apart, but repealing upon being squeezed onto one another...

R. Feynman

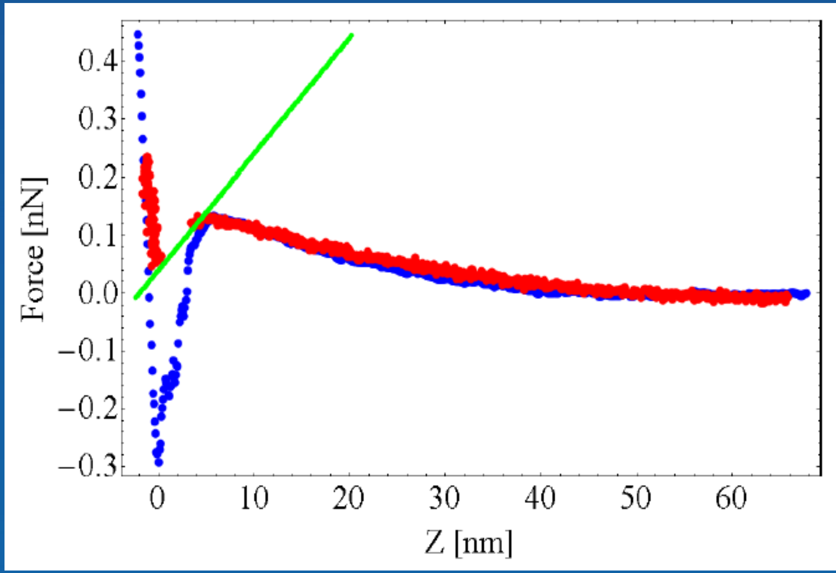
The Jump to contact

IN AIR



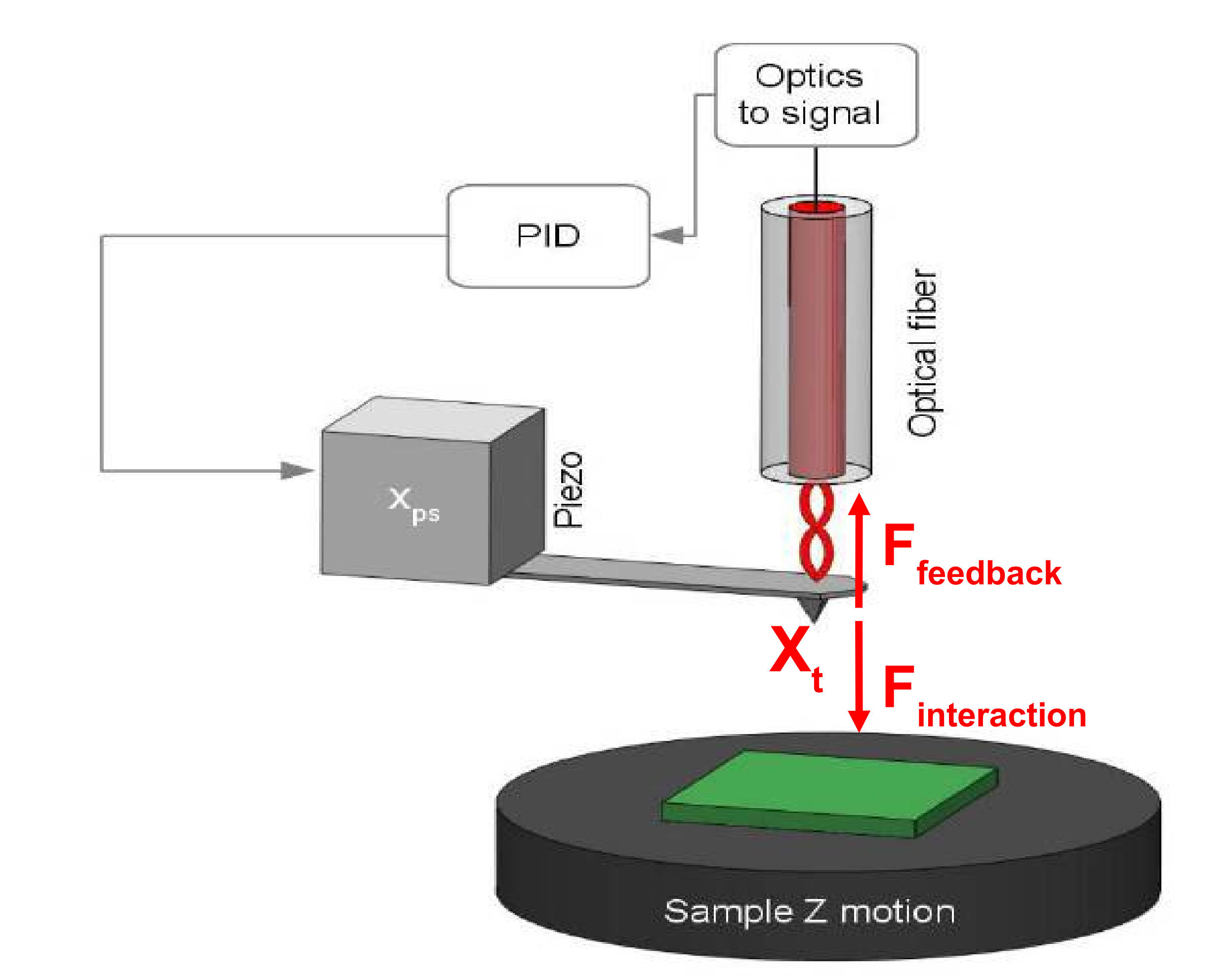
Force between a silicon probe and hydrophilic silicon native oxide surface in air

IN LIQUID



Force between a silicon nitride probe and mica in deionized water

SETUP


Optics to signal
PID
Optical fiber
Piezo
X_{ps}
$F_{feedback}$
X_t
$F_{interaction}$
Sample Z motion

THE IDEA

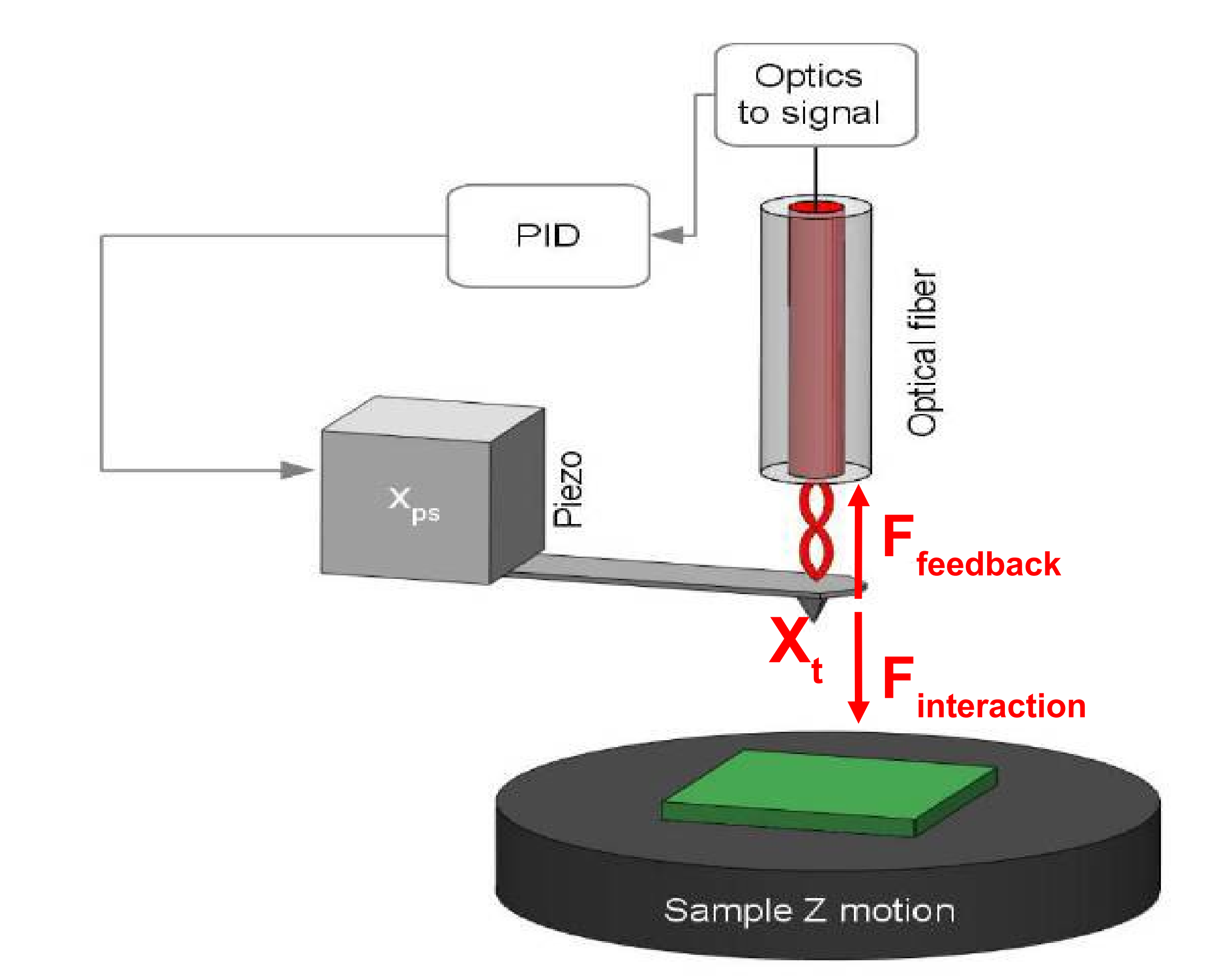
$$\sum_i \vec{F}_{i/tip} = \vec{0}$$

Sum of the force acting on the tip equal to zero

$$F_{feedback} = F_{interaction}$$

Position of the tip fixed in space

SETUP

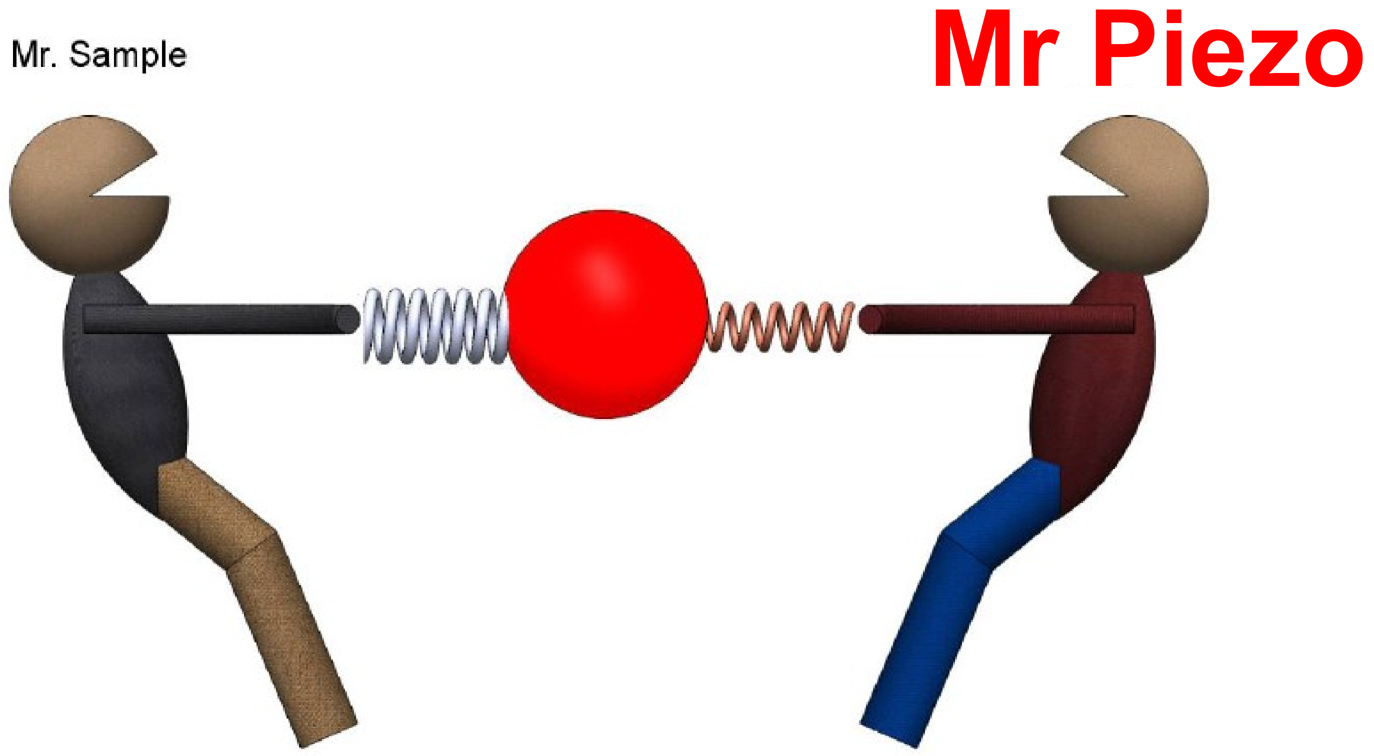


Where

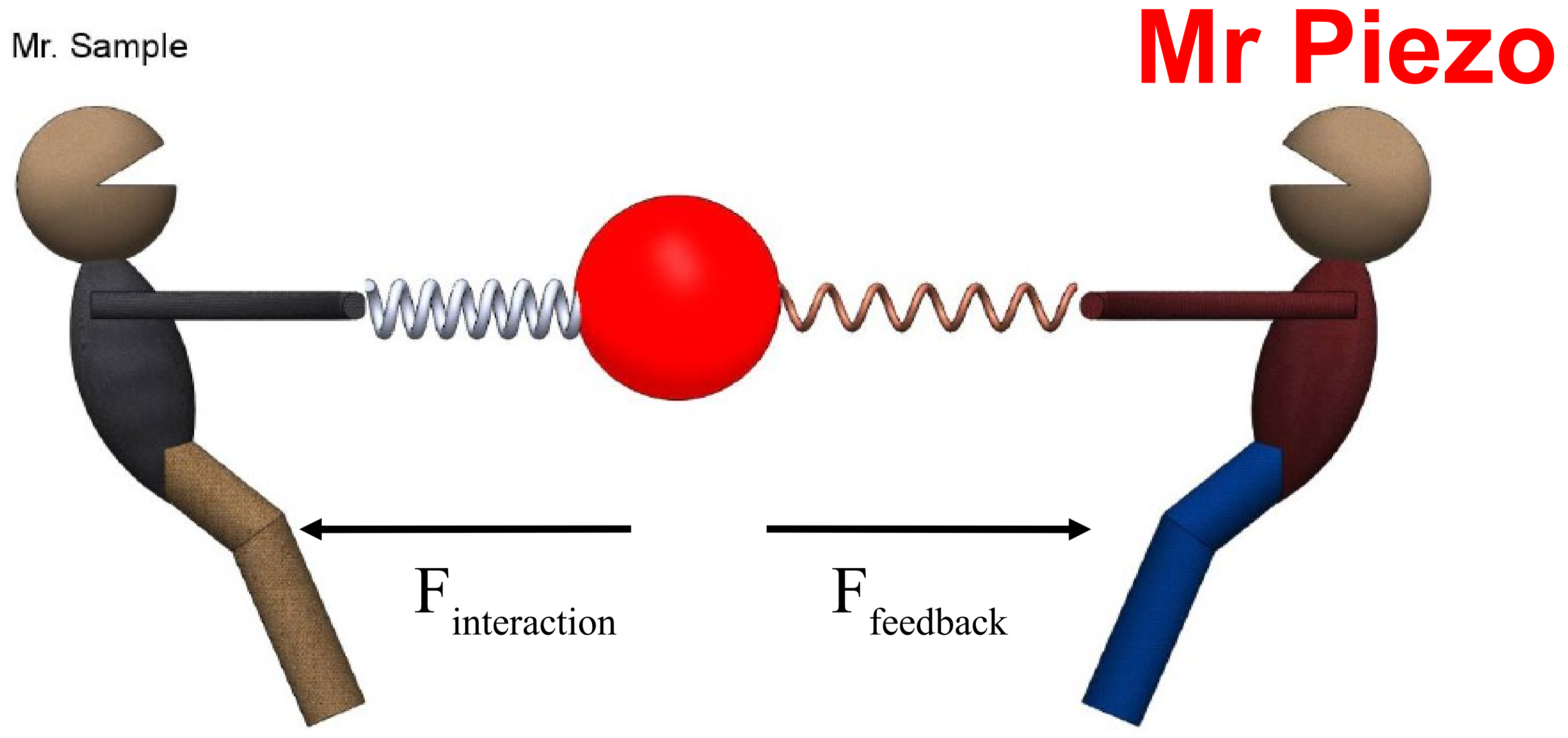
$$F_{feedback} = F_{interaction}$$

X_t is kept constant by a feedback loop

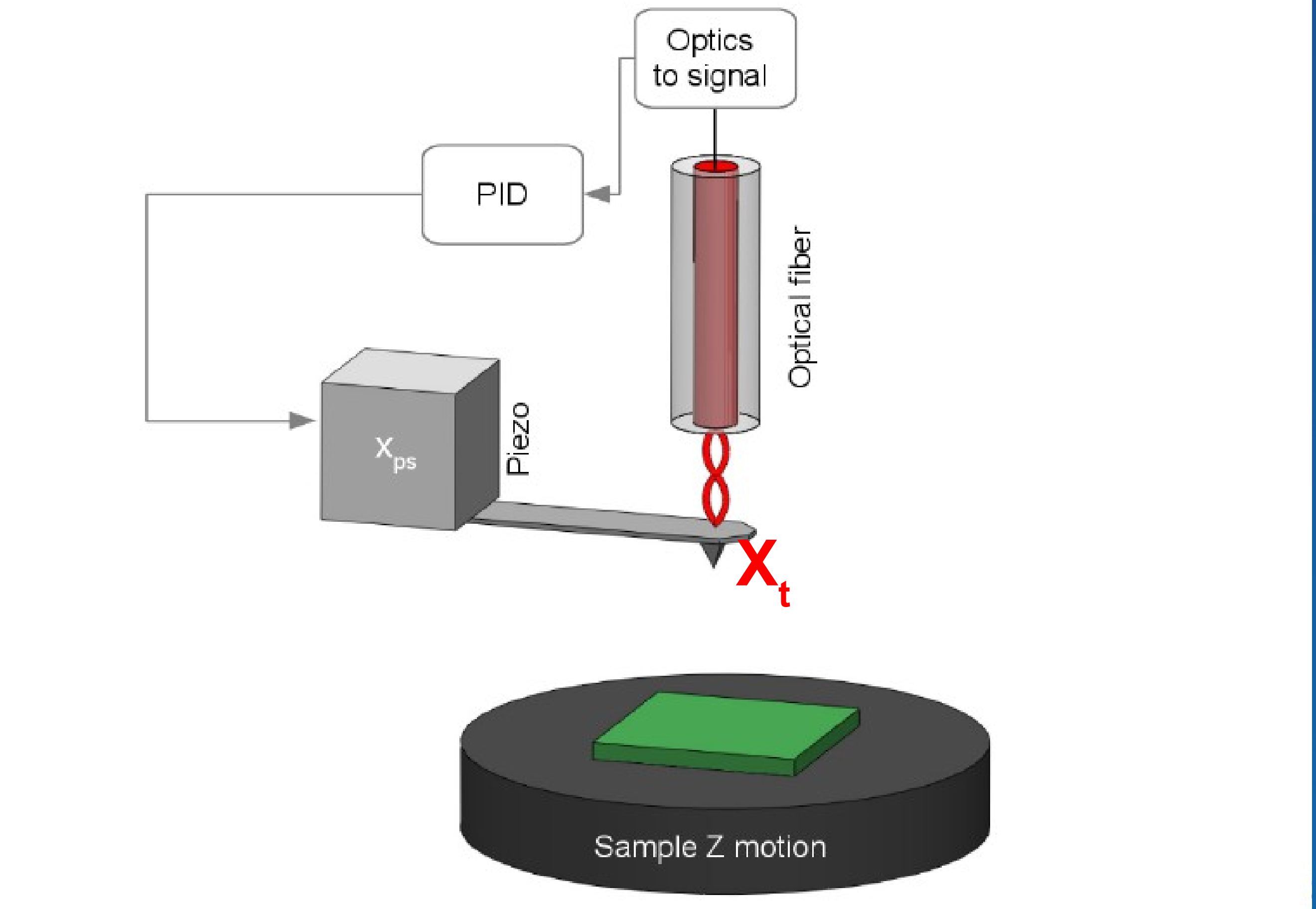
SETUP



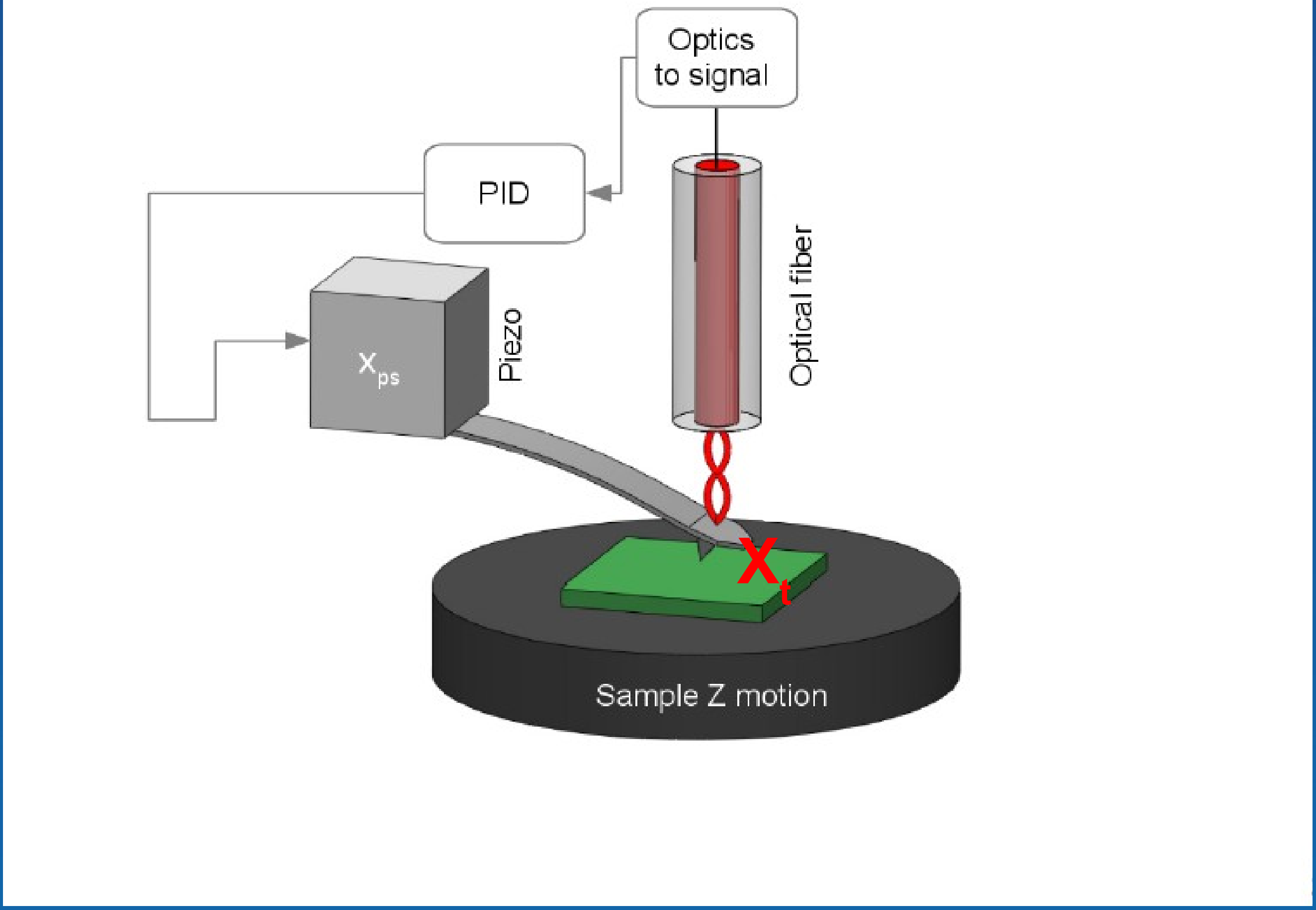
SETUP



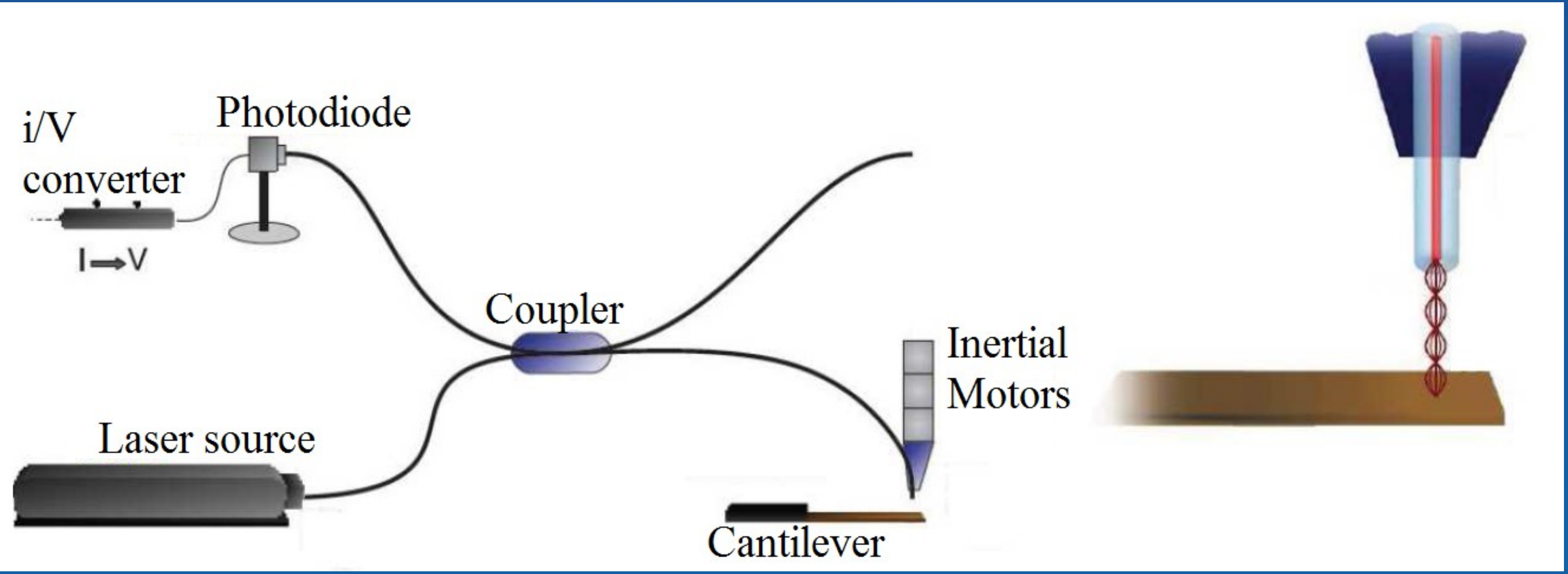
SETUP


Optics
to signal
PID
Optical fiber
X_{ps}
Piezo
X_t
Sample Z motion

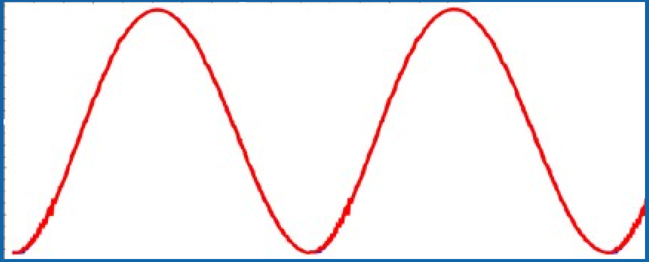
SETUP


Optics
to signal
PID
X_{ps}
Piezo
Optical fiber
X_t
Sample Z motion

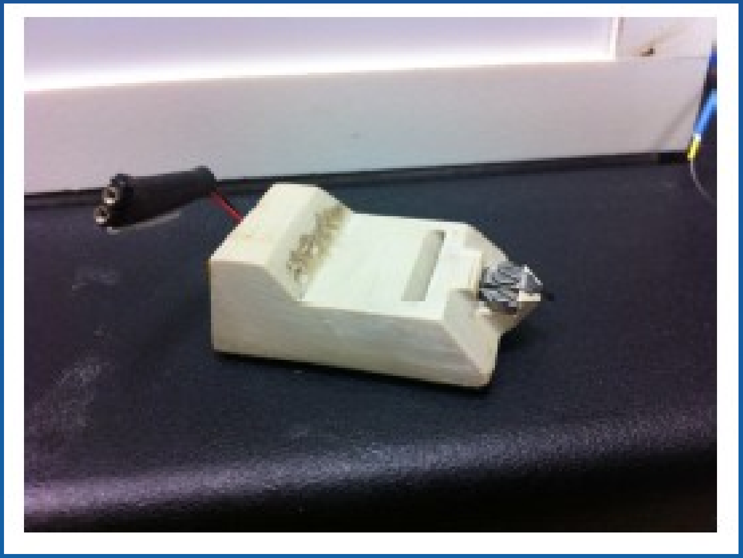
Fabry-Perot Interferometer

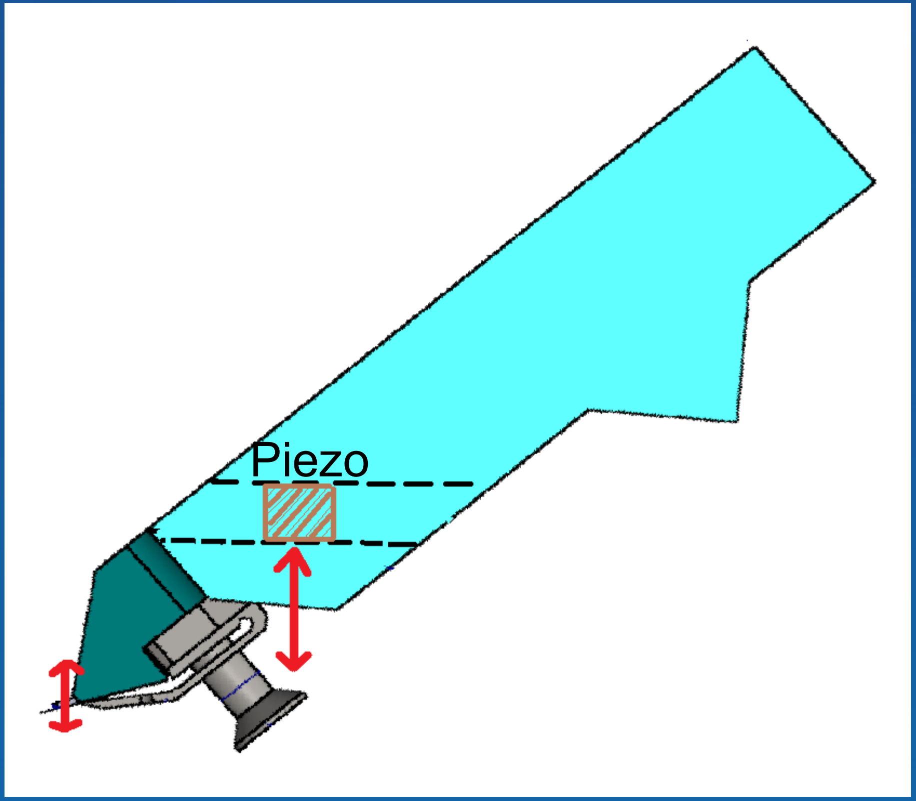


$$I(d) = I_0 + \Delta I_0 sin\left(\frac{4\pi}{\lambda}d + \phi\right)$$



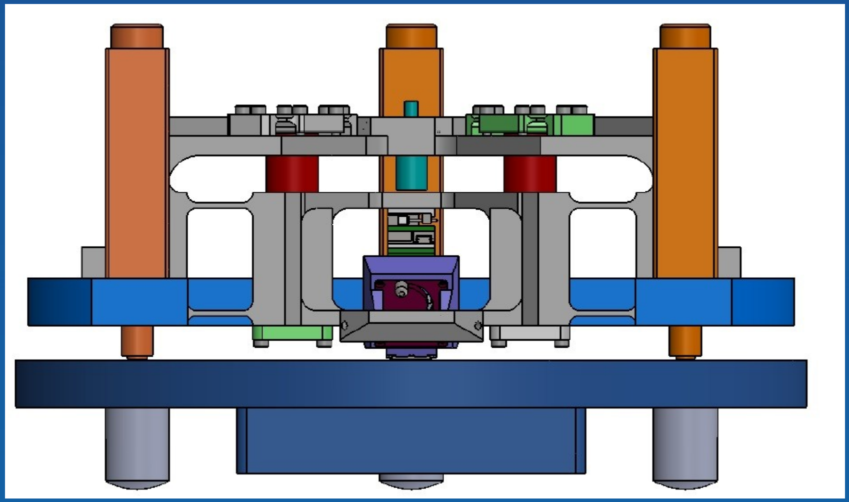
Cantilever holder

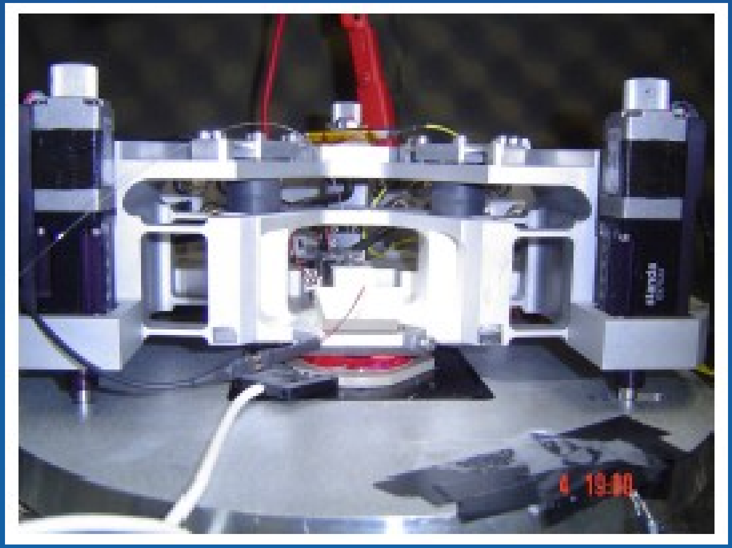




Cantilever base displacement ≈ 5 nm/V applied

MICROSCOPE



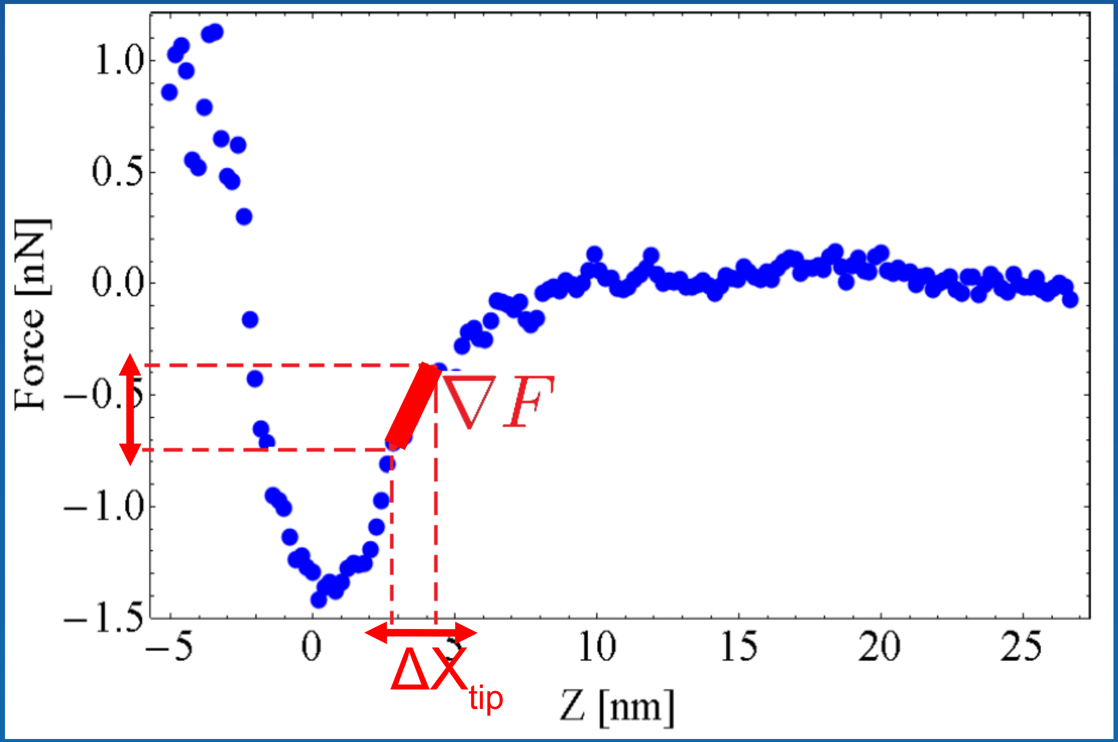


Michal Hrouzek PhD Thesis, 2004-2007

Mario S. Rodrigues Post-doc 2010-2011

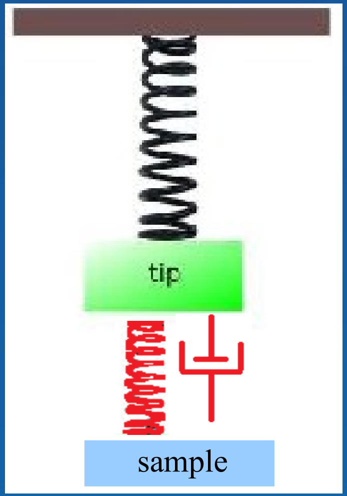
DYNAMIC FFM

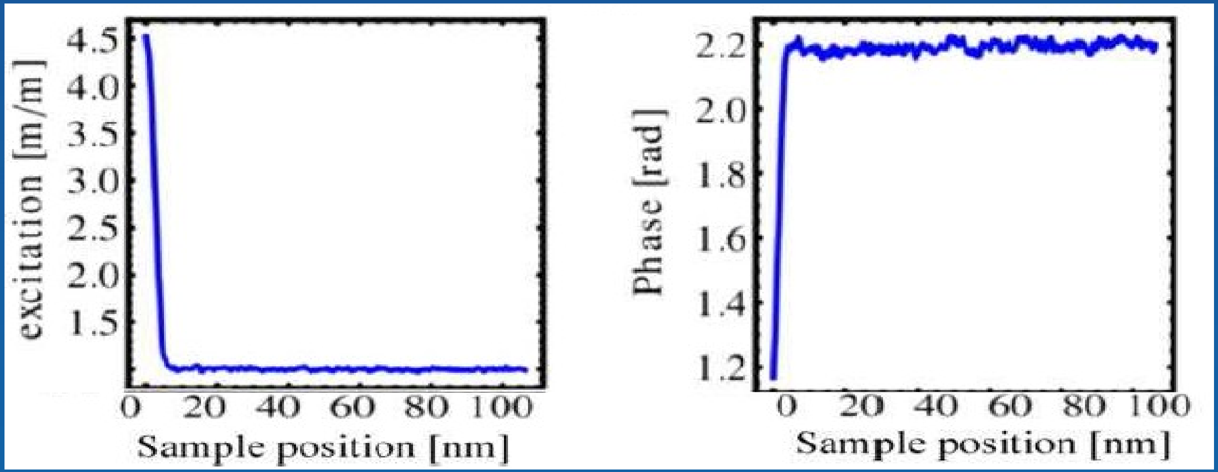
Tip oscillation $X_{tip}(t) = \overline{X_{tip}} + \Delta X_{tip} \cos(\omega t)$

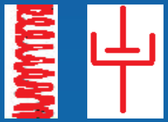


DYNAMIC FFM

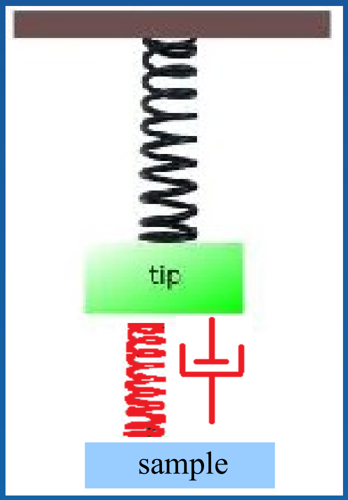
RAW DATA

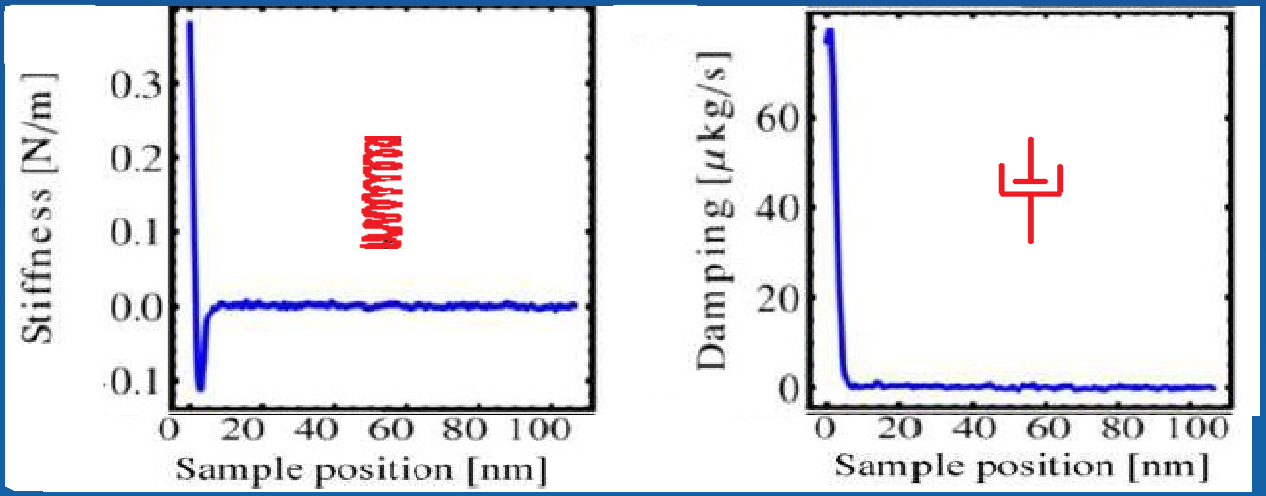




RAW DATA →  ?

Force gradient & Dissipation

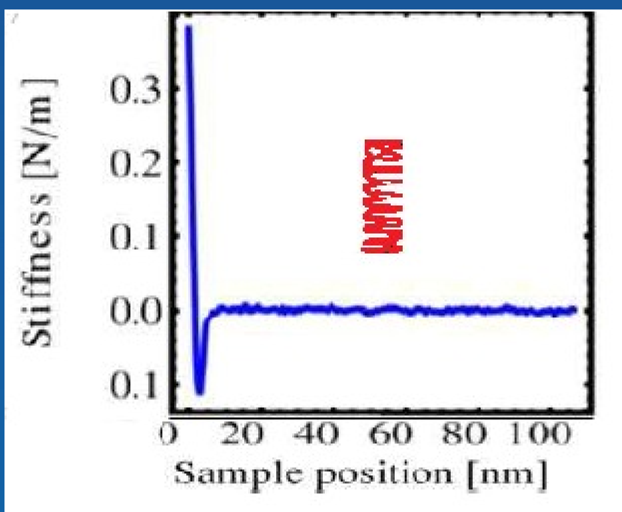


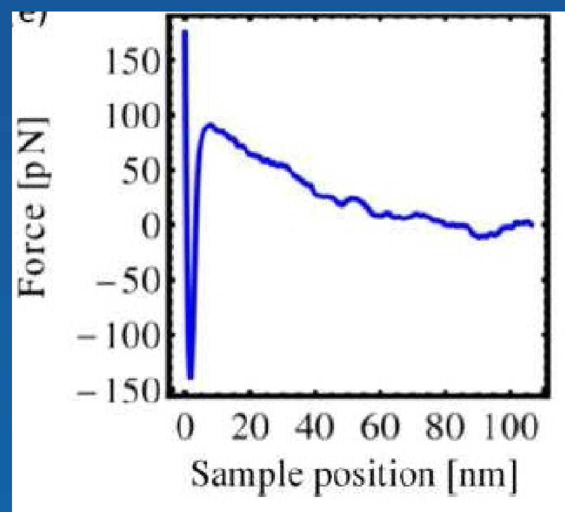


Force

$$\nabla F$$

$$F_{static} = \int \nabla F \, dz$$



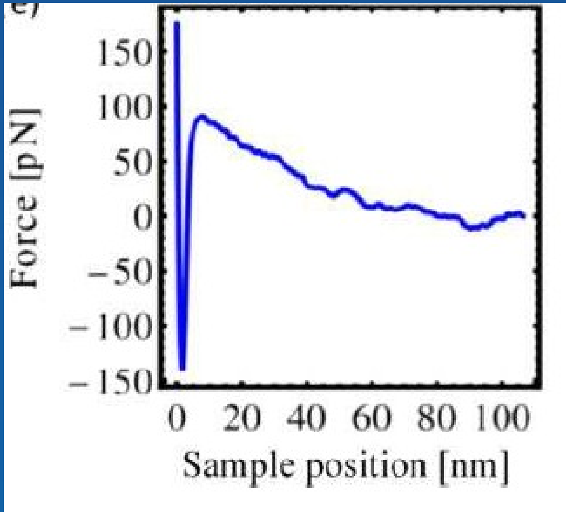


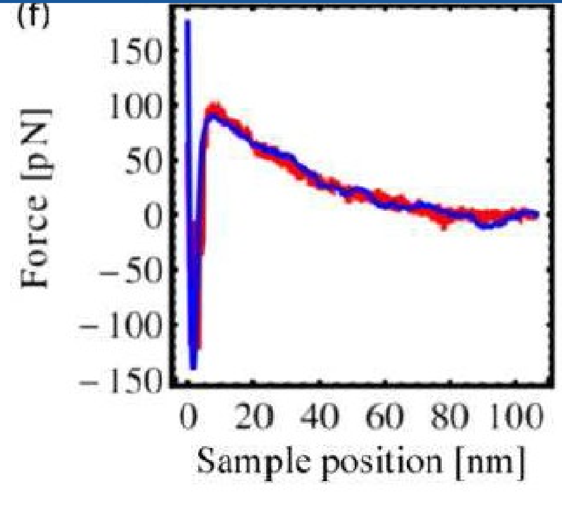
M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, Applied Physics Letters 101, 203105 (2012)

Force

$$F_{static} = \int \nabla F \, dz$$

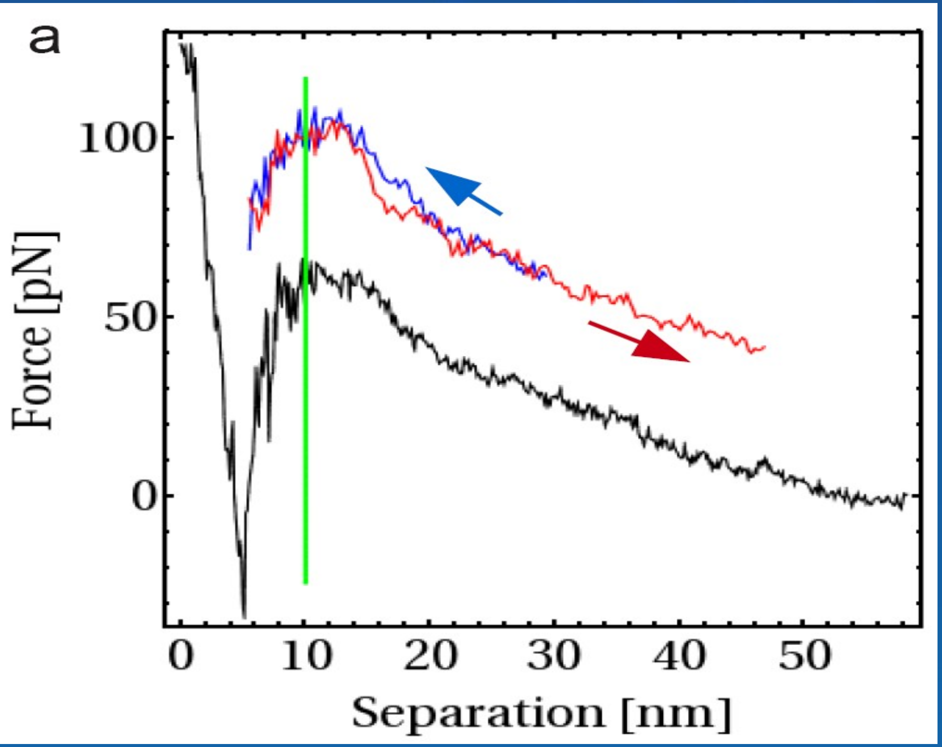
$$F_{static}$$





M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, Applied Physics Letters 1 0 1 , 203105 (2012)

Solid/liquid interface

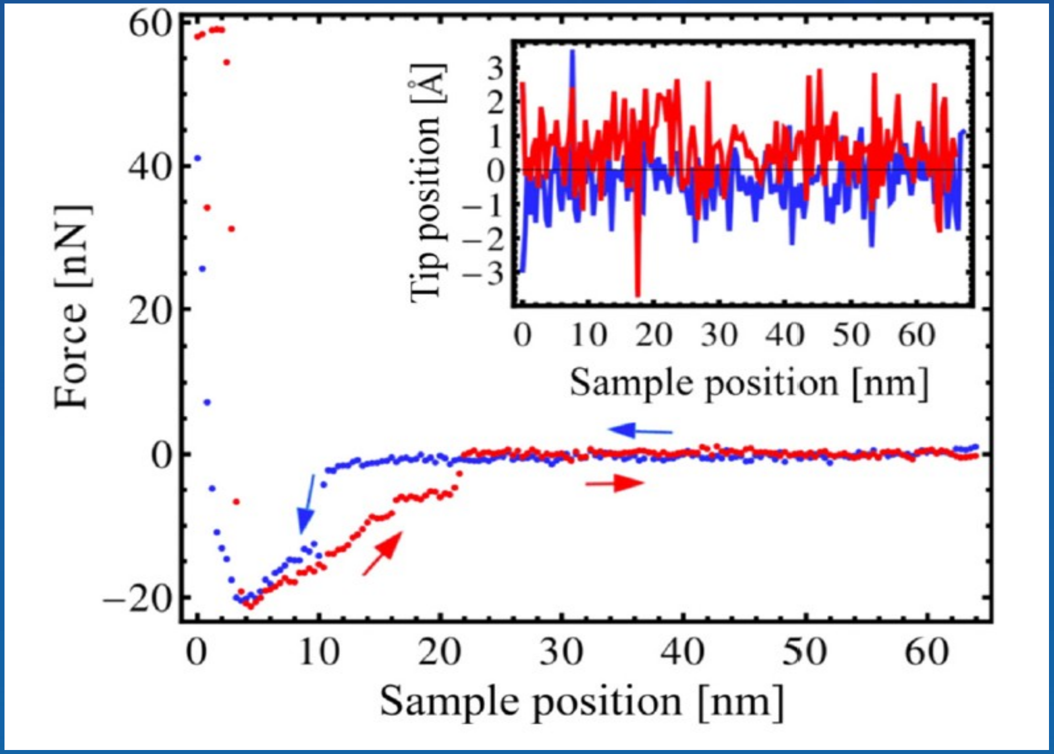


NO HYSTERESIS

Approach – Retract force curves between a silicon nitride probe and mica in deionized water

M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, Applied Physics Letters 1 0 1 , 203105 (2012)

Solid/air interface



HYSTERESIS DUE TO THE SYSTEM

Approach – Retract force curves between a silicon probe and hydrophilic silicon native oxide surface in air

M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, Applied Physics Letters 1 0 1 , 203105 (2012)

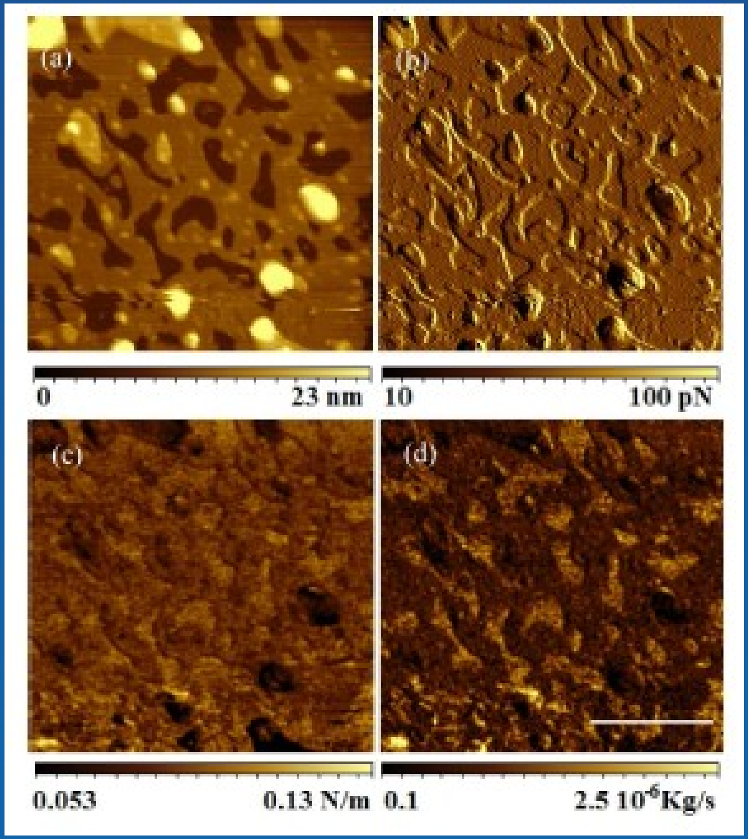
Imaging modes

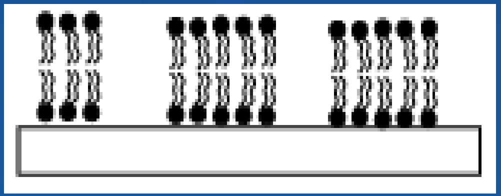
Mode	Tip position	Force	Force gradient	Dissipation	Interaction nature
Constant force	constant	constant	recorded	recorded	repulsive
Constant phase	constant	recorded	"partially constant"	"partially constant"	attractive or repulsive
Constant excitation	constant	recorded	"partially constant"	"partially constant"	attractive or repulsive

Imaging modes applied to the study of soft biological samples

Phospholipids DSPE

Page 73 of the manuscript





a) Topography
b) Force
c) Stiffness
d) Dissipation

Excitation frequency of the tip not linked to resonance

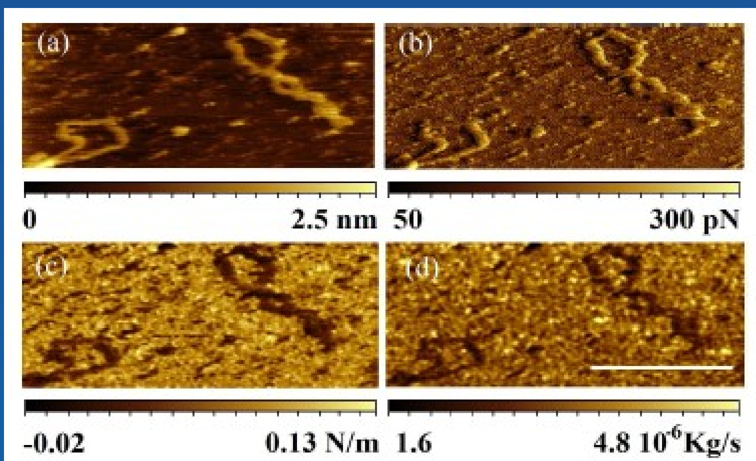
Young modulus E ≈ 7 MPa

L. Costa, M. S. Rodrigues, E. Newman, C. Zubieta, J. Chevrier, and F. Comin, Journal of Molecular Recognition, 26: 689-693 (2013)

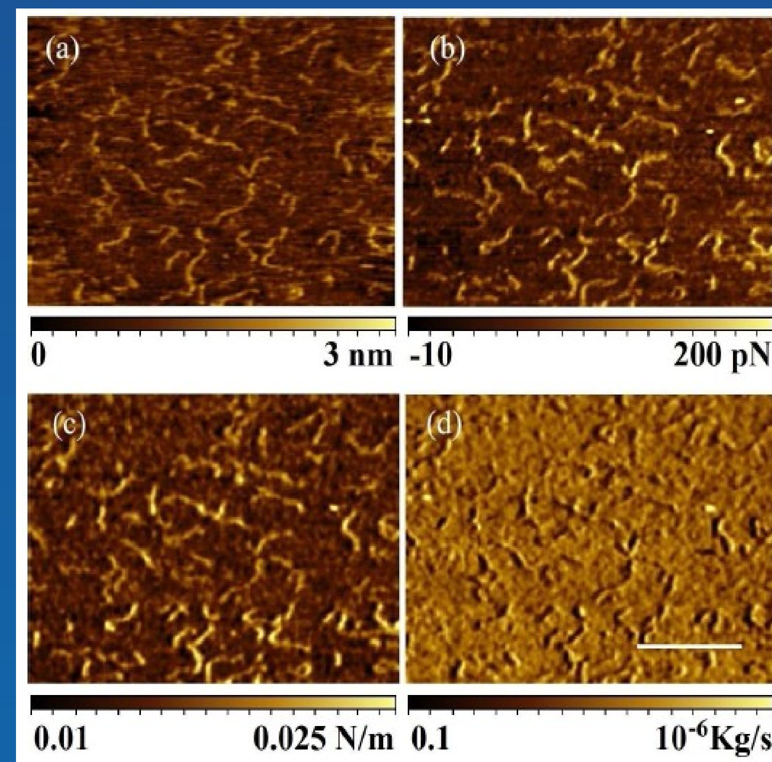
DNA

Pages 76-77 of the manuscript

Constant Force



Constant Dissipation



a) Topography
b) Force
c) Stiffness
d) Dissipation

Excitation frequency of the tip not linked to cantilever resonance

L. Costa, M. S. Rodrigues, E. Newman, C. Zubieta, J. Chevrier, and F. Comin, Journal of Molecular Recognition, 26: 689-693 (2013)

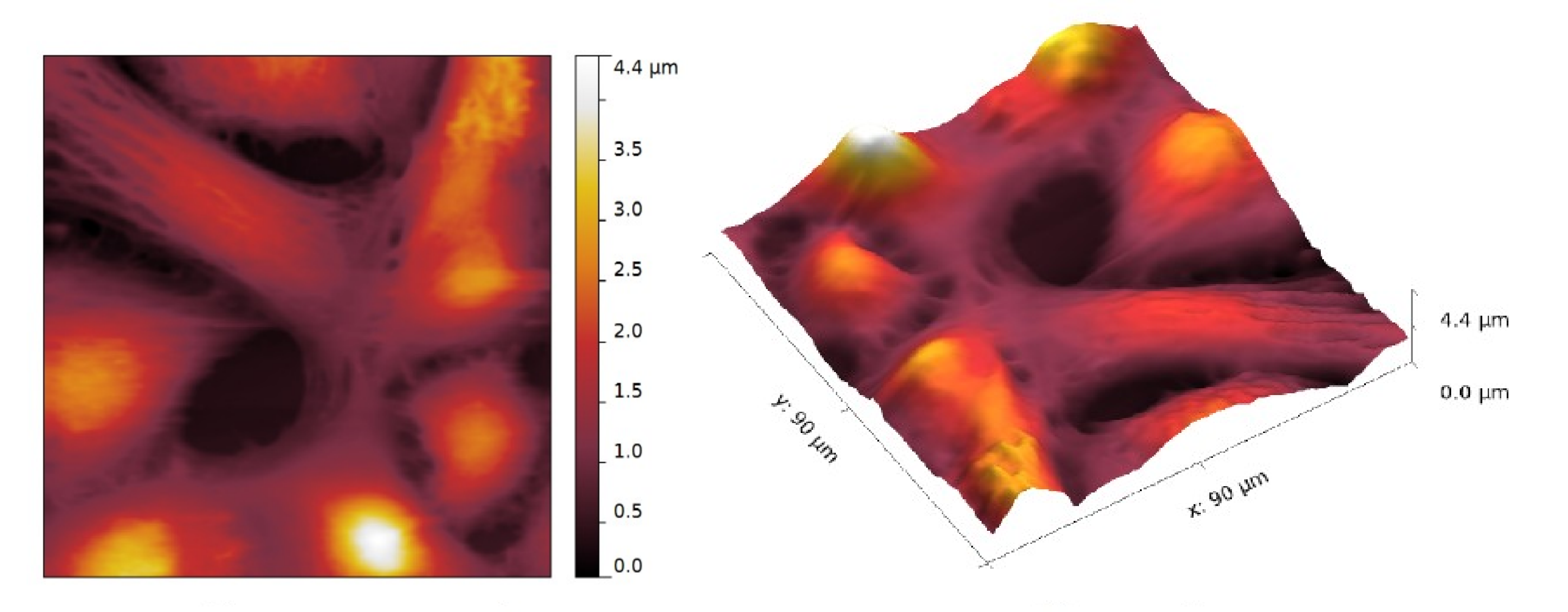
Living Cells PC12

TUNABLE EXCITATION FREQUENCY

↓

Viscoelastic spectroscopy

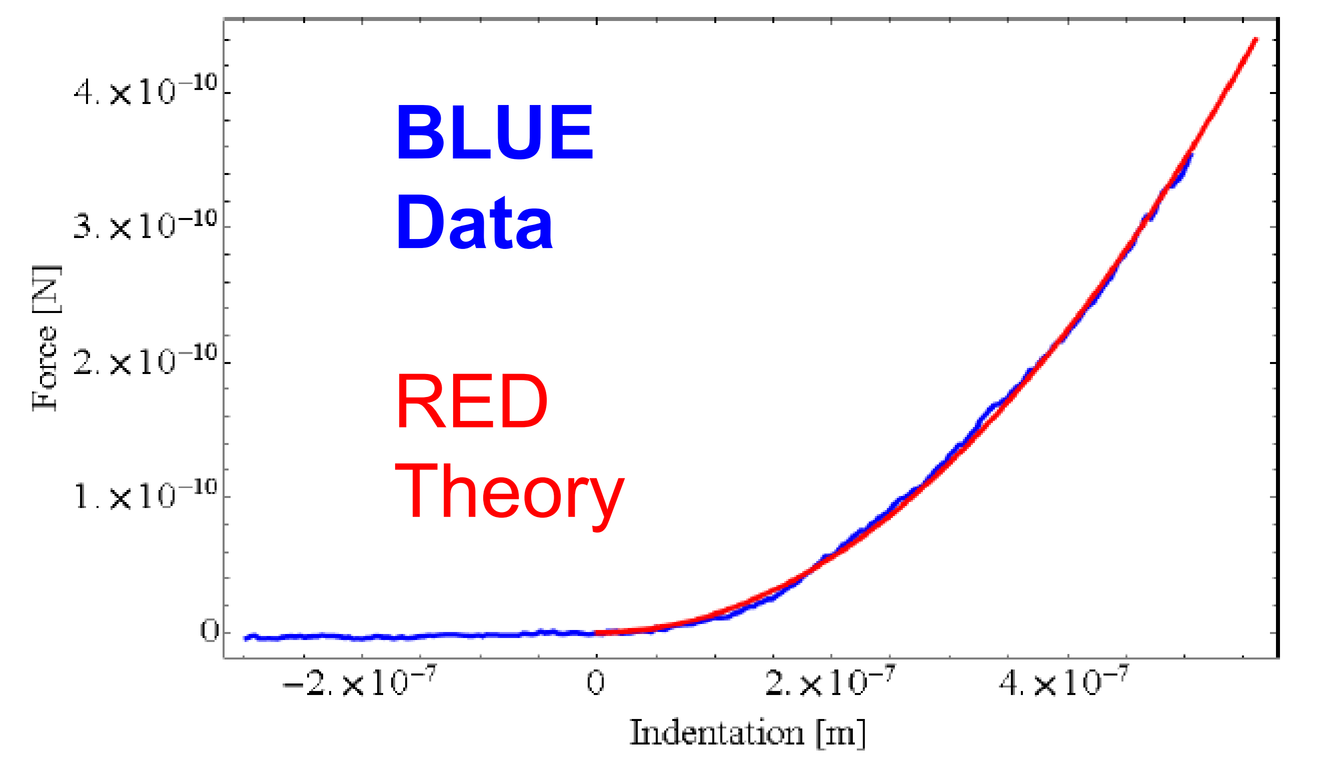
Candidate: **Living cell PC12,** in MEM - 1% Strepto-Penicillin



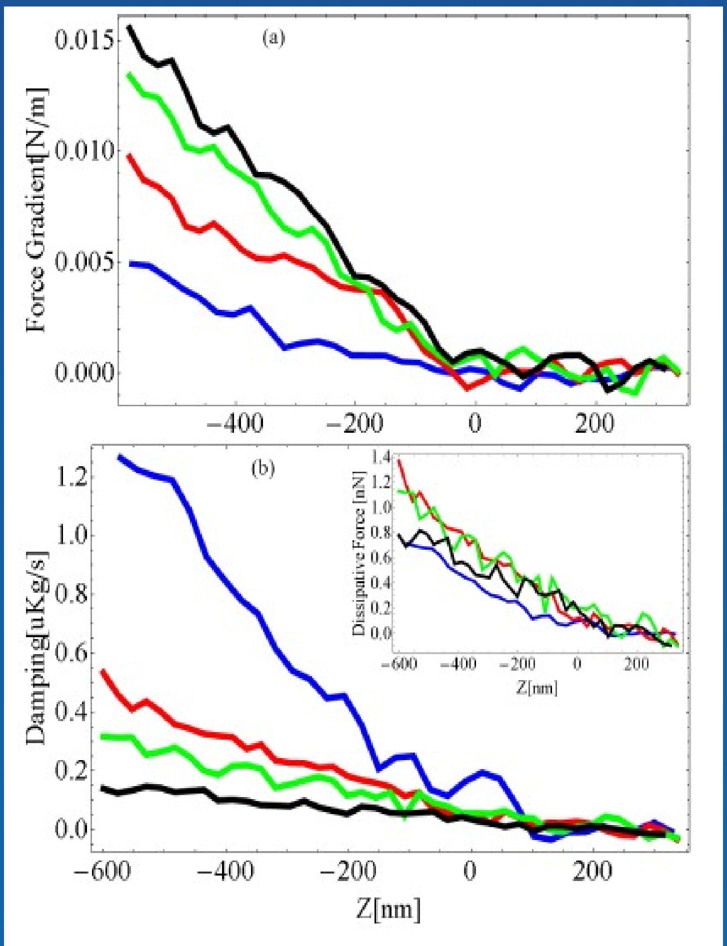
Static indentation

Quasi-static conditions: Young modulus ≈ 1 kPa

$$F = \frac{3E\tan\theta}{4(1-\nu^2)}\delta^2$$



FFM indentation

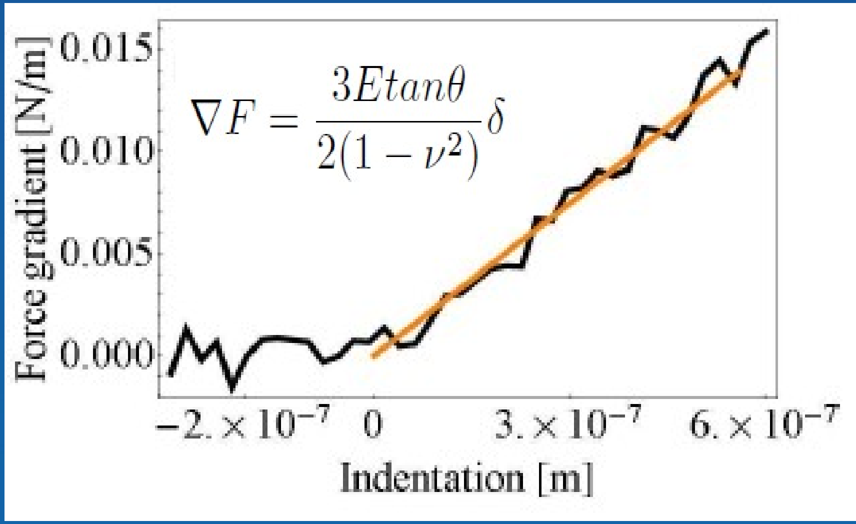


Blue = 1.13 kHz

Red = 5.13 kHz

Green = 7.13 kHz

Black = 11.13 kHz

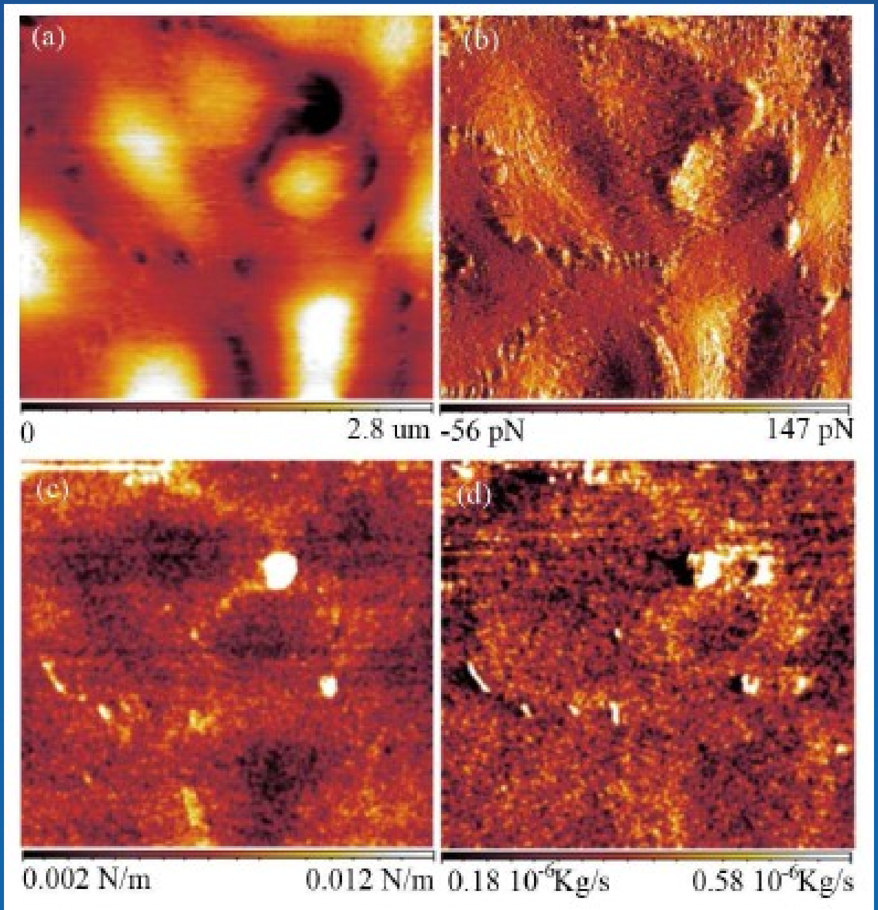


At 10 kHz, Young modulus ≈ 30 kPa

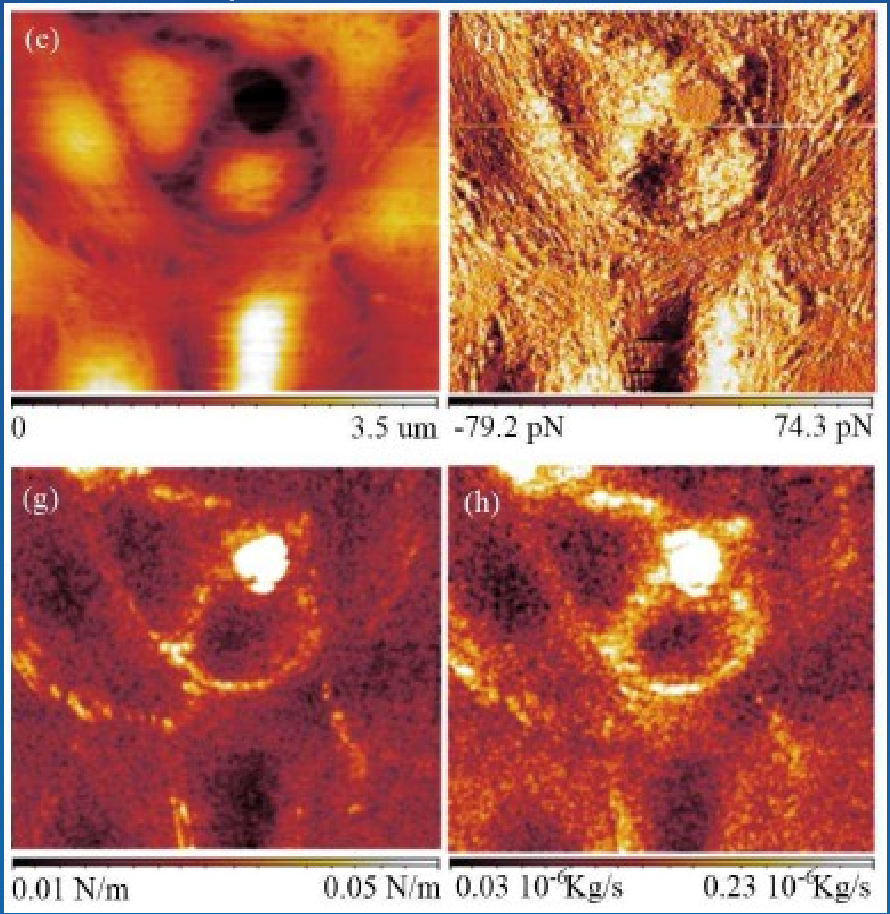
L. Costa, M. S. Rodrigues, N. Benseny-Cases, V. Mayeux, J. Chevrier, and F. Comin, submitted to PlosOne (2013)

Mapping mechanical properties

Page 94 of the manuscript

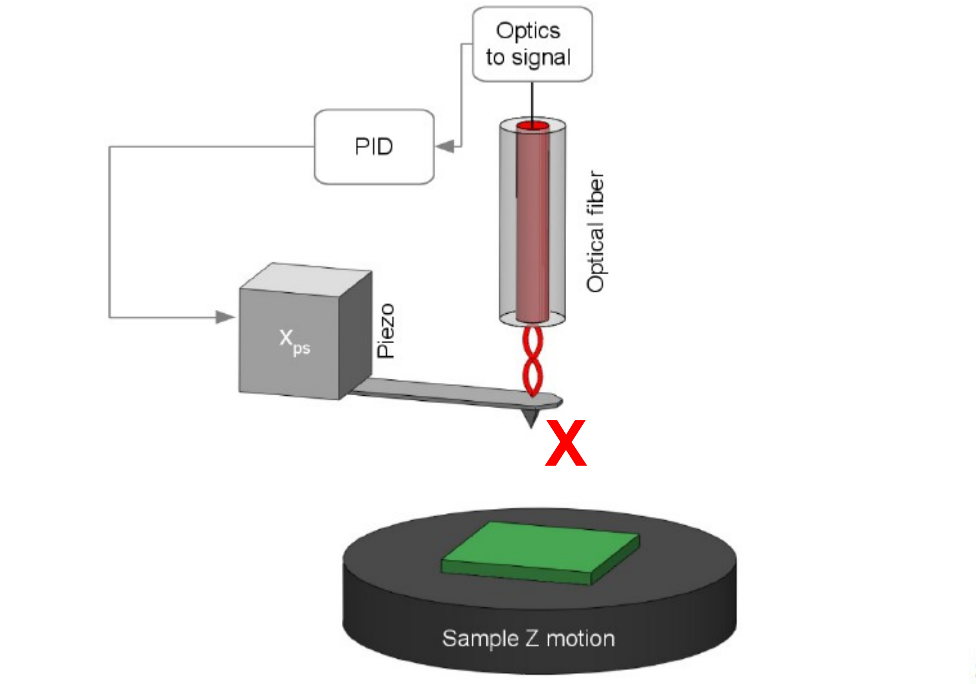


ω = 2.25 kHz



ω = 13.25 kHz

Feedback loop



Motion of the tip described by

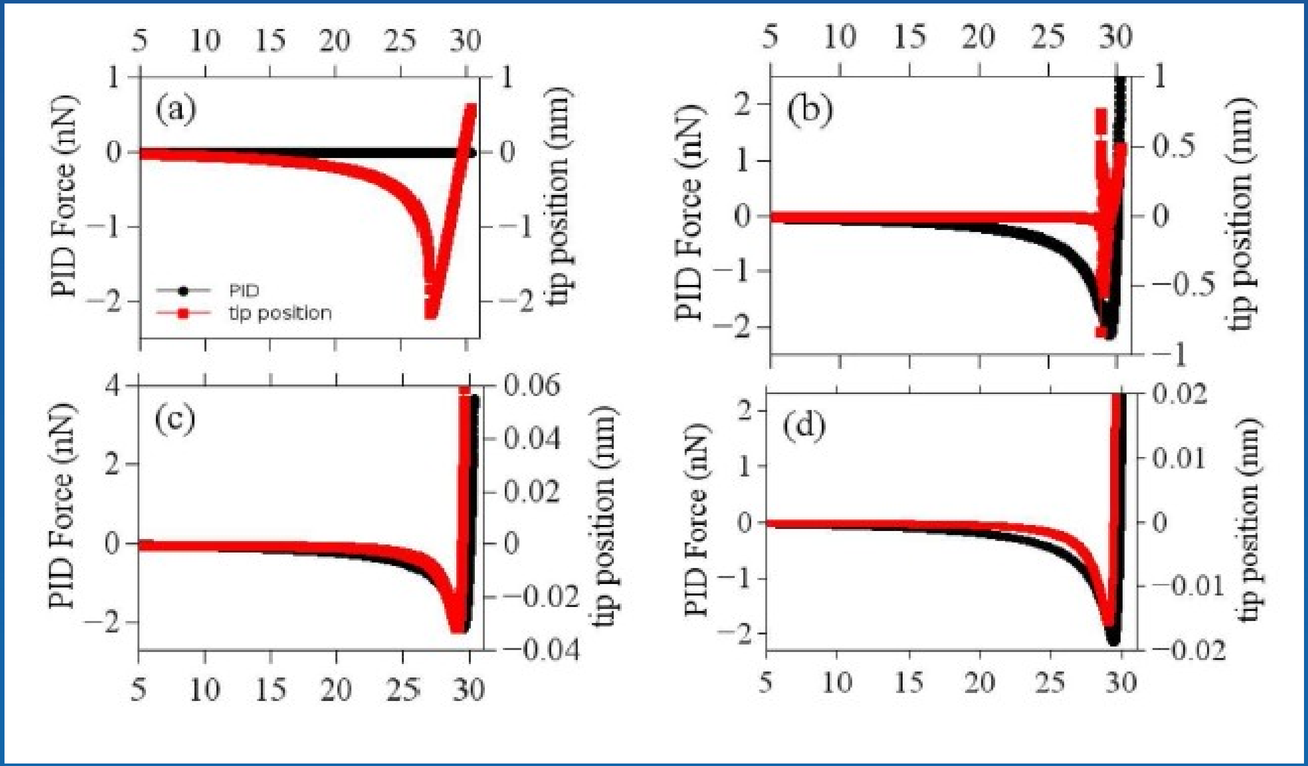
$$m\ddot{x} + \gamma\dot{x} + kx = F_{control} + F_{tip-sample}$$

$$F_{control} = -g_P x + -g_D \dot{x} - g_I \int_0^t x \, dt$$

M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, submitted to Journal of applied Physics (2013)

Simulations

Lever stiffness = 1 N/m   attractive force gradient = -1 N/m



a) no gains

b) just Integral gain

c) Proportional gain equal to lever stiffness (fast approach) + Integral gain

d) Proportional gain equal to lever stiffness (slow approach) + Integral gain

M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, submitted to Journal of applied Physics (2013)

Stability conditions

$$F_{ts} = F_{ts,0} - k_{ts}x - \gamma_{ts}\dot{x}$$

$$k_t = k + k_{ts}$$

$$\omega_0^2 = \frac{k}{m} \qquad Q = \frac{\omega_0}{\Delta\omega}$$

Stability conditions

$g_P > -k_t$ Mechanical stability of the tip

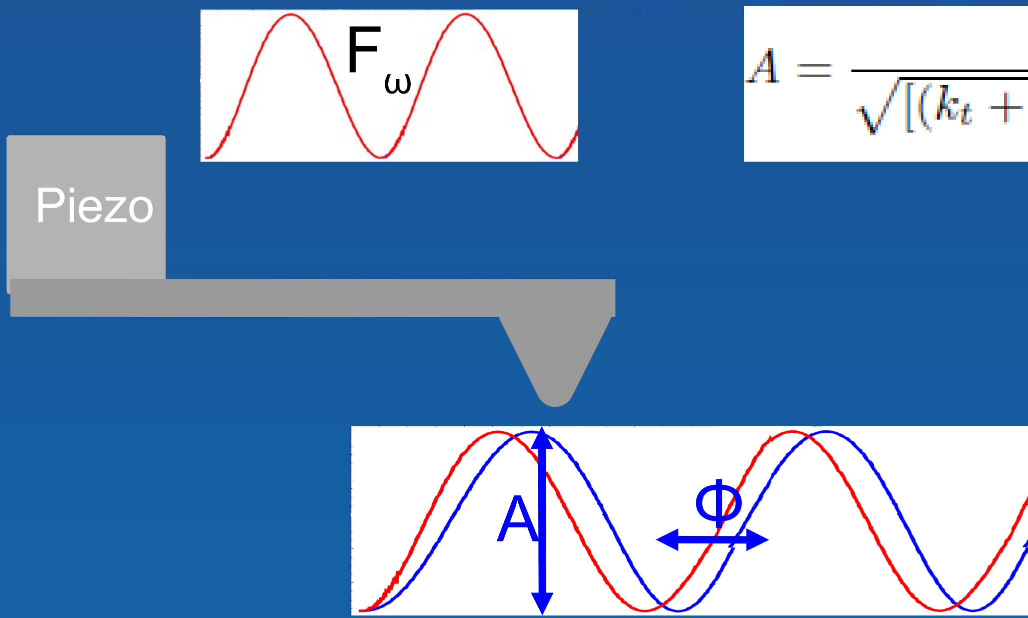
$$g_I < \frac{\omega_0}{3Q}(k_t + g_P) \qquad under - damped$$

$$g_I < Q\omega_0 \frac{(k_t + g_P)^2}{k} \qquad over - damped$$

Speed

M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, submitted to Journal of applied Physics (2013)

Dynamic FFM



$$A = \frac{F_\omega}{\sqrt{[(k_t + g_P) - m\omega^2]^2 + [(\gamma_t + g_D)\omega - \frac{g_I}{\omega}]^2}}$$

$$\phi = \arctan\left[\frac{(\gamma_t + g_D)\omega - \frac{g_I}{\omega}}{(k_t + g_P) - m\omega^2}\right]$$

Cantilever Transfer Function depends on PID gains

F_ω is the harmonic excitation

M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, submitted to Journal of applied Physics (2013)

Master equations

Force gradient

$$k_{ts} = F_{\omega\infty}\left[n cos(\phi) - cos(\phi_{\infty})\right]$$

Dissipation

$$\gamma_{ts} = \frac{F_{\omega\infty}}{\omega}\left[n sin(\phi) - sin(\phi_{\infty})\right]$$

$$F_{ts} = F_{ts,0} - k_{ts}x - \gamma_{ts}\dot{x}$$

$$n = F_{\omega}/F_{\omega\infty}$$

$$F_{\omega\infty} cos(\phi_{\infty}) = k - m\omega^2$$

$$F_{\omega\infty} sin(\phi_{\infty}) = \gamma\omega$$

M. S. Rodrigues, L. Costa, J. Chevrier, and F. Comin, submitted to Journal of applied Physics (2013)

Limits

Speed: defined by the maximum integral gain employed

$$g_I = \frac{\omega_0}{3Q}(k_t + g_P) \qquad under - damped$$

$$g_I = Q\omega_0 \frac{(k_t + g_P)^2}{k} \qquad over - damped$$

Force sensitivity: given by the lever stiffness. No difference between AFM and FFM The FFM simply let you measure attractive forces where AFM cannot.

Force gradient sensitivity: given by the effective lever stiffness. No difference between AFM and FFM.

Jump to contact: avoided for force gradient 5 times stiffer than the cantilever

Perspectives (1)

Time – dependent interaction forces

$$F_{static} \neq \int \nabla F(\omega = \omega_R) dz$$

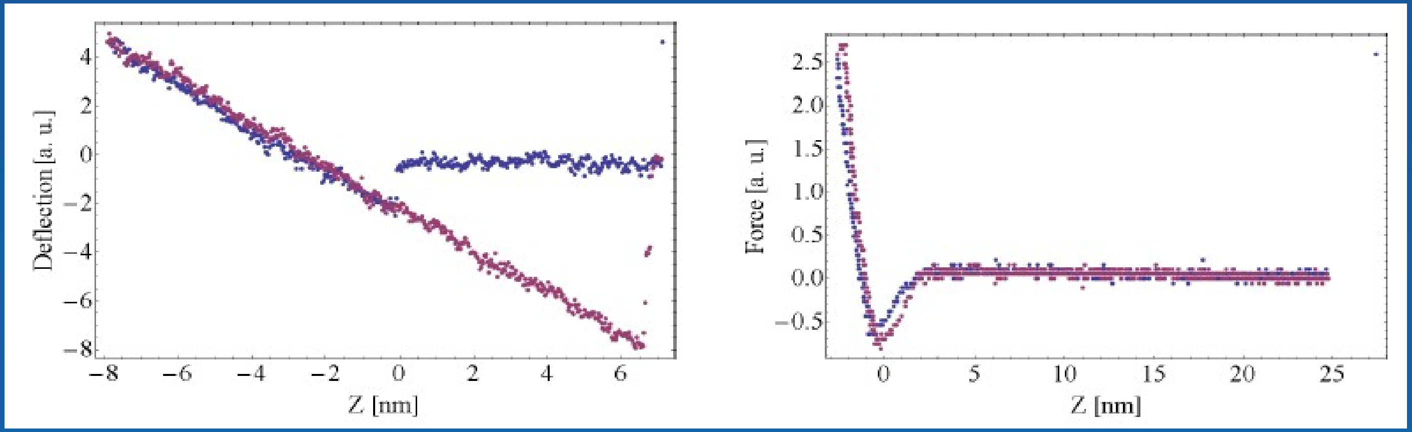
- Viscoelastic materials

- Capillary condensates

- Ligand-Receptor binding

Perspectives (2)

FFM with optical beam deflection systems



Measure the lever angle ⟶ counteract on the cantilever base angle

Surface Science Lab. Team

(past & present)

ESRF:
Chloe Zubieta
Sriarsha Puranik
Veronique Mayeaux
Harald Muller
Irina Snigireva
Nuria Benseny-Cases
Lin Zhang
Pascal Dideron
Pascal Bernard
SCM group

EMBL:
Emily Newman
Aurelian Dordor

External help:
P.E. Mihliet
Neil Thomson
Jean-Luc Pellequer

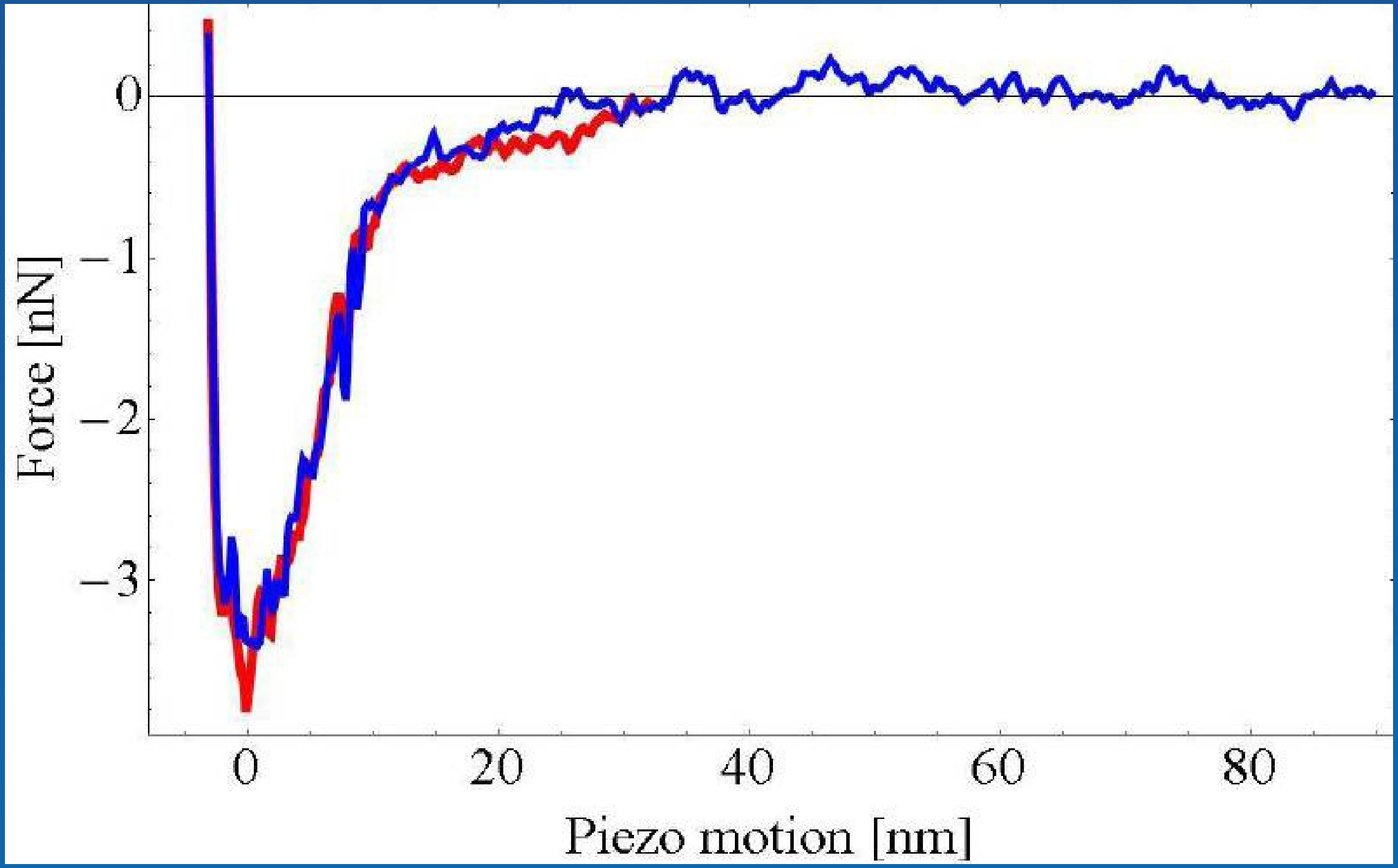
ILL:
Giovanna Fragneto
Jess Webster

My EUROPEAN COLLEAGUES
My FRENCH FRIENDS

..... and you for your kind attention

Additional Slides

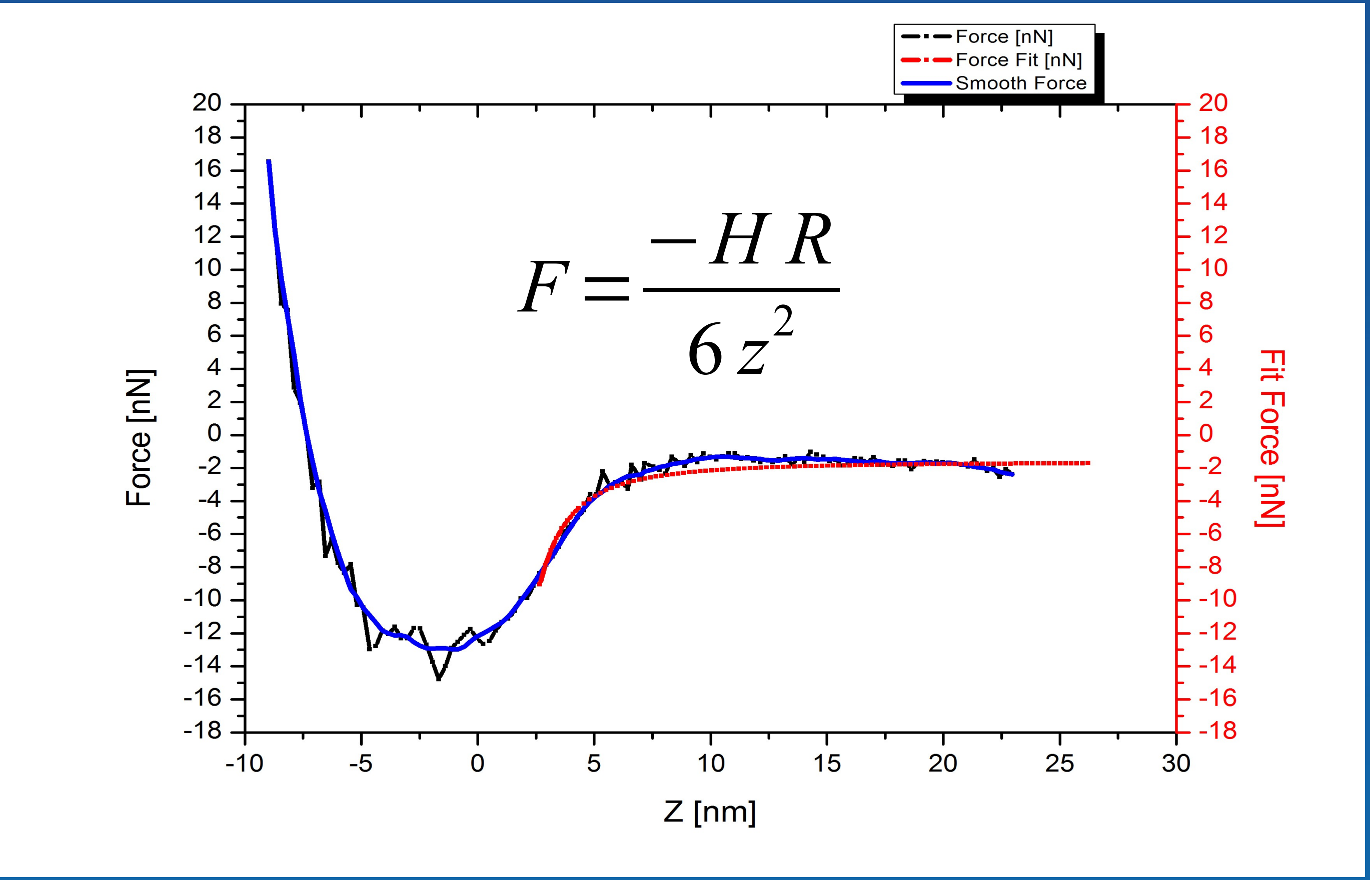
Approach-Retract on HOPG



Additional Slides

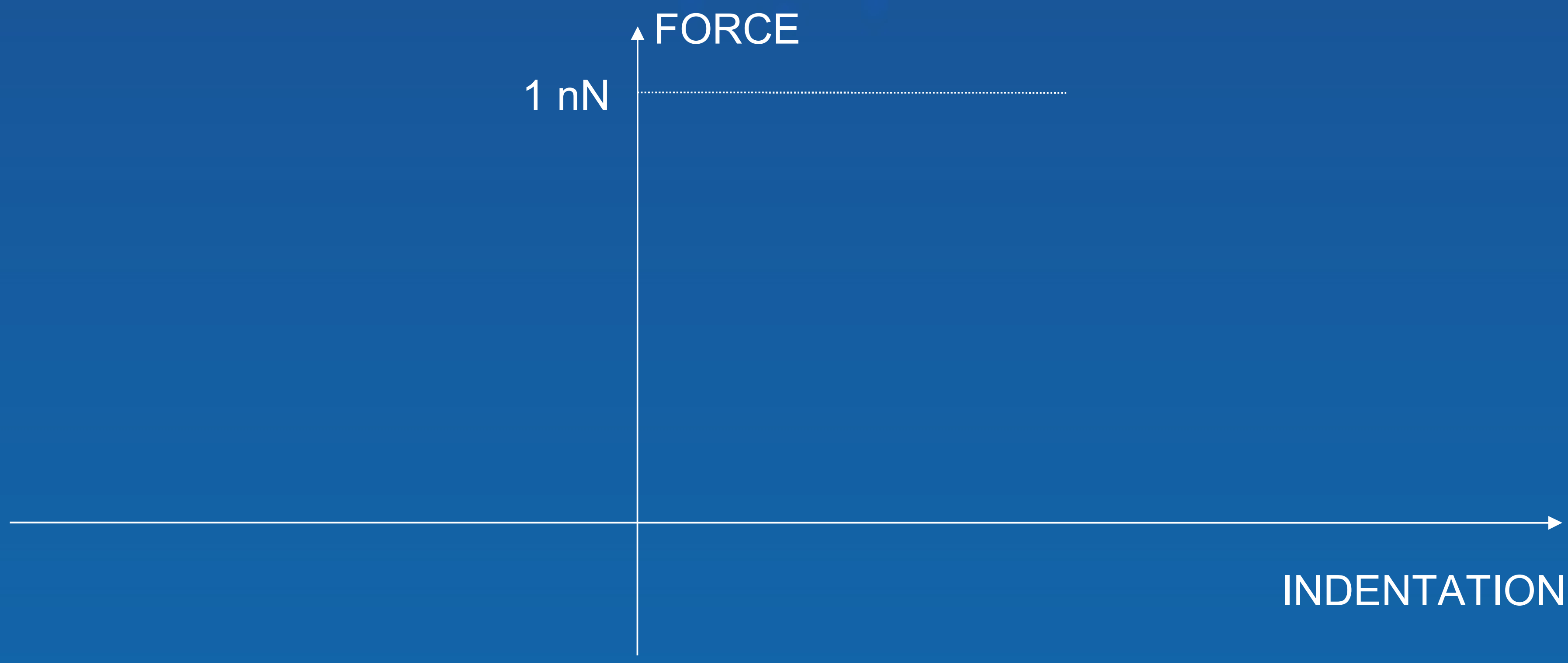
Science Surface Lab.

Fit for Van der Waals

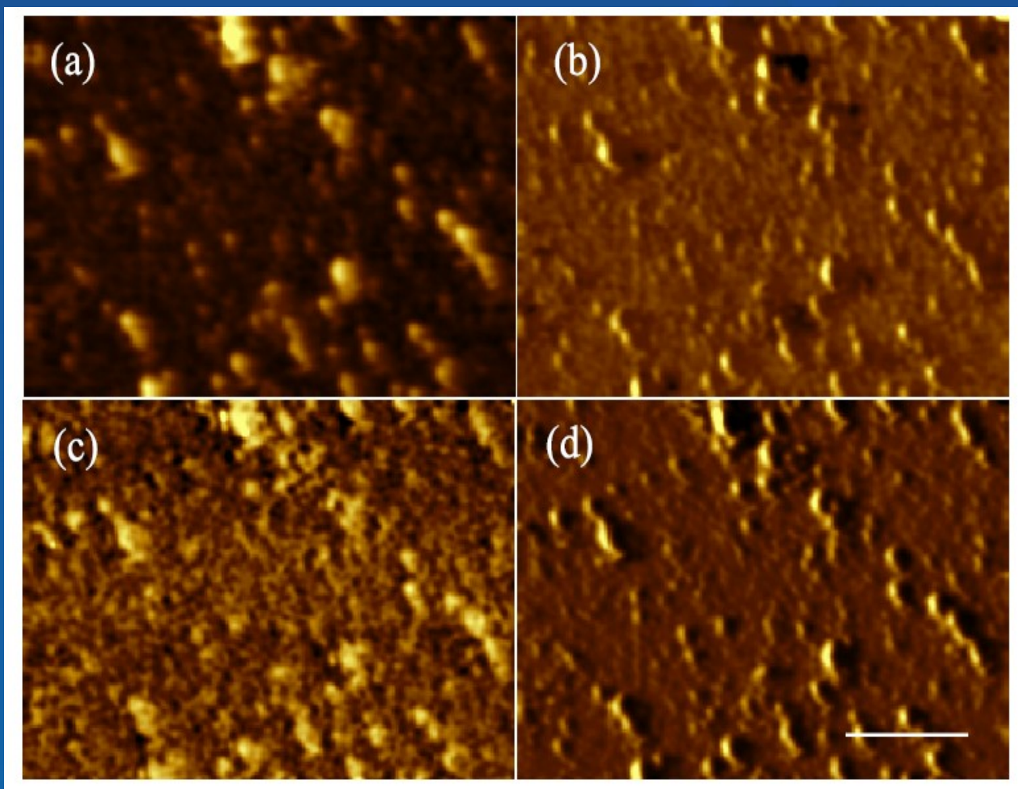


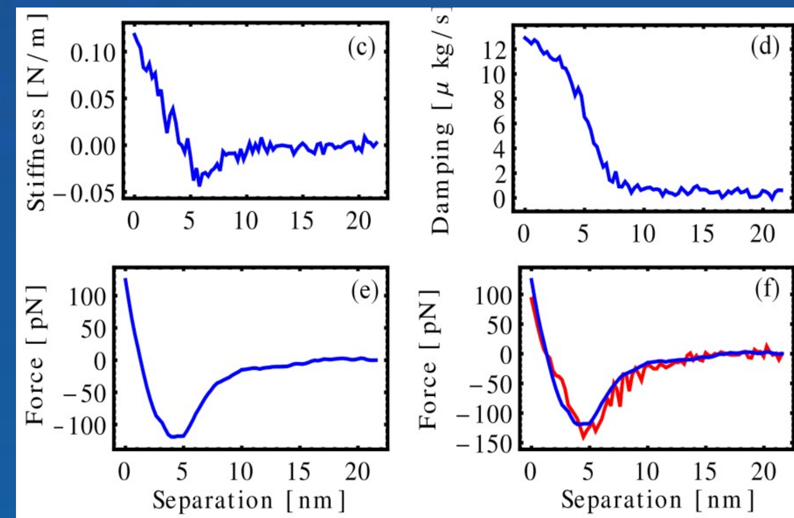
Additional Slides

Rupture cell membrane



Additional Slides





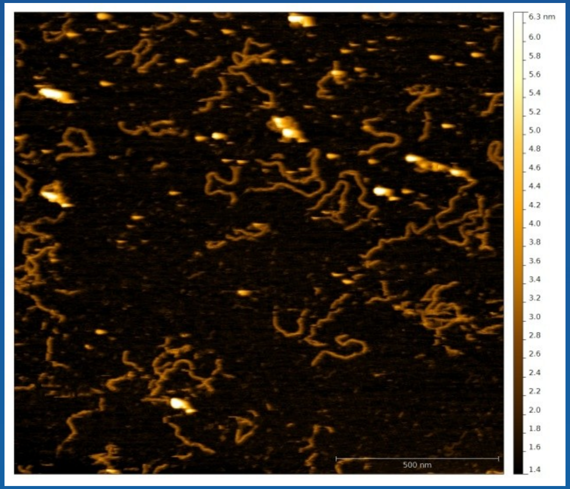
Tank Binding Kinase (TBK1) and Optineurin (OPTN) protein complexes on mica in 20 mM HEPES and 5 mM $MgCl_2$

(a) topography, (b) force, (c) stiffness, (d) damping

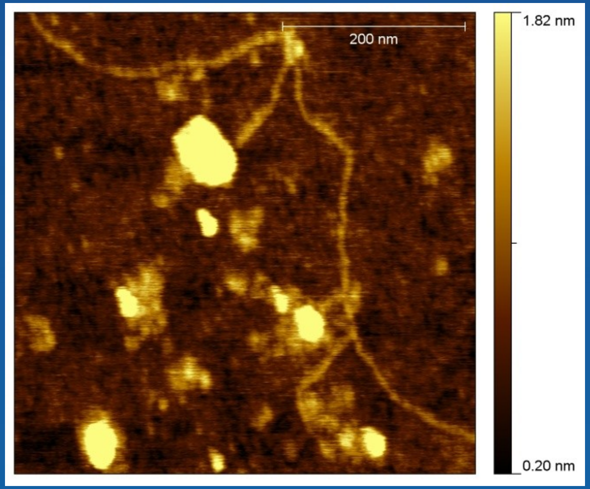
The full color scale is a) 24nm, b) -300pN, c) -0.04 N/m, d) 5 μkg/s

Additional Slides

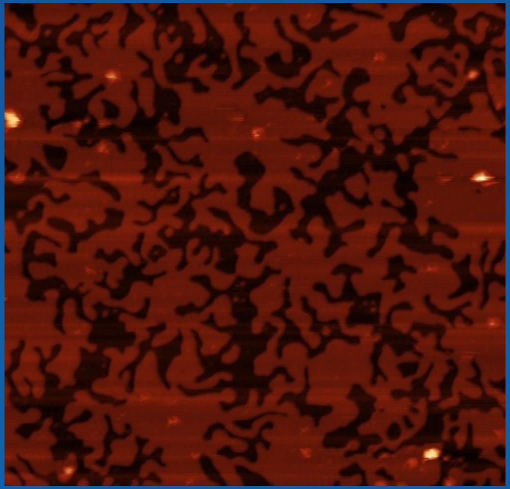
Biomolecules images with a commercial AFM (Asylum MFP3D)



DNA



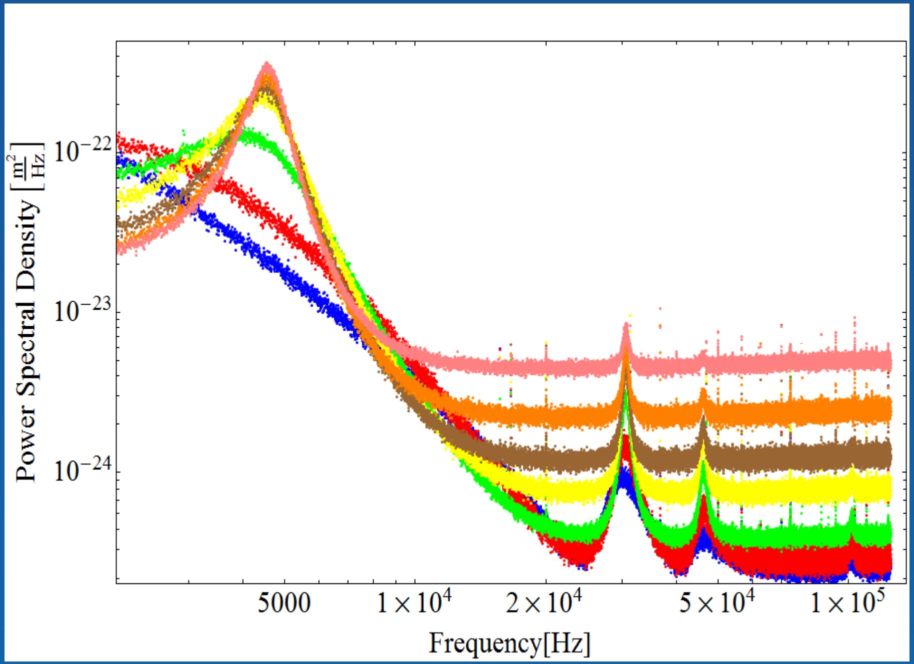
Proteins
TBK1 OPTN

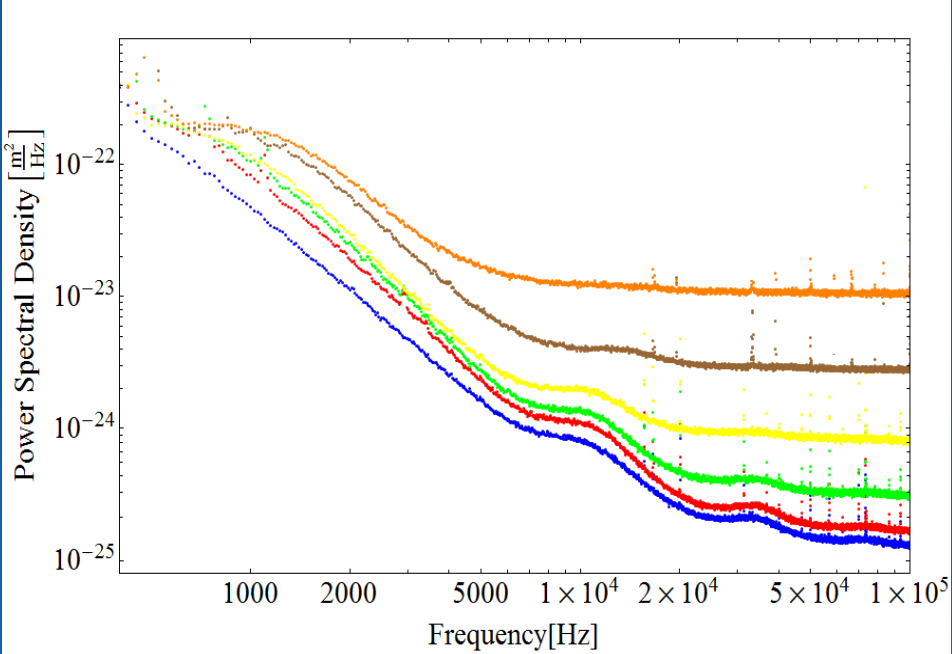


LIPIDS

Additional Slides

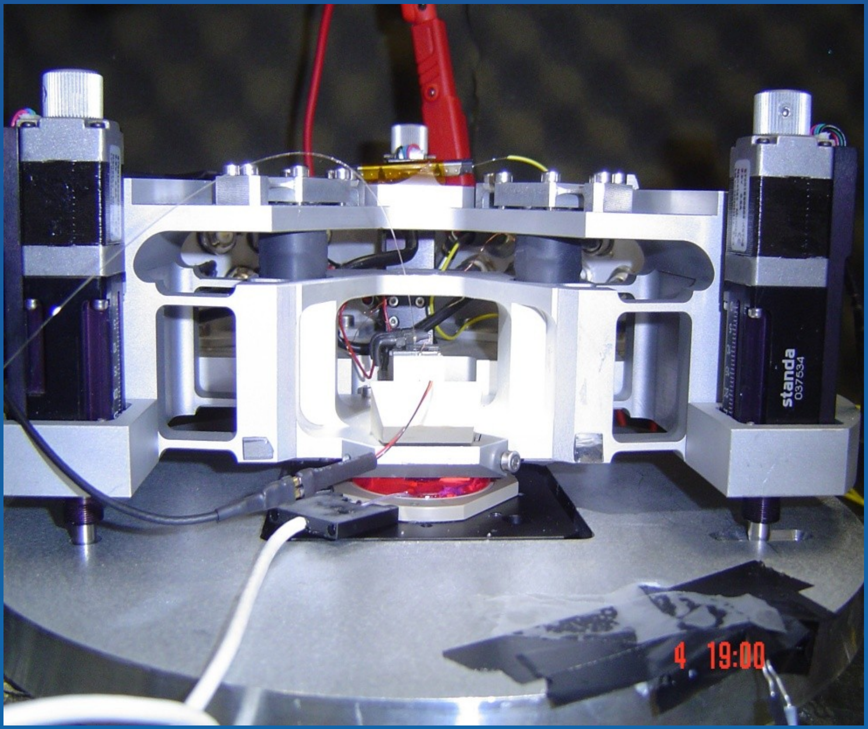
Tip power spectral density in air and liquid as a function of the tip-fiber distance

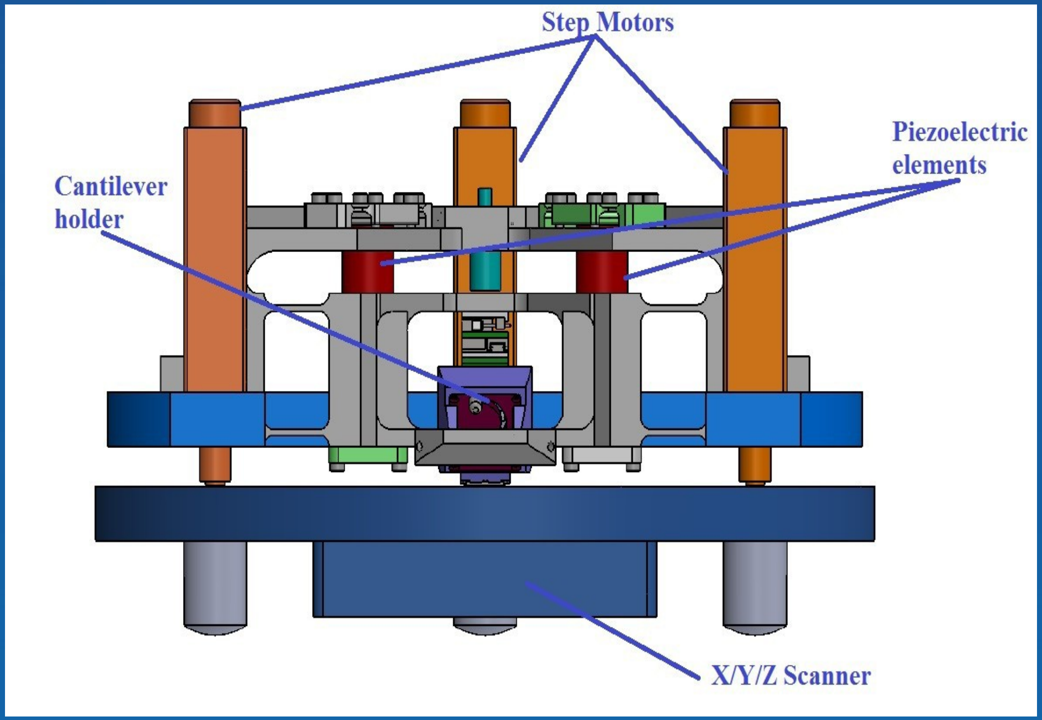




Additional Slides

The Force Feedback Microscope





Additional Slides

Another FFM image on PC12 living cells