The Force Feedback Microscope: an AFM for soft condensed matter

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Outline

- Motivation

- The Force Feedback Microscope

- Results

- Theory

- Conclusions
...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

Motivation
Instrumentation
Results
Theory
Conclusions

The general idea
Setup
The fiber optic based Interferometer
The Microscope
Dynamic FFM
THE IDEA

\[ \sum_i F_{i/tip} = 0 \]

Sum of the force acting on the tip equal to zero

\[ F_{\text{feedback}} = F_{\text{interaction}} \]

Position of the tip fixed in space
Where
\[ F_{\text{feedback}} = F_{\text{interaction}} \]

\( X_t \) is kept constant by a feedback loop.
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SETUP

Mr. Sample

Mr Piezo

European Synchrotron Radiation Facility
20/01/2014
Luca Costa
SETUP

Mr. Sample

F_{interaction}

Mr. Piezo

F_{feedback}
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SETUP
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


Mario S. Rodrigues Post-doc 2010-2011
**DYNAMIC FFM**

Tip oscillation

\[ X_{\text{tip}}(t) = X_{\text{tip}} + \Delta X_{\text{tip}} \cos(\omega t) \]
DYNAMIC FFM

RAW DATA

RAW DATA → 🤔
Force gradient & Dissipation

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**Force**

\[ F_{static} = \int \nabla F \, dz \]

<table>
<thead>
<tr>
<th>Sample position [nm]</th>
<th>Stiffness [N/m]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.1</td>
</tr>
<tr>
<td>20</td>
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<td>100</td>
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</tbody>
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<table>
<thead>
<tr>
<th>Sample position [nm]</th>
<th>Force [pN]</th>
</tr>
</thead>
<tbody>
<tr>
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<td>-150</td>
</tr>
<tr>
<td>20</td>
<td>0</td>
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</tr>
</tbody>
</table>

Force

\[ F_{\text{static}} = \int \nabla F \, dz \]

**Results**

- The fiber optic based Interferometer
- The Microscope
- Dynamic FFM

Solid/liquid interface

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

Solid/air interface

HYSTERESIS DUE TO THE SYSTEM

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

Imaging modes applied to the study of soft biological samples
Phospholipids DSPE

Excitation frequency of the tip not linked to resonance

Young modulus $E \approx 7$ MPa

DNA

Constant Force

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

Excitation frequency of the tip not linked to cantilever resonance

Constant Dissipation

(a) 
(b) 
(c) 
(d) 

Living Cells PC12
TUNABLE EXCITATION FREQUENCY

Viscoelastic spectroscopy
Candidate: Living cell PC12, in MEM - 1% Strepto-Penicillin
Static indentation

Quasi-static conditions: Young modulus \( \approx 1 \text{ kPa} \)

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

**BLUE**
Data

**RED**
Theory
At 10 kHz, Young modulus $\approx 30$ kPa

Blue $= 1.13$ kHz
Red $= 5.13$ kHz
Green $= 7.13$ kHz
Black $= 11.13$ kHz

At 10 kHz, Young modulus $\approx 30$ kPa
Mapping mechanical properties

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\( \omega = 2.25 \text{ kHz} \)

\( \omega = 13.25 \text{ kHz} \)
Feedback loop

Motion of the tip described by

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

\[ F_{\text{control}} = -g_P x - g_D \dot{x} - g_I \int_0^t x \, dt \]
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

Stability conditions

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

\[ k_t = k + k_{ts} \]

\[ \omega_0^2 = \frac{k}{m} \quad Q = \frac{\omega_0}{\Delta\omega} \]

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Stability conditions

\[ g_P > -k_t \]  Mechanical stability of the tip

\[ g_l < \frac{\omega_0}{3Q}(k_t + g_P) \]  under - damped

\[ g_l < Q\omega_0\frac{(k_t + g_P)^2}{k} \]  over - damped

Dynamic FFM

Cantilever Transfer Function depends on PID gains
\( F_\omega \) is the harmonic excitation

\[ A = \frac{F_\omega}{\sqrt{[(k_t + gP) - m\omega^2]^2 + [(\gamma + gD)\omega - \frac{gI}{\omega}]^2}} \]

\[ \phi = \arctan \left[ \frac{(\gamma + gD)\omega - \frac{gI}{\omega}}{(k_t + gP) - m\omega^2} \right] \]

Master equations

**Force gradient**

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

**Dissipation**

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

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

\[ \eta = \frac{F_{\omega}}{F_{\omega \infty}} \]

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

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

Limits

**Speed:** defined by the maximum integral gain employed

\[
g_I = \frac{\omega_0}{3Q} (k_t + gP) \quad \text{under-damped}
\]

\[
g_I = Q \omega_0 \frac{(k_t + gP)^2}{k} \quad \text{over-damped}
\]

**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

\[ F_{\text{static}} \neq \int \nabla F(\omega = \omega_R) dz \]

- 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
Approach-Retract on HOPG

![Graph showing force vs. piezo motion](attachment://graph.png)
Additional Slides

Fit for Van der Waals

\[ F = \frac{-HR}{6z^2} \]
Additional Slides

Rupture cell membrane

FORCE

1 nN

INDENTATION
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
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