The utilization of AFM’s tip-sample interaction for the surface morphology imaging and mechanical properties mapping

Andrzej Sikora
The utilization of AFM’s tip-sample interaction for the surface morphology imaging and mechanical properties mapping

Starting from the principles...

SPM is about the tip–sample interaction observation

Force

Mechanical properties
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Starting from the principles...
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Detecting tip-sample interaction

Retraction

e) Adhesion
\[ F = -3 \pi R \gamma \]
f) Capillary force
\[ F = 4 \pi R \gamma \cos \theta \]
g) Polymer extension
\[ F(x) = \frac{kT}{a} L \left( \frac{x}{Na} \right) \]
h) Binding
\[ F = \frac{U - kT \ln(\tau / \tau_0)}{A} \]
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Enabling semicontact / phase imaging
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**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 derivative

Magonov S.N.S., Elings V., Whangbo M.-H.
Phase imaging and stiffness in tapping-mode atomic force microscopy

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

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

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

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

\[
E_{dis} = \int F_{ts} \frac{dz}{dt} \, dt 
\]

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
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Phase imaging used for topography imaging / alcane
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Phase imaging – polymerization map
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Phase imaging – polymerization map
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**Phase imaging vs force spectroscopy**
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Phase imaging – there are issues...
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Phase imaging – there are issues...
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Every tap you make...
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Using DMT model to analyze signal...

time domain

- 10 GPa
- 5 GPa
- 1 GPa

FFT
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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
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Testing the approach on PS-LDPE

2 MPa

0.5 MPa
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...and multiplying the solution
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Interesting differences can be revealed

<table>
<thead>
<tr>
<th>Topography</th>
<th>1. harmonic</th>
<th>3. harmonic</th>
<th>5. harmonic</th>
<th>7. harmonic</th>
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<tr>
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<td><img src="c.png" alt="Image" /></td>
<td><img src="d.png" alt="Image" /></td>
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</table>

<table>
<thead>
<tr>
<th>Phase imaging</th>
<th>9. harmonic</th>
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<th>13. harmonic</th>
<th>15. harmonic</th>
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<tr>
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<td><img src="g.png" alt="Image" /></td>
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<td><img src="i.png" alt="Image" /></td>
<td><img src="j.png" alt="Image" /></td>
</tr>
</tbody>
</table>

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)
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Different sensitivities can be seen for acquired harmonic maps
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Example of stiffer surfaces...

1. Silicon (approx. 110 GPa)
2. Aluminum (approx. 69 GPa)
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...looking at harmonic maps

topography

phase imaging

1. harmonic

3. harmonic

5. harmonic

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

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

**Harmonic oscillator**

\[ F_p \cos(\omega t) \]

\[ k \]

\[ \gamma \]

\[ \text{masa} \]

\[ F_{ts} \]

**Setup transmittance**

\( \omega_0 \) – resonance pulsation

\( k \) – spring constant

\( Q \) – resonance quality
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**How to remove low frequency flexural resonance**

Sarioglu A.F., Solgaard O.
Cantilevers with integrated sensor for time-resolved force measurement in tapping-mode atomic force microscopy
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**Flexural and torsional mechanical response of the cantilever**

flexural oscillations
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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
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So maybe T-shaped probe...

Sahin O., Magonov S., Su C., Quate C.F., Solgaard O.
An atomic force microscope tip designed to measure time-varying nanomechanical forces
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...so we could acquire interaction force

\[ s = CG(f_{\text{drive}} + f_{\text{tip}}) \]

\[ s_X = C_X G_X (f_{\text{drive}} + f_{\text{tip}}) = H_X (f_{\text{drive}} + f_{\text{tip}}) \]

\[ s_Y = C_Y G_Y f_{\text{tip}} = H_Y f_{\text{tip}} \]

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

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

Sahin O., Atalar A., Quate C.F., Solgaard O.
Harmonic cantilevers and imaging methods for atomic force microscopy
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
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So finally it works like this
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Implementation using standard AFM

Atomic Force Microscope
- TR+TL-BR+BL high bandwidth path
- BL+TL-BR+TR low bandwidth path

AFM controller NanoDrive
- software signal crosspoint

PXi module
- 2.53 GHz Intel Core 2 Duo T9600 dual-core processor
- 60 MS/s real time sampling rate ADC

LAN interface
USB interface
multi-signal connector
analogue signals
digital signals

out aux 1
out aux 2
SYNC
ADC
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Raw torsional signal
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...acquired in various spots
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Reconstruction of the force spectroscopy curve

Sikora A., Bednarz Ł.,
Mapping of the surface’s mechanical properties due to analysis of torsional cantilever bending in dynamic force microscopy
Nanoscience and Technology / Acoustic Scanning Probe Microscopy, Springer 2012
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Reconstruction of the force spectroscopy curve

2 MPa
0.5 MPa
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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
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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)
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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
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**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
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I can’t get no interaction...

da) 

c) 

e) 

b) 

d) 

f) 

too close  OK  too far
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Epoxy resin with silica nanofiller

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
<th>Weight%</th>
<th>Atomic%</th>
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<tbody>
<tr>
<td>C K</td>
<td>54.30</td>
<td>65.82</td>
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<tr>
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<td>Mg K</td>
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<td>Al K</td>
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<td>Si K</td>
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<tr>
<td>Cl K</td>
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<tr>
<td>Totals</td>
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</tbody>
</table>
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**Calcium deposits from human vains**

- **Topography**
- **Phase imaging**
- **Adhesion**
- **Peak force**
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Manganese cathode materials for lithium ion batteries

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Indication of the different indentation of the surface

topography

topography correction map
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Tip-sample interaction information may have metrological application

Sikora A., Bednarz L.:
Direct measurement and control of peak tapping forces in atomic force microscopy for improved height measurements
Measurement Science and Technology, 22 (9), p. 94005, 2011
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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

Łukasz Bednarz, M.Sc.
Thank you for your attention