An introduction to Atomic Force Microscopy
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An introduction to
Atomic Force Microscopy

Olivier ARNOULD

WOOD TEAM
Laboratory of Mechanics and Civil Engineering (LMGC)
CNRS/Université de Montpellier
France
Outline

- Brief history and basic principles of scanning probe microscopy
- AFM principle and typical set-up
  - Tip probe-sample surface interactions
  - Tip and cantilever
  - Deflection sensor and scanner
  - Whole set-up
- Contact mechanics
- Operating modes
  - Contact
  - Intermittent
  - Force-distance curve and mechanical characterisation modes
- Image analysis and artefacts
- Sample preparation
Scanning Probe Microscopy

IDEA: To measure (and use) the local (near field) probe (sharp tip)-sample interactions to map the surface morphology (topography) and physical properties

- ✓ Image resolution limited by probe-sample interaction volume, not by diffraction!
- ✓ Interaction can modify surface → nanolithography possible
- ❌ Scanning technique (quite) slow
- ❌ Limited maximum image size

All data in SPM are subjected to interpretation
Optical stylus (optical/light lever) profilometer by Schmalz in 1929
≈ 25 nm vertical resolution! (magnification > 1000x)
Scanning Tunnelling Microscopy (STM) by Binnig and Rohrer in 1981 (Nobel Prize in 1986)

**Drawback**
need conductive material and high vacuum!

HOPG atoms view by STM [www.ntmdt.com]
History - SPM timeline

1972  Russel Young's topografiner
1981  First STM results in the lab by Binnig, Rohrer and Gerber
1982  First publication - PRL
1984  Near field Optical Microscope
1985  Invention of AFM at Stanford by Binnig, Gerber and Quate
1986  Nobel Prize to Ruska, Binnig and Rohrer
1987  Dynamic AFM by Wickramasinghe
1987  First commercial instruments – spin-off from Quate Group in Stanford (Park Scientific) and Hansma Group in UCSB (Digital Instruments)
1989  IBM in atoms, by Don Eigler
1990  First microfabrication of AFM tips by Albrecht et al.

Topographic map of ruled diffraction grating

Constant current or constant height imaging
History - SPM timeline

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Image of Xenon atoms moved on Nickel (110) surface using an STM tip

Topography of CaIrSn₄ (110)
Scanning Probe Microscopy

**SPM**

- **STM** (Scanning Tunneling Microscopy)
  - Tunneling current

- **AFM** (Atomic Force Microscopy)
  - Local interaction forces (Van der Waals, elastic, electrostatic, friction, adhesion…)

- **SNOM** (Scanning Near-field Optical Microscopy)
  - Evanescent optical waves

**Other Techniques**

- Nanoindentation
  - Mechanical modes

- **EFM** (Electrostatic Force Microscopy)

- Conductive AFM (C-AFM)

- **SThM** (Scanning Thermal Microscopy)

- **Chemical Force Microscopy**

**A question of probe tip (apex)!”**
Brief history and basic principles of scanning probe microscopy

**AFM principle and typical set-up**
- Tip probe-sample surface interactions
- Tip and cantilever
- Deflection sensor and scanner
- Whole set-up

Contact mechanics

Operating modes
- Contact
- Intermittent
- Force-distance curve and mechanical characterisation modes

Image analysis and artefacts

Sample preparation
Probe tip-sample interactions

- Tip (feels the surface)

Apex radius $R \approx 2\text{-}200\text{nm}$

Height $H \approx 10\mu\text{m}$

Coating (Pt, CoCr, $W_2C$, diamond, etc), functionalization, …

→ www.nanoandmore.com/afm-probes
The Lennard-Jones potential

\[ F = \frac{\partial V}{\partial r} \]

Energy (a.u.)

Repulsive: \( +A/r^{12} \)
Short range coulomb interaction (Pauli repulsion) - Strong

Attractive: \( -B/r^{6} \)
Van der Waals dipole-dipole electrostatic - Weak

Well depth

Equilibrium

 Probe tip-sample interactions
Probe tip-sample interactions

Capillary force

\[ F_{\text{cap}} \approx \frac{4\pi R \gamma \cos \theta}{(1 + d/h)} \]
\[ \cos \theta = \frac{\cos \theta_1 + \cos \theta_2}{2} \]

Water meniscus

Surface tension of water: \( \gamma = 72 \text{mJ/m}^2 \)

Köber et al, Small 2010, 6, 2725


Max at snap (pull) out

\[ F_{\text{cap}} = 4\pi R \gamma \]
Probe tip-sample interactions

\[ F_{t/s} = F_{E/M} + F_{cap} + F_{VdW} + F_{rep} \]

Tip-surface Gap (z)

**Electrostatic force** \((1/z^n; 1<n<2)\) : Long range interaction => electric modes

\[ F_{\text{elect}} = \frac{1}{2} \frac{\partial C}{\partial z} V^2 \leq \frac{1}{2} \frac{\varepsilon_0 \varepsilon_r S}{z^n} V^2 \]

**Capillary Force**

\[ F_{\text{cap}} = f(\gamma, R, \theta_{p/eau}, \theta_{eau/surf}, z) \]

**Van der Waals Force**

\[ F_{VdW}(z) = \frac{HR}{6z^2} \]

**Repulsive Force** \((m>3)\)

\[ F_{\text{repulsion}}(z) = \frac{A}{z^m} \]
AFM principle and typical set-up

- Cantilever (spring, load sensor)
  - Stiffness $k_C$: 0.01 – 100 N/m
  - Length $L$: 100-300 µm
  - Width $w$: 10-50 µm
  - Thickness $e$: 0.3-4 µm
  - Coating

$\rightarrow$ www.nanoandmore.com

+ mounting on the holder 😱

Calibration!
AFM principle and typical set-up

- Photodetector (deflection sensor)
  Photodiode + geometry + laser position sensitivity ≈ 10 nm/V
  → minimum measurable deflection < 0.1 nm!

Calibration!

(A+B) - (C+D) = 0  
no deflection

(A+B) - (C+D) > 0  
deflected up
AFM principle and typical set-up

- **Scanner**

Typical piezo-response ≈ 0.1 nm/V (0.01-10 nm/V)

mV control → displacement step < 0.1 nm!
AFM principle and typical set-up

The information collected by the SPM is stored as a two-dimensional file of integer numbers $a_{ij}$ (square matrix in general)

Commonly $256 \times 256$ or $512 \times 512$ elements

The image spatial resolution is conditioned by the digitalisation

Example: Image size $10 \mu m$  \[ \Delta X = \frac{10\mu m}{511} = 0.02\mu m = 20nm \]

Slow-scan axis

Fast-scan axis

Trace/Retrace

Up/Down

Rotation
AFM principle and typical set-up

- Whole set-up
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Contact mechanics

- **(Depth-sensing) Nanoindentation**
  - The depth of penetration $h$ is measured during load application mainly with a Berkovich indenter (3-sided pyramid)

![Nanoindentation curve on electrodeposited Ni](www.microstartech.com)

![Diagram of Nanoindentation](image)
Contact mechanics

- (Depth-sensing) Nanoindentation

\[
E^* = \frac{\sqrt{\pi}}{2} \frac{dP}{dh} \frac{1}{\sqrt{A_c}}
\]

With \( E^* \) or \( M \) reduced/contact/indentation modulus

\[
\frac{1}{E^*} = \frac{1-v_i^2}{E_i} + \frac{1-v^2}{E}
\]

for linear **ISOTROPIC** material

- Case of the Berkovich indenter

  [A.C. Fischer-Cripps, *Vacuum*, 2000]

Berkovich

\[
A = 24.5 h^2 \quad \alpha = 70.3^\circ
\]

Cone

\[
A = \pi \tan^2 \alpha h^2
\]

\[
h_c = h_t - \varepsilon P \left( \frac{dP}{dh} \right)^{-1} \quad \text{with} \quad \varepsilon = \left( \frac{2(\pi - 2)}{\pi} \right) 0.75
\]

cone

Berkovich
Contact mechanics – NI vs AFM

NEAR SURFACE measurements
\[ \rightarrow \text{sample surface preparation}! \]

Sink-in
[Jakes et al., 2009; Stanzl-Tschegg et al., 2009]

\[ \delta < 10 \text{ nm} \]
\[ \delta \approx 400 \text{ nm} \]

Elastically affected

S1

\[ S2 \approx 4 \mu m \]

S3
Contact mechanics without adhesion

\[ F = \frac{4}{3} M R_{eq}^{1/2} \delta^{3/2} \]

\[ k_N = \left( 6 M^2 R_{eq} F \right)^{1/3} \]

\[ \delta \ll R \]

Non conforming

\[ \delta \approx R \]

\[ \delta \gg R \]

**LINEAR ELASTIC HOMOGENEOUS and ISOTROPIC materials**

\[ F = \frac{2}{\pi} M \tan \theta \delta^2 \]

\[ k_N = \left( \frac{32}{\pi^3} M \tan \theta F \right)^{1/2} \]

\[ F = 2MR \delta \]

\[ k_N = 2MR \]

\[ \frac{1}{M} = \frac{1 - \nu_{Tip}^2}{E_{Tip}} + \frac{1 - \nu_{Sample}^2}{E_{Sample}} \]
Contact mechanics + adhesion (capillary, Van der Waals, …)

**DMT Model:**

\[
k_{N_{DMT}} = \left(6M^2R (F + F_{ad_{DMT}})\right)^{\frac{1}{3}}
\]

Adhesive force \( F_{ad_{DMT}} = 2\pi Rw_{ad} \)

Used for hard samples, low adhesion force and low tip radius R

**JKR Method:**

\[
k_{N_{JKR}} = 2Ma_{JKR} \quad \frac{1-\left(\frac{a_{0_{JKR}}}{a_{JKR}}\right)^{\frac{3}{2}}}{1-\frac{1}{6}\left(\frac{a_{0_{JKR}}}{a_{JKR}}\right)}
\]

with

\[
b_{JKR} = \left[\frac{3RF_{ad_{JKR}}}{4M}\left(\sqrt{F_{ad_{JKR}}} + \sqrt{F_{ad_{JKR}} + F}\right)^2\right]^{\frac{1}{3}}
\]

\[
a_{0_{JKR}} = \left[\frac{3RF_{ad_{JKR}}}{M}\right]^{\frac{1}{3}}
\]

\[
F_{ad_{JKR}} = \frac{3}{2}\pi Rw_{ad}
\]

At pull-off \( a \neq 0 \) and \( F = -F_{ad} \)

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

For a typical S2-layer with MFA~0° [Jäger et al, 2011]

\[ M_L \approx 19 \text{ GPa whereas } E_L \approx 45 \text{ GPa} \]

\[ (E_t \approx E_r \approx 12 \text{ GPa}, \nu_{tl} \approx \nu_{rl} \approx 0.028, \nu_{rt} \approx 0.28 \]

\[ G_{tl} \approx G_{rL} \approx 2.5 \text{ GPa, } G_{rt} \approx 2 \text{ GPa) } \]

Kevlar fibre: \( M// \approx 15-20 \) / \( E// \approx 80 \text{ GPa} \) [Arnould et al, 2017]

in particular case…

→ transversely isotropic // contact surface

[Hanson, J. Appl. Mech., 1992]

\[ M_3 = 2 \sqrt{\frac{C_{11}C_{33} - C_{13}^2}{C_{11}}} \left( \frac{1}{C_{44}} + \frac{2}{\sqrt{C_{11}C_{33} + C_{13}}} \right)^{-1} \]

1=2: \( C_{22}, C_{23}, G_{13}=G_{23} \)

[Delafargue et al., Int. J. Sol. Struct., 2004]
Contact mechanics – Fibre cell wall anisotropy

Nanoindentation on Spruce (S2-AMF? + CCML ~ 5-10GPa)
[Wimmer et al., Wood Sci. Tech., 1997]

Nanoindentation on Spruce (S2 + CCML~7-12GPa)

Nanoindentation on Pine (S2)
[Tze et al., Comp. A, 2007]

Anisotropic indentation model (7,5-15%)
[Jäger et al., Comp. A, 2011]

Nanoindentation on Spruce
[Konnerth et al., J. Mater. Sci., 2009]
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Van der Waals force, electrostatic force, contact stiffness, adhesion force, …

Scanning at constant force/height

[B. Nysten, 2007]
Contact mode

- Constant height or force and friction

±Fast scan speed in constant force mode (topography/closed-loop feedback)
Limited topography in height force mode
Tip wear, effect of sample elasticity
~Poor resolution (surface deformation, large tip surface contact area)

CD disk surface
[www.ntmdt.com]

PS-PMMA polymer blend

Cantilever with low stiffness (0.01-1 N/m)
Contact mode

- **Force Modulation (FMM) ~ DMA**
  - Few nm oscillations @ few kHz, longitudinal or transversal!
  - Cantilever vibration amplitude and phase-shift related to surface elastic and viscoelastic properties
  - Same limitations as F-d curves but better resolution (≈100 nm on polymer) and lower imaging time (scan rate <0.5 line/s)

Topography and force modulation images (900 nm scans) of a two-phase block copolymer (softer = black)

[www.afmuniversity.org] [www.veeco.com]
IDEA: To probe the local elastic stiffness of the tip-sample system by means of cantilever’s resonance frequency in contact mode at reduce applied force.

\[ F = F_0 + F_{\text{excitation}} \times \sin \omega t \]

if \( F_{\text{excitation}} \) is small \( \Rightarrow \) \( \approx \) LINEAR contact stiffness

if \( F_0 \) is small \( \Rightarrow \) Good spatial resolution + Hertz theory validity

Point-mass model [Rabe et al, 1996]

\[ f_0 = \frac{1}{2\pi} \sqrt{\frac{k_c + k_N}{m^*}} \quad \text{with} \quad m^* = \frac{1}{4} m + m_{\text{tip}} \]

Amplitude of vibration and resonance frequency depend on the contact stiffness \( k_N \)… but how?
Contact Resonance-AFM (CR-AFM)

Cantilever dynamic and response

[Rabe et al, 1996]
Contact Resonance-AFM principle with Dual Resonance Frequency Tracking

[Arinero and Lévêque, Rev. Phys. Instr., 2003]

[Gannepalli et al., Nanotechnol. 2011]

Sinusoidal excitation

Sample holder + US piezo

Lock-in amplifier

acquisition frequency sweep

DRFT

Mapping

Calibration $f_i > M$

frequency spectra
Chestnut tension wood

[Arnould and Arinero, Comp. A, 2015, 74: 79-76]

Effect of the topography and sample orientation

[Konnerth et al, J. Mater. Sci., 2009]

Veeco Enviroscope, CR-AFM, Nanoworld ARROW FMR, 2,8 N/m, 55 nm (adh), 180 nN
Chestnut tension wood

[Arnould and Arinero, Comp. A, 2015]

Effect of sample orientation
[Konnerth et al, J. Mater. Sci., 2009]
Topography (nm)

Simplified model
AMF(S2) ~20° et tilt ~ -20°

Cutting angle / MFA $\alpha$ (°)

[Shaune et al, Holzforschung, 1994]
Topography (nm)

Simplified model
AMF(S2) $\sim 20^\circ$ et tilt $\sim -20^\circ$

Cutting angle / MFA $\alpha$ ($^\circ$)

Contact modulus (GPa)
Abraiona

Starch granules inside a wheat endosperm (SEM)

Occurrence of “lubricated” interface in soft wheat

Tablet of pure gluten before/after abrasion (SEM)

Chichti et al., Plant Sci., 2015, 239: 1-8
**Contact mode**

- **Scanning Thermal Microscopy (SThM)**
  
  [Konnerth *et al.*, Holzforschung, 2008]

  \[ R \approx 100 \text{ nm} \]

  Scan rate 0.1-0.5 Hz

  Spruce wood

  **Probe Current**

  CCM-mode

  **SThM Error**

  TCM-mode

  Low      high

  20µm

  embedding material (SPURR)

  PRF adhesive
Van der Waals force, electrostatic force, contact stiffness, adhesion force, …

[B. Nysten, 2007]
Intermittent contact/tapping™/...

- Knocking on the surface!
  - Linear harmonic oscillator approximation (free)

\[ \omega_0 = \sqrt{\frac{k_C}{m_{\text{eff}}}} \]

\[ k_C > 10 \text{N/m} \]

\[ f_0 > 100 \text{kHz} \]

<table>
<thead>
<tr>
<th>Media</th>
<th>Liquid</th>
<th>Air</th>
<th>High vacuum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q</td>
<td>10</td>
<td>400</td>
<td>10^4</td>
</tr>
</tbody>
</table>

\[ f_0/Q = 2f_1 \]
Scan at constant vibration amplitude = topography + stiffness contrast
→ soft (topography) $A_s/A_0 > 0.8 + A_0↓$ / hard (elasticity) $A_s/A_0 < 0.4 + A_s↑$ tapping

Linear harmonic oscillator approximation in a force gradient

What are we measuring?

- Linear harmonic oscillator approximation in a force gradient
  \[ \Delta \varphi \approx \frac{Q}{k_c} \frac{\partial F}{\partial z} \text{ if } \omega \sim \omega' \sim \omega_0 \text{ and } \frac{\partial F}{\partial z} \ll \frac{k_c}{Q} \]
  Pure elastic material with Hertz: \( F = \frac{4}{3} E^* \sqrt{Rz}^{3/2} \rightarrow \frac{\partial F}{\partial z} \approx 2E^*a \)

- Phase-lag proportional to the contact stiffness in certain condition
  \( k_N \ll k_c/Q + A_0 \uparrow \text{ and } A_s \downarrow \) → phase images…
  but more generally “proportional” to any dissipation (capillarity, viscoelasticity, adhesion, …)
  difficult to interpret quantitatively!

- High resolution (<10nm) and \(+slow\) imaging (cantilever with high frequency)
- Less tip and sample damages, ~no friction, …
Intermittent contact/tapping\textsuperscript{TM}/…

Spruce tracheid wood cell wall, AFM probe and condition?

Intermittent contact/tapping™/...

Tapping mode (phase image)
\( R \approx 4.25 \text{ nm} \)
\( k_C \approx 40 \text{ N/m} \)
\( f_0 = 300 \text{ kHz} \)
Spruce pulp fiber

[Fahlén and Salmén, Biomacromolecules, 2005]
Van der Waals force, electrostatic force, contact stiffness, adhesion force, …
- Decoupling load/displacement

![Graph showing deflection vs. Z for hard and soft samples]

[www.veeco.com]
Requirements:

- Cantilever stiffness

\[
\frac{d}{z} = \frac{1}{1 + \frac{k_c}{k_N}} \\
\begin{align*}
k_N \gg k_C & \rightarrow d = z \\
k_N \ll k_C & \rightarrow d = 0
\end{align*}
\]

- Calibration of the laser/photodetector
- Calibration of the cantilever stiffness
- Measurements of the real tip apex shape + **suitable** contact model…
- Non-normal load (shear) due to cantilever tilt + tip sliding?
- Limited resolution (>100nm on polymer) due to large tip surface contact area

Mapping (Force-Volume mode) of the elastic properties is very time consuming (256 x 256 points = 18h)

→ Force Modulation Mode… PeakForce QNM, QI, etc
Fd - Indentation

- Peak Force Quantitative NanoMechanical (QNM)
  
  [www.bruker-axs.com]
  
  force curve recording at some kHz with sinusoidal displacement

- Calibration!
  
  DMT modulus
  Dissipation (hysteresis loop)
  Adhesion force
Kevlar® Dupont™ K48 fibres

[Arnould et al, Ind. Crops Prod, 2017]


Topography

Contact modulus

Core:
$E_l = 84 \text{ GPa} / E_t = 3 \text{ GPa}$

Skin:
$E_l = 85 \text{ GPa} / E_t = 0,2 \text{ GPa}$

$G_{lt} = 1,8 \text{ GPa} / G_{tt} = 1,2 \text{ GPa}$

$\nu_{lt} = 0,6 / \nu_{tt} = 0,25$

$M_{\text{core NI}} = 22,8 \pm 1,7 \text{ GPa}$

$(150 \text{ nm @ } 1 \mu\text{N/s} + 20 \text{ s} + 10 \mu\text{N/s})$

$M_{\text{core calc}} \approx 21 \text{ GPa}$

$M_{\text{skin calc}} \approx 17 \text{ GPa}$

MultiMode 8, PF-QNM, Bruker RTESPA-525, 139 N/m, 32 nm (HOPG), 200 nN
Flax fibres for bio-composites


$E_i$ fibre $\approx$ 50-60 GPa !

$M_{Ni}^{fibre} = 21.3 \pm 2.2$ GPa

(150 nm @ 1 $\mu$N/s + 20 s - 10 $\mu$N/s)

Effect of processing temperature and fibres confinement in the composite matrix


Indentation modulus

$140^\circ$C

$250^\circ$C

MultiMode 8, PF-QNM, Bruker RTESPA-525, 139-200 N/m, 9-50 nm (HOPG), 200 nN
Developing flax fibres


Indentation modulus (GPa)

MultiMode 8, PF-QNM
Bruker RTESPA-525
139 N/m, 32 nm (HOPG)
200 nN
Developing flax fibres

[Goudenhoof et al, Fibers, 2018, 6(6): 9p]

Average thickness
Gn : 2.0 ± 0.5 µm
Average modulus
Gn : 13.0 ± 2.5 GPa

Average thickness
Gn+G : 3.2 ± 1.3 µm
Average modulus
Gn : 14.0 ± 1.8 GPa

Average thickness
G : 5.0 ± 1.5 µm
Average modulus
G : 19.5 ± 2.9 GPa

MultiMode 8, PF-QNM Bruker RTESPA-525, 139 N/m, 15-35 nm (HOPG) 200 nN
Quality of data vs. Resolution

[Leclere, 2011]
AFM frequency and elastic modulus range...

[Leclere, 2017]
Van der Waals force, electrostatic force, contact stiffness, adhesion force, …
Adhesion force spectroscopy

- Influence of surface roughness and tip geometry


Jin and Kasal, R. Soc. open sci. 2016, 3: 160248

Inert condition (N₂, 0% RH)

Spruce wood
Adhesion force spectroscopy

- Variability in surface polarity and aging

Frybort et al, Colloids and Surfaces A, 457 (2014) 82–87

23°C and 50% RH

Beech wood
Adhesion force spectroscopy

- Effect of surface treatment

TM-AFM phase image of (a) raw flax fibre, (b) NaOH (10%) treated flax fibre, (c) Enzyme treated (18 h) flax fibre.
Adhesion force spectroscopy

- Tip functionalisation - Chemical force microscopy

Adhesion force spectroscopy

- Tip functionalisation - Molecular force microscopy

Fungus *Aspergillus fumigatus*
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AFM artifacts

- Images dilation due to the “finite” tip shape

Flater et al, Ultramicroscopy, 2014, 146: 130-143

http://www.nanophys.kth.se/nanophys
AFM artifacts

- Images dilation due to tip shape default, dirt or wear

Flater et al, Ultramicroscopy, 2014, 146: 130-143
http://www.fc.up.pt/pessoas/peter.eaton/artifacts/artifacts.html

http://www.nanophys.kth.se/nanophys/facilities/nfl/afm/
AFM artifacts

- Images dilation due to tip contamination

- Optical interference

AFM artifacts

- Distortion due to non-linear piezo-scanner response + aging/creep
- Background bow/tilt
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Sample preparation...

Without embedding in a resin: no sample modifications but ±measurement artifacts

Dehydration in ethanol and embedding in resin (LR-White, SPURR, ...) or PEG

±no thermo-chemical modification of the cell wall (extractives...)?
Resin penetration / inclusion time?
Cell wall water content?

Microtome/Grinding/FIB...??!!
Sample surface effect

- Effect of sample local slope
  [Heinze et al, Ultramicroscopy, 2018, to appear...]

Topography

Starch granules

Raw indentation modulus

Local angle/cantilever $\phi$ ($^\circ$)

Corrected
**Effect of sample roughness**

Stan and Cook, Nanotechnology, 2008, 19, 235701

Au film \(\approx\) <110>, \(k_C = 14.8 \pm 0.5\) N/m, 350 nN