An introduction to Atomic Force Microscopy

Olivier Arnould

Chemical imaging of plant cell walls, PhD-course
June 4-8 2018, University of Copenhagen

An introduction to
Atomic Force Microscopy

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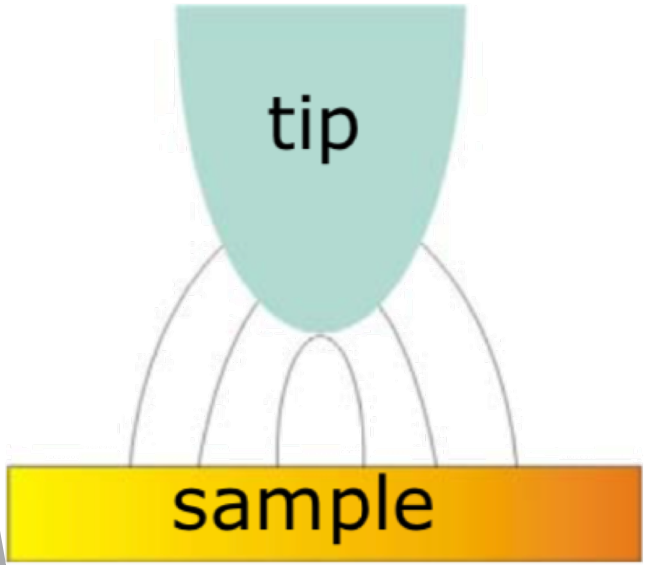
Outline

- 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!

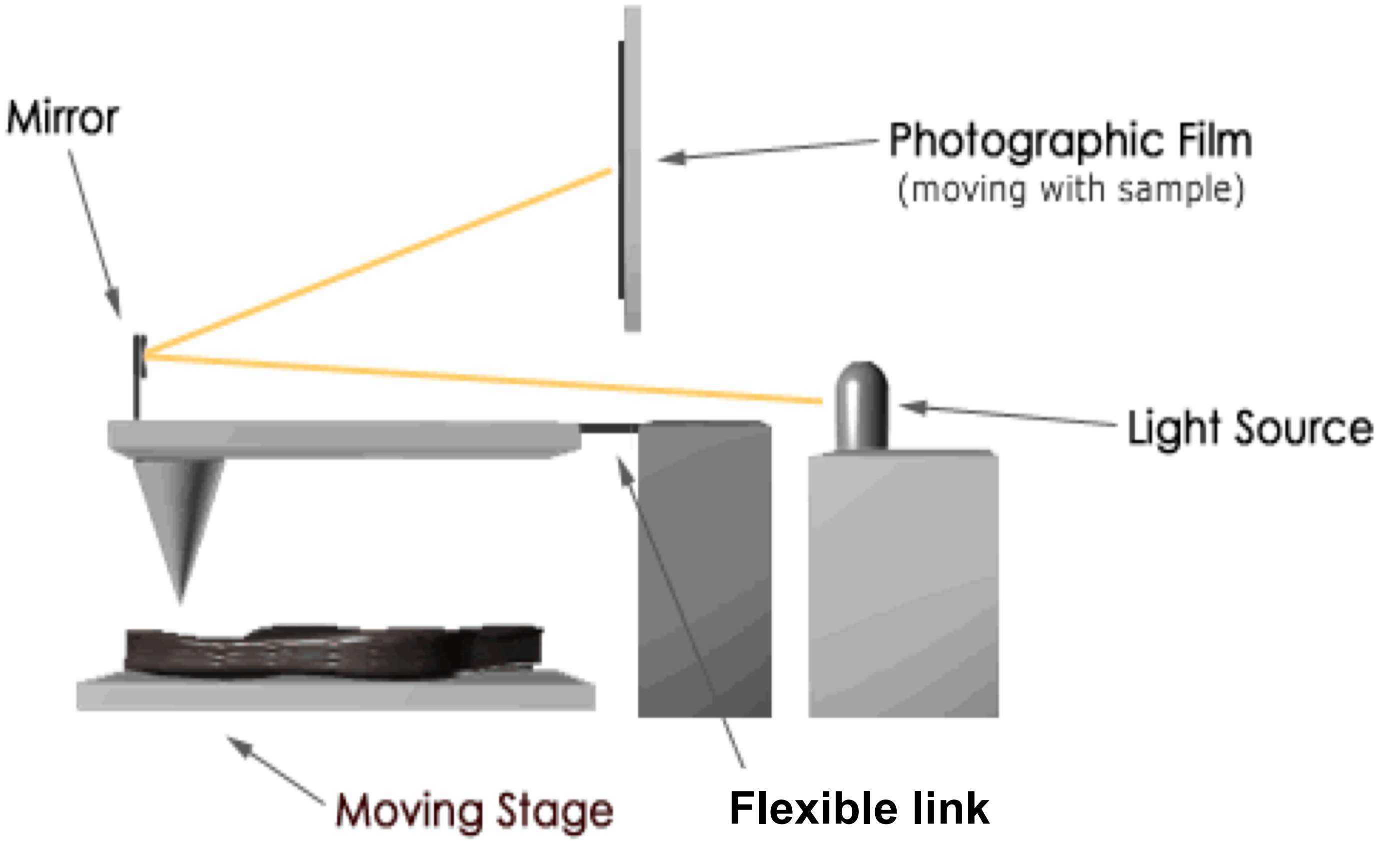
✓ Interaction can modify surface
→ nanolithography possible

✗ Scanning technique (quite) slow

✗ Limited maximum image size

All data in SPM are subjected to interpretation

History - Forerunners

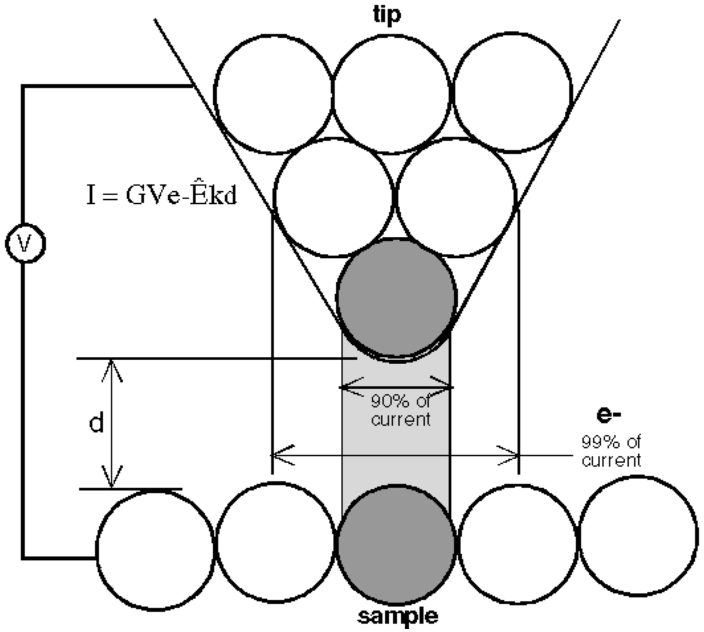


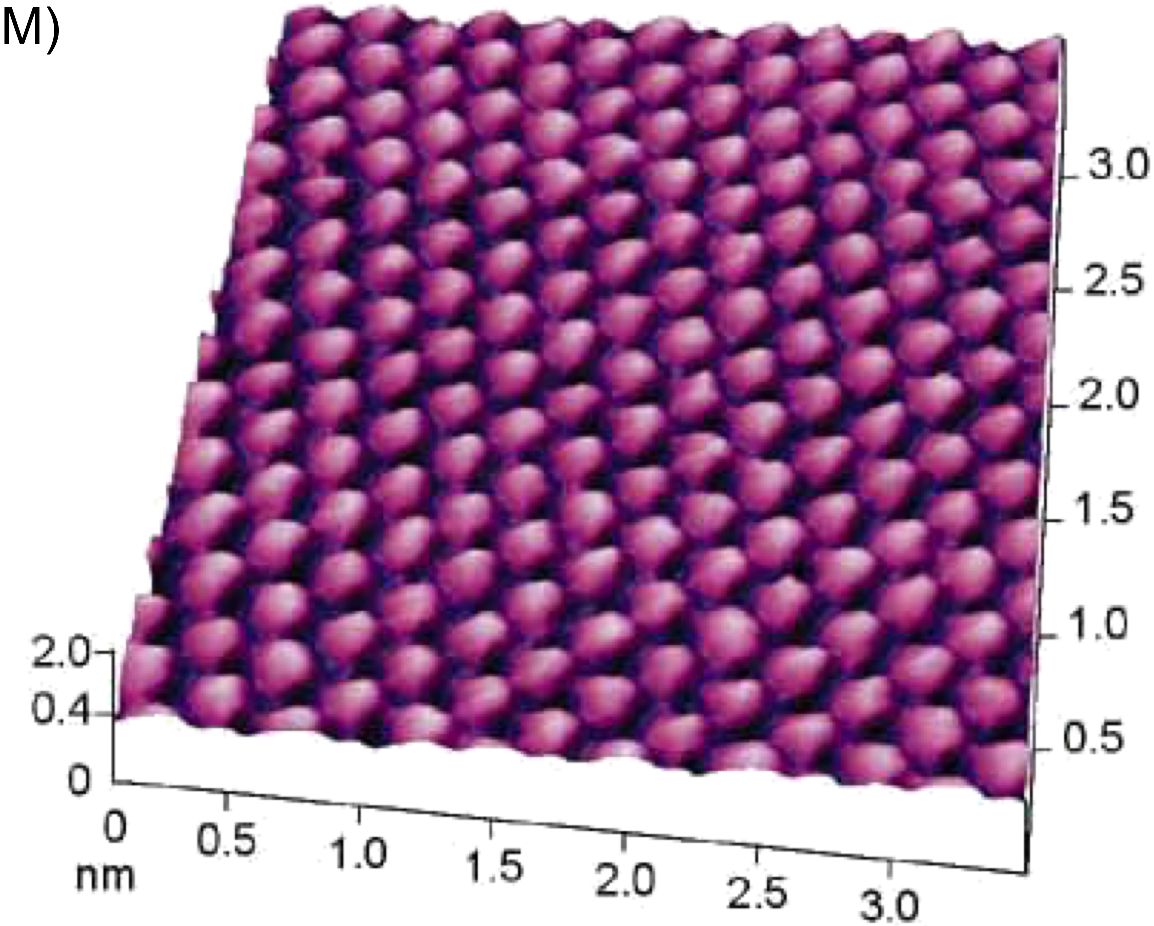
Optical stylus (optical/light lever) profilometer by Schmalz in 1929
≈ 25 nm vertical resolution! (magnification > 1000x)

[www.afmuniversity.org]

History - Forerunners

Scanning Tunnelling Microscopy (STM)
by Binnig and Rohrer in 1981
(Nobel Prize in 1986)



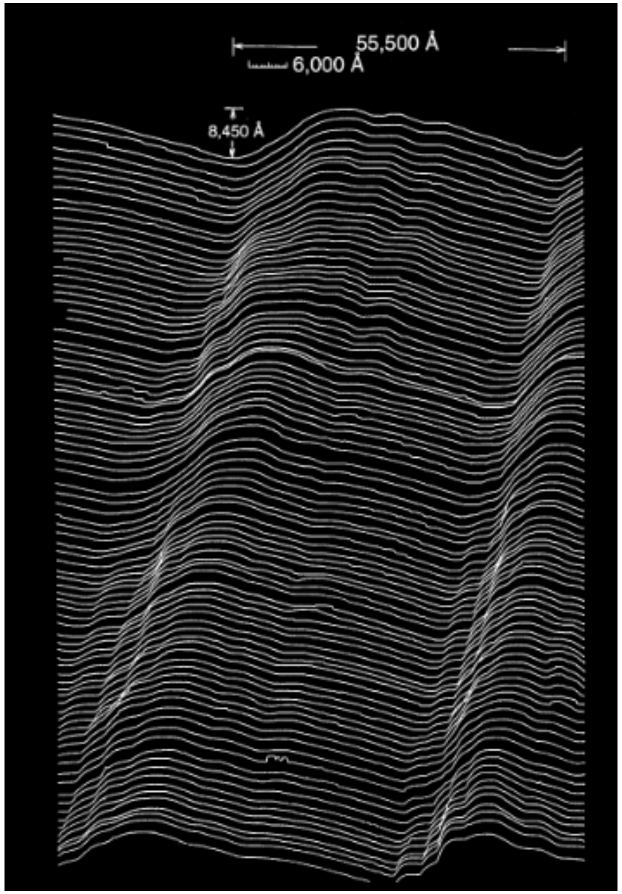


HOPG atoms view by STM
[www.ntmdt.com]

<u>Drawback</u>
need conductive material
and high vacuum!

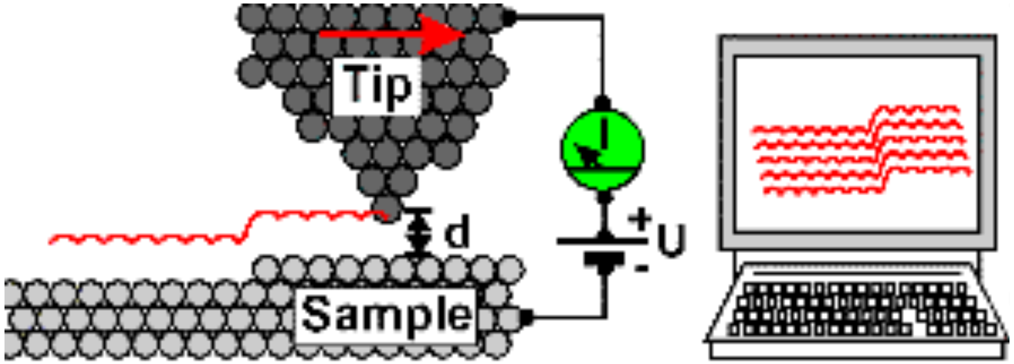
History - SPM timeline

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.

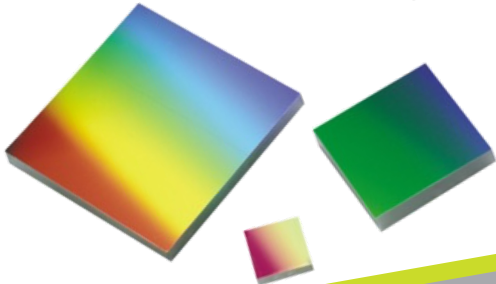


Topographic map of ruled diffraction grating

Rev. Sci. Instr. 43(7), 1972



Constant current or constant height imaging



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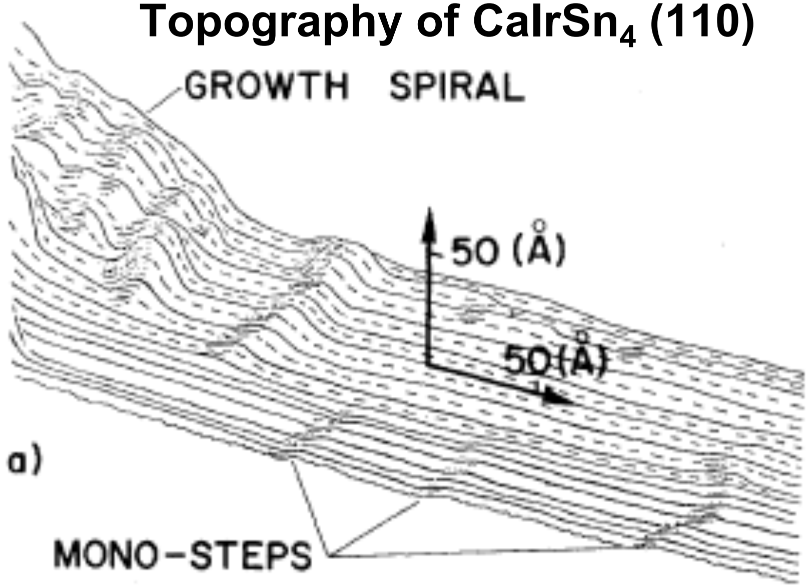
Topography of $CaIrSn_4$ (110)



Phys. Rev. Lett., 49(1), 1982

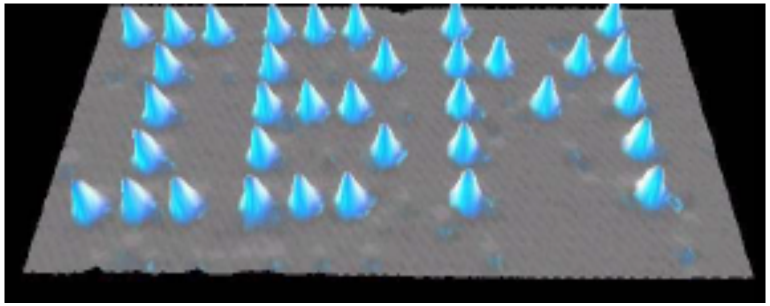
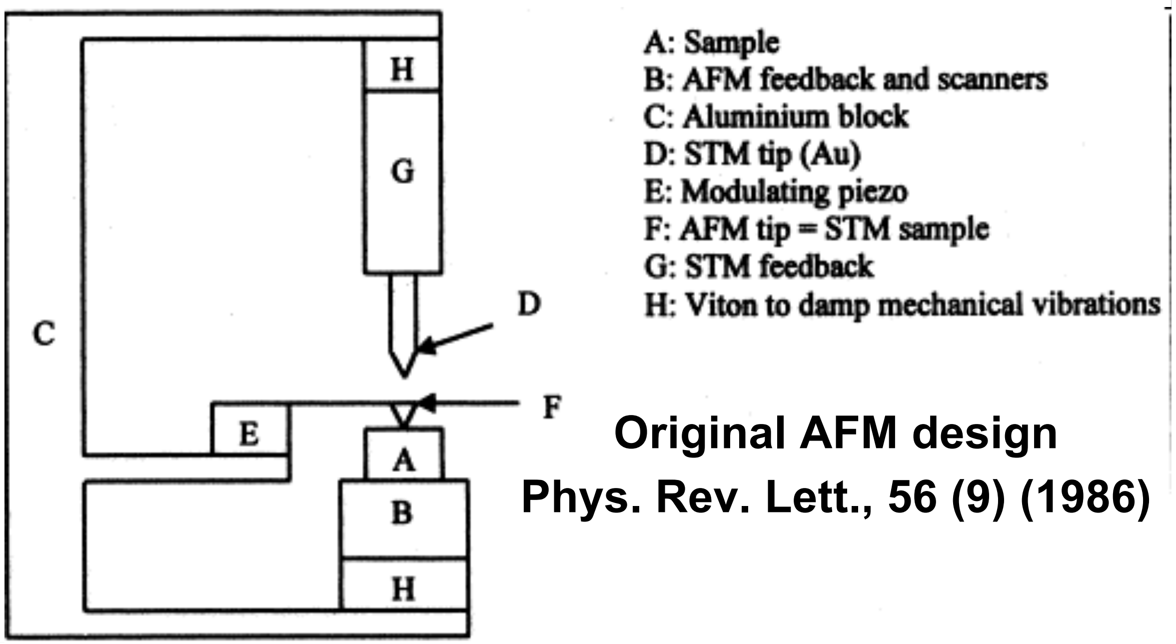


Image of Xenon atoms moved on Nickel (110) surface using an STM tip



Original AFM design
Phys. Rev. Lett., 56 (9) (1986)

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

- Nanoindentation Mechanical modes
- Electrostatic Force Microscopy (EFM)
- Conductive AFM (C-AFM)
- Scanning Thermal Microscopy (SThM)
- Chemical force microscopy

A question of probe tip (apex)!

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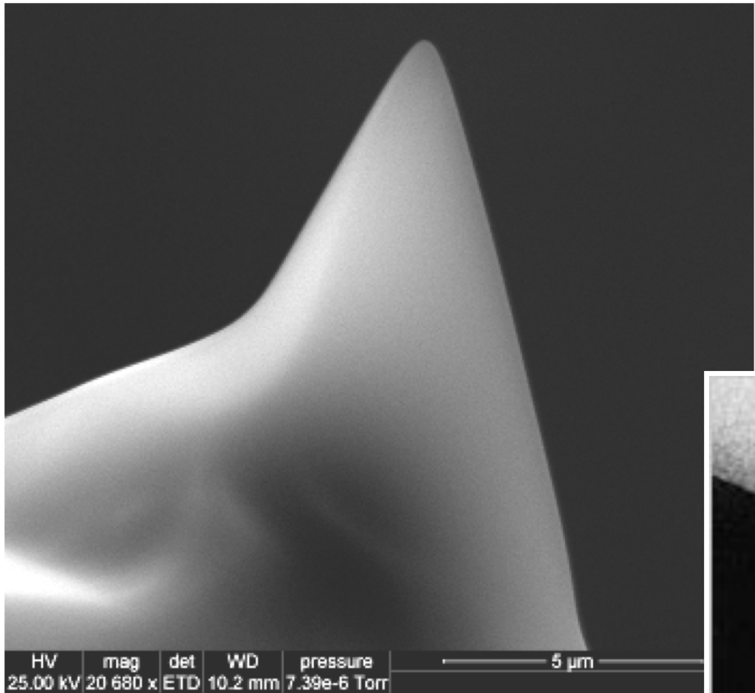
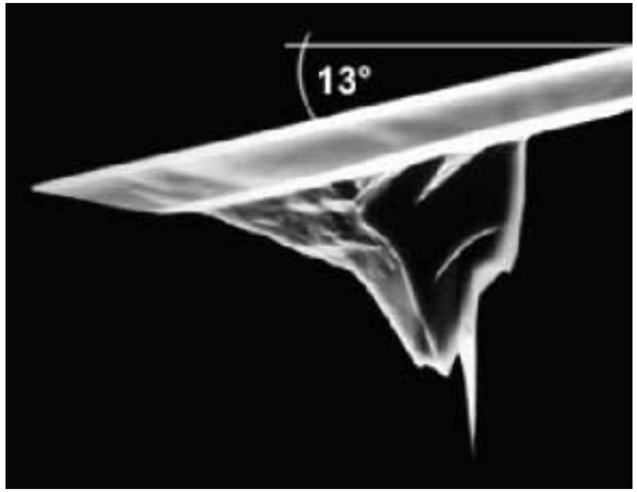
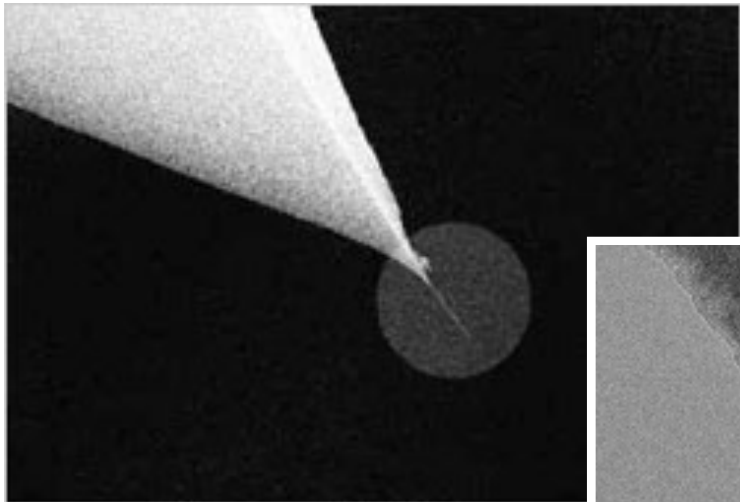
Probe tip-sample interactions

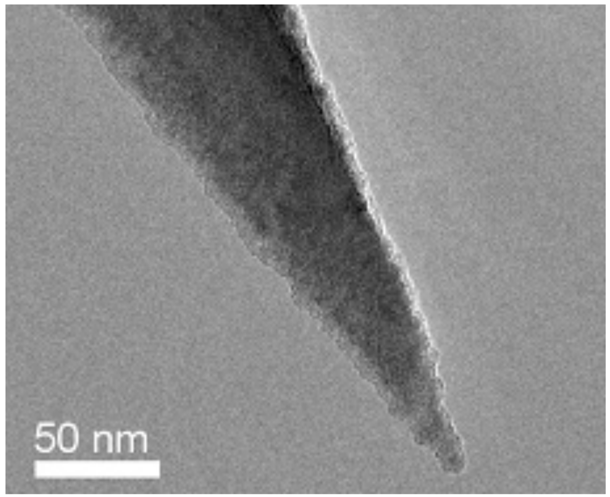
- Tip (feels the surface)



Calibration!







Apex radius $R \approx$ 2-200nm

Height $H \approx$ 10µm

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

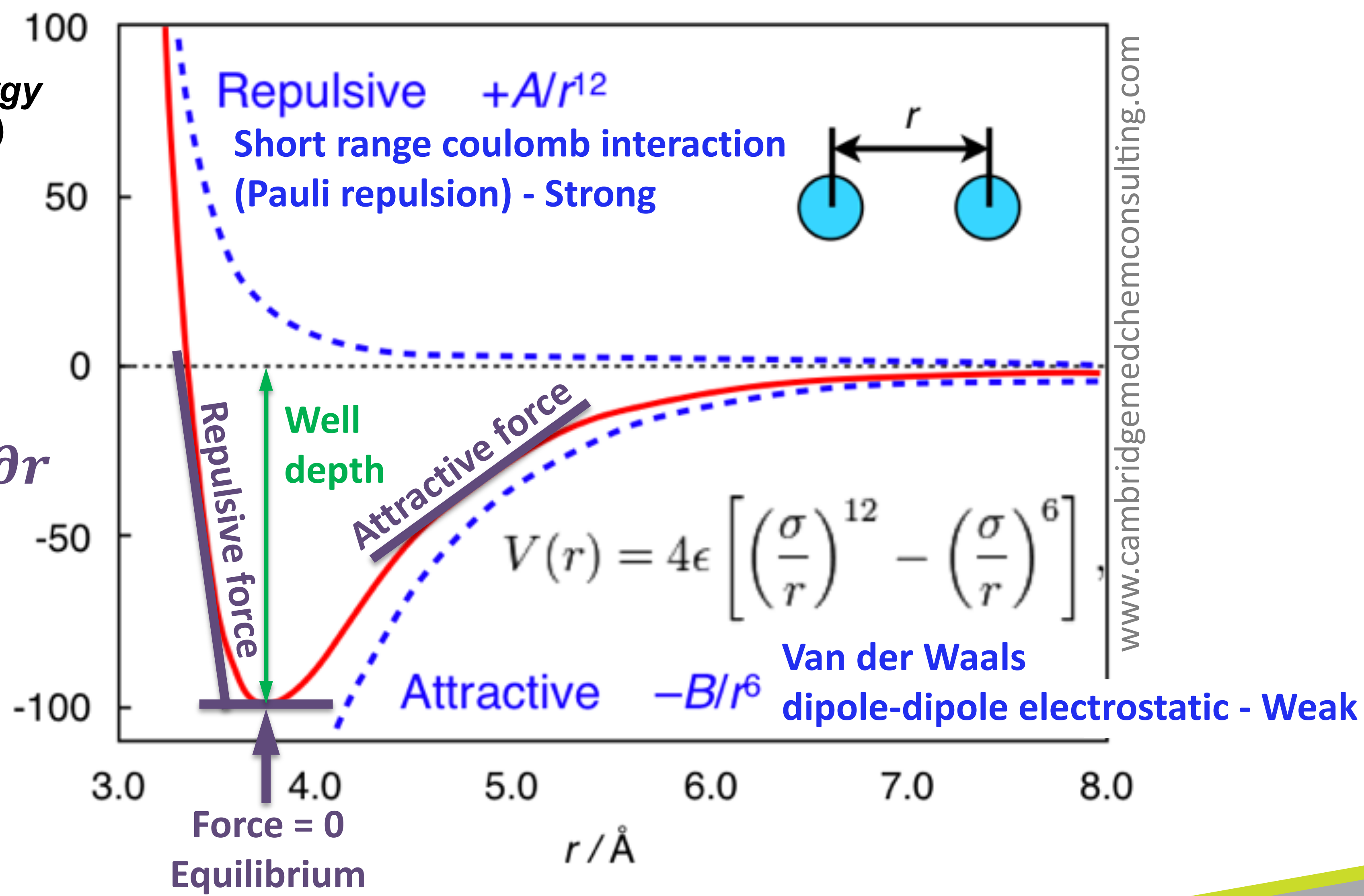
⇢ **www.nanoandmore.com/afm-probes**

[NanoAndMore, NT-MDT, BudgetSensor, …]

Probe tip-sample interactions

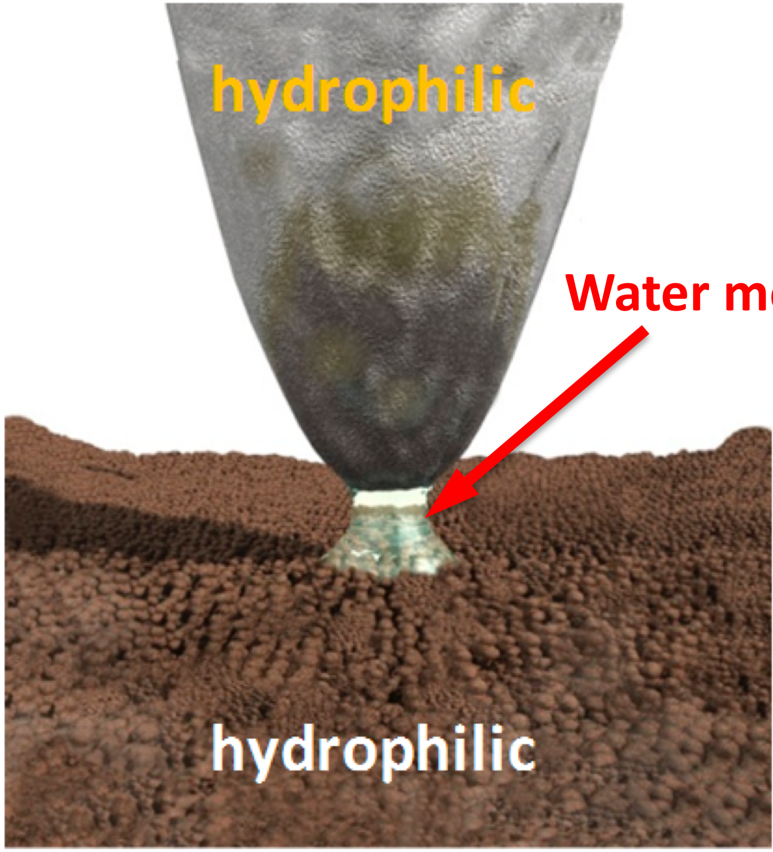
The Lennard-Jones potential

$$F = {}^{\partial V}/_{\partial r}$$

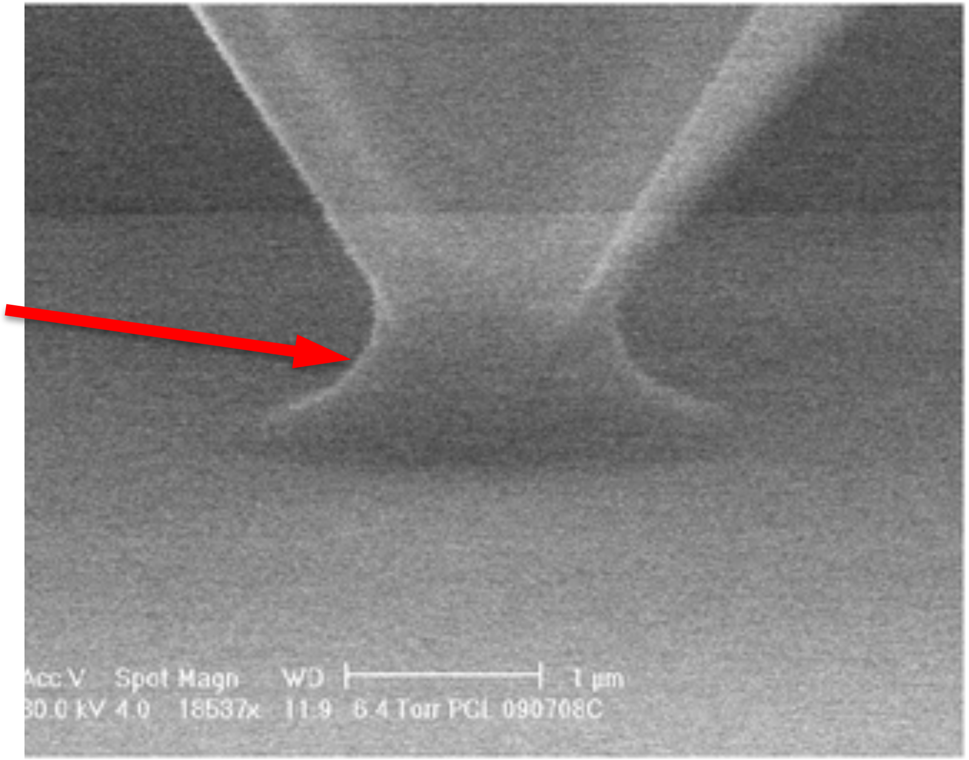


Probe tip-sample interactions

Capillary force



Köber et al, Small 2010, 6, 2725



van Honschoten et al, Chem. Soc. Rev., 2010, 39, 1096-1114

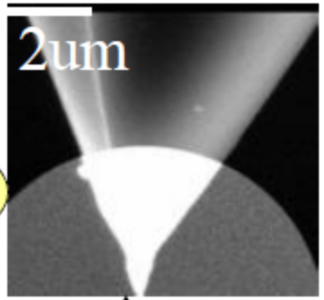
$$F_{cap} \approx \frac{4\pi R \gamma_L \cos\theta}{(1+d/h)} \qquad \cos\theta = \frac{\cos\theta_1 + \cos\theta_2}{2}$$

Surface tension of water: $\gamma = 72 mJ/m^2$

Max at snap (pull) out

$$\boxed{F_{cap} = 4\pi R\gamma}$$

Probe tip-sample interactions



$$\vec{F}_{t/s} = \vec{F}_{E/M} + \vec{F}_{cap} + \vec{F}_{VdW} + \vec{F}_{rép}$$

Tip-surface Gap (z)

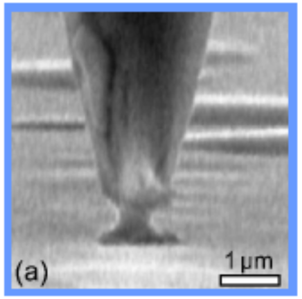
Electrostatic force ($1/z^n$; $1<n<2$) : Long range interaction => electric modes

$$F_{elect} = \frac{1}{2}\frac{\partial C}{\partial z}V^2 \prec \frac{1}{2}\frac{\varepsilon_0 \varepsilon_r S}{z^n}V^2$$

z > 20nm

Capillary Force

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



z> 5nm

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

Van der Waals Force

z< 0.5nm

Repulsive Force (m>3)

$$F_{répulsion}(z) = \frac{A}{z^m}$$

AFM principle and typical set-up

- Cantilever (spring, load sensor)

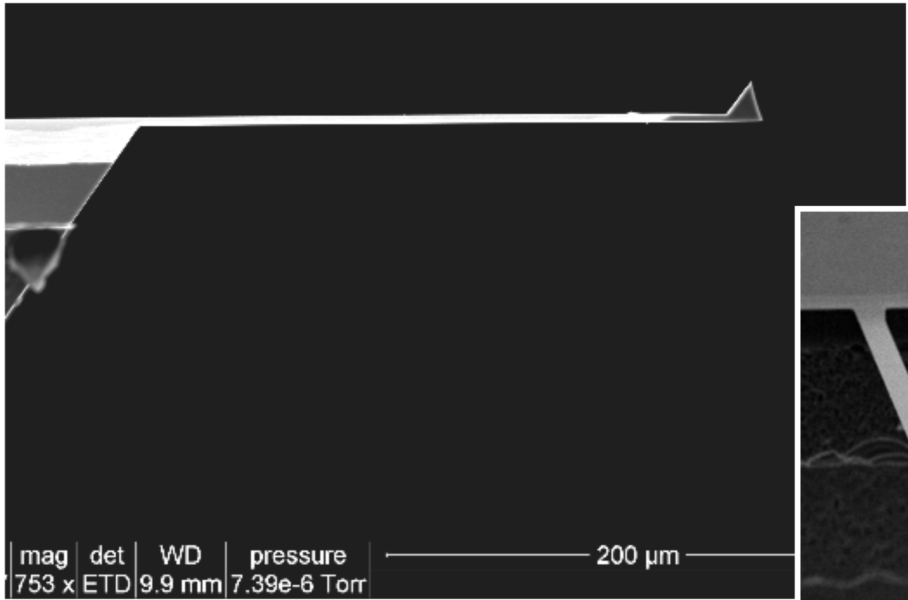
Stiffness k_C: 0.01 – 100 N/m

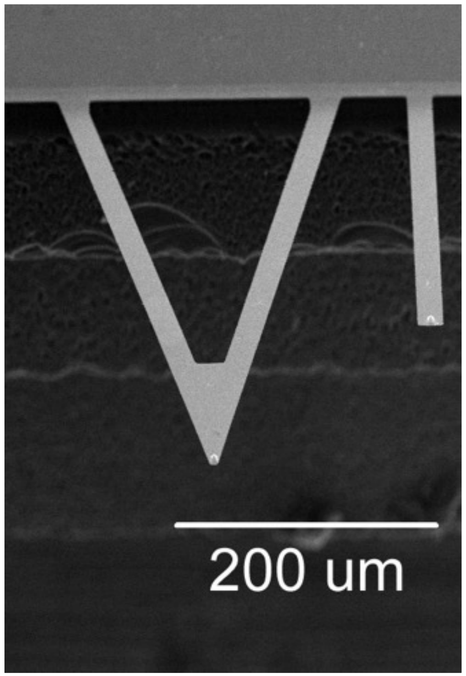
Length L: 100-300 µm

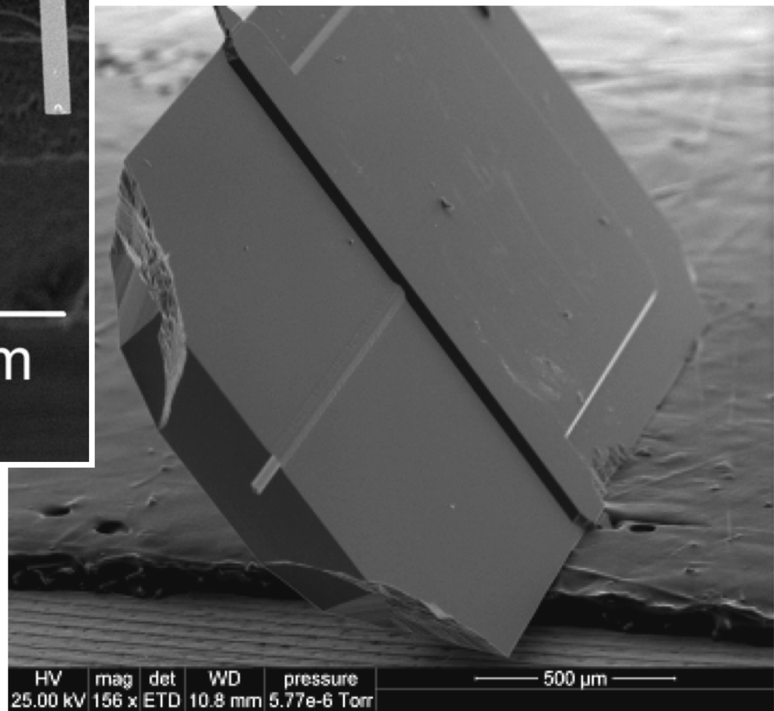
Witdth w: 10-50 µm

Thickness e: 0.3-4 µm

Coating







[NanoAndMore, NT-MDT, BudgetSensor, ...]

⇢ **www.nanoandmore.com**

+ mounting on the holder 😱

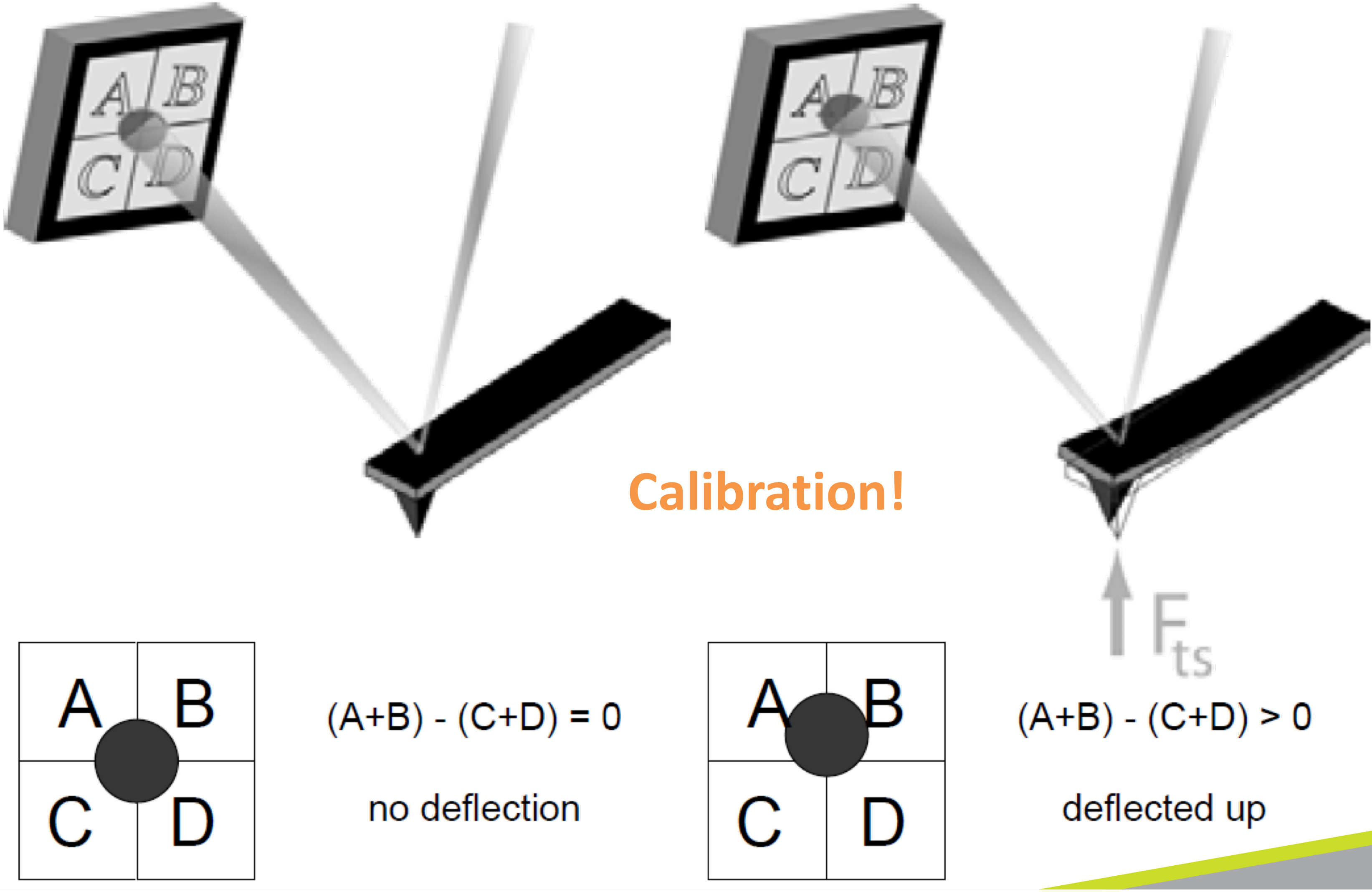
Calibration!

AFM principle and typical set-up

- Photodetector (deflection sensor)

Photodiode + geometry + laser position sensitivity ≈ x10 nm/V

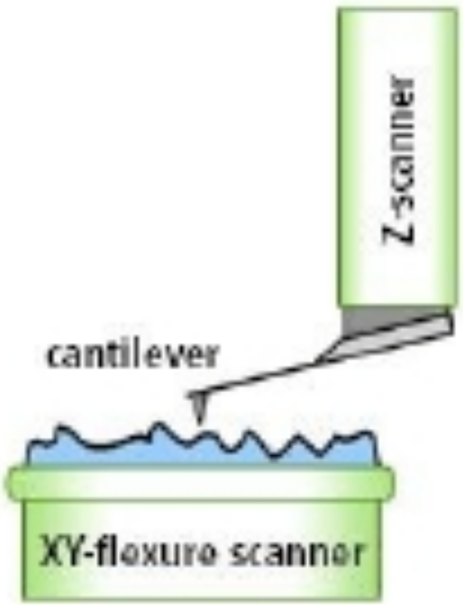
⇢ minimum measurable deflection < 0.1 nm!

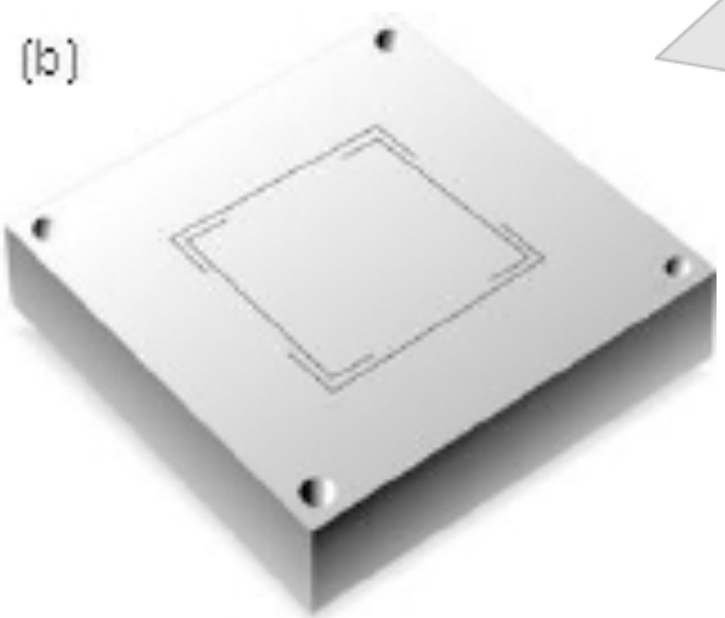


AFM principle and typical set-up

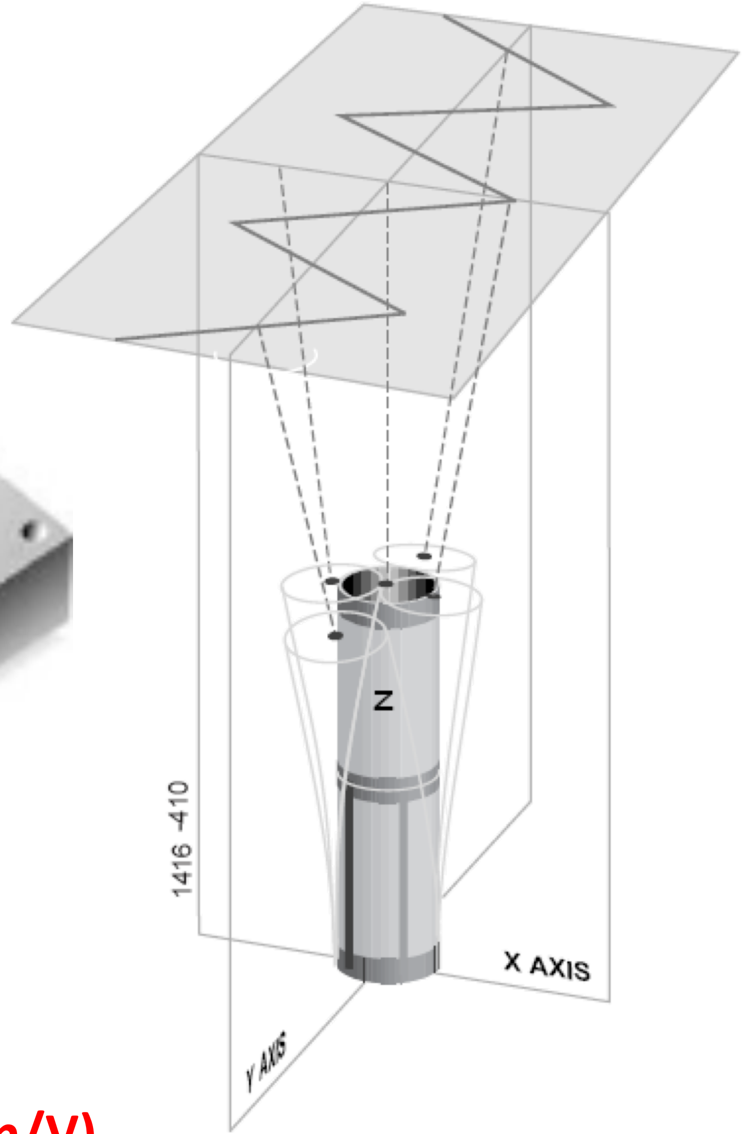
- Scanner

Calibration!





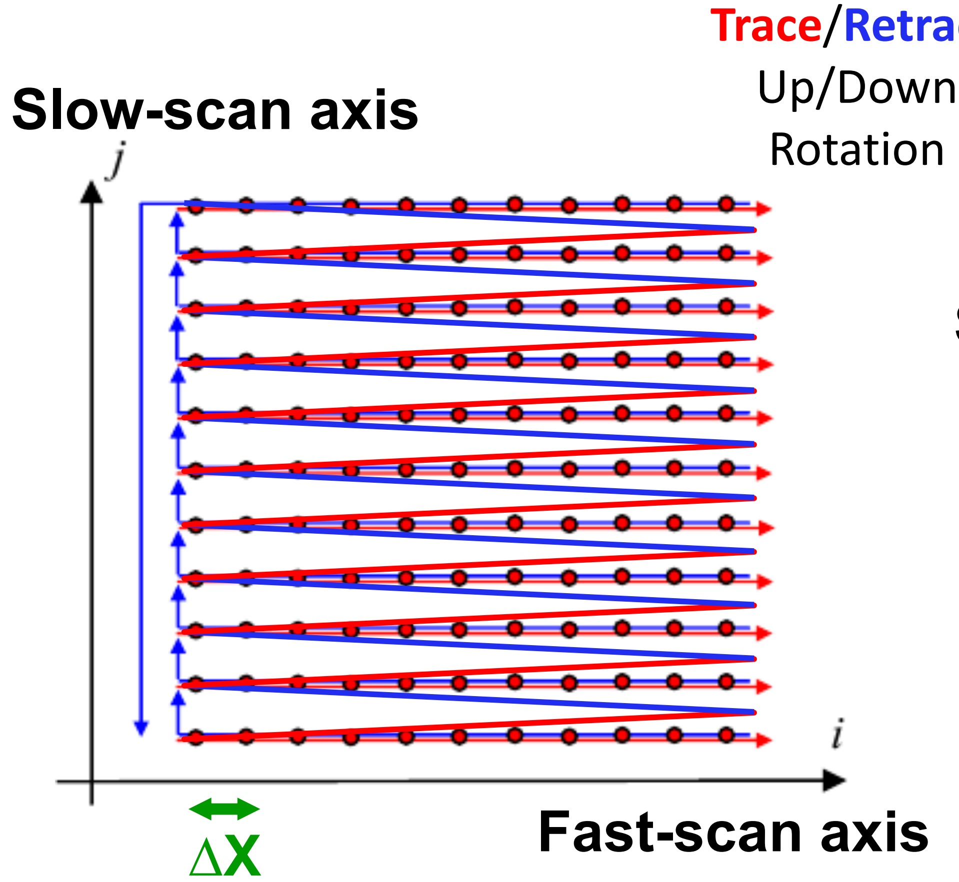
[www.parkafm.com]



[www.veeco.com]

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 × 256 or 512 × 512 elements

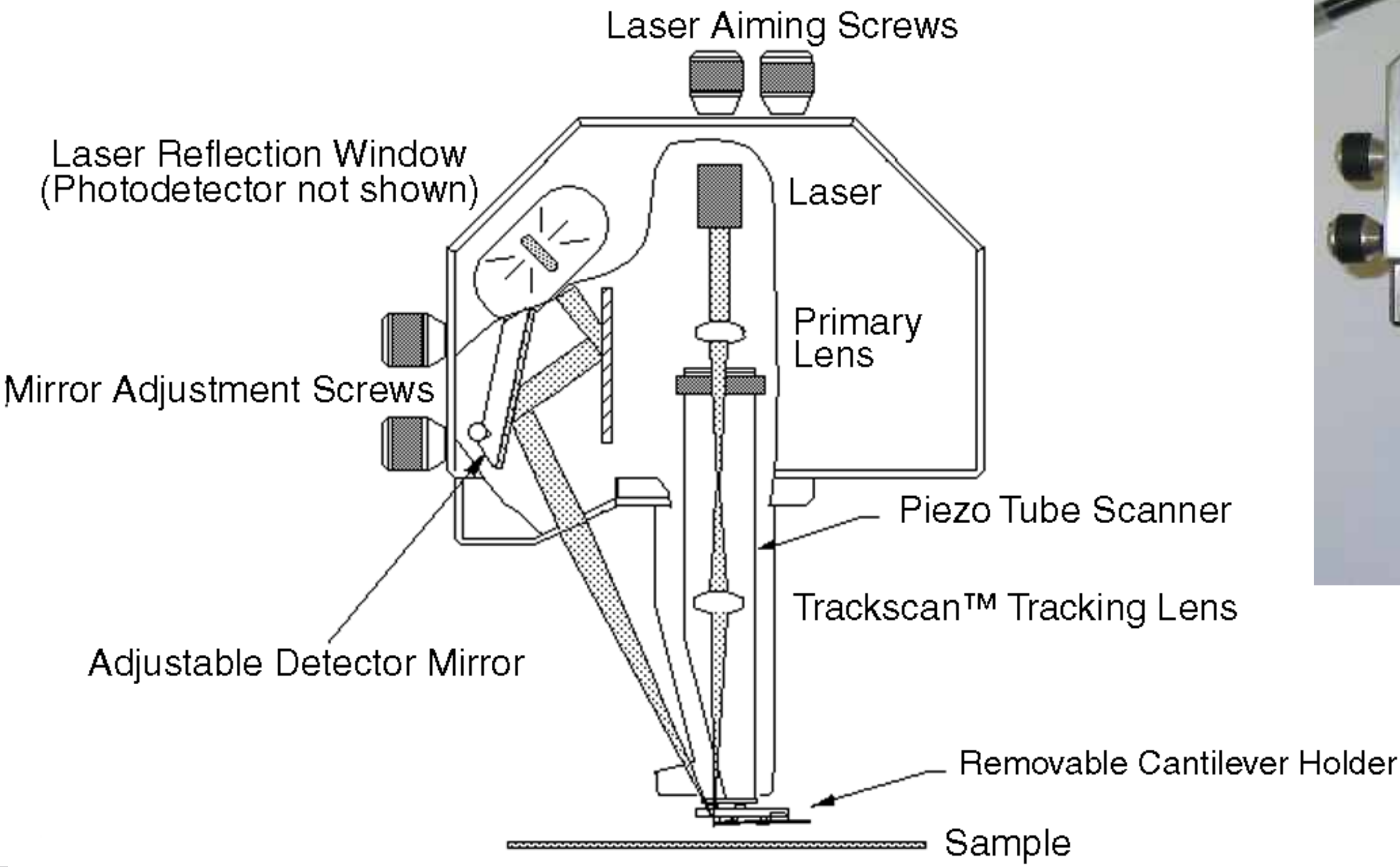
The image spatial resolution is conditionned by the digitalisation

Example: **Image size 10 µm** $\Delta X = \dfrac{10\mu m}{511} = 0.02\mu m = 20nm$

512 × 512 elements

AFM principle and typical set-up

- Whole set-up



[www.veeco.com]

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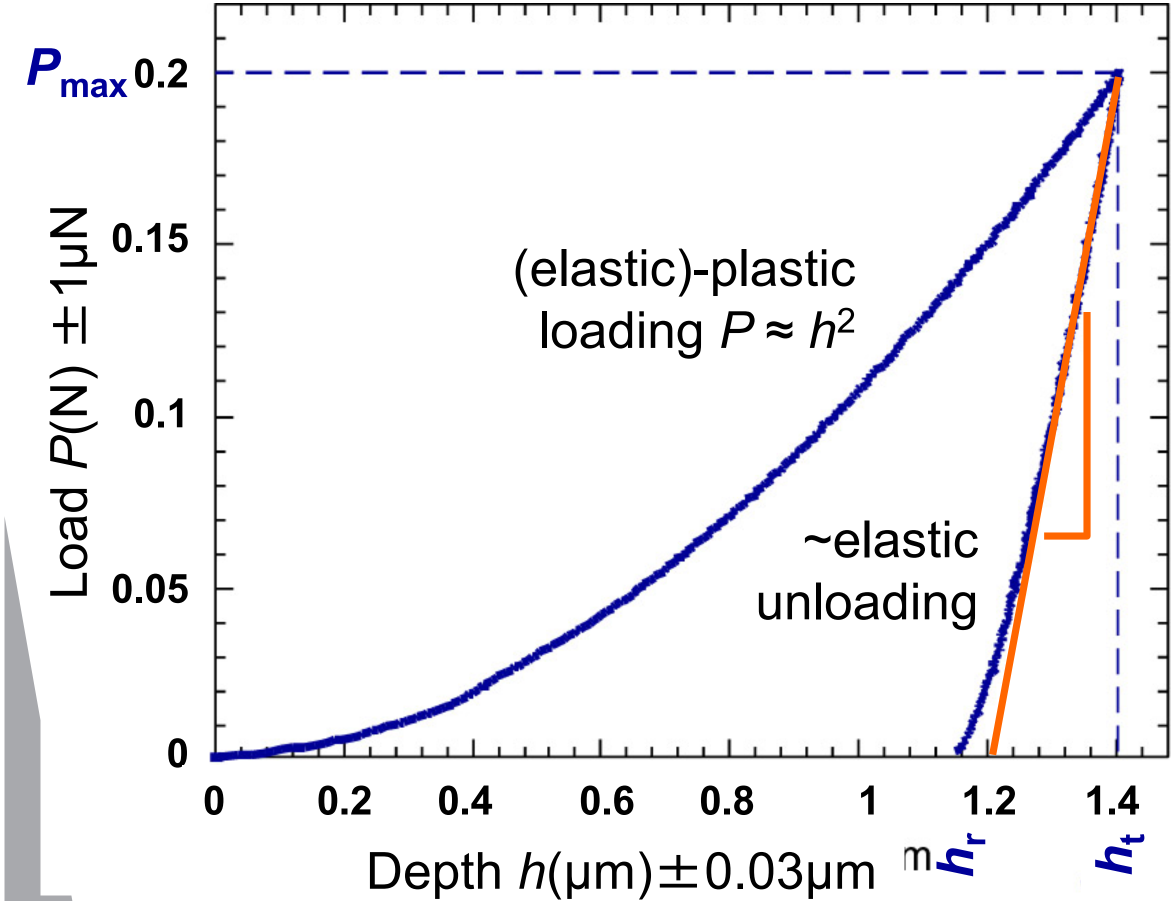
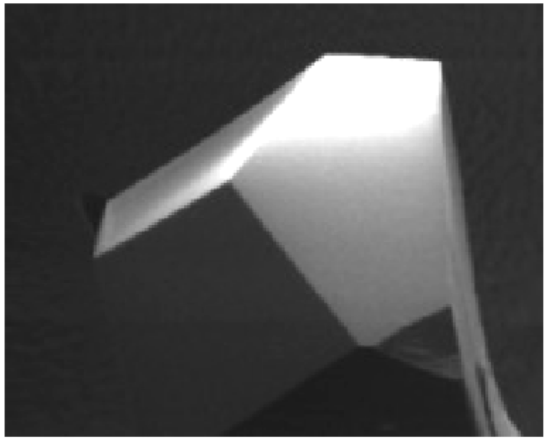
Contact mechanics

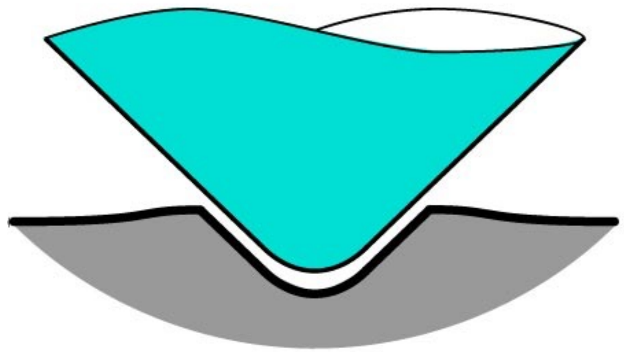
- (Depth-sensing) Nanoindentation
  - ➲ The depth of penetration h (δ!) is measured during load application mainly with a Berkovich indenter (3-sided pyramid)



Nanoindentation curve on electrodeposited Ni



[www.microstartech.com]



Contact mechanics

- (Depth-sensing) Nanoindentation

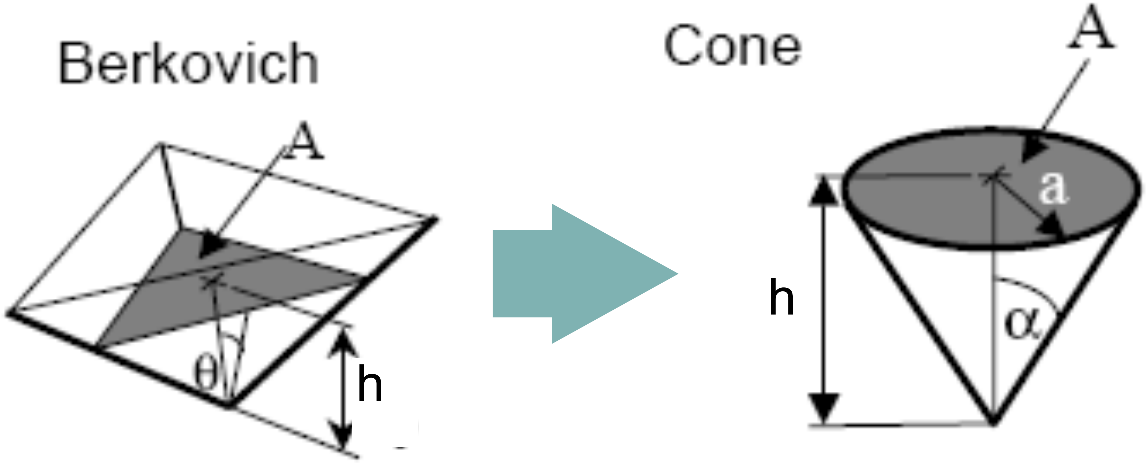
$$\boxed{E^* = \frac{\sqrt{\pi}}{2}\frac{dP}{dh}\frac{1}{\sqrt{A_c}}}$$

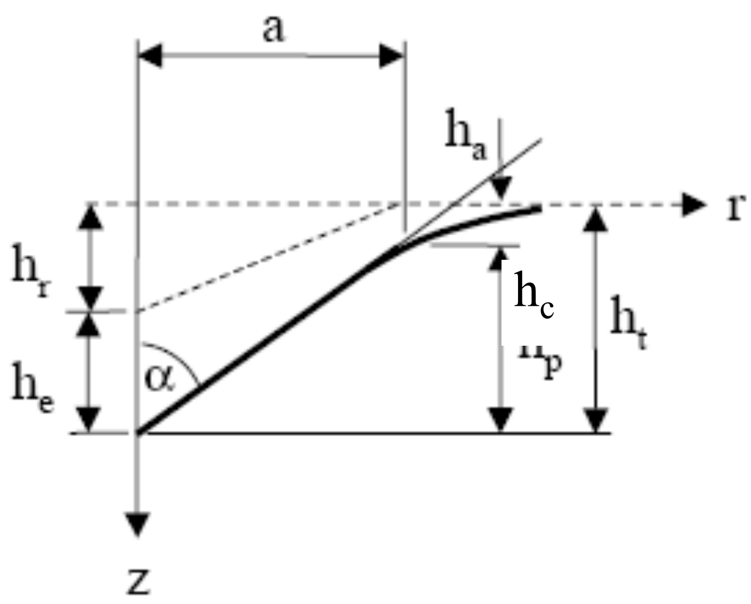
With E* or M reduced/contact/indentation modulus

$$\frac{1}{E^*} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu^2}{E}$$ for linear ***ISOTROPIC*** material

- Case of the Berkovich indenter

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



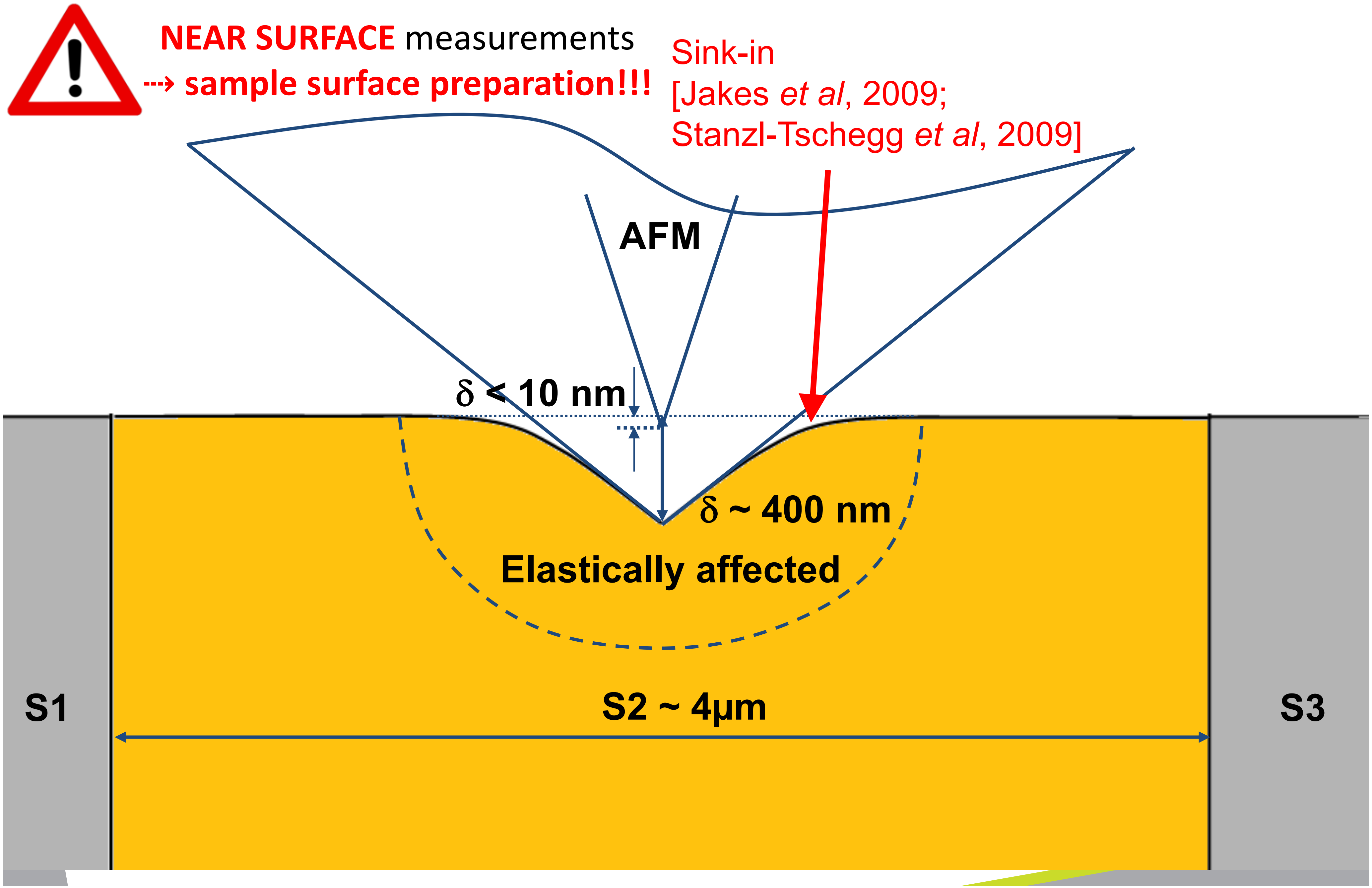


$A = \boxed{24.5}h^2$ $\alpha = 70.3°$ $A = \boxed{\pi \tan^2 \alpha}\, h^2$

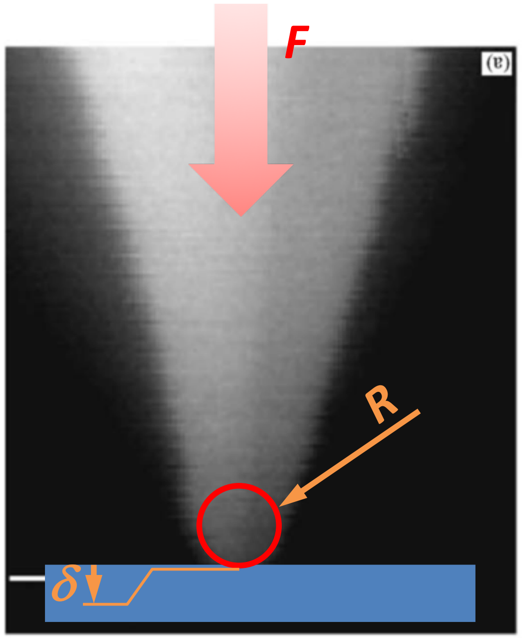
$$h_c = h_t - \varepsilon P\left(\frac{dP}{dh}\right)^{-1} \quad \text{with } \varepsilon = \begin{cases}\left(\dfrac{2(\pi-2)}{\pi}\right) & \text{cone}\\ 0.75 & \text{Berkovich}\end{cases}$$

Contact mechanics – NI vs AFM

NEAR SURFACE measurements
⇢ **sample surface preparation!!!**



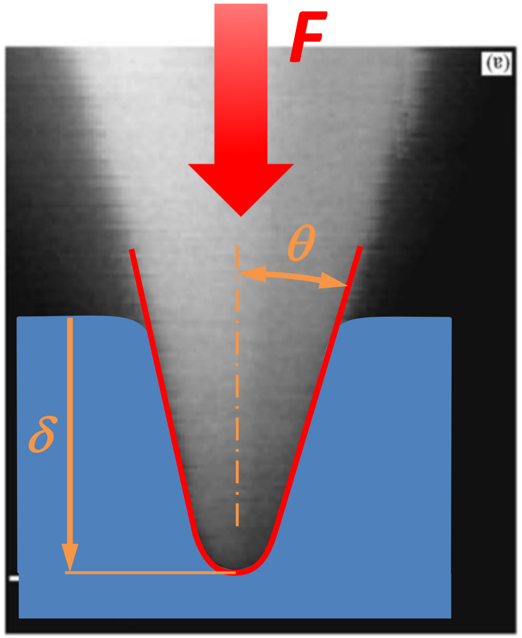
Contact mechanics without adhesion



✓ **Hertz (1885)**

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

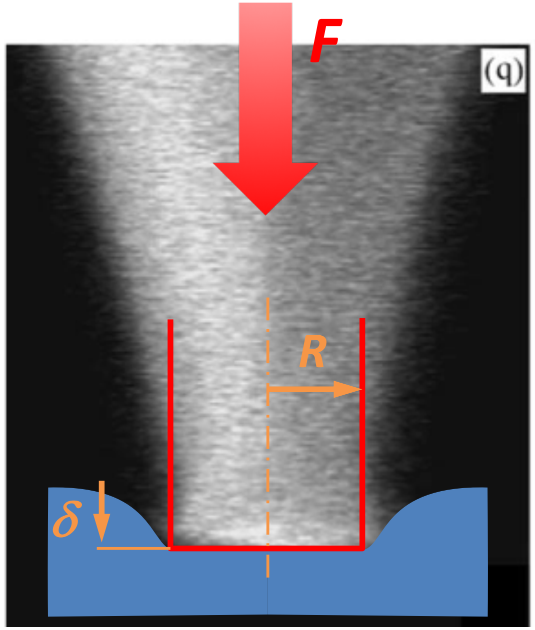
$$k_N = \left(6\, M^2 R_{eq} F\right)^{1/3}$$



✓ **Sneddon (1965)**

$$F = \frac{2}{\pi} M \tan\theta\, \delta^2$$

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



✓ **Flat punch (??)**

$$F = 2MR\,\delta$$

$$k_N = 2MR$$

δ << R **δ ≈ R??!!** **δ >> R**

Non conforming

E^*_{sample} << E^*_{tip}

M: Reduced/contact modulus

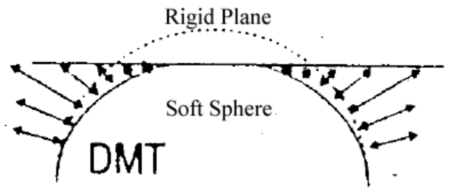
$$\frac{1}{M} = \frac{1-\nu_{Tip}^2}{E_{Tip}} + \frac{1-\nu_{Sample}^2}{E_{Sample}}$$

LINEAR ELASTIC HOMOGENEOUS and *ISOTROPIC* materials

[Kopycinska-Müller et al, Ultramicroscopy, 2006]

Contact mechanics + adhesion (capillary, Van der Waals, …)

✓ **DMT Model:**
- **Adhesive model**
- **Hertz model + offset of applied load**

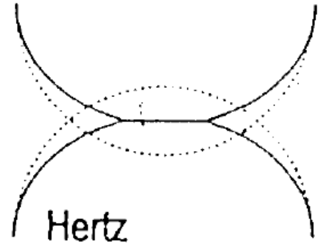


$$k_{N_{DMT}} = \left(6M^2R\left(F + F_{ad_{DMT}}\right)\right)^{1/3}$$

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

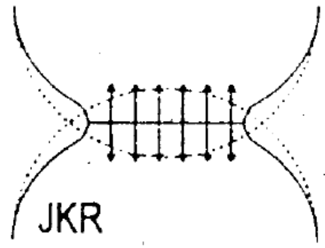
Used for hard samples, low adhesion force and low tip radius R

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



Hertz's theory is no longer valid: no direct relation between F and δ

$$k_{N_{JKR}} = 2Ma_{JKR}\,\frac{1-\frac{1}{2}\left(\frac{a_{0_{JKR}}}{a_{JKR}}\right)^{3/2}}{1-\frac{1}{6}\left(\frac{a_{0_{JKR}}}{a_{JKR}}\right)^{3/2}} \quad \text{with} \quad \begin{cases} a_{JKR} = \left[\dfrac{3R}{4M}\left(\sqrt{F_{ad_{JKR}}} + \sqrt{F_{ad_{JKR}} + F}\right)^2\right]^{1/3} \\ a_{0_{JKR}} = \left[\dfrac{3RF_{ad_{JKR}}}{M}\right]^{1/3} \\ F_{ad_{JKR}} = \frac{3}{2}\pi R w_{ad} \end{cases}$$



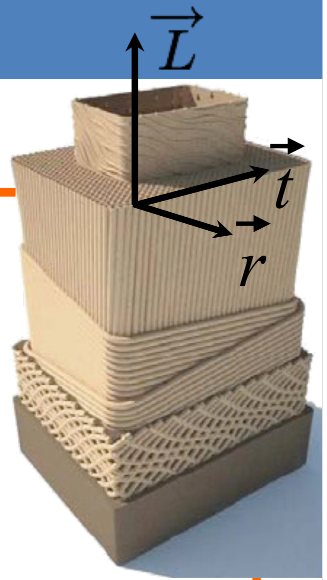
[R.M. Overney]

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

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

Contact mechanics

- Elastic anisotropy



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

$M_L \approx 19$ GPa whereas $E_L \approx 45$ GPa

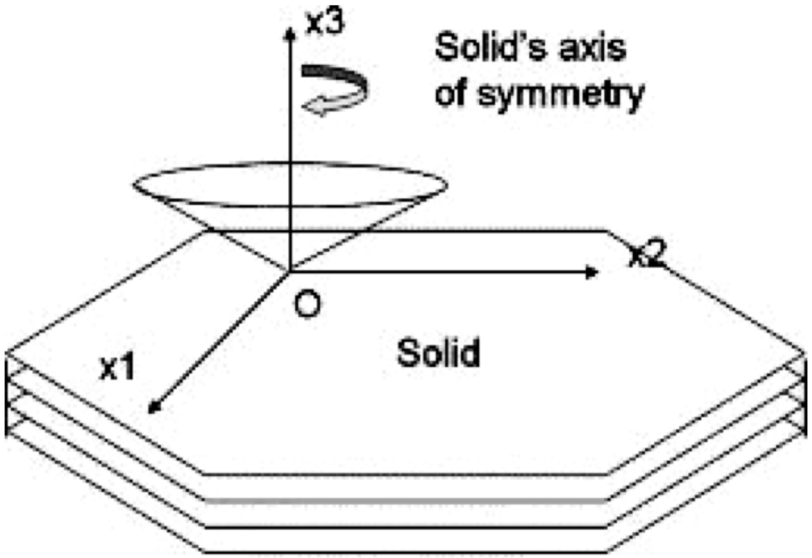
($E_t \approx E_r \approx 12$ GPa, $\nu_{tL} \approx \nu_{rL} \approx 0.028$, $\nu_{rt} \approx 0.28$
$G_{tL} \approx G_{rL} \approx 2.5$ GPa, $G_{rt} \approx 2$ GPa)

Kevlar fibre: M// ≈ 15-20 / E// ≈ 80 GPa [Arnould et al, 2017]

in particular case…
→ transversely isotropic // contact surface
[Hanson, J. Appl. Mech., 1992]

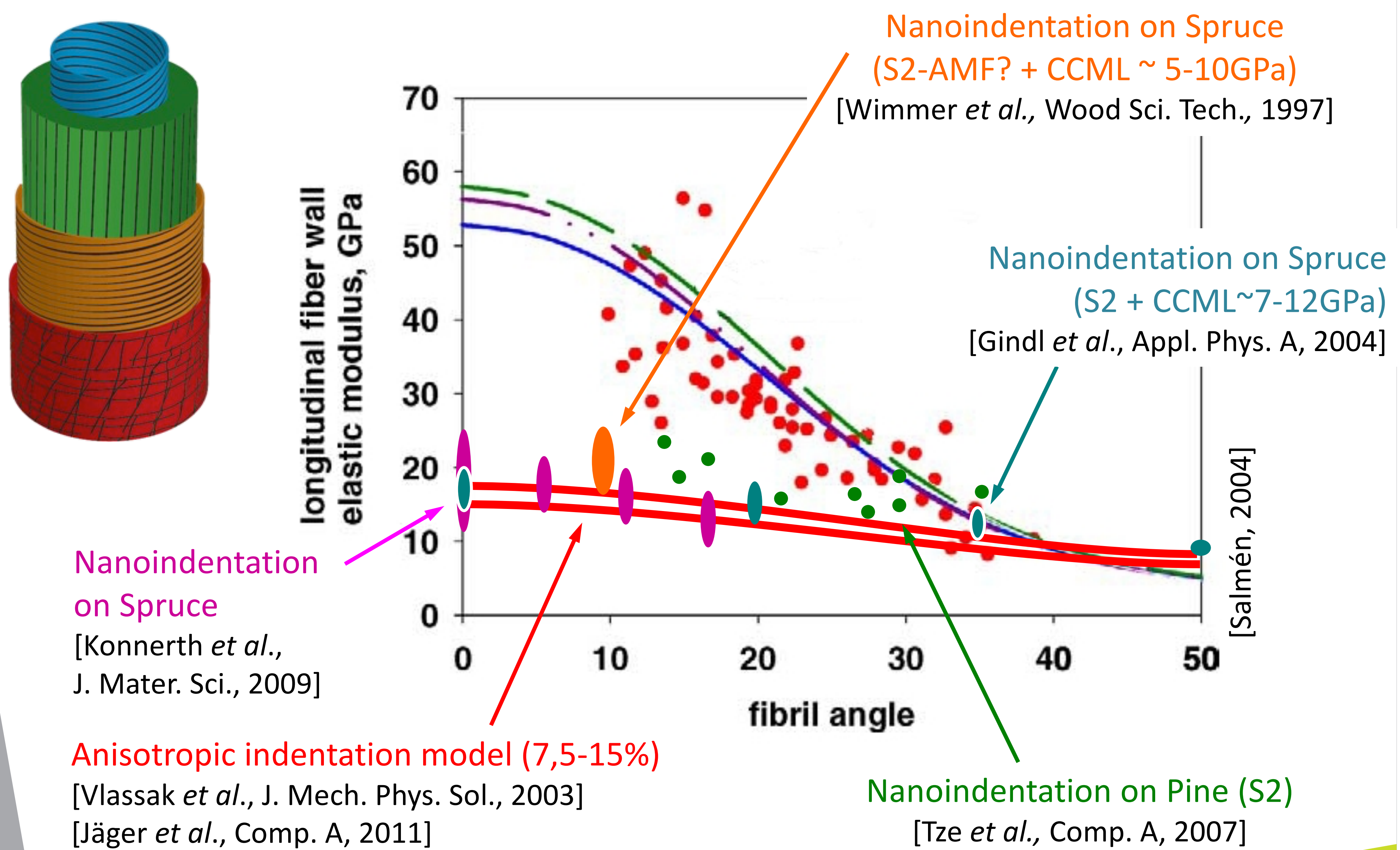
$$M_3 = 2\sqrt{\frac{C_{11}C_{33} - C_{13}^2}{C_{11}}\left(\frac{1}{C_{44}} + \frac{2}{\sqrt{C_{11}C_{33}} + C_{13}}\right)^{-1}}$$

1=2: C_{22} C_{23} $G_{13}=G_{23}$



[Delafargue *et al.*, Int. J. Sol. Struct., 2004]

Contact mechanics – Fibre cell wall anisotropy



[Eder, Arnould et al, Wood Sci. Technol., 2013]

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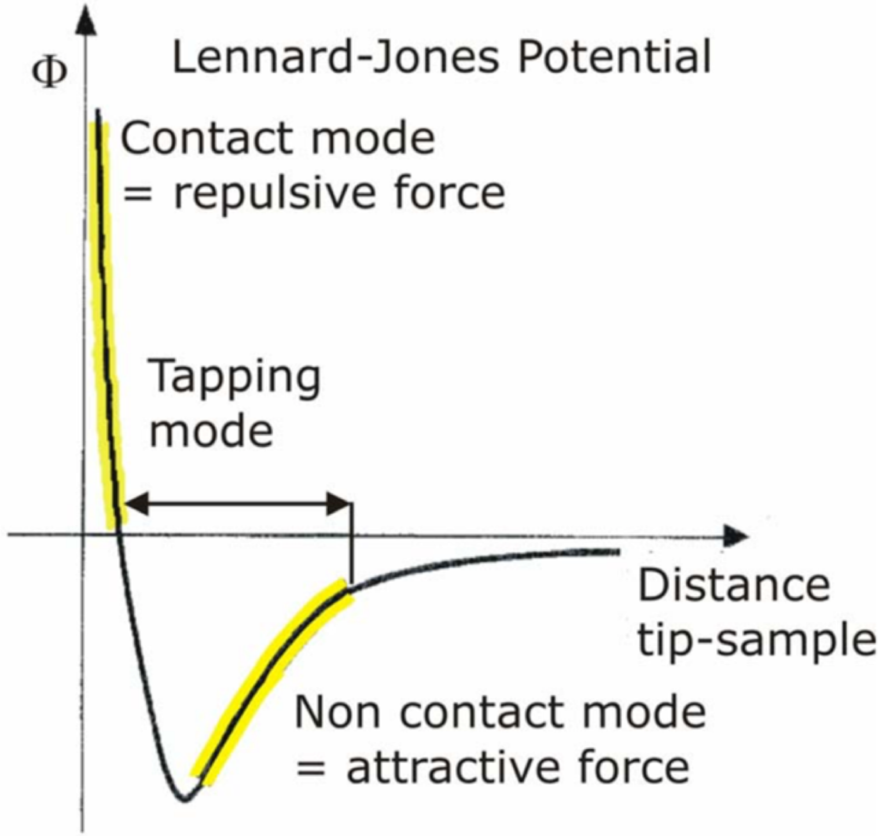
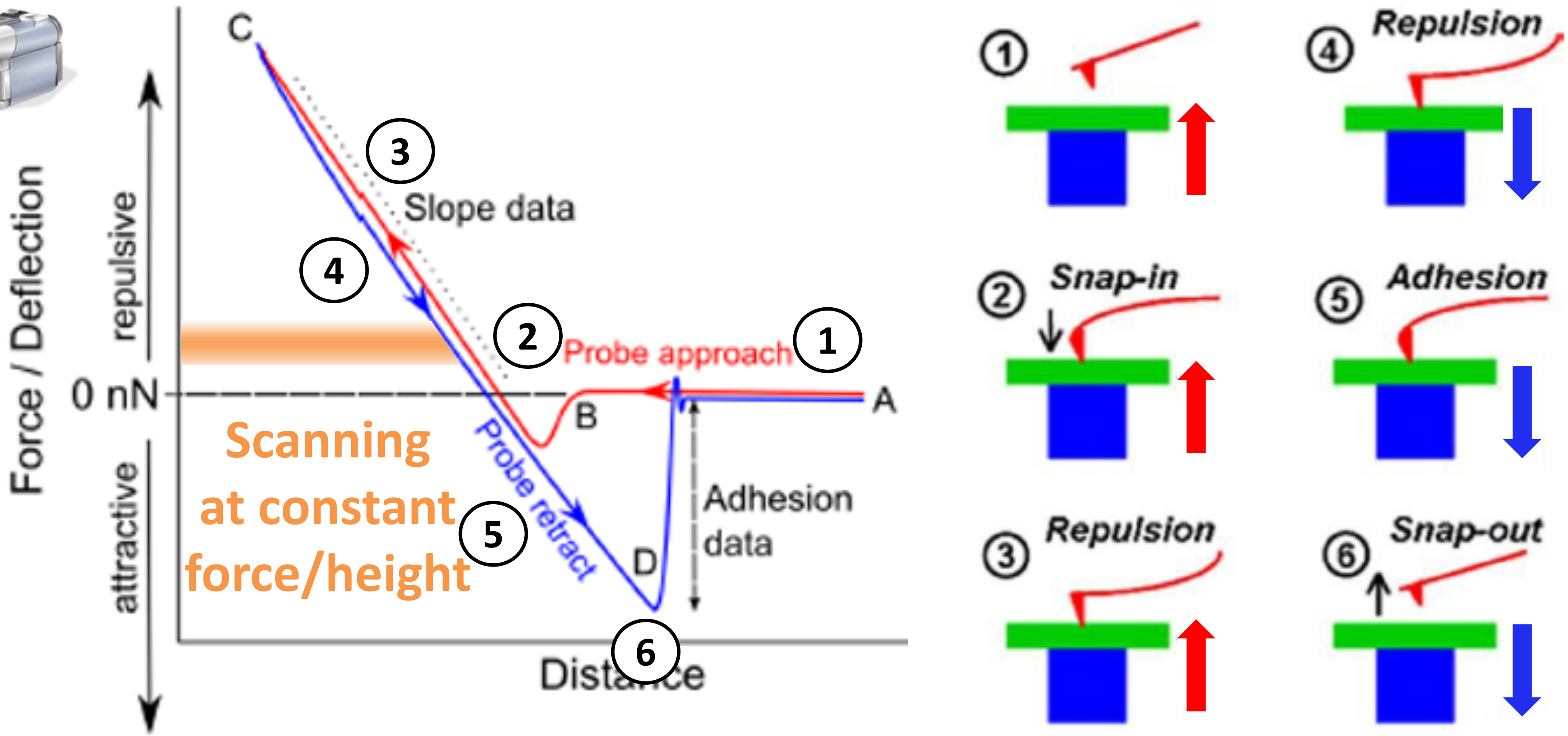
  - Deflection sensor and scanner
  - Whole set-up
- Contact mechanics
- Operating modes (some)
  - Contact
  - Intermittent
  - Force-distance curve and mechanical (
- Image analysis and artefacts
- Sample preparation



Force-distance curve

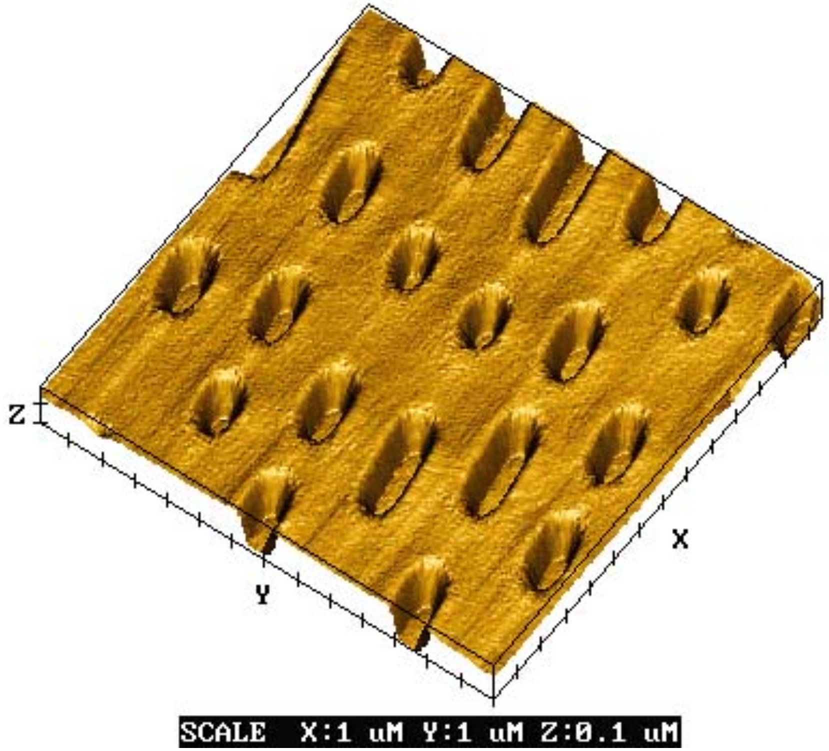
- Van der Waals force, electrostatic force, contact stiffness, adhesion force, …



[B. Nysten, 2007]

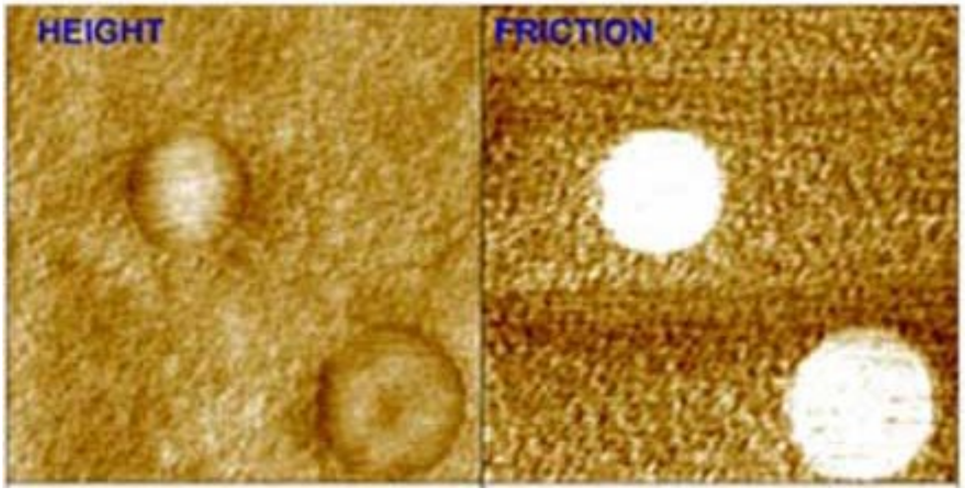
Contact mode

- Constant height or force and friction



CD disk surface
[www.ntmdt.com]

±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)

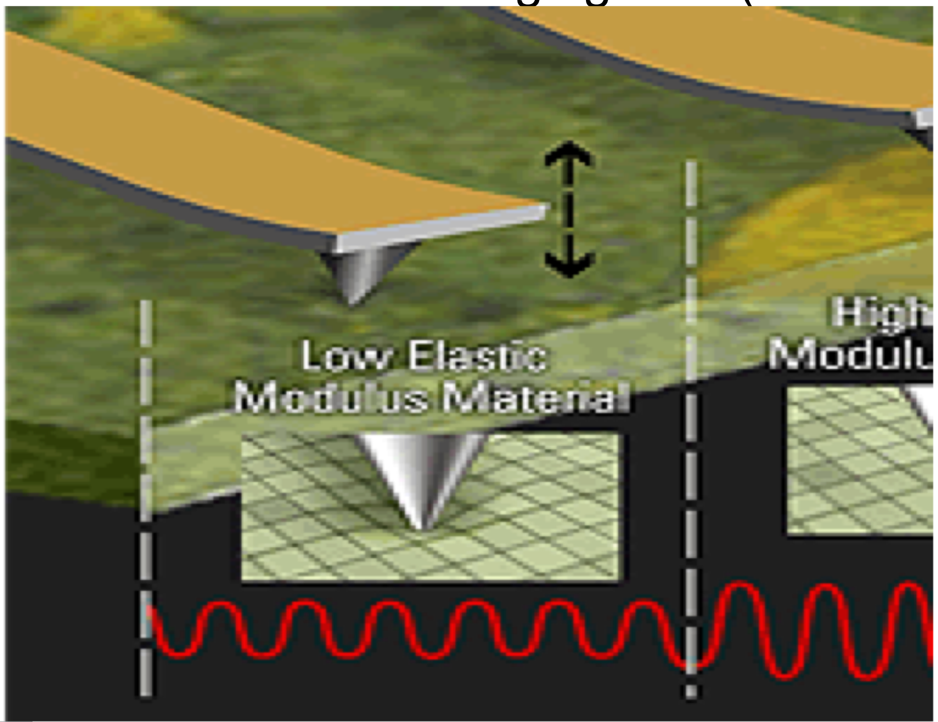


PS-PMMA polymer blend
[Feldman et al., *Langmuir*, 1998]

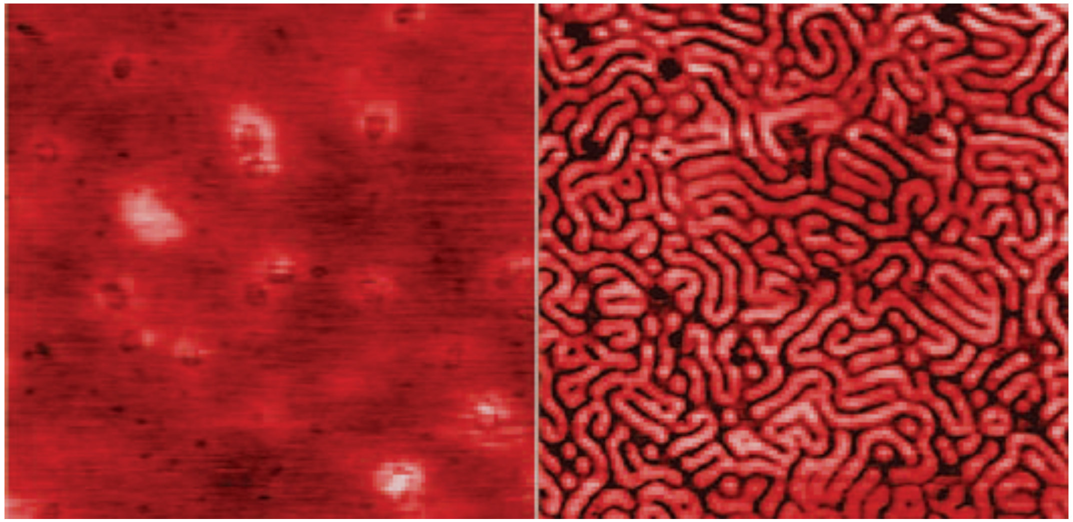
Cantilever with low stiffness (0.01-1 N/m)

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)



[www.afmuniversity.org]

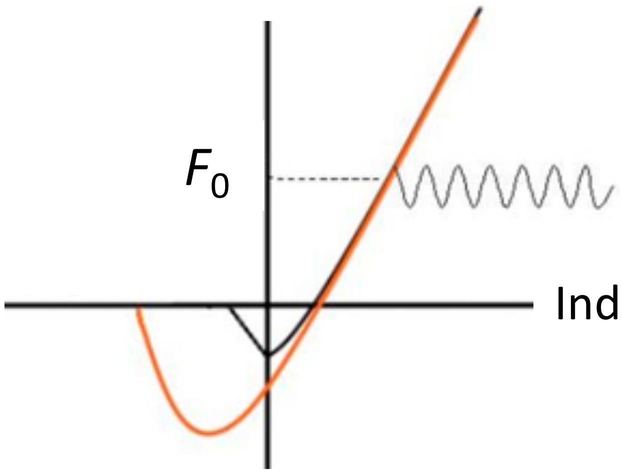


Topography and force modulation images (900 nm scans) of a two-phase block copolymer (softer = black)

[www.veeco.com]

Contact Resonance-AFM (CR-AFM)

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



Applied force is modulated $F = F_0 + F_{excitation} \times \sin\omega t$

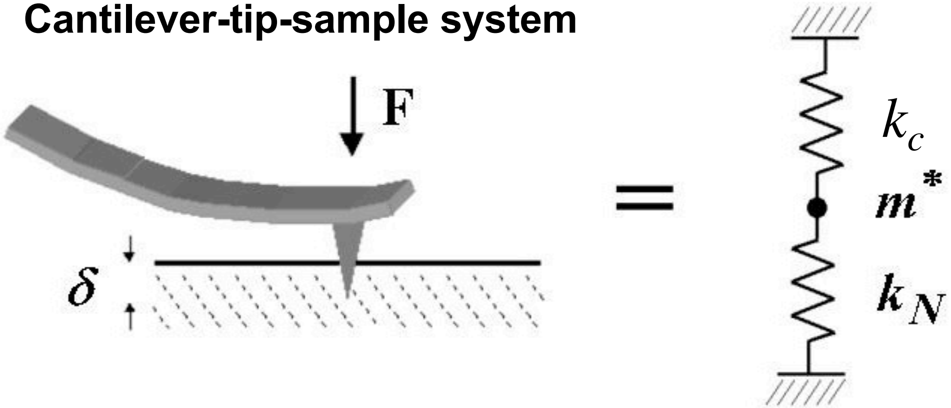
if $F_{excitation}$ is small → ≈<u>LINEAR</u> contact stiffness

if F_0 is small → Good spatial resolution + Hertz theory validity

Point-mass model **[Rabe et al, 1996]**

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

Cantilever-tip-sample system

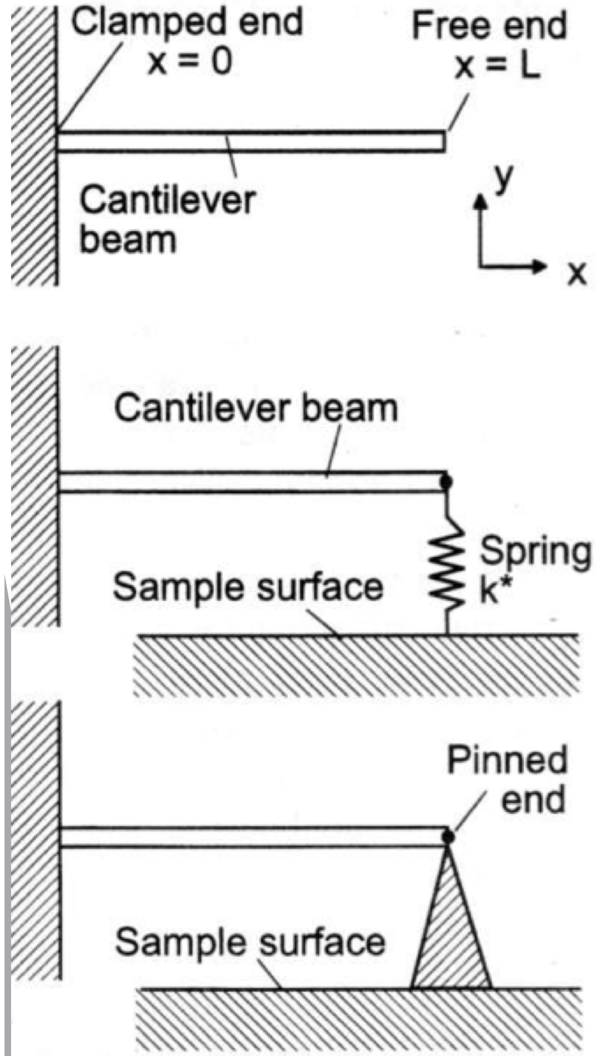


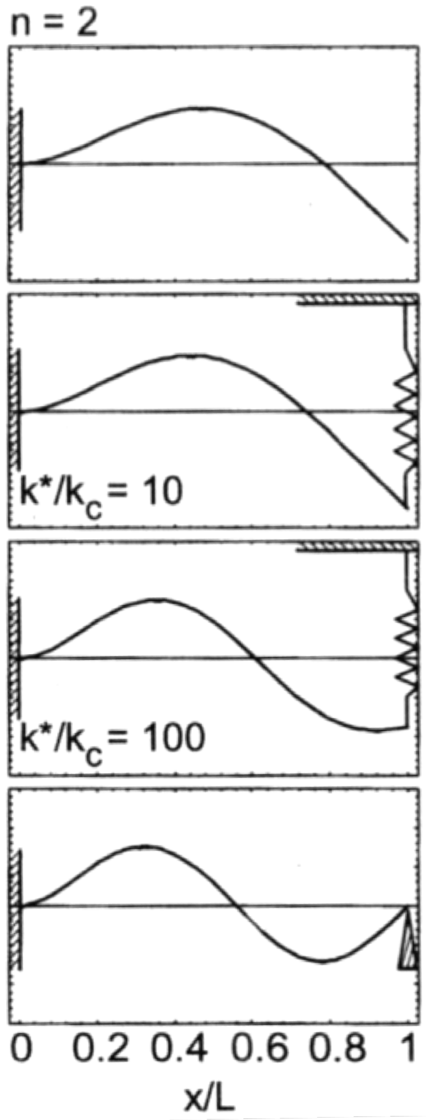
Amplitude of vibration and resonance frequency depend on the contact stiffness k_N… but how?

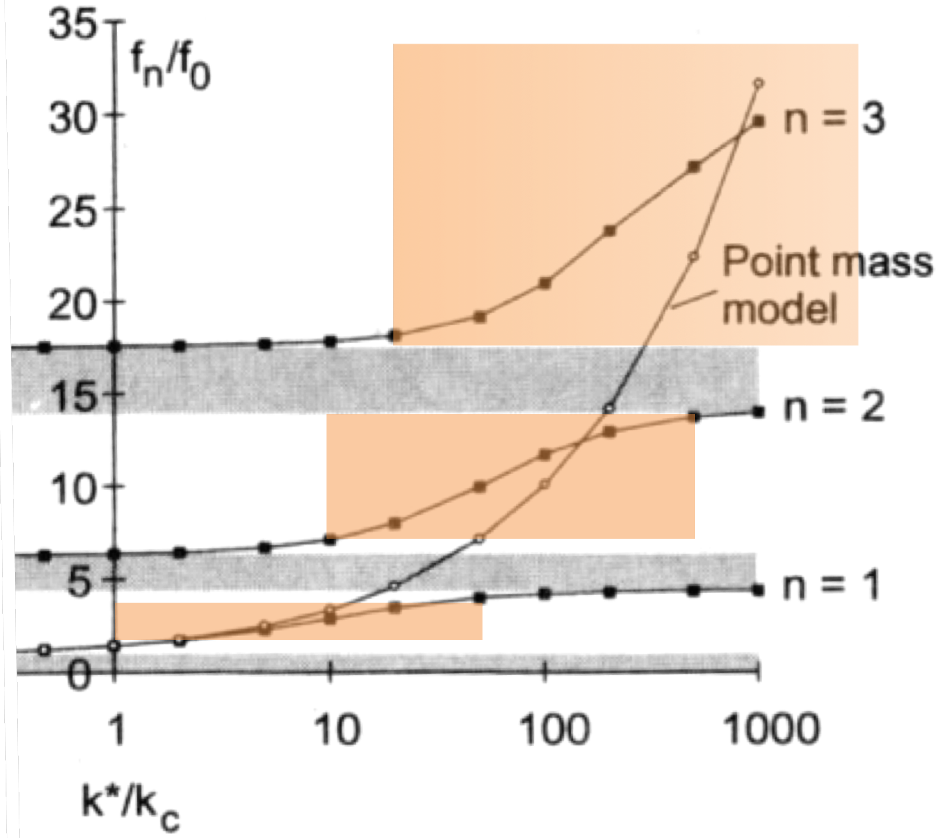
Contact Resonance-AFM (CR-AFM)

Cantilever dynamic and response

[Rabe *et al*, 1996]

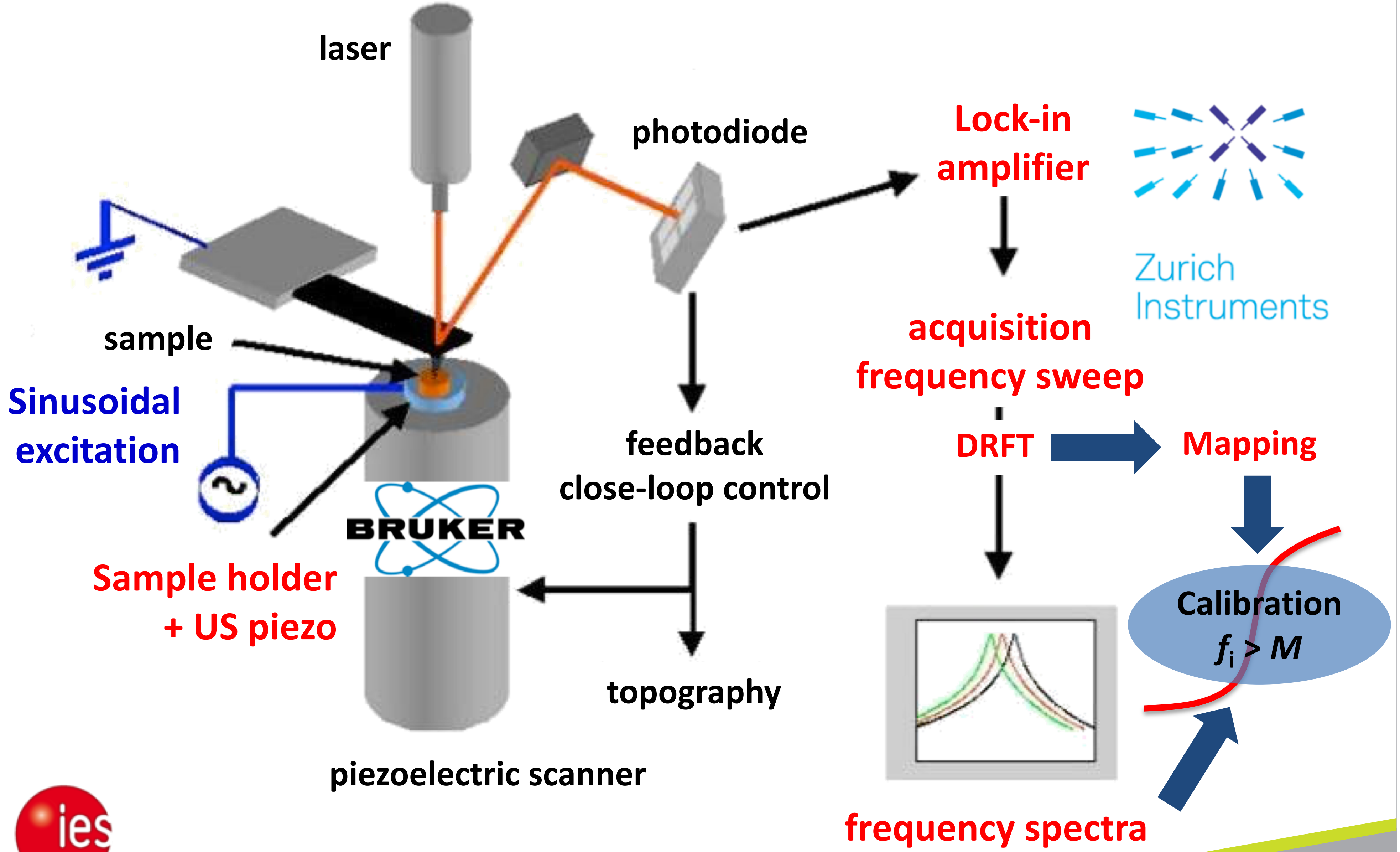




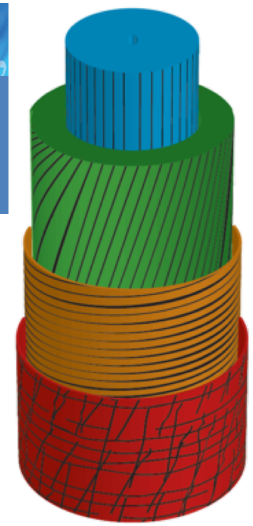


Contact Resonance-AFM principle with Dual Resonance Frequency Tracking

[Arinero and Lévêque, Rev. Phys. Instr., 2003]
[Gannepalli *et al.*, Nanotechnol. 2011]



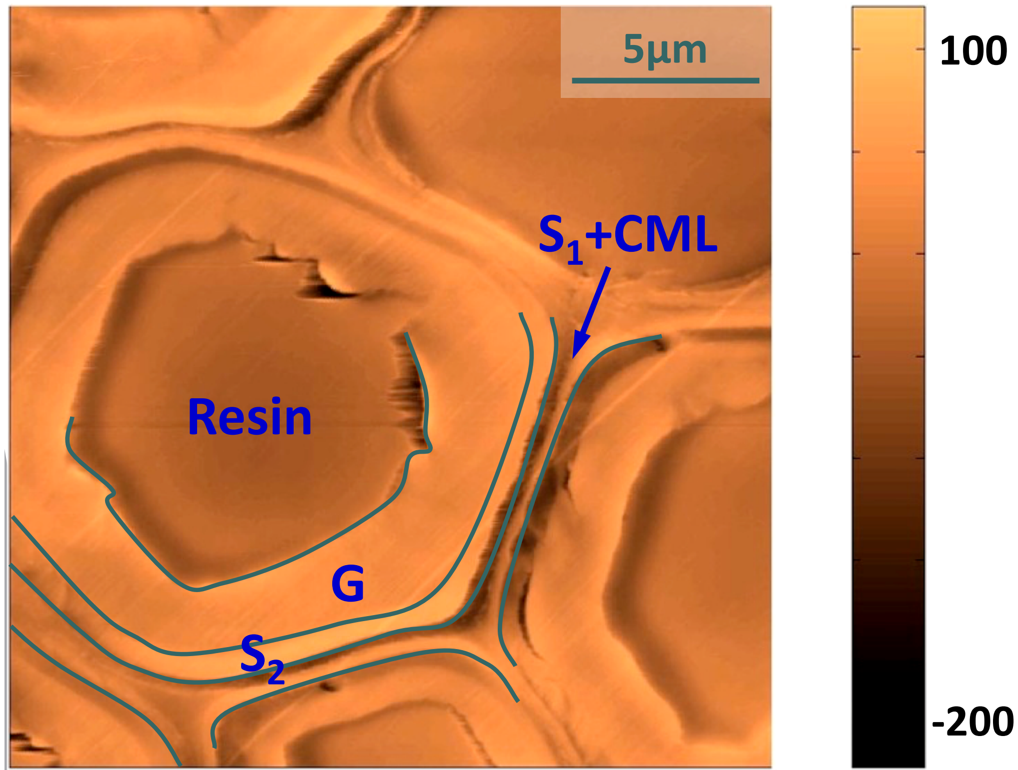
Chestnut tension wood



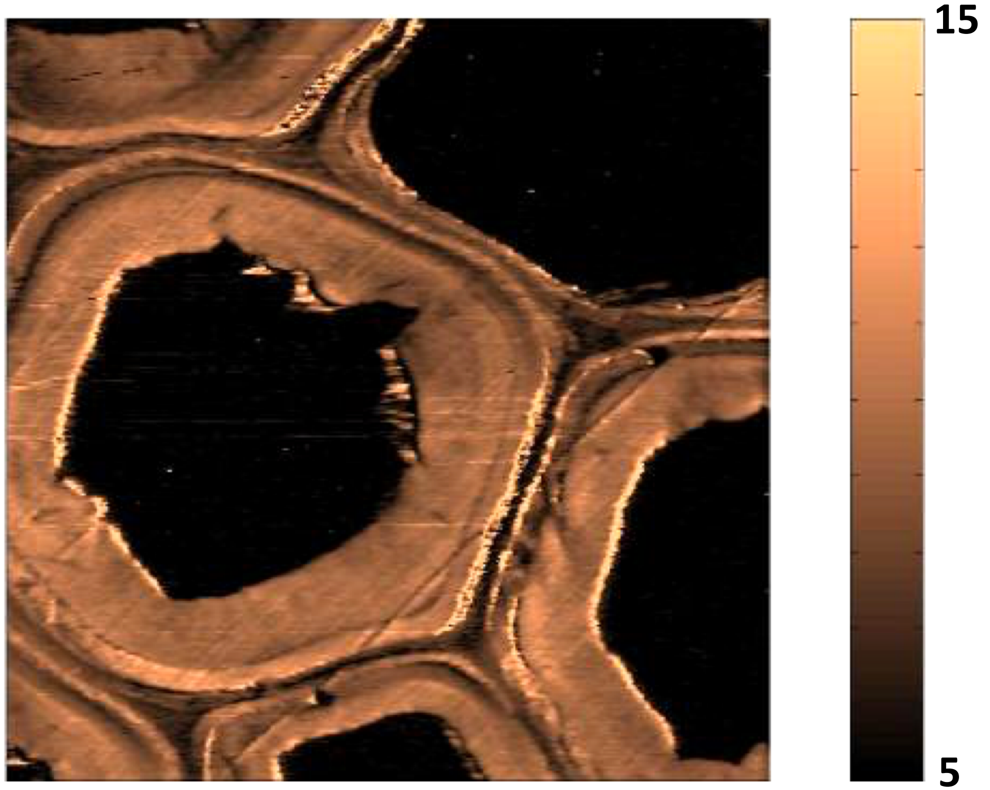
[Arnould and Arinero, Comp. A, 2015, 74: 79-76]

Effect of the topography and sample orientation

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



Topography (nm)



Contact modulus (GPa)

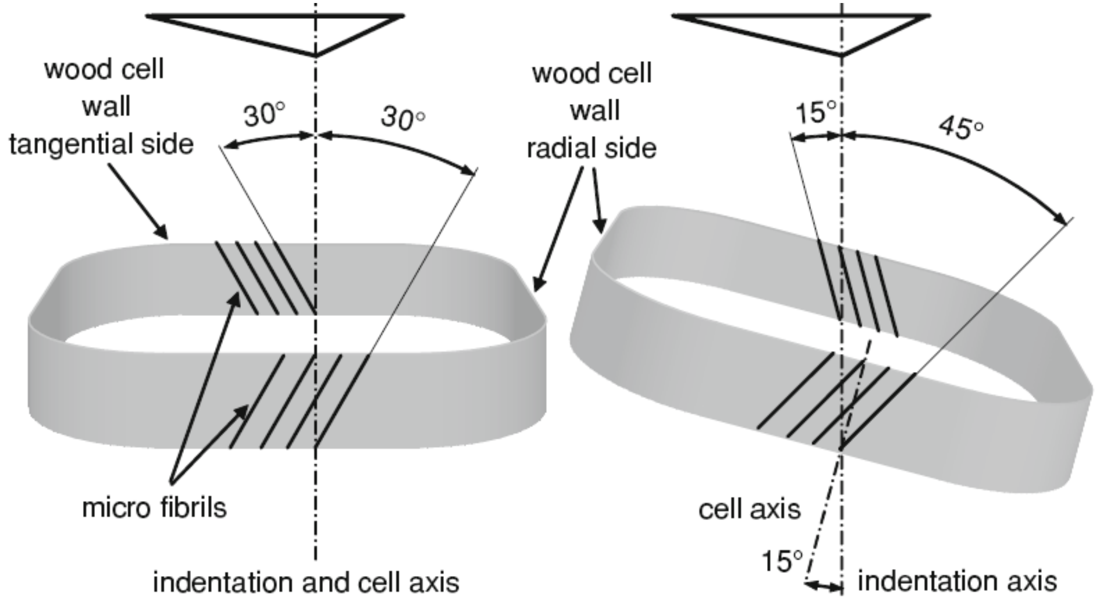
Veeco Enviroscope, CR-AFM, Nanoworld ARROW FMR, 2,8 N/m, 55 nm (adh), 180 nN

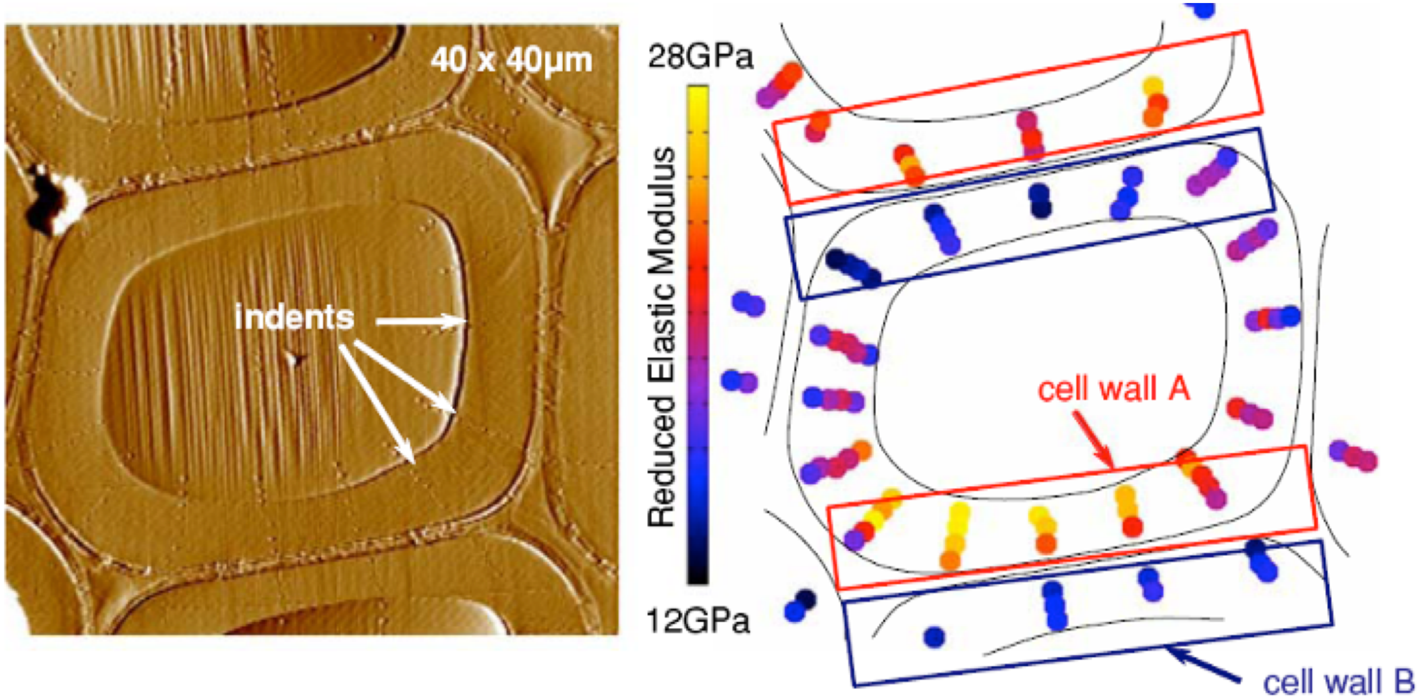
Chestnut tension wood

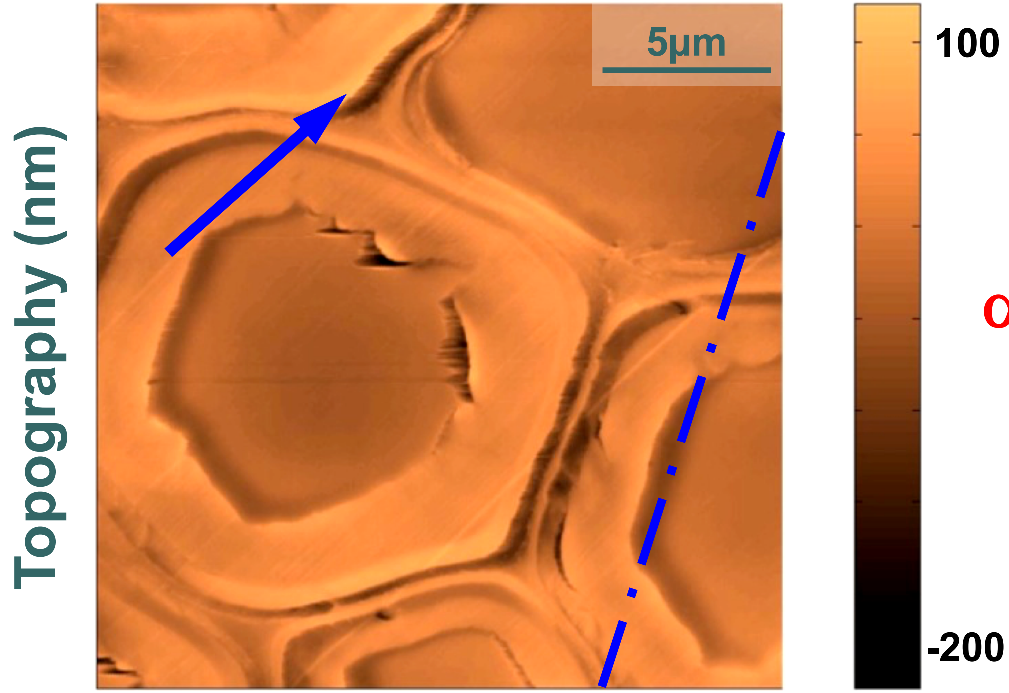
[Arnould and Arinero, Comp. A, 2015]

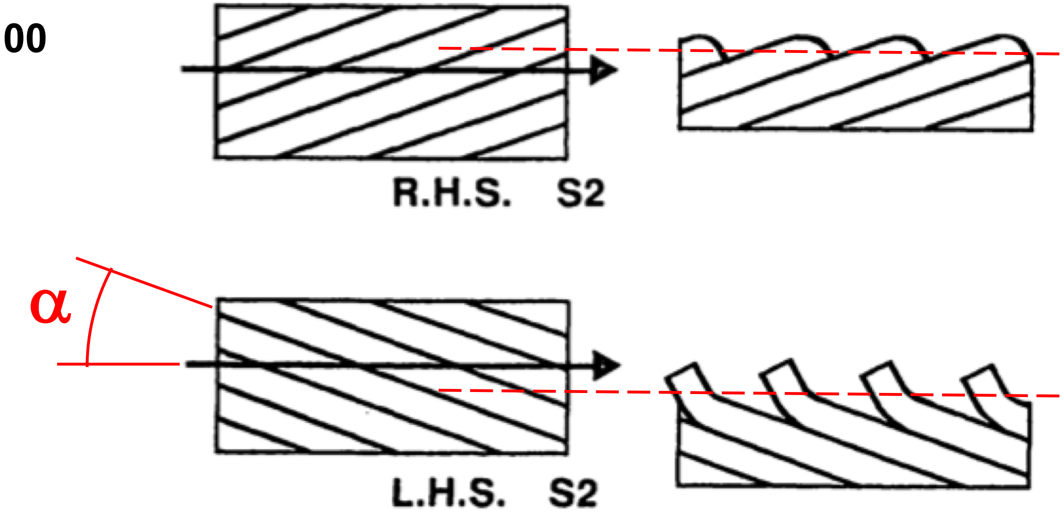
Effect of sample orientation

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



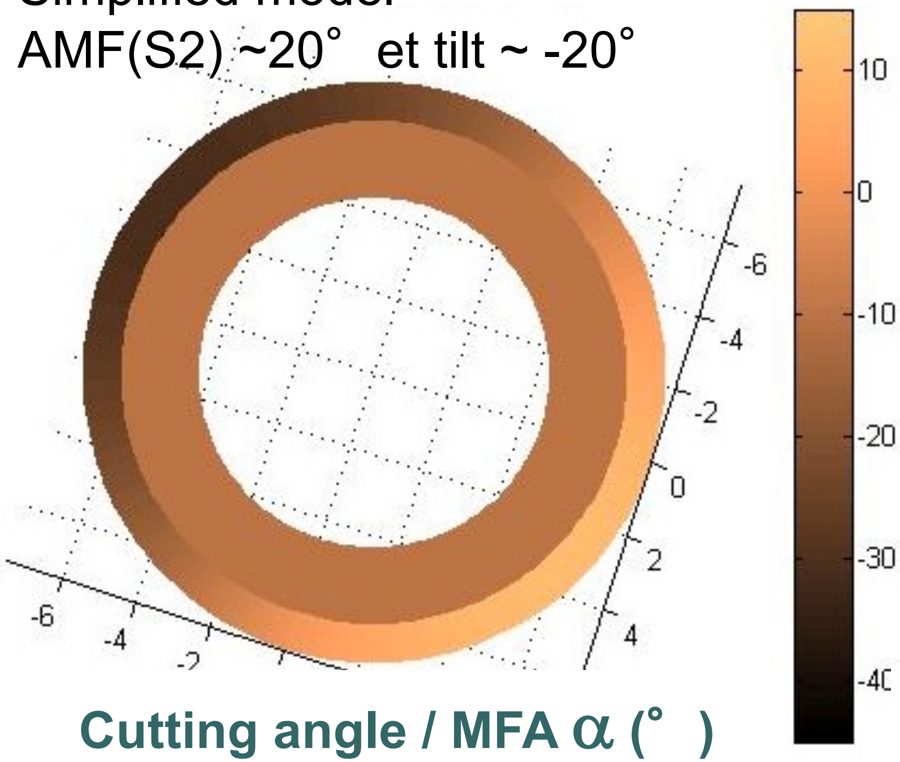




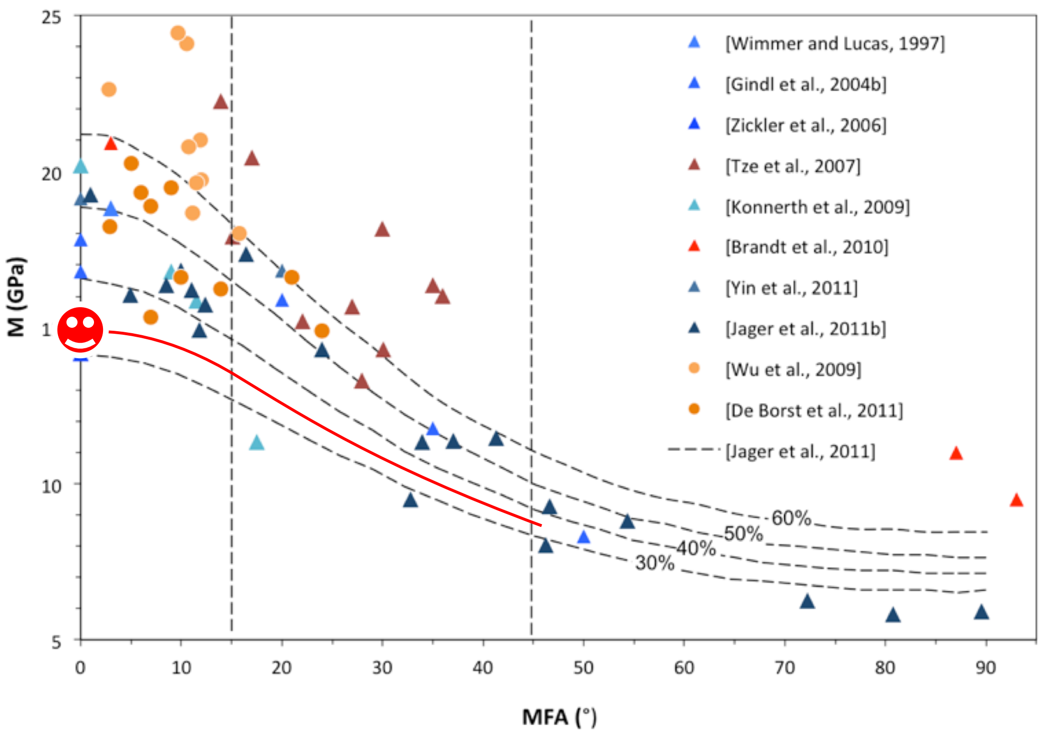


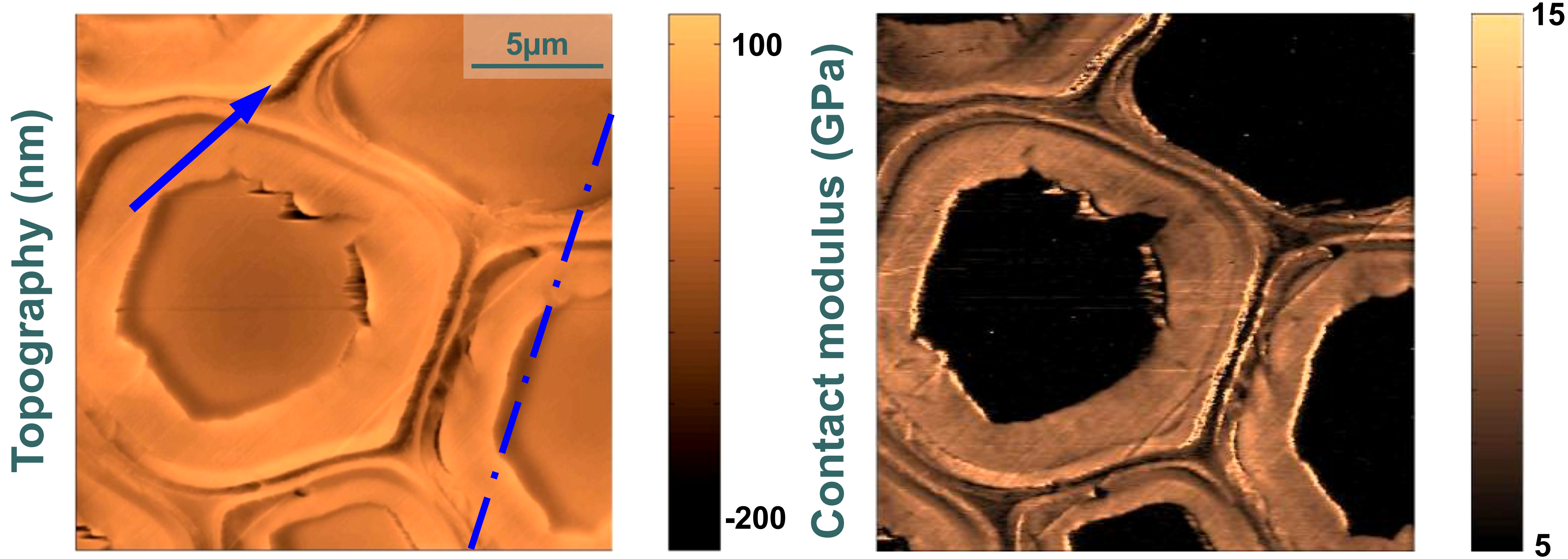
[Shaune et al, Holzforschung, 1994]

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

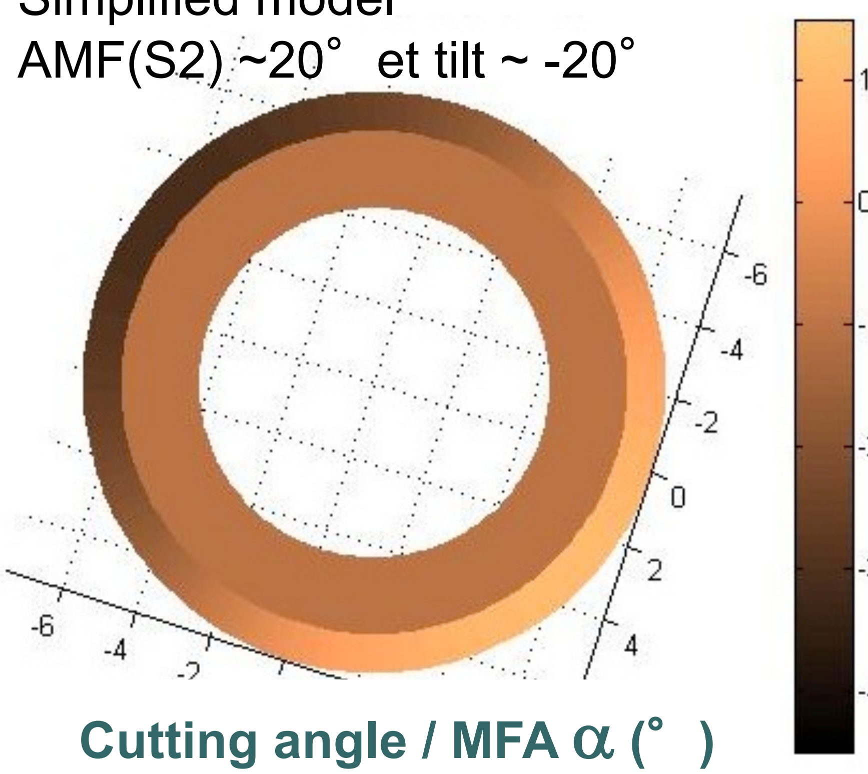


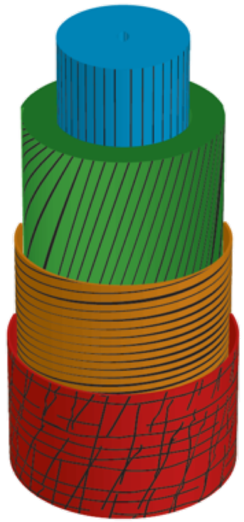
Cutting angle / MFA α (°)

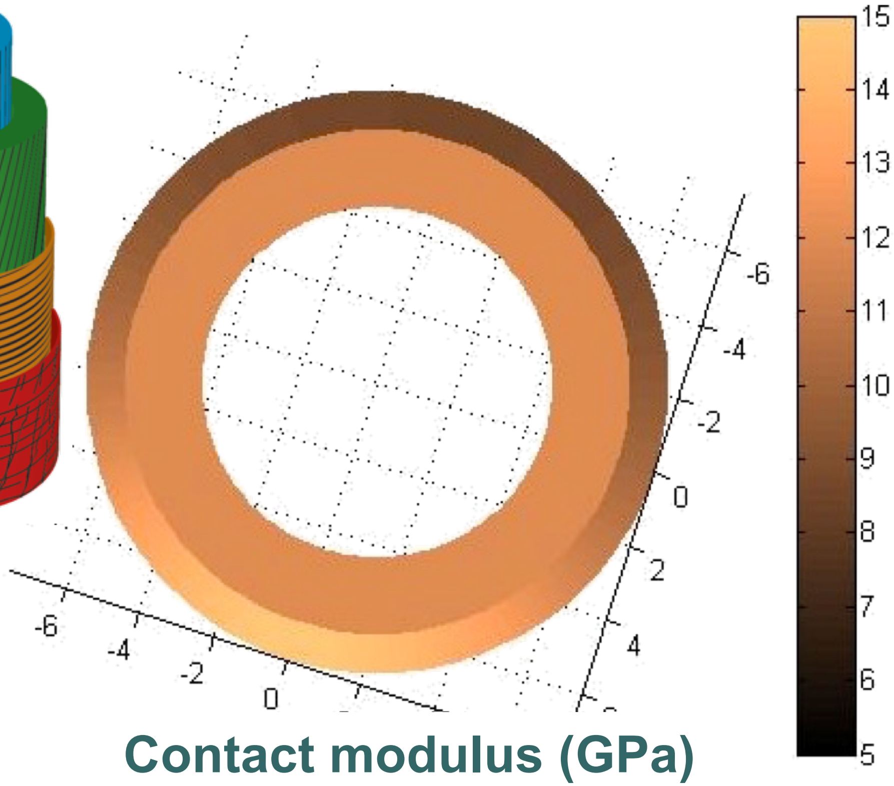




Simplified model
AMF(S2) ~20° et tilt ~ -20°

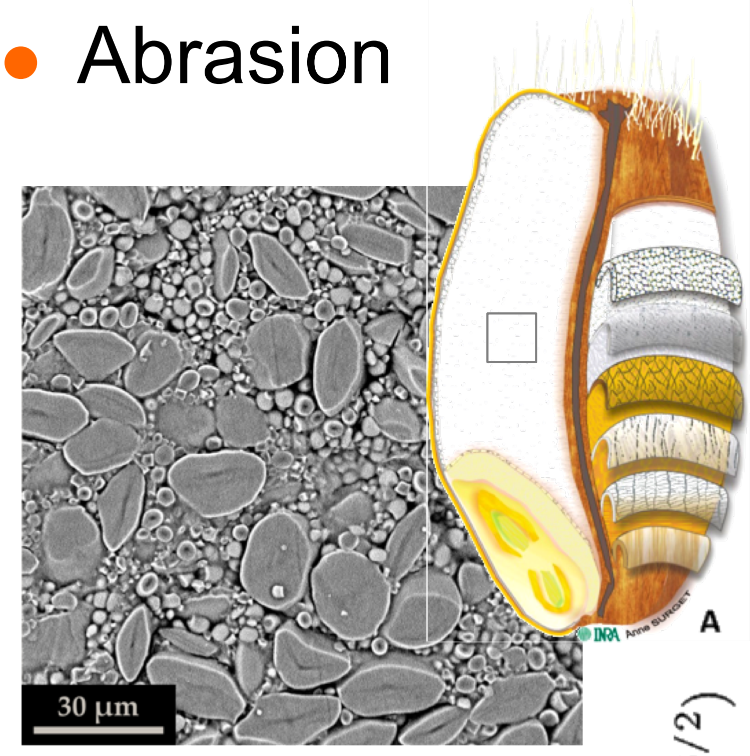




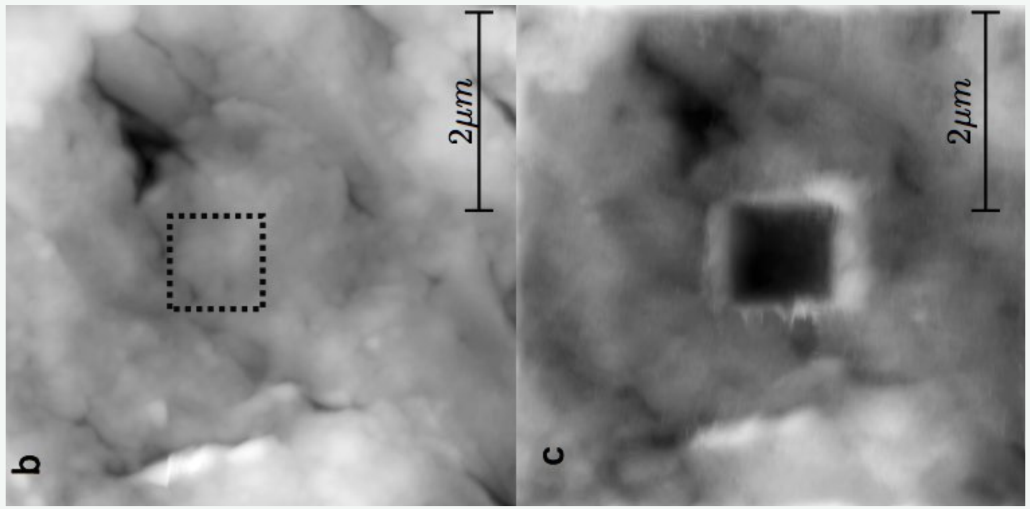


Contact mode

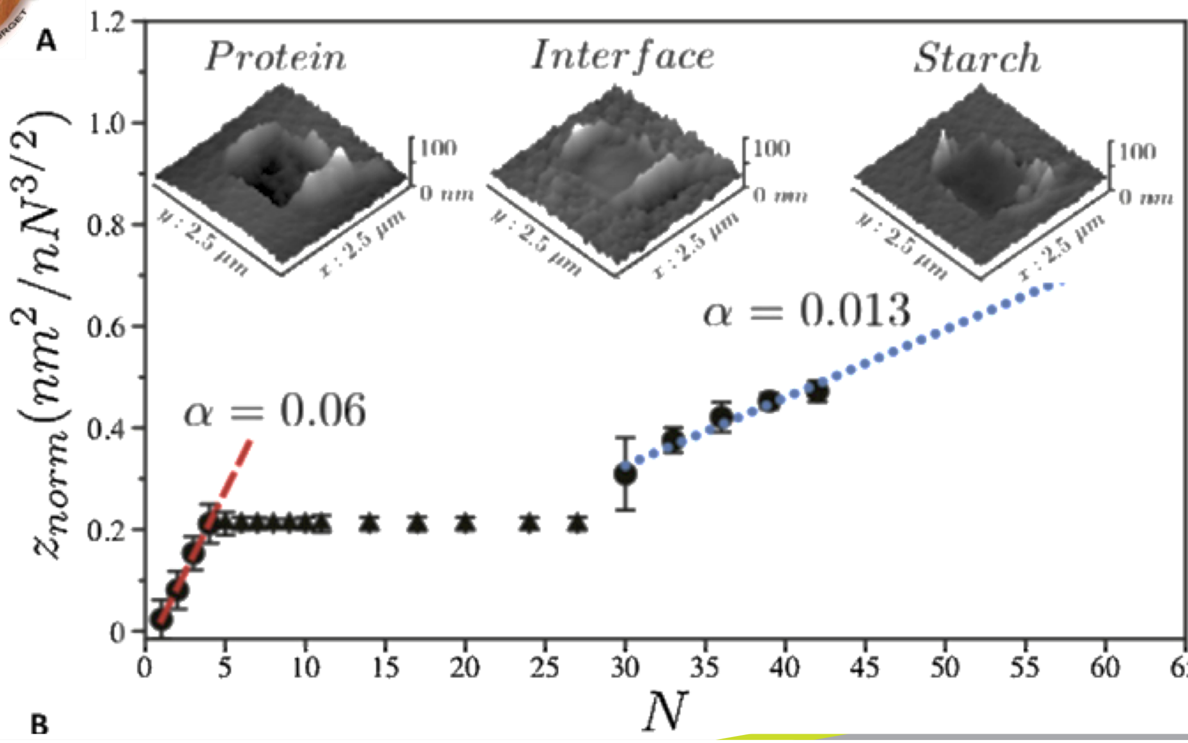
- Abrasion



Starch granules inside a wheat endosperm (SEM)



Tablet of pure gluten before/after abrasion (SEM)



Occurrence of “lubricated” interface in soft wheat

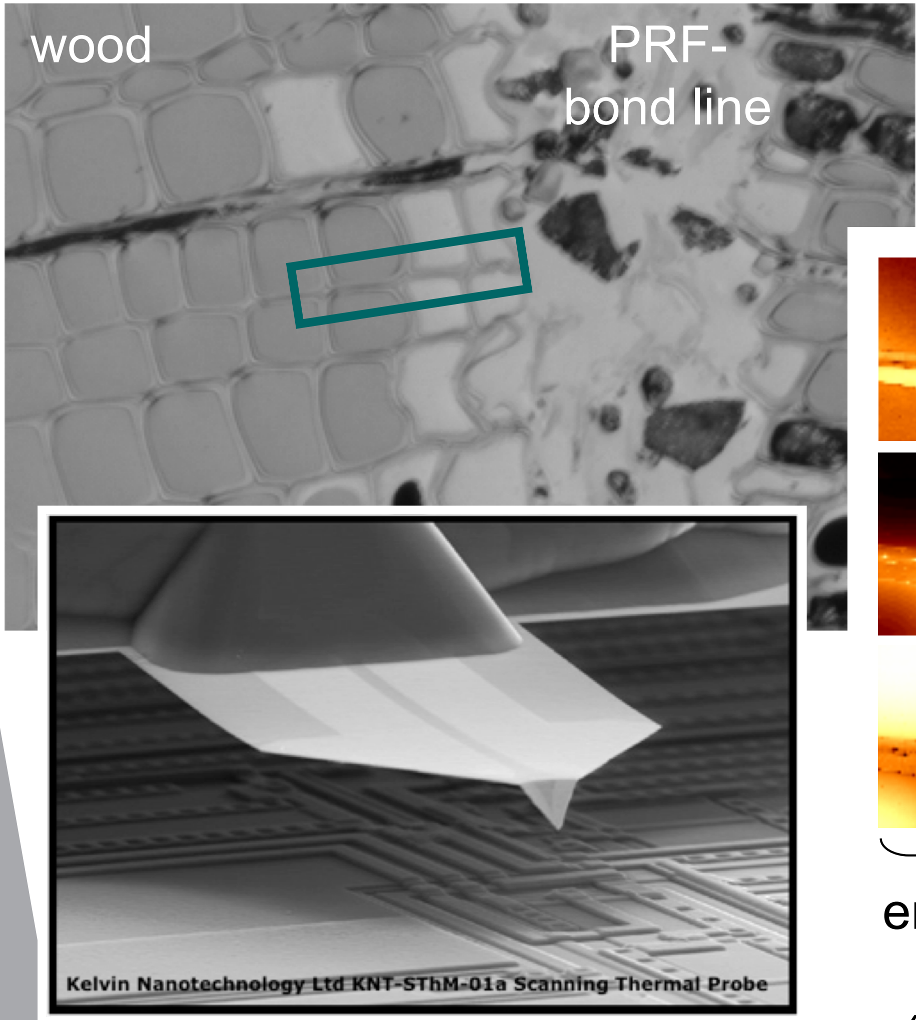
Chichti et al, Eur. Polym. J., 2013, 49(12): 3788-3795
Chichti et al, Plant Sci., 2015, 239: 1-8

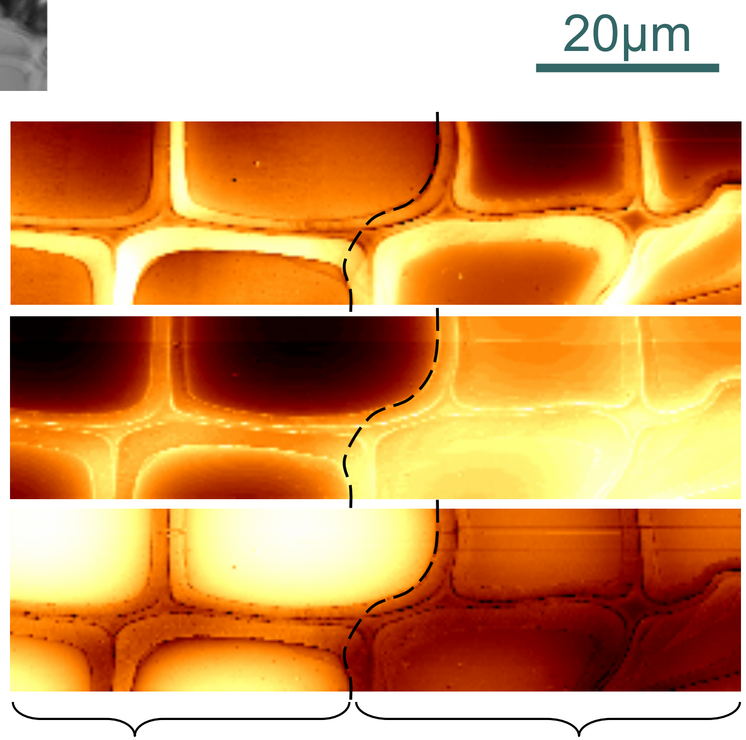
Contact mode

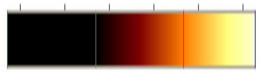
- Scanning Thermal Microscopy (SThM)

[Konnerth *et al*., Holzforschung, 2008]

$R \approx 100$ nm
Scan rate 0.1-0.5 Hz
Spruce wood

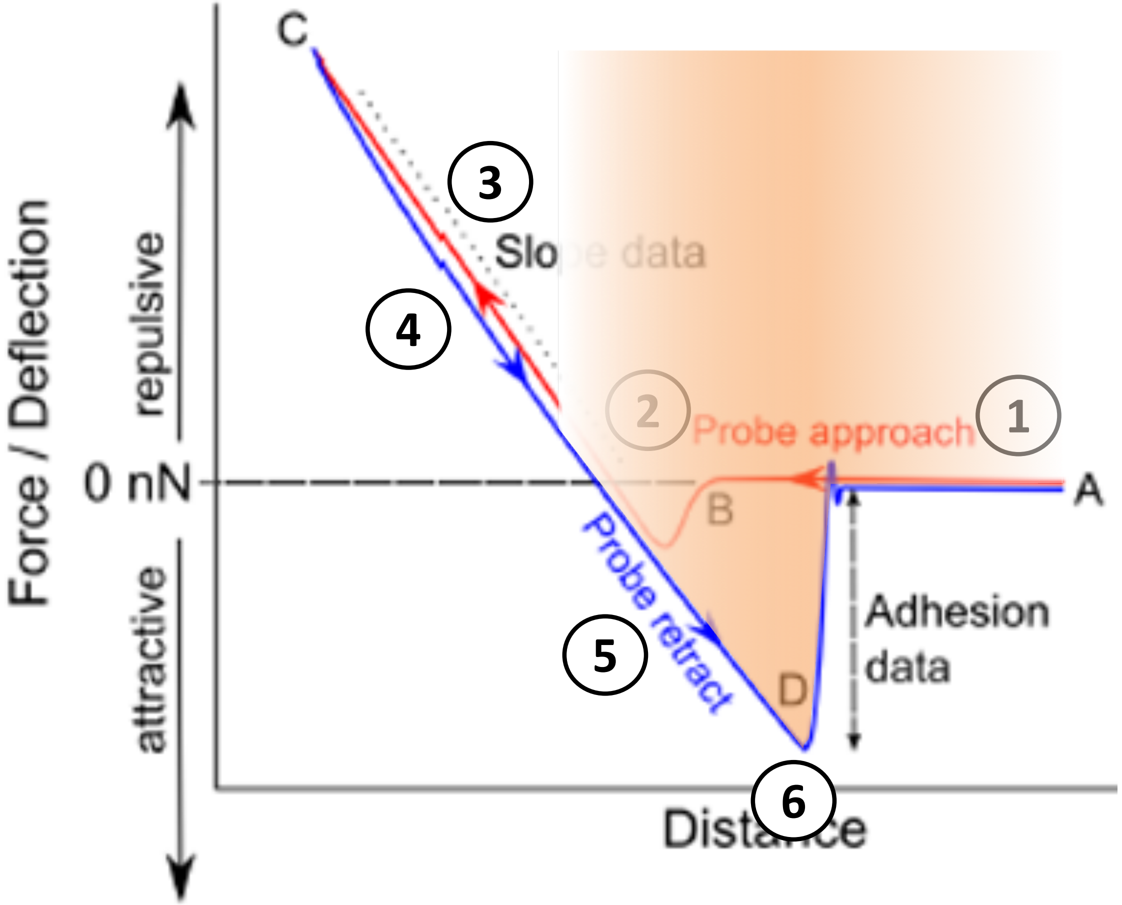


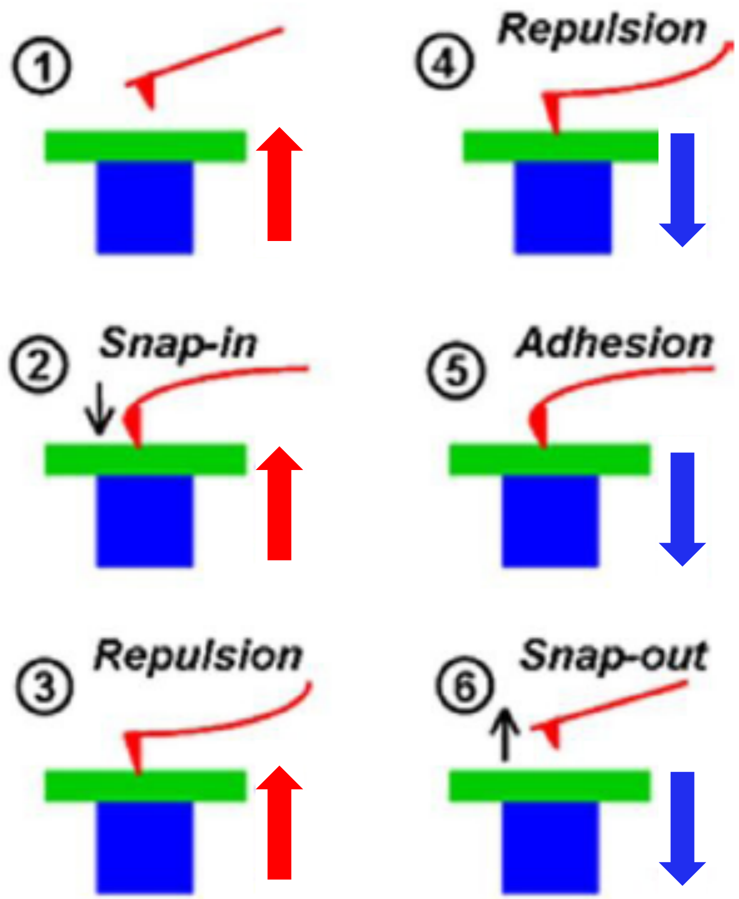




Force-distance curve

- Van der Waals force, electrostatic force, contact stiffness, adhesion force, …

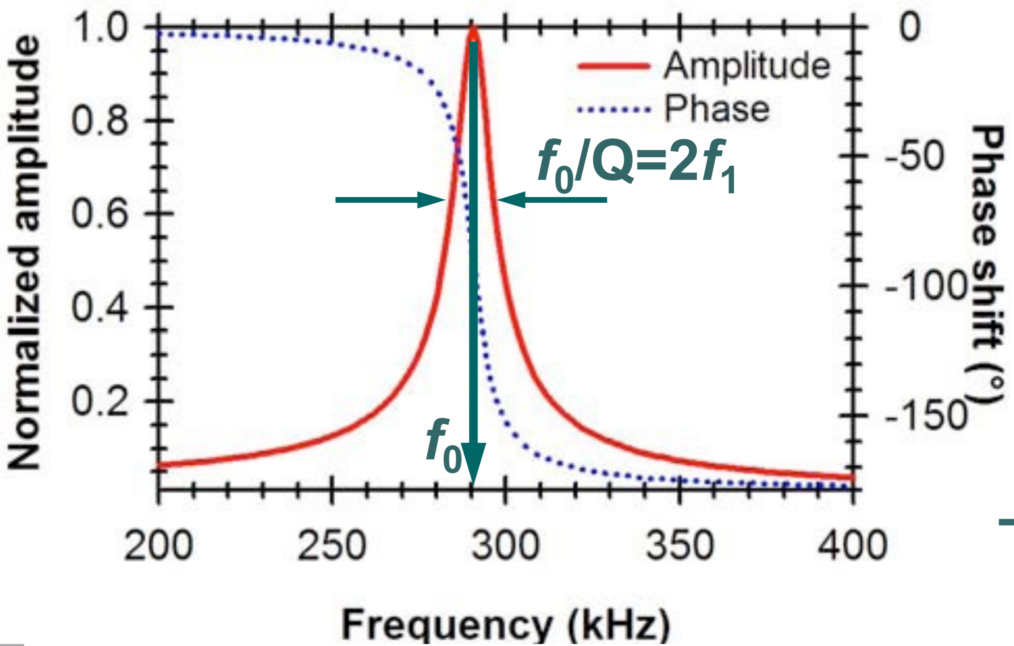




[B. Nysten, 2007]

Intermittent contact/tapping™/…

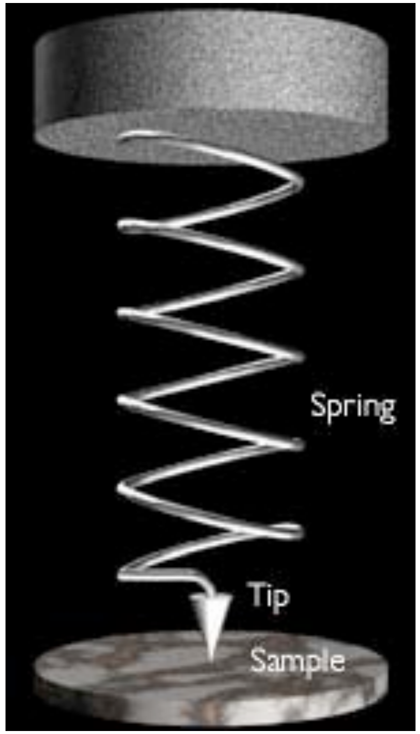
- Knocking on the surface!
  - ➲ Linear harmonic oscillator approximation (free)



$$\omega_0 = \sqrt{\frac{k_C}{m_{\mathrm{eff}}}}$$

$k_C > 10\mathrm{N/m}$

$f_0 > 100\mathrm{kHz}$



Media	Liquid	Air	High vacuum
Q	10	400	10^4

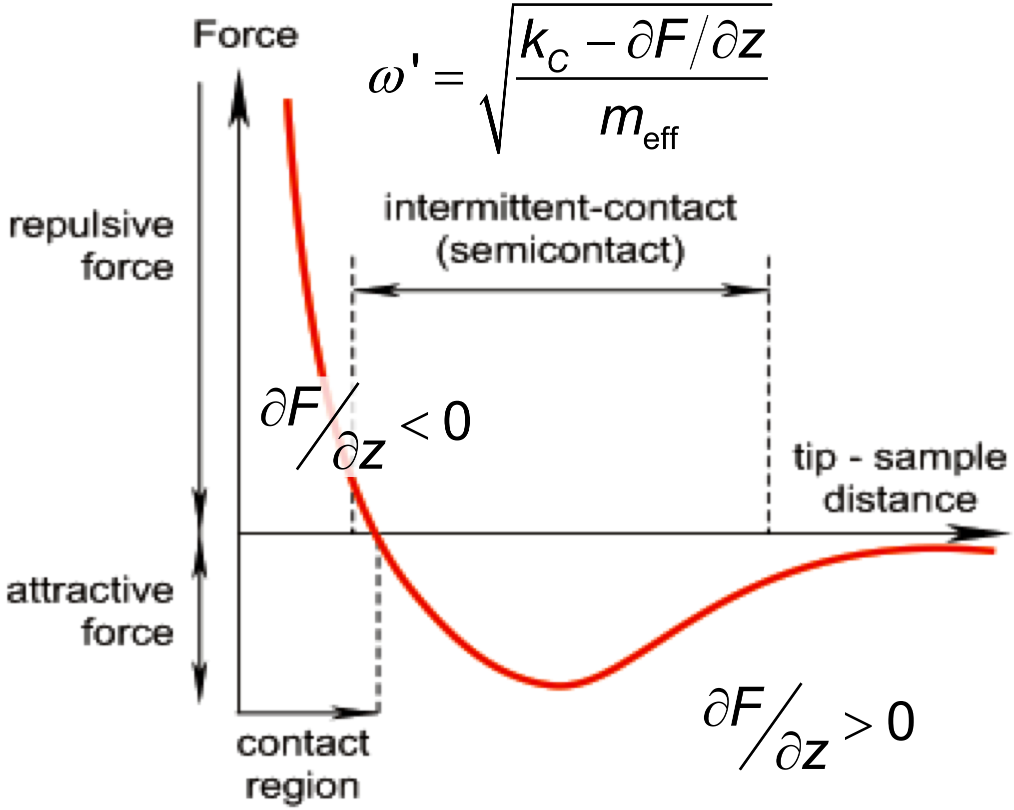
Intermittent contact/tapping™/…

- Scan at constant vibration amplitude = topography + stiffness contrast

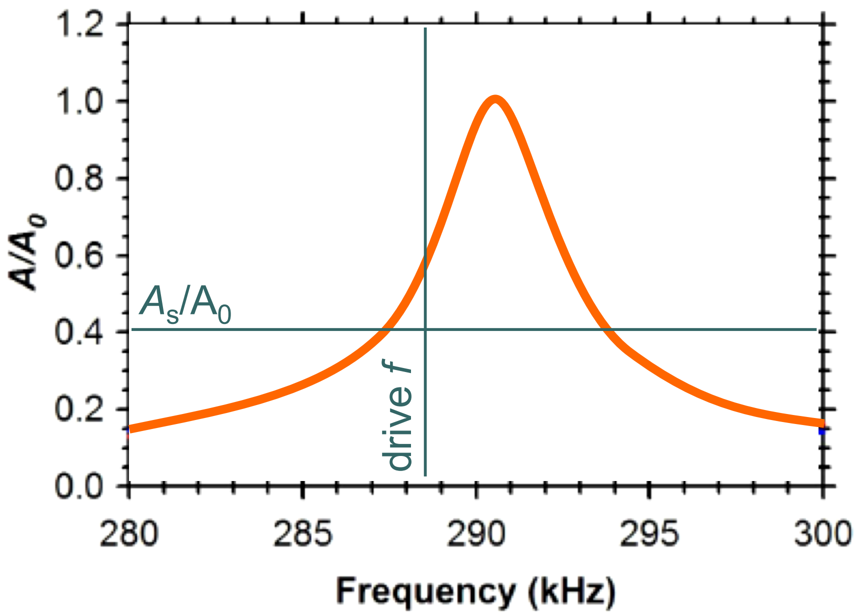
→ soft (topography) $A_s/A_0 > 0.8 + A_0\downarrow$ / hard (elasticity) $A_s/A_0 < 0.4 + A_s\uparrow$ tapping

- Linear harmonic oscillator approximation in a force gradient
  [Magonov et al., *Surf. Sci. Lett.*, 1997; Nysten, 2007]

$$\omega' = \sqrt{\frac{k_C - \partial F/\partial z}{m_{\mathrm{eff}}}}$$



$$\frac{\Delta\omega}{\omega_0} \approx \frac{1}{2k_C}\frac{\partial F}{\partial z}$$



Intermittent contact/tapping™/…

- What are we measuring?

➲ Linear harmonic oscillator approximation in a force gradient
[Magonov et al., Surf. Sci. Lett., 1997; Nysten, 2007]

$$\Delta\varphi \approx \frac{Q}{k_C}\frac{\partial F}{\partial z} \text{ if } \omega \sim \omega' \sim \omega_0 \text{ and } \frac{\partial F}{\partial z} \ll \frac{k_C}{Q}$$

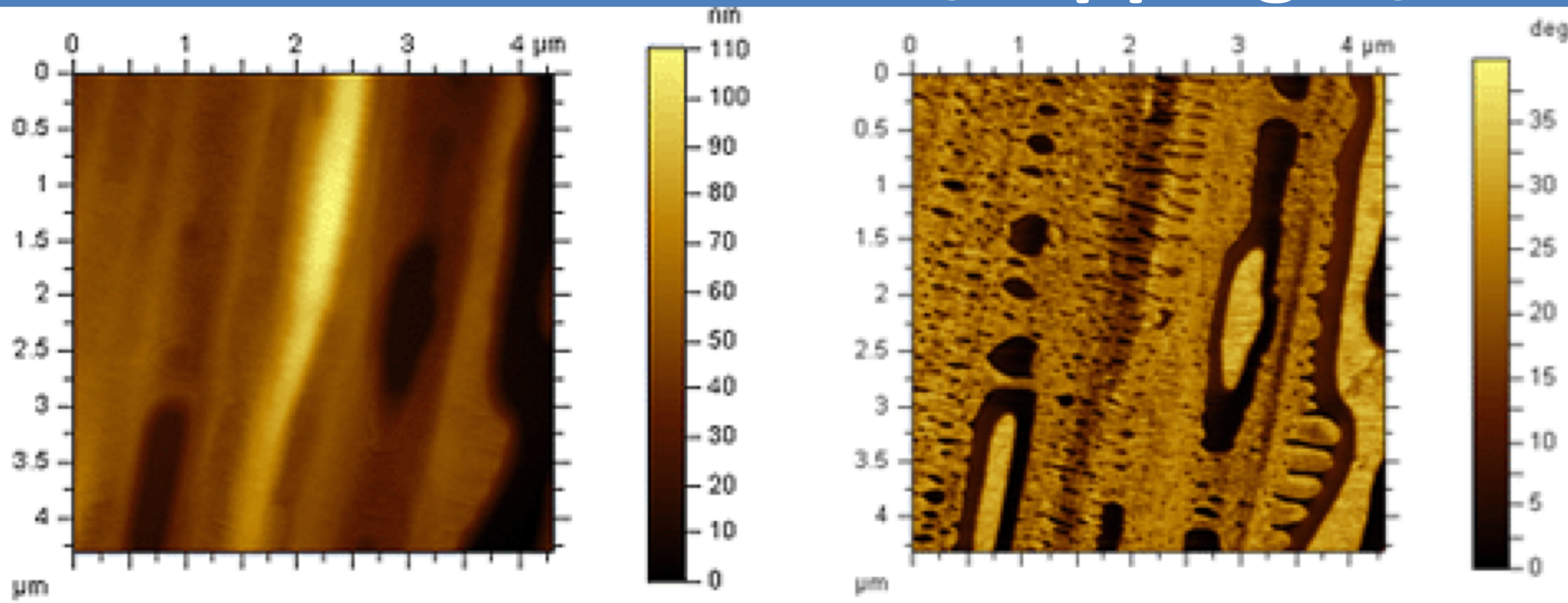
Pure elastic material with Hertz: $F = \frac{4}{3}E^*\sqrt{R}z^{3/2} \rightarrow \frac{\partial F}{\partial z} \approx 2E^*a$ ➡ $\boxed{\Delta\varphi \approx \frac{Q}{k_C}2E^*a}$

➲ Phase-lag proportional to the contact stiffness in certain condition (k_N<<k_C/Q + A_0↑ and A_s↓) → phase images…
but more generally "proportional" to any dissipation (capillarity, viscoelasticity, adhesion, …) ➡ difficult to interpret quantitatively!

➲ High resolution (<10nm) and <u>±slow</u> imaging (cantilever with high frequency)

➲ Less tip and sample damages, ~no friction, …

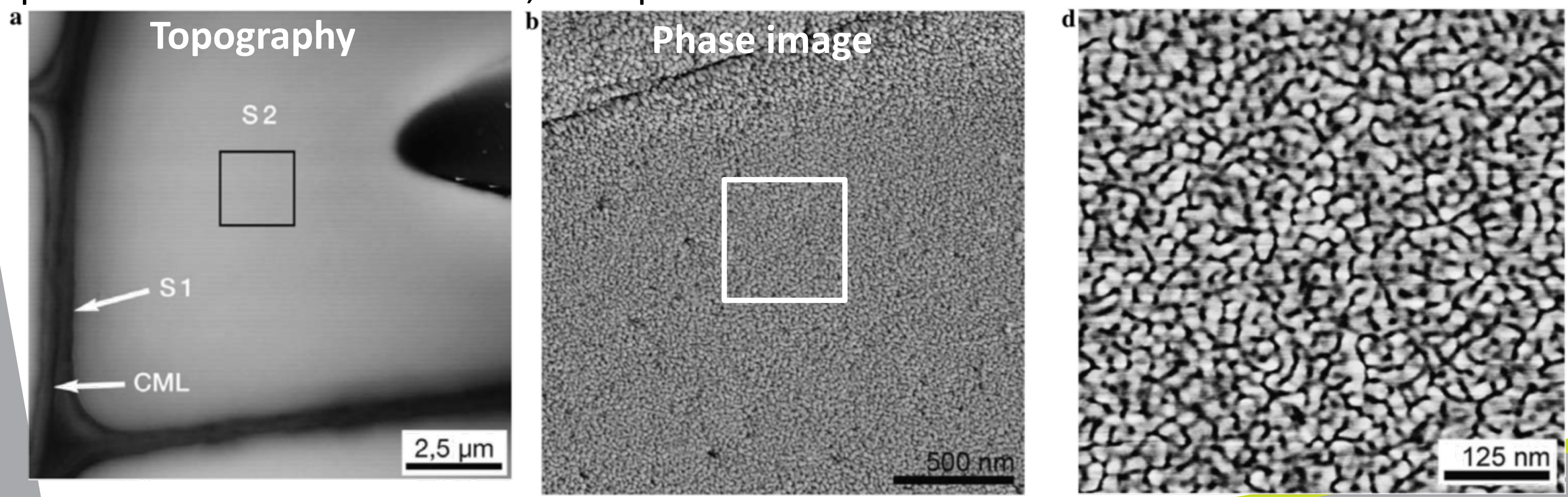
Intermittent contact/tapping™/…



(Figure 6a & b) Topographic (Left) and phase image (Right) of polydiethysiloxane polymer

[www.afmuniversity.com]

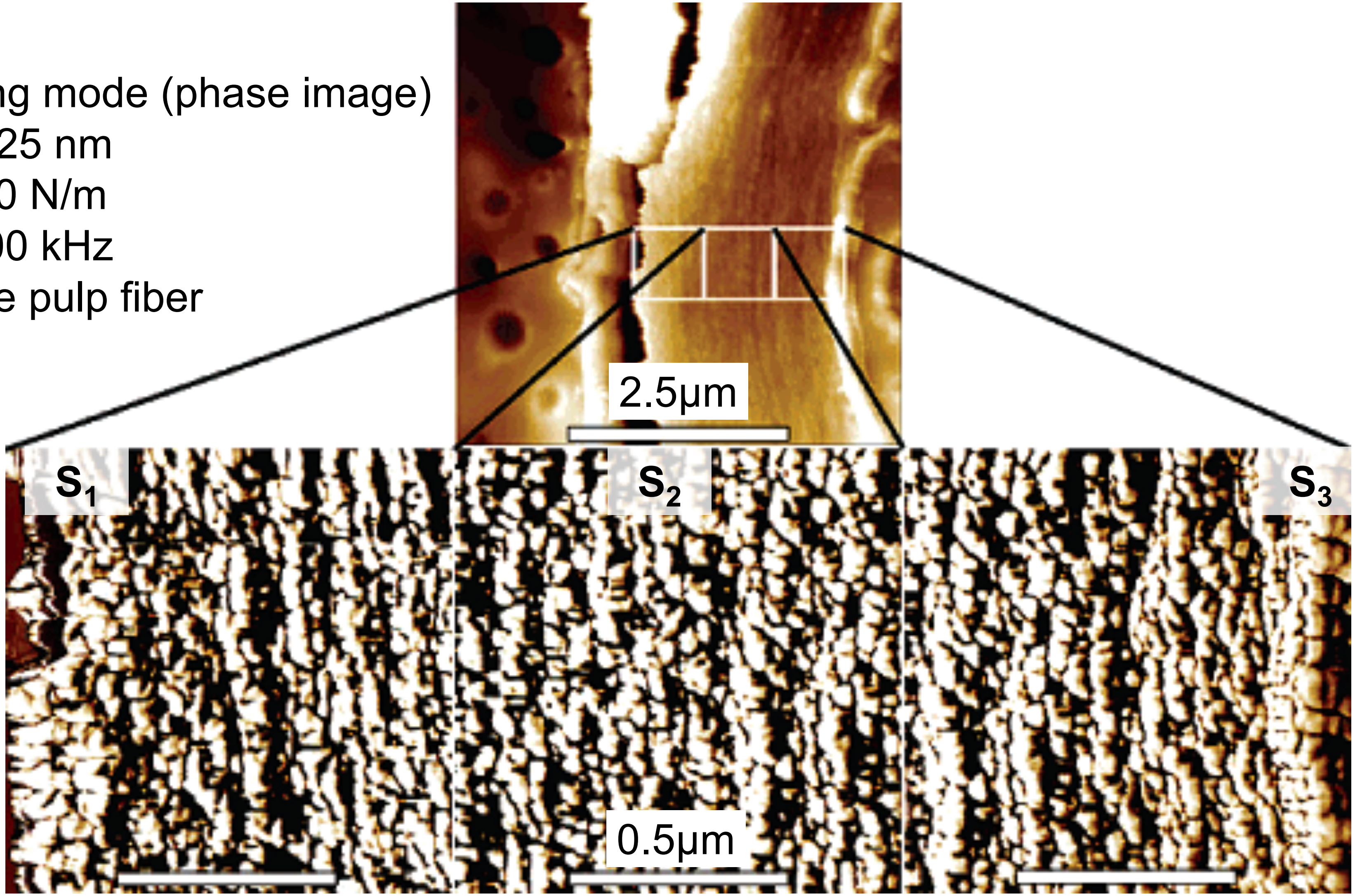
Spruce tracheid wood cell wall, AFM probe and condition?



Zimmermann et al, J. Ultrastruct. Biol., 2006, 156 (2): 363-369

Intermittent contact/tapping™/…

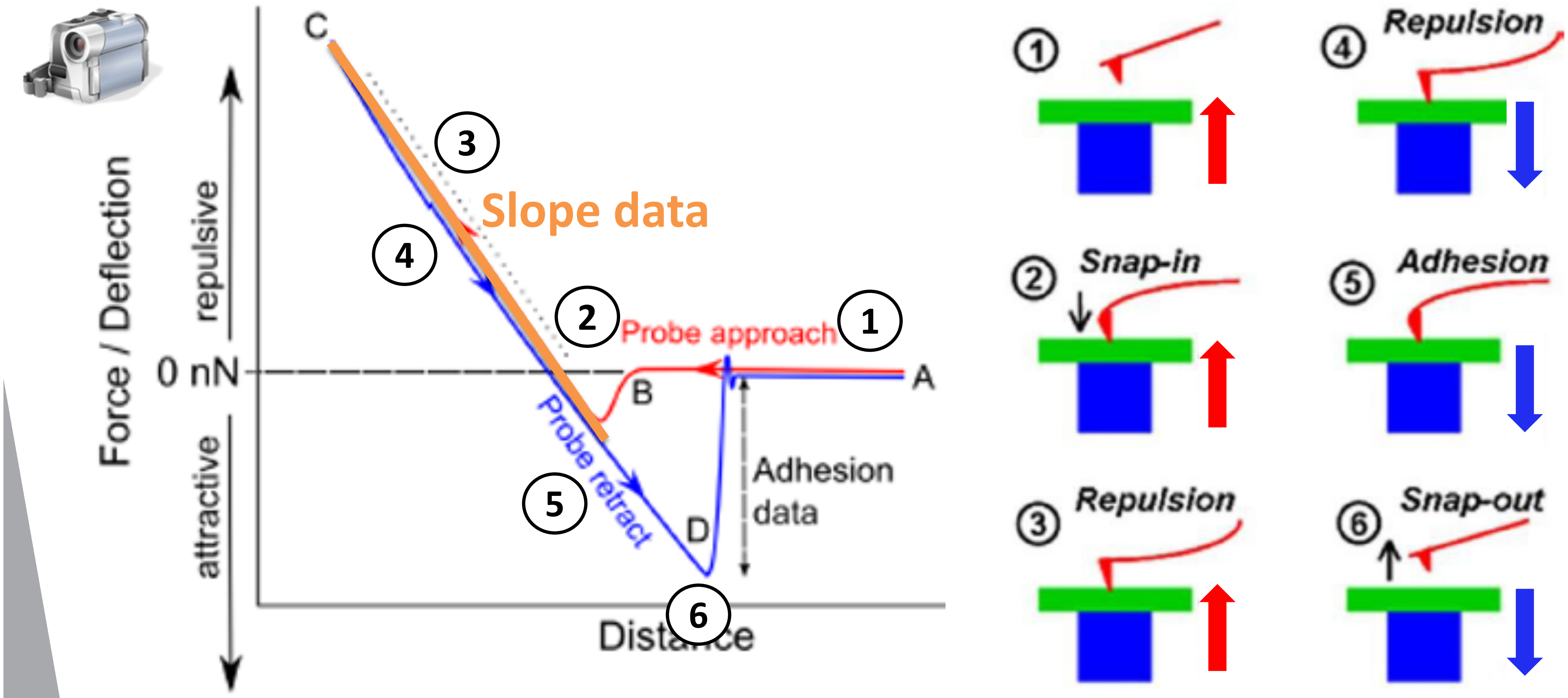
Tapping mode (phase image)
$R \approx 4.25$ nm
$k_C \approx 40$ N/m
$f_0 = 300$ kHz
Spruce pulp fiber



[Fahlén and Salmén, Biomacromolecules, 2005]

Force-distance curve

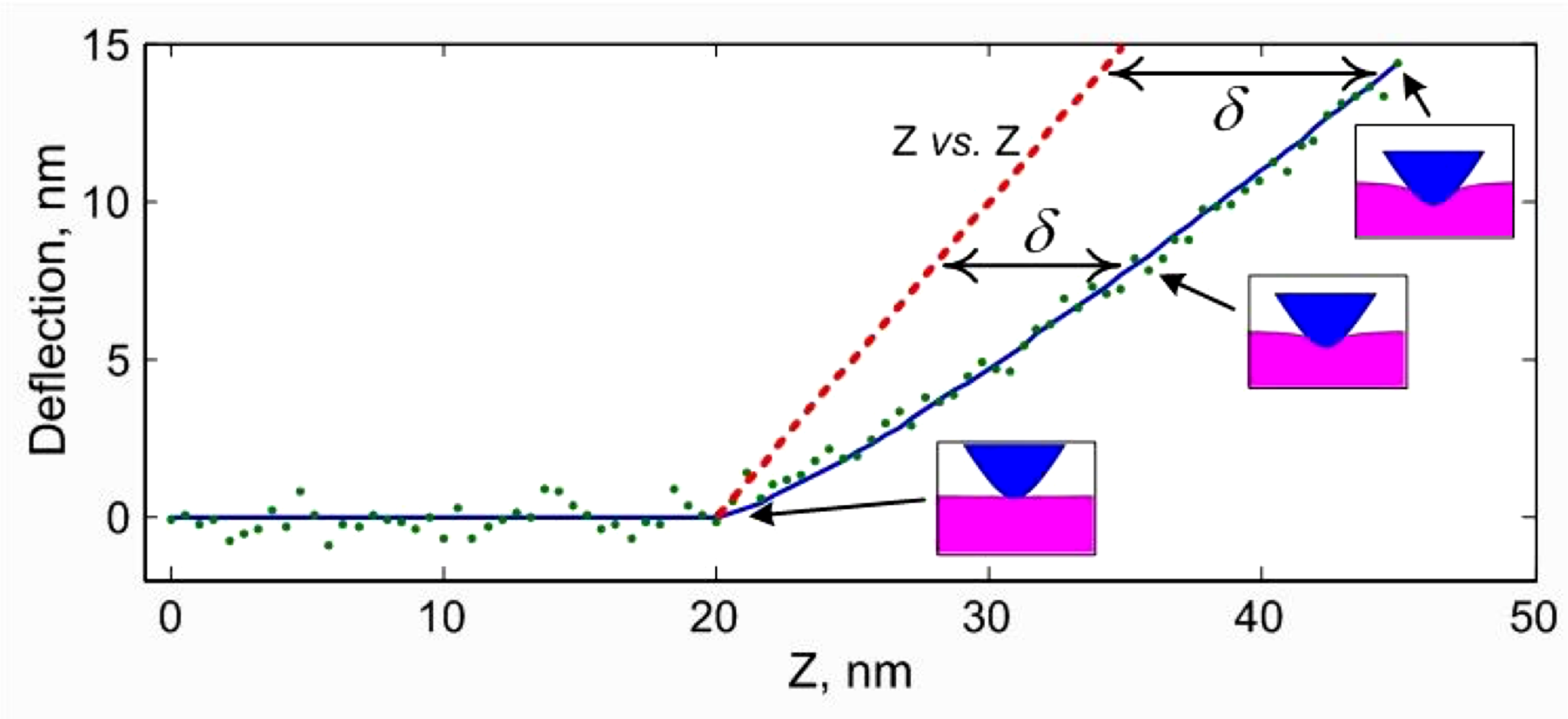
- Van der Waals force, electrostatic force, contact stiffness, adhesion force, …



[B. Nysten, 2007]

Fd - Indentation

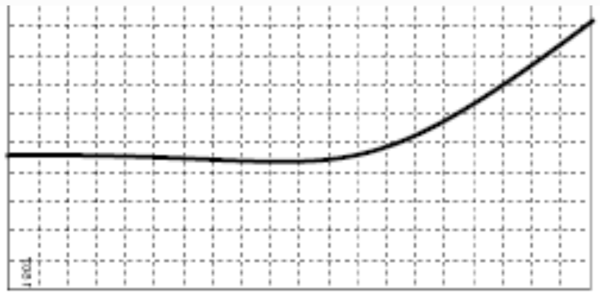
- Decoupling load/displacement



[www.chem.duke.edu/~boris/research/elasticity/]

Hard sample

[www.veeco.com]



Soft sample

Fd - Indentation

- Requirements:
  - Cantilever stiffness

$$\frac{d}{z} = \frac{1}{1+\frac{k_C}{k_N}}$$

$$k_N \gg k_C \rightarrow d = z$$

$$k_N \ll k_C \rightarrow d = 0$$

$$k_N \sim k_C$$

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

with

$$F = k_c d = k_c \chi V_{A-B}$$

- 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

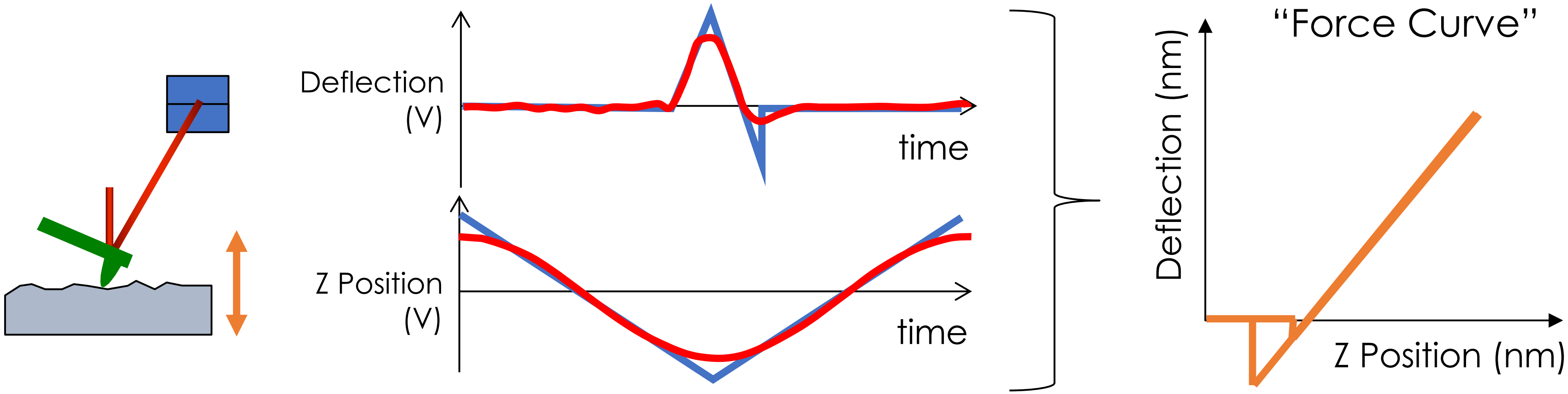
---

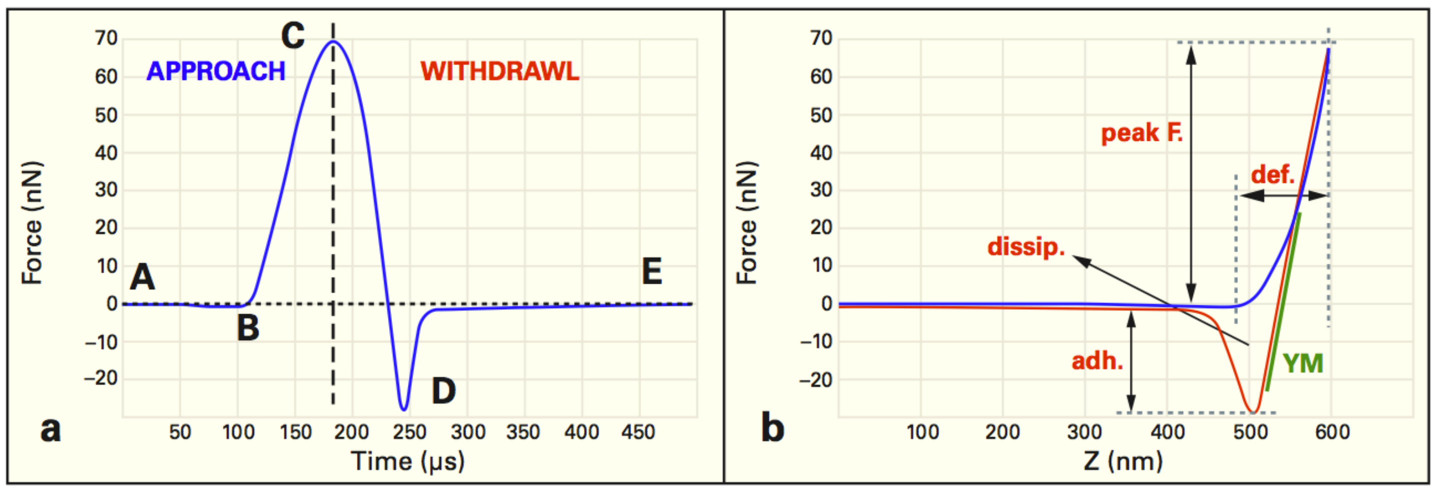
- Mapping (Force-Volume mode) of the elastic properties is very time consuming (256 x 256 points = 18h)

→ 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



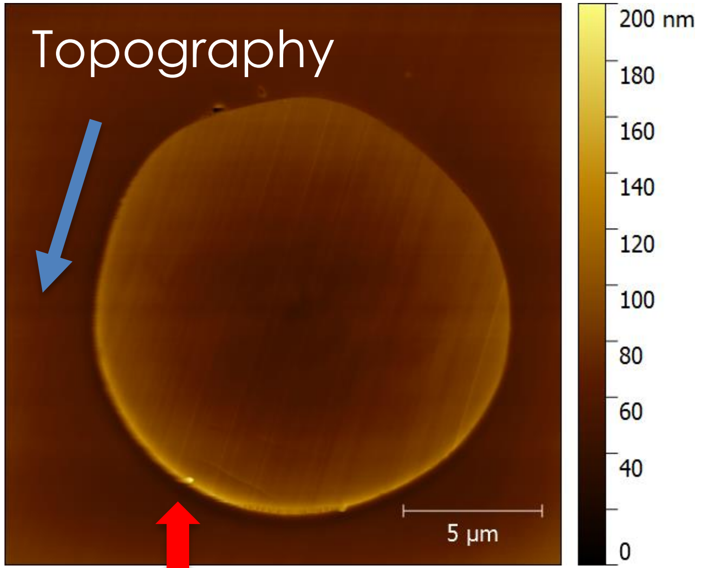


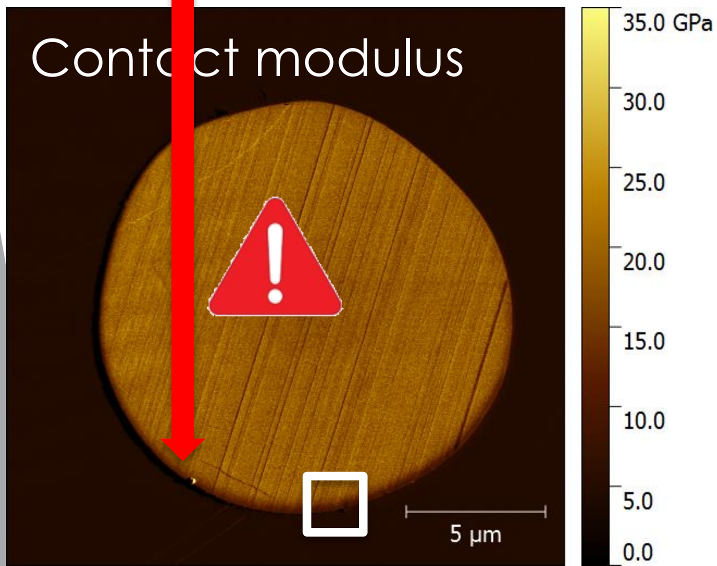
Calibration!

DMT modulus
Dissipation (hysteresis loop)
Adhesion force

Kevlar® Dupont™ K48 fibres

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





[Wollbrett-Blitz et al, J. Polym. Sci., 2016]

Core:

E_l = 84 GPa / E_t = 3 GPa

Skin:

E_l = 85 GPa / E_t = 0,2 GPa

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

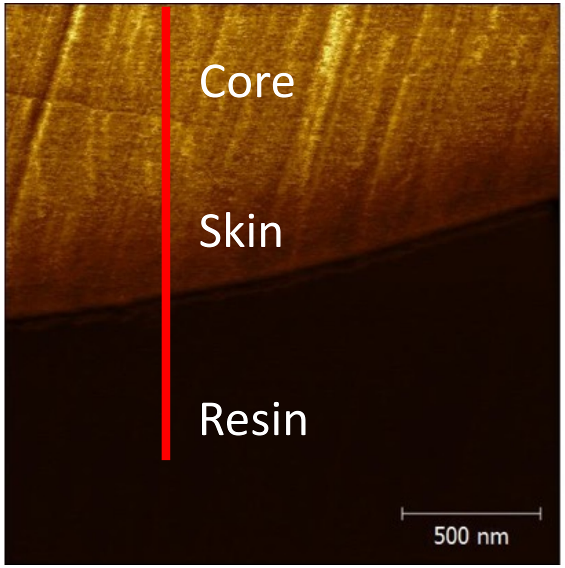
ν_{lt} = 0,6 / ν_{tt} = 0,25

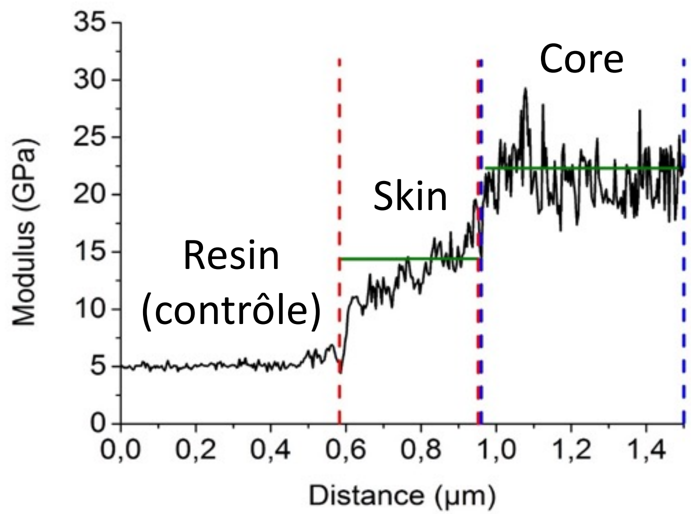
$M_{core\ NI}$ = 22,8 ± 1,7 GPa

(150 nm @ 1 µN/s + 20 s + 10 µN/s)

$M_{core\ calc}$ ≈ 21 GPa

$M_{skin\ calc}$ ≈ 17 GPa



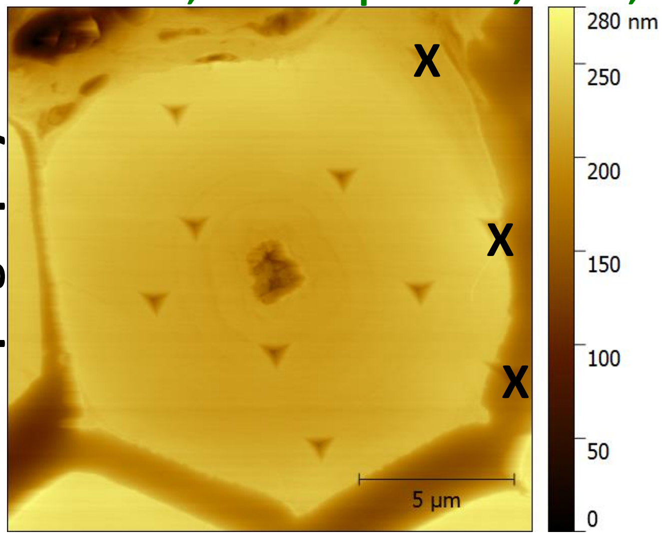


MultiMode 8, PF-QNM, Bruker RTESPA-525, 139 N/m, 32 nm (HOPG), 200 nN

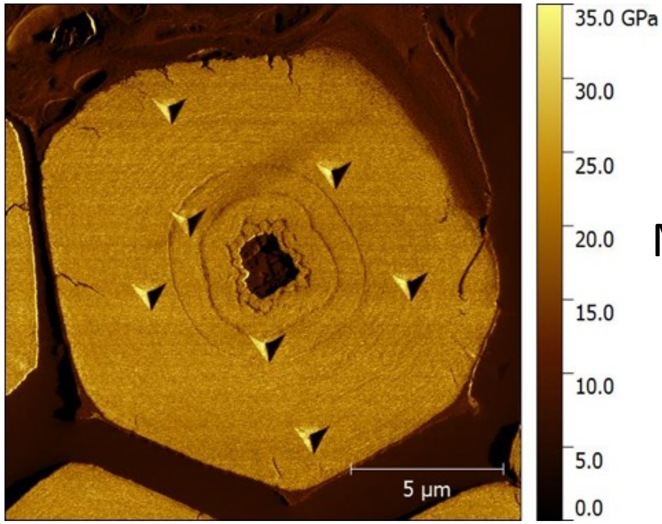
Flax fibres for bio-composites

[Arnould et al, Ind. Crops Prod., 2017, 97: 224-228]

Topography



Indentation modulus



E_l fibre ≈ 50-60 GPa !

$M_{NI\ fibre}$ = 21.3 ± 2.2 GPa

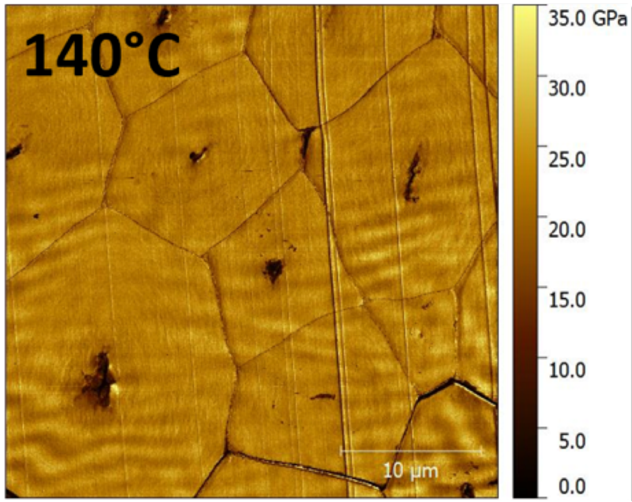
(150 nm @ 1 µN/s

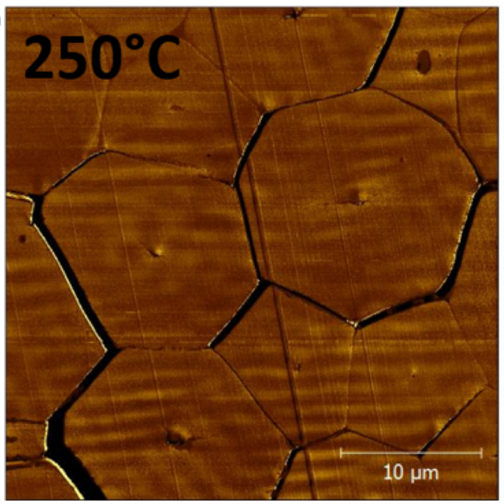
+ 20 s - 10 µN/s)

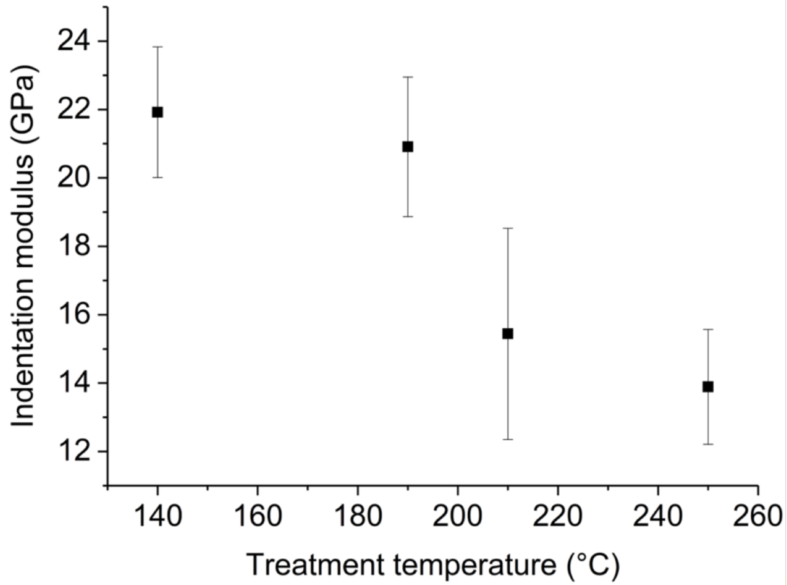
Effect of processing temperature and fibres confinement in the composite matrix

[Sinsicalco et al, Polym. Test., 2018, 69:91-99]

Indentation modulus



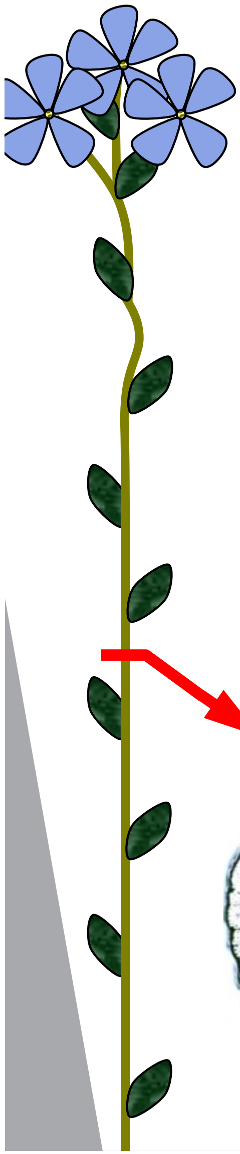


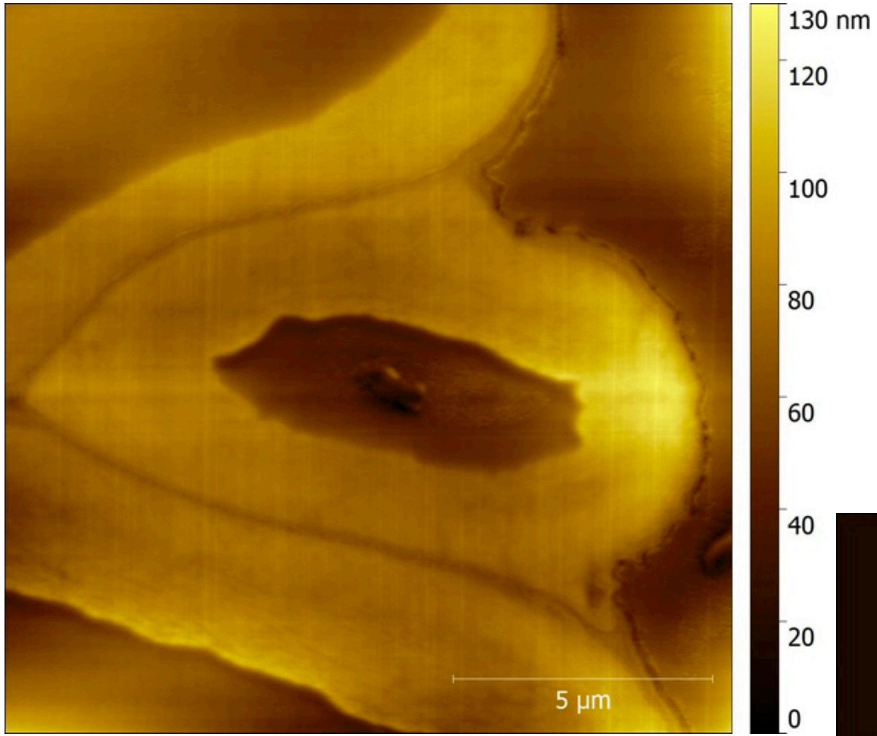


MultiMode 8, PF-QNM, Bruker RTESPA-525, 139-200 N/m, 9-50 nm (HOPG), 200 nN

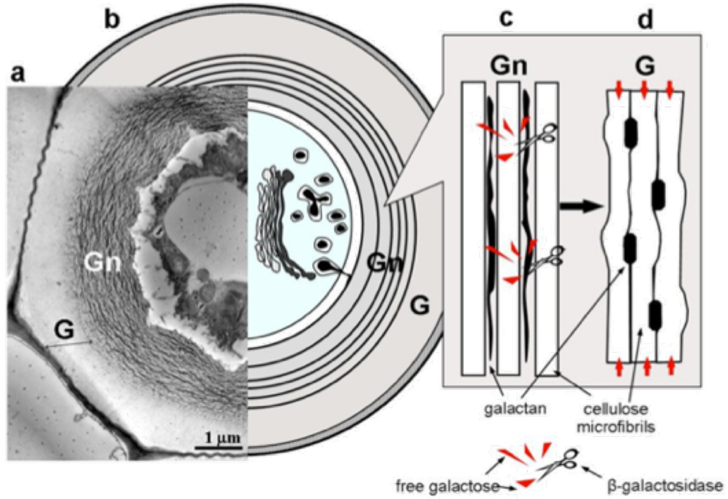
Developping flax fibres

[Arnould et al, Ind. Crops Prod., 2017, 97: 224-228]



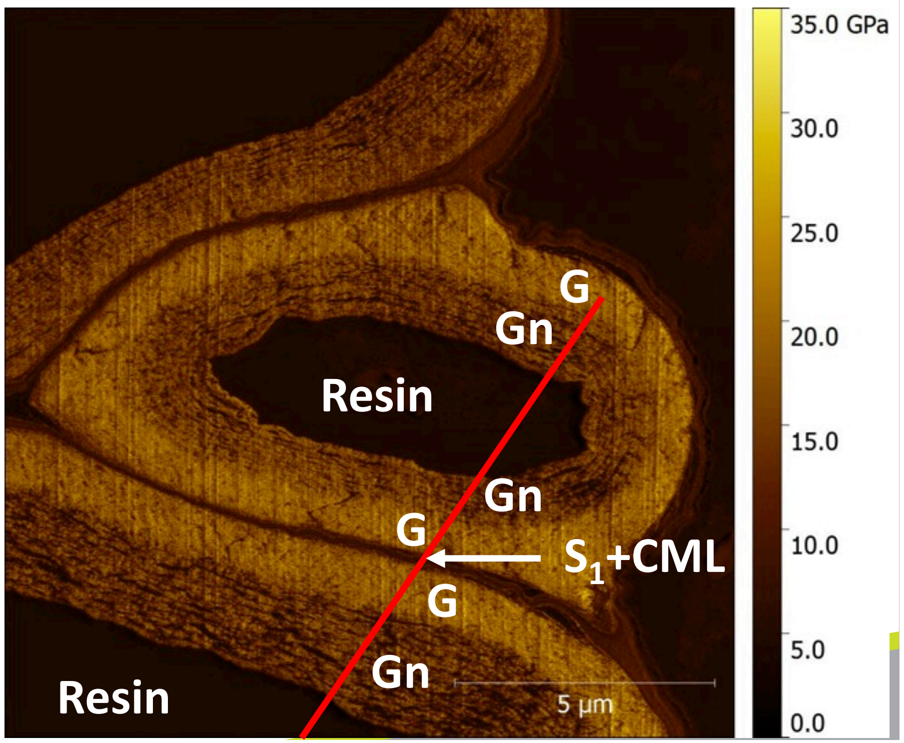


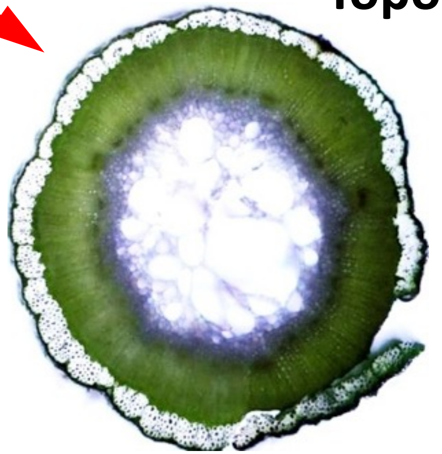
Topography (nm)



[Mikshina et al, InTech, 2013]

Indentation modulus (GPa)





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

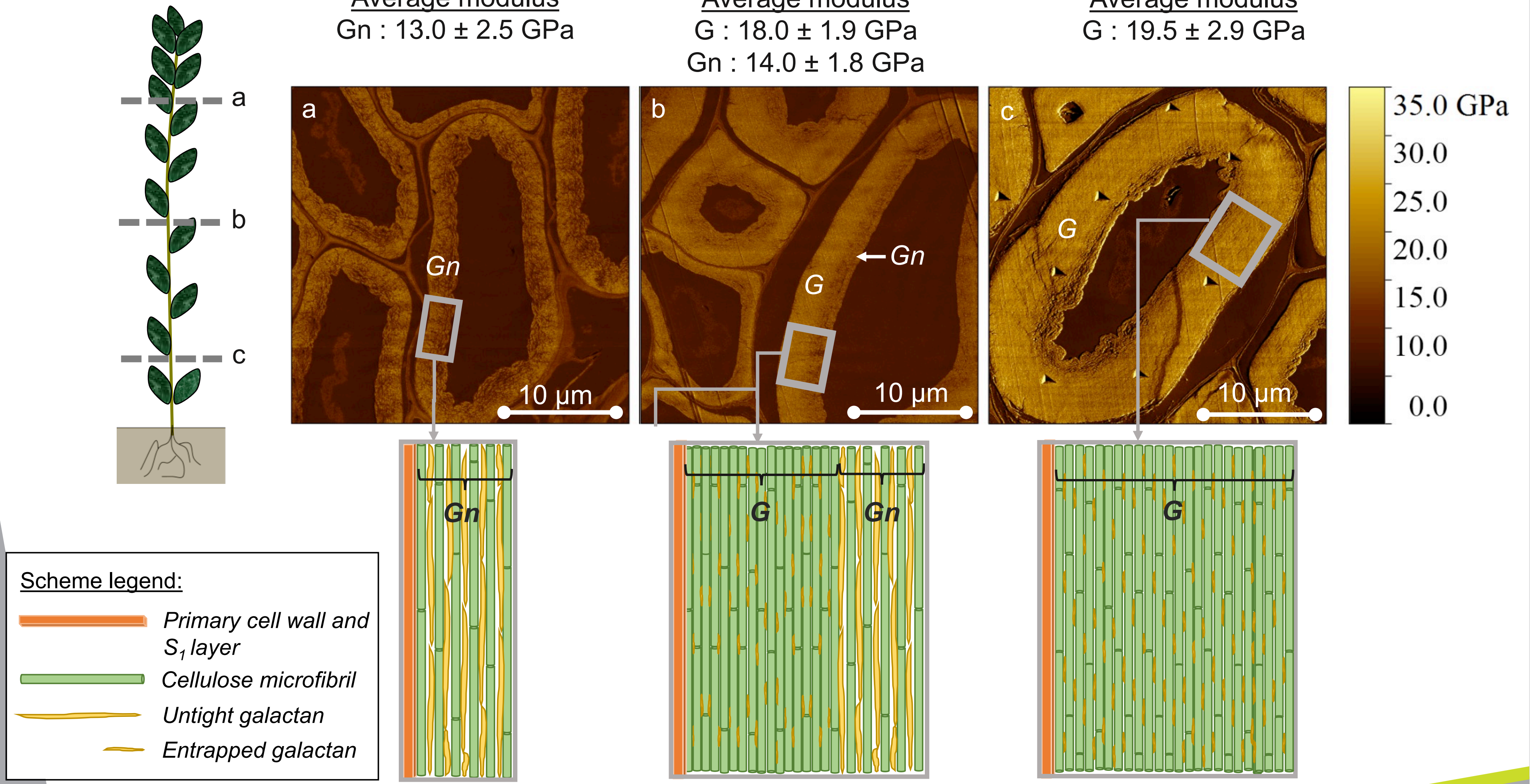
Developping flax fibres

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

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

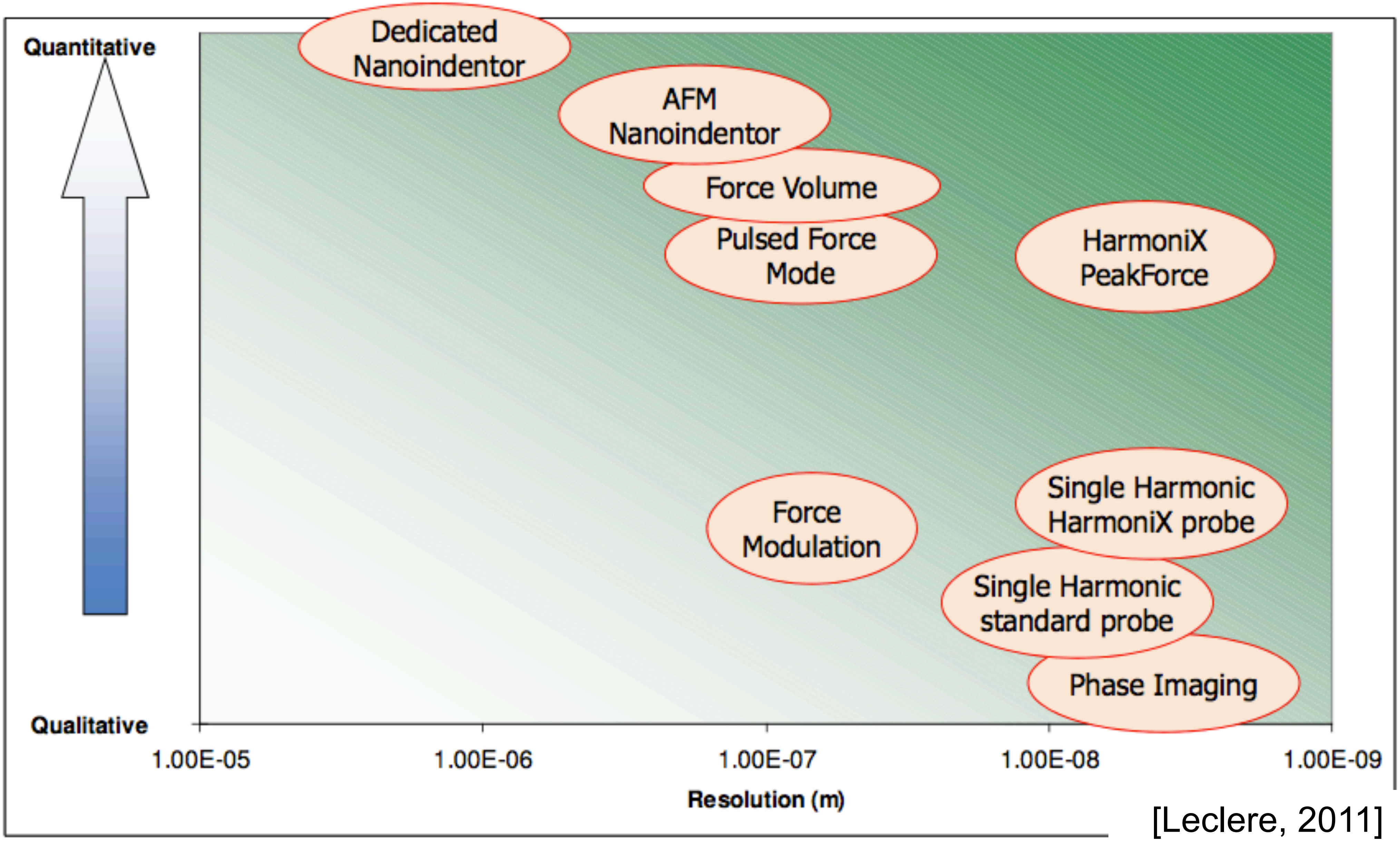
Average thickness
Gn+G : 3.2 ± 1.3 µm
Average modulus
G : 18.0 ± 1.9 GPa
Gn : 14.0 ± 1.8 GPa

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



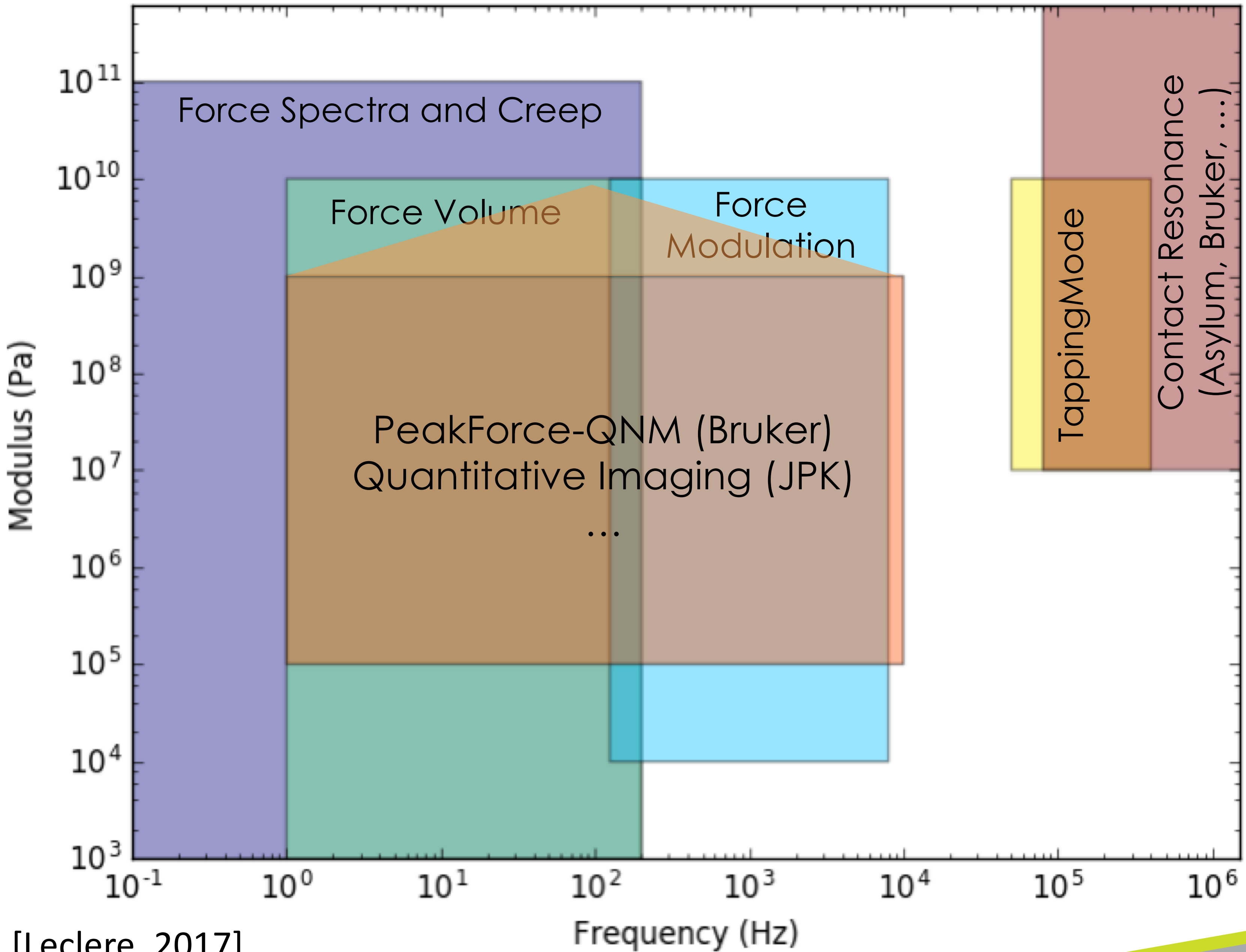
MultiMode 8, PF-QNM Bruker RTESPA-525, 139 N/m, 15-35 nm (HOPG) 200 nN

Quality of data vs. Resolution



[Leclere, 2011]

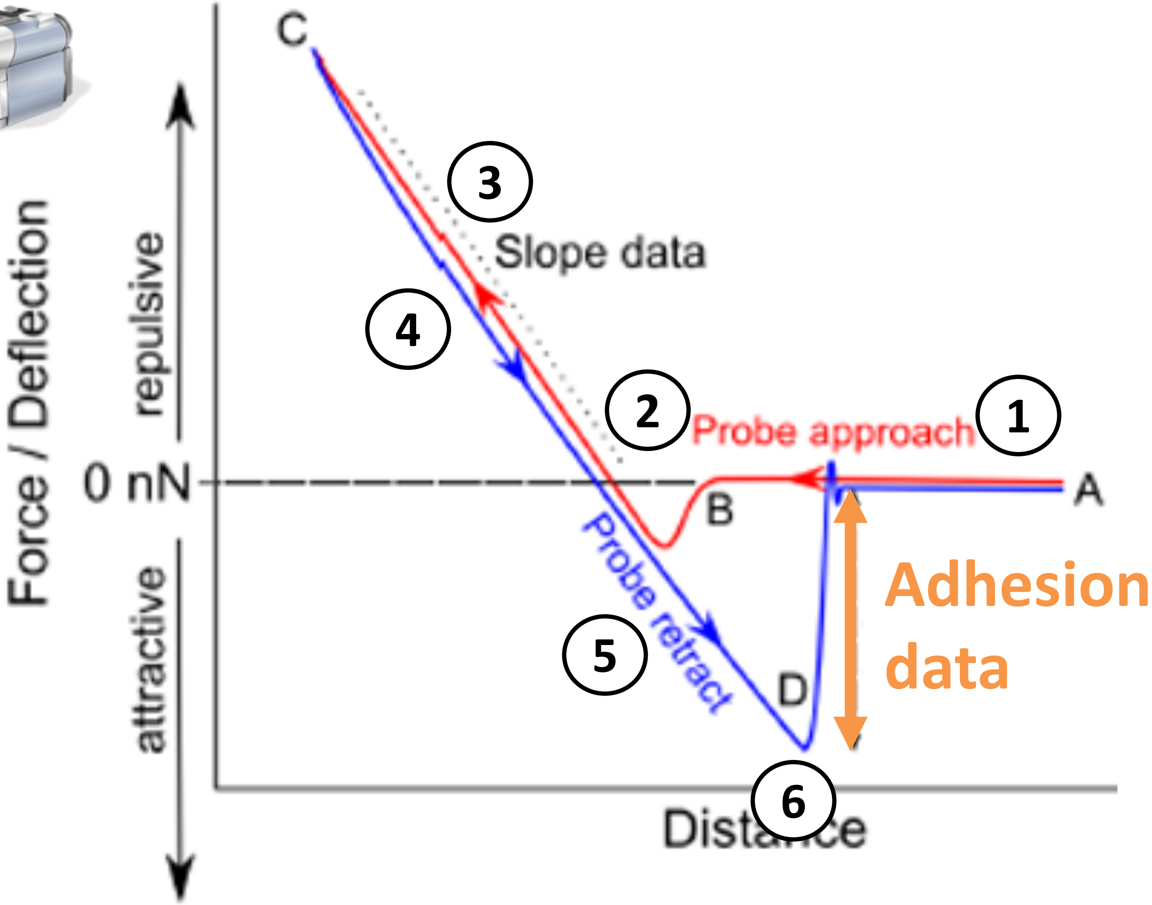
AFM frequency and elastic modulus range…

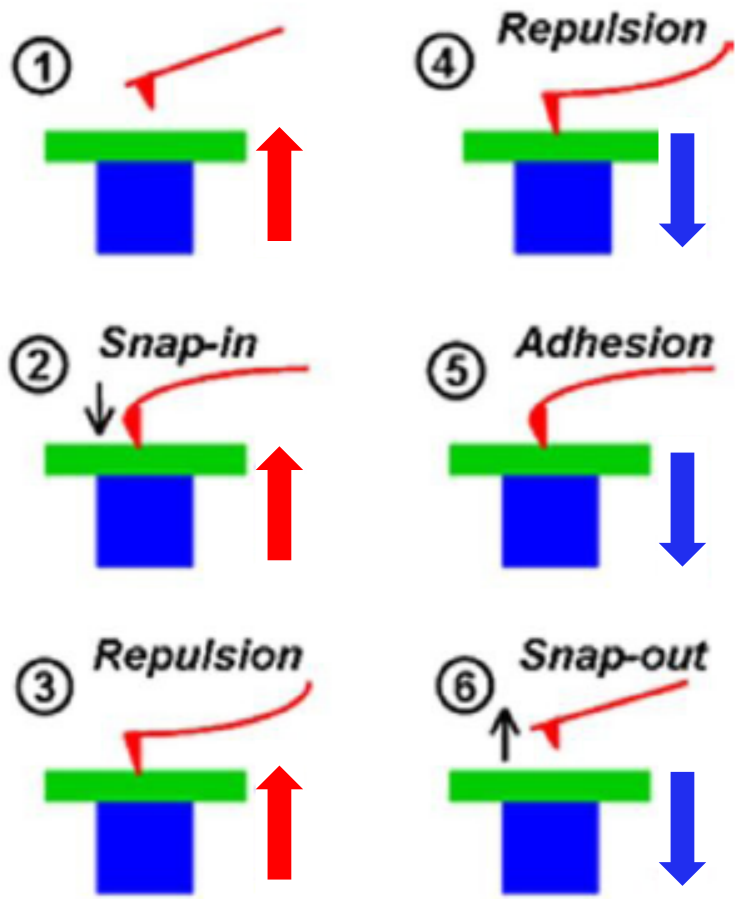


[Leclere, 2017]

Force-distance curve

- Van der Waals force, electrostatic force, contact stiffness, adhesion force, …

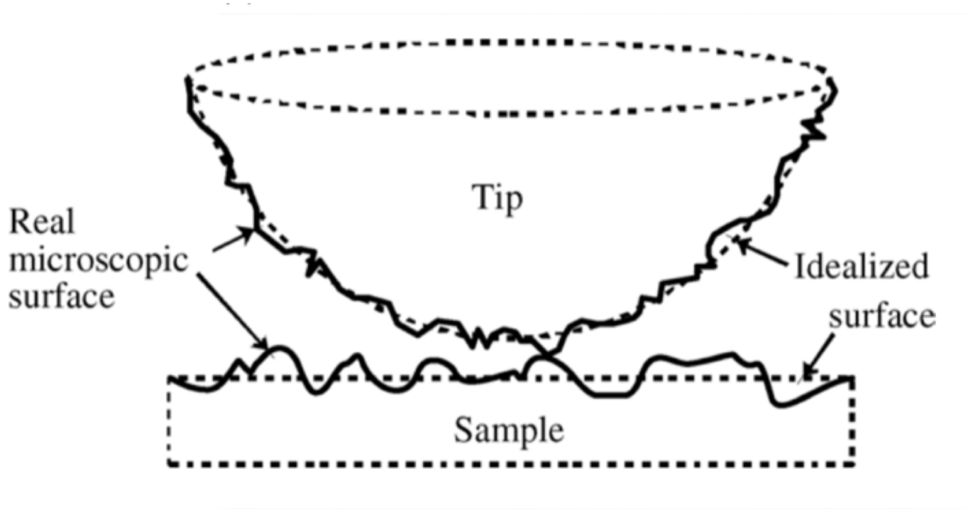




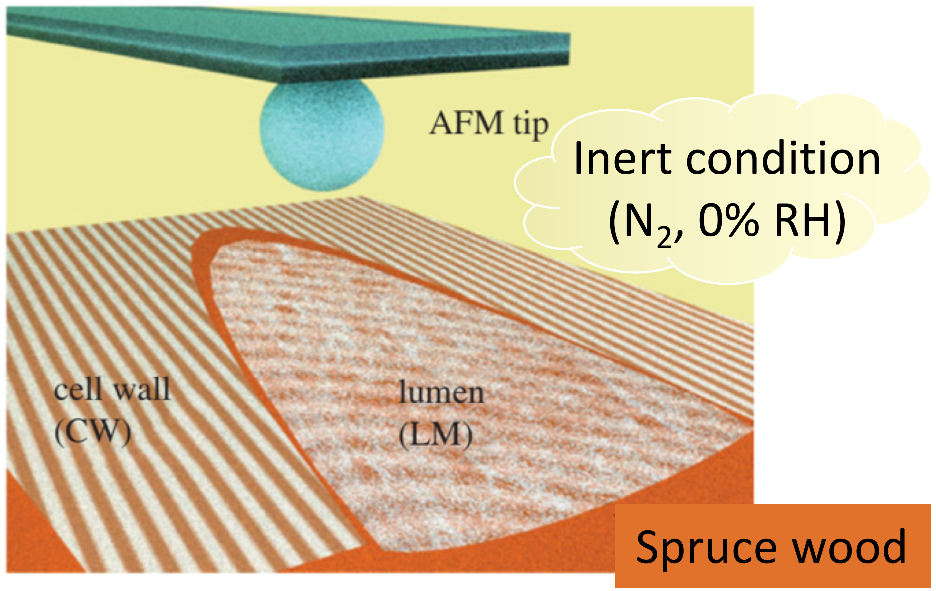
[B. Nysten, 2007]

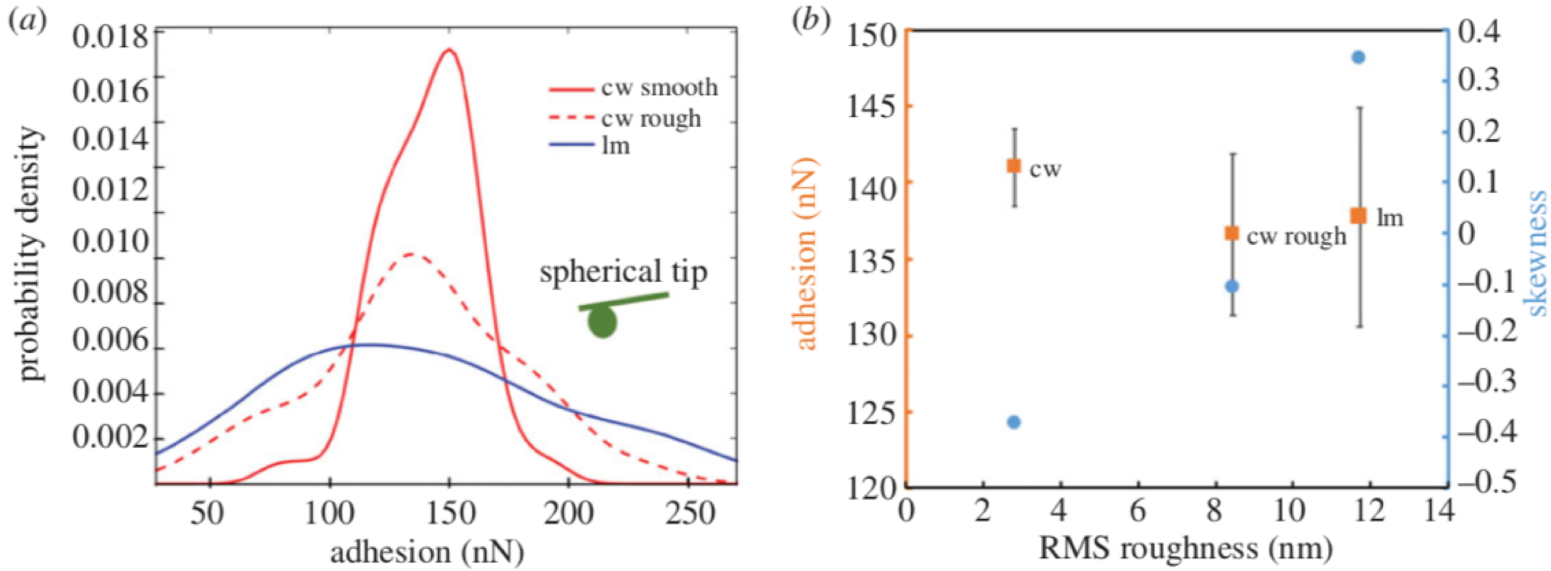
Adhesion force spectroscopy

- Influence of surface roughness and tip geometry



Butt et al, Surf. Sci. Rep. 2005, 59, 1–152





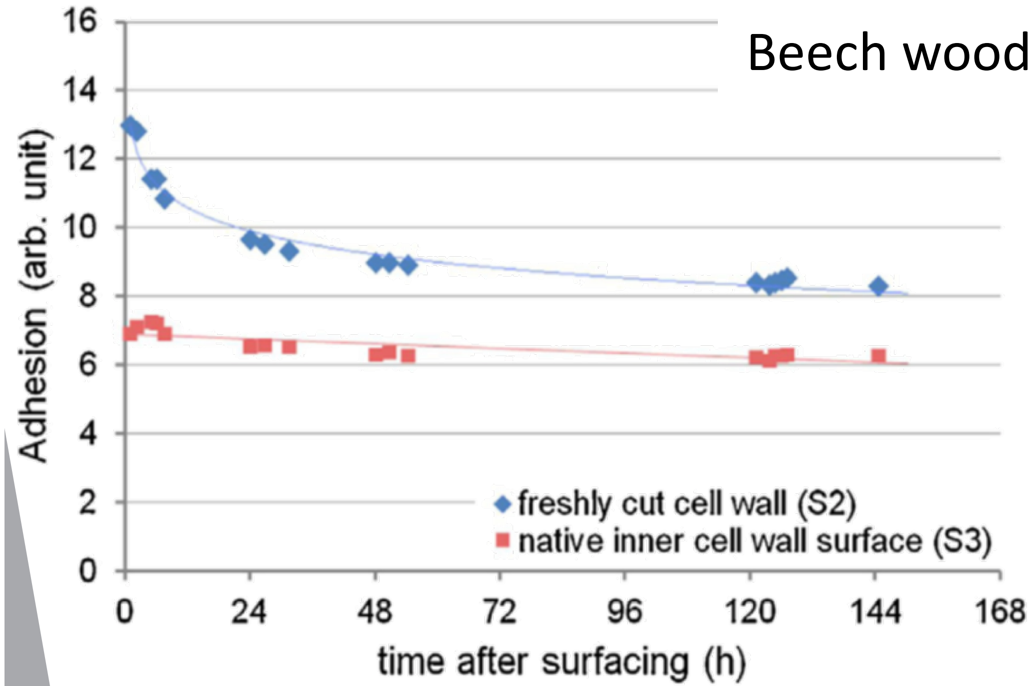
Jin and Kasal, R. Soc. open sci. 2016, 3: 160248

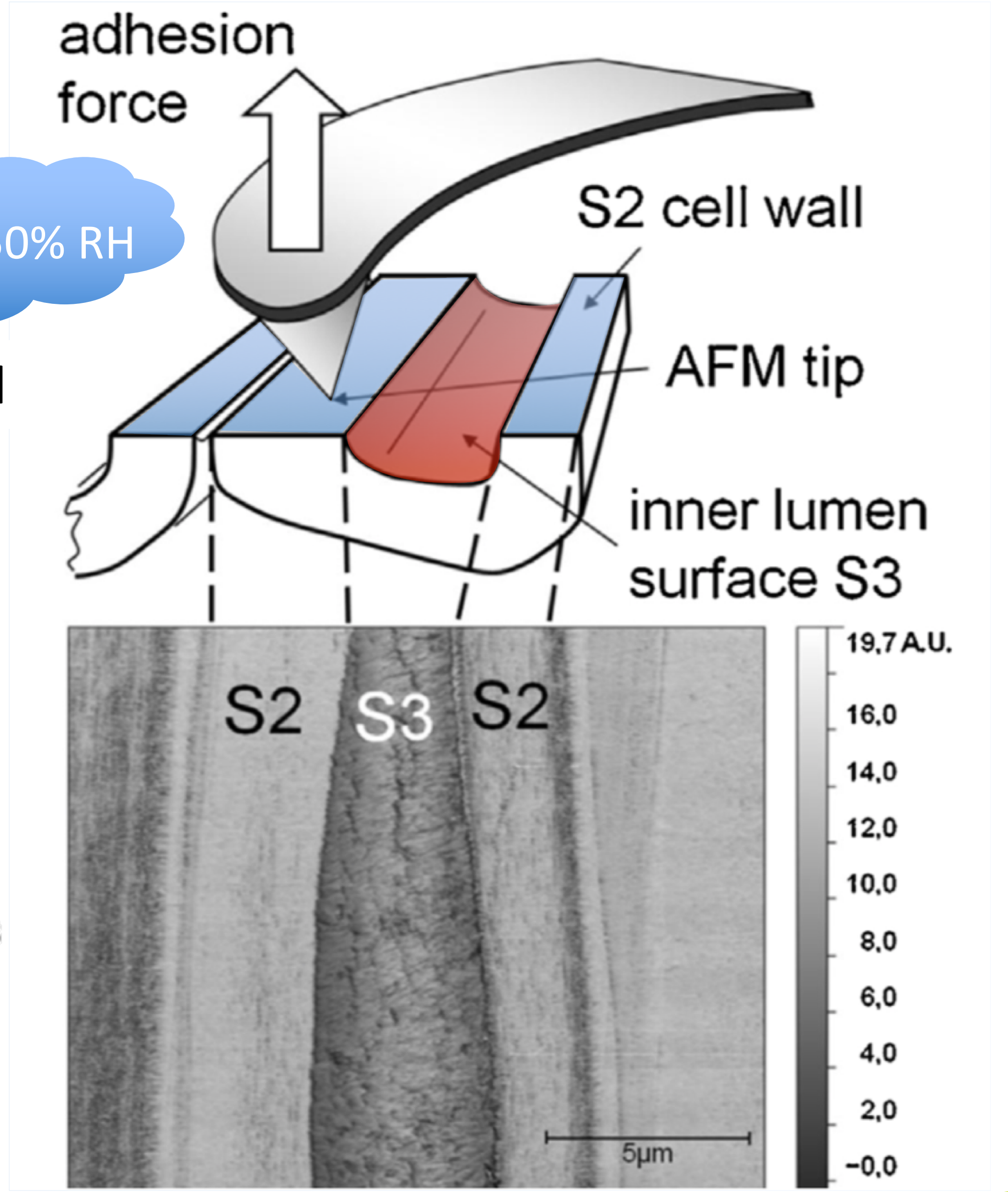
Adhesion force spectroscopy

- Variability in surface polarity and aging

23°C and 50% RH

Beech wood





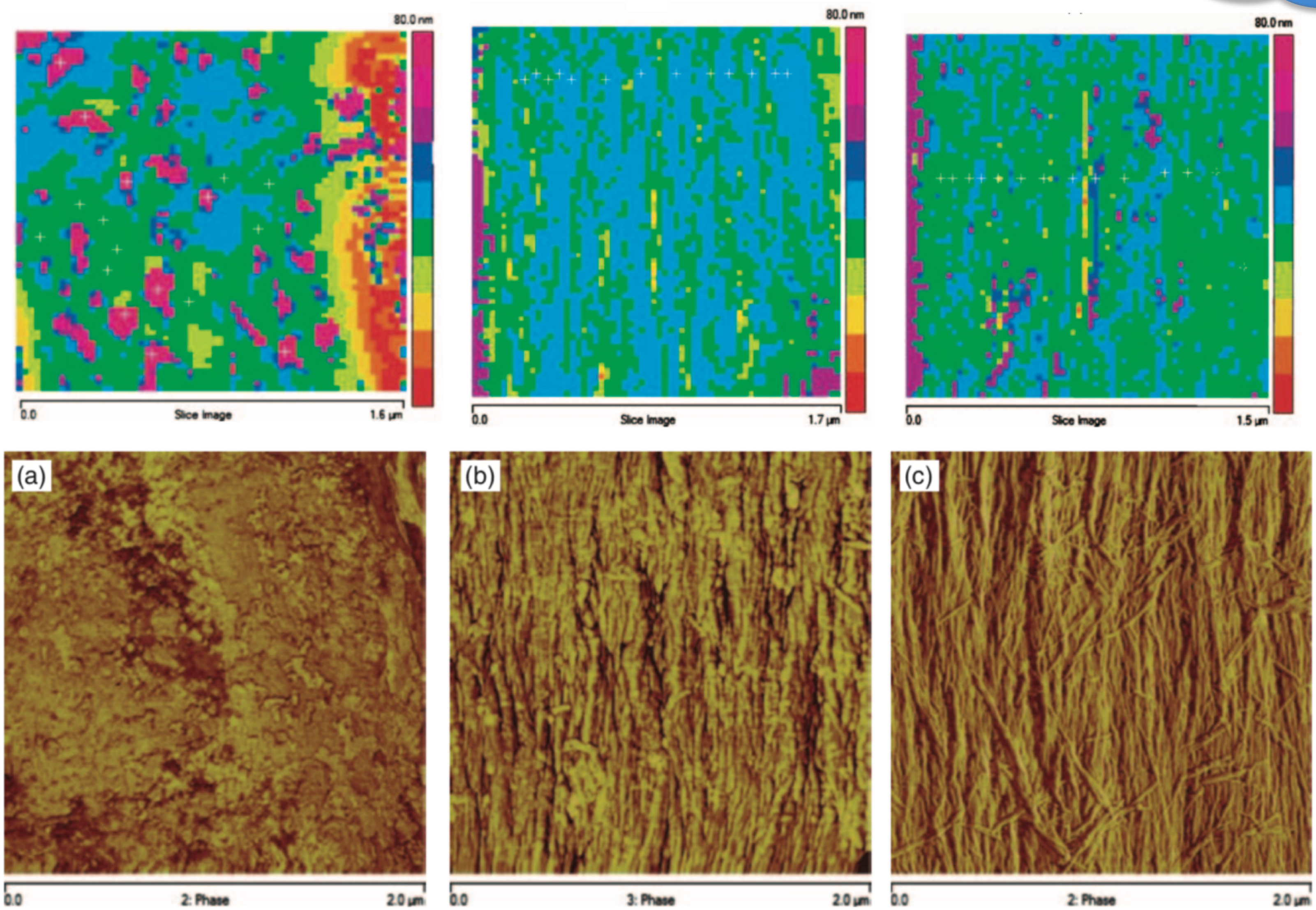
Frybort et al, Colloids and Surfaces A, 457 (2014) 82–87

Adhesion force spectroscopy

Room conditions

- Effect of surface treatment

Flax fibres

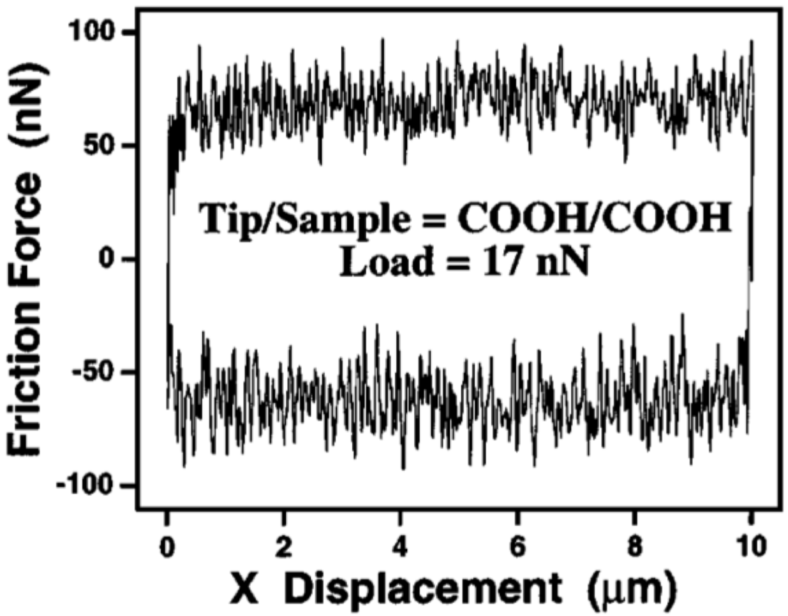


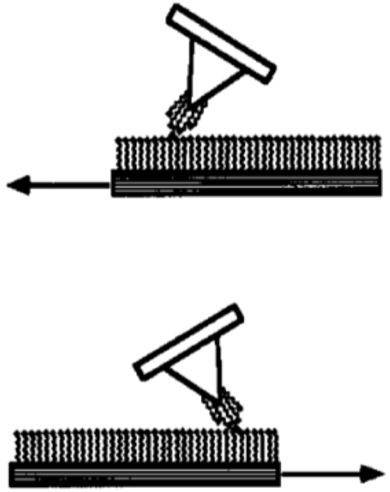
TM-AFM phase image of (a) raw flax fibre, (b) NaOH (10%) treated flax fibre, (c) Enzyme treated (18 h) flax fibre.

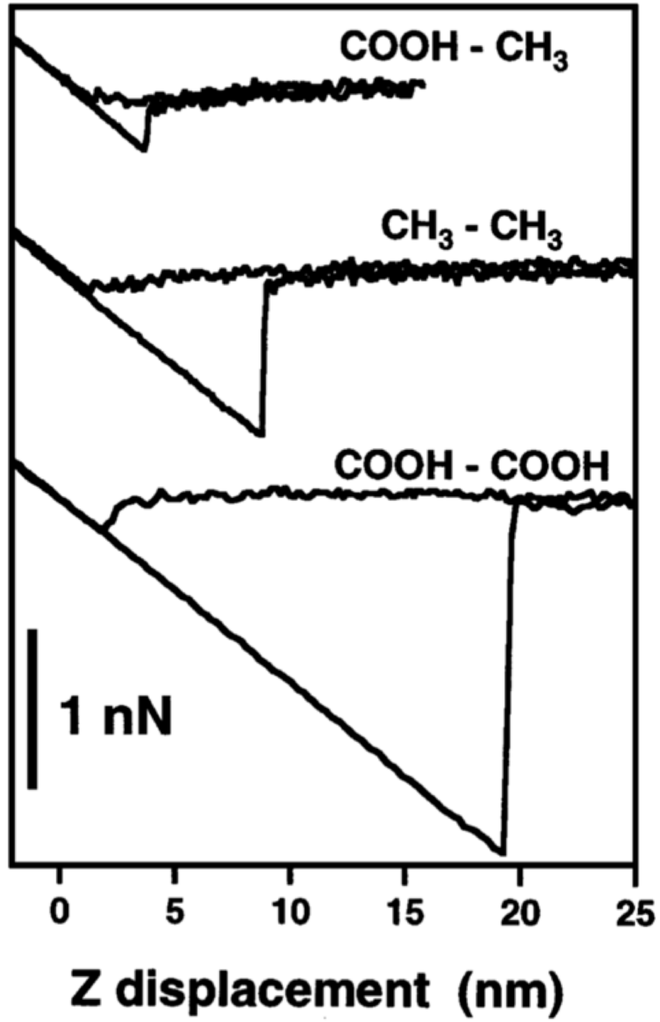
Raj et al, J. Scann. Probe Microsc. 2009, 4(2), 1-7

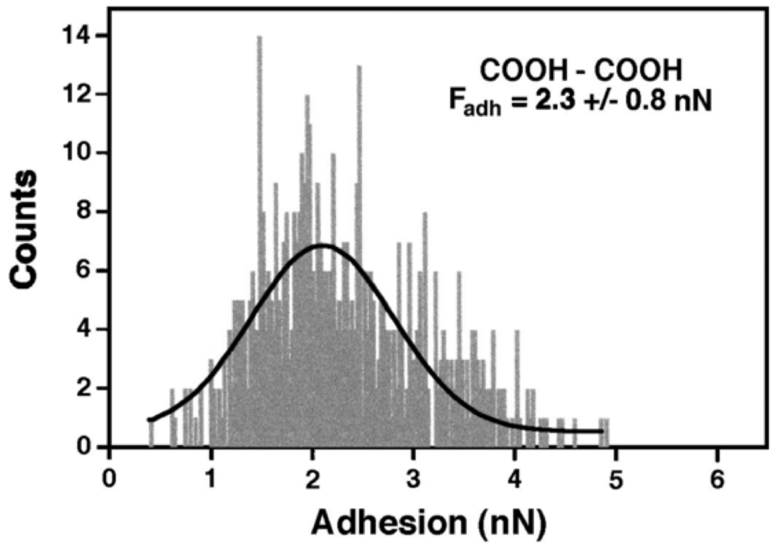
Adhesion force spectroscopy

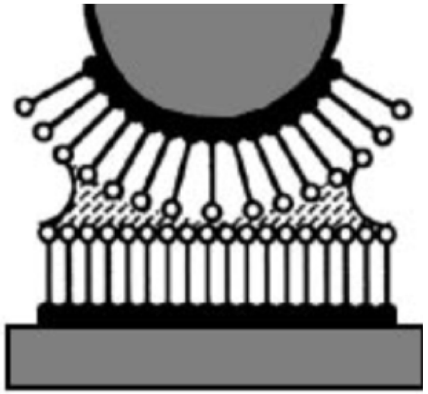
- Tip functionalisation - Chemical force microscopy







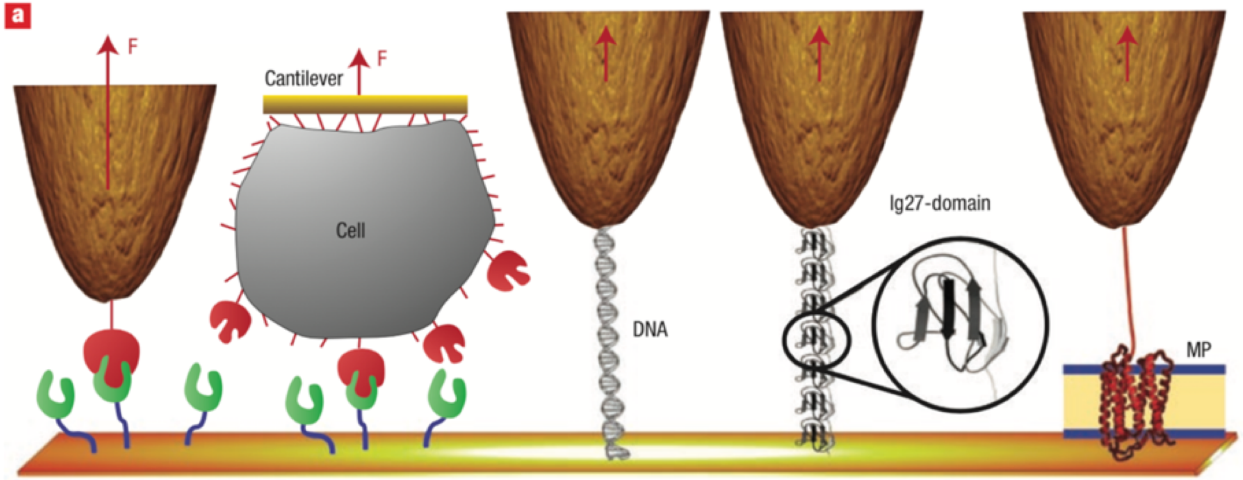


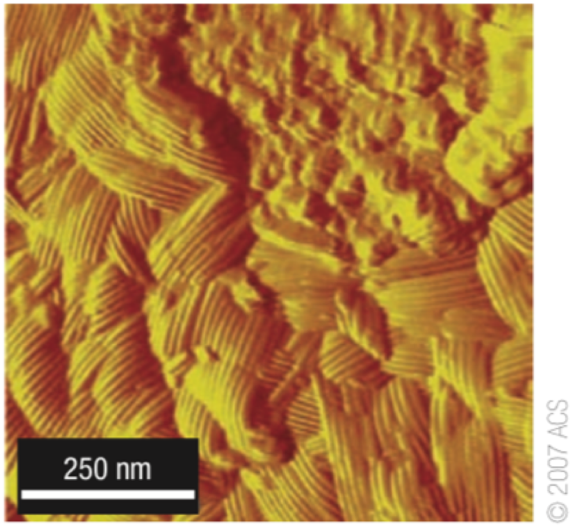


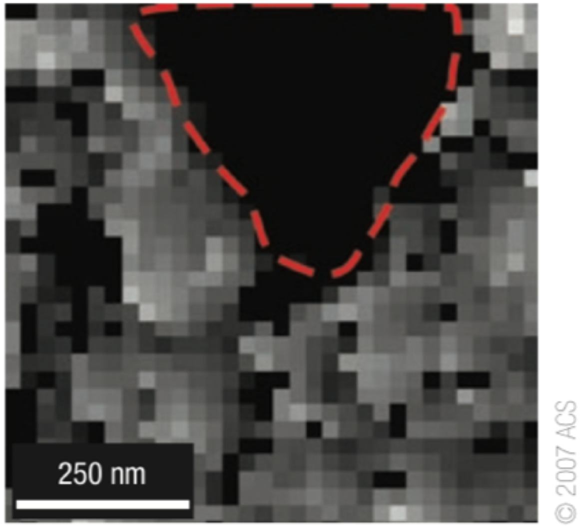
Noy et al, Annu. Rev. Mater. Sci. 1997. 27:381–421

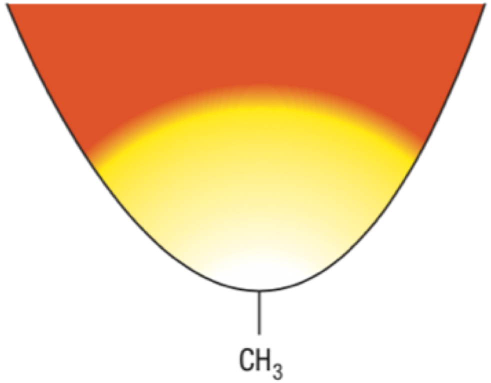
Adhesion force spectroscopy

- Tip functionalisation - Molecular force microscopy









Fungus *Aspergillus fumigatus*

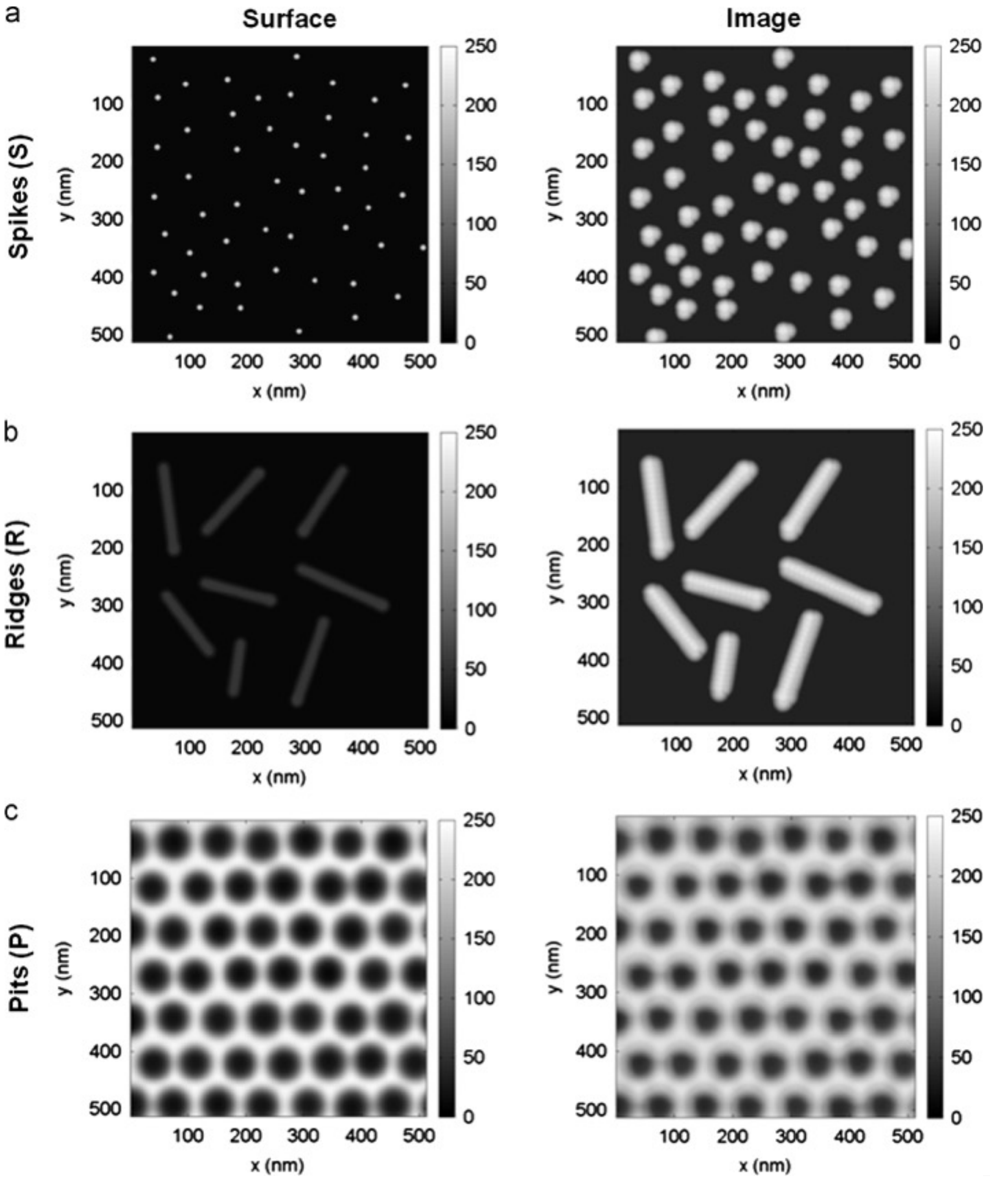
Müller et al, Nature Nanotech. 2008, 3: 261-269

Outline

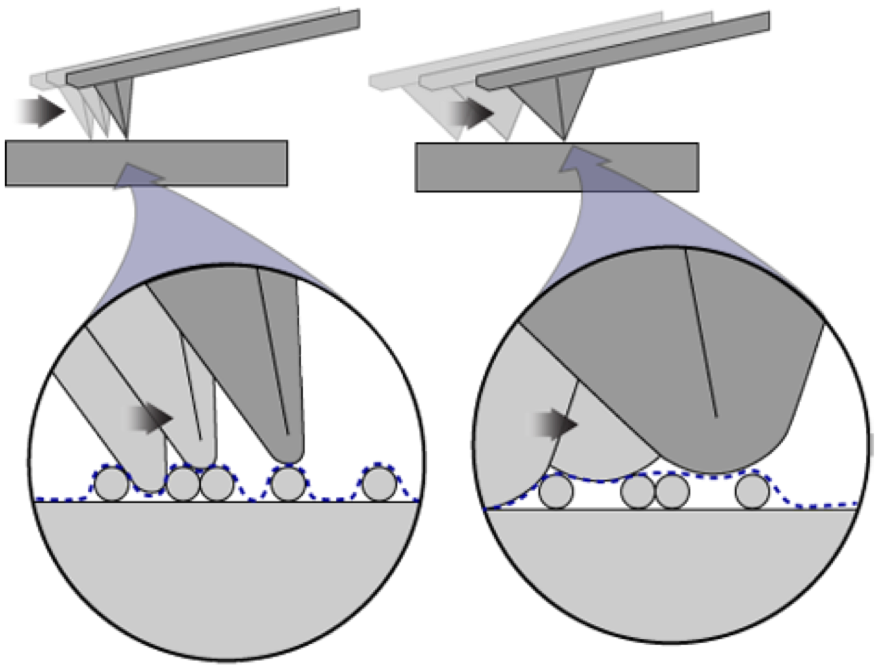
- Brief history and basic principles of scanning probe microscopy
- AFM principle and typical set-up
  - Tip probe-sample surface interactions
  - Tip and cantilever
  - Deflection sensor and scanner
  - Whole set-up
- Contact mechanics
- Operating modes
  - Contact
  - Intermittent
  - Force-distance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

AFM artifacts

- Images dilation due to the “finite” tip shape



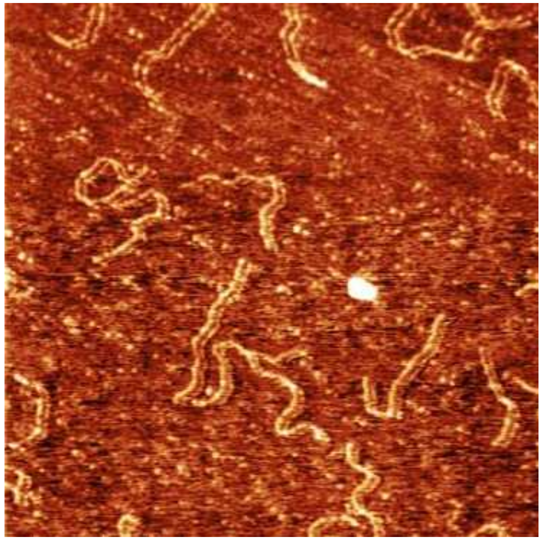
Flater et al, Ultramicroscopy, 2014, 146: 130-143

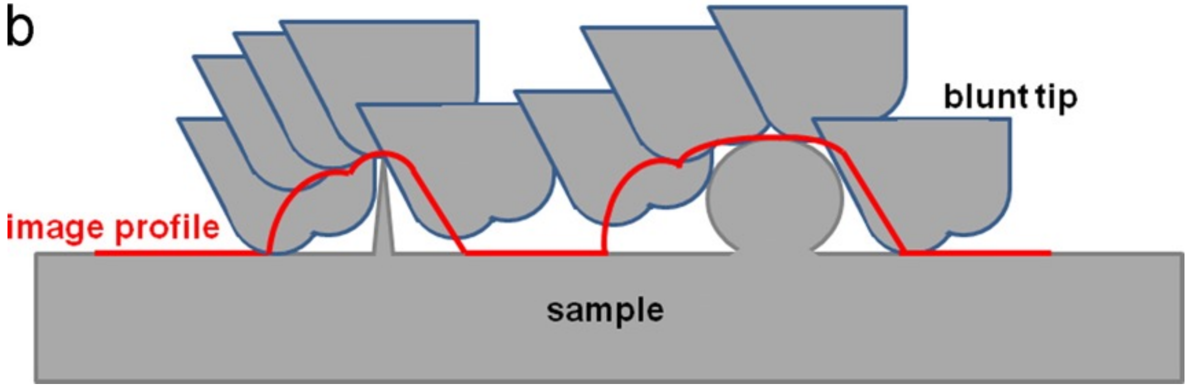


http://www.nanophys.kth.se/nanophys

AFM artifacts

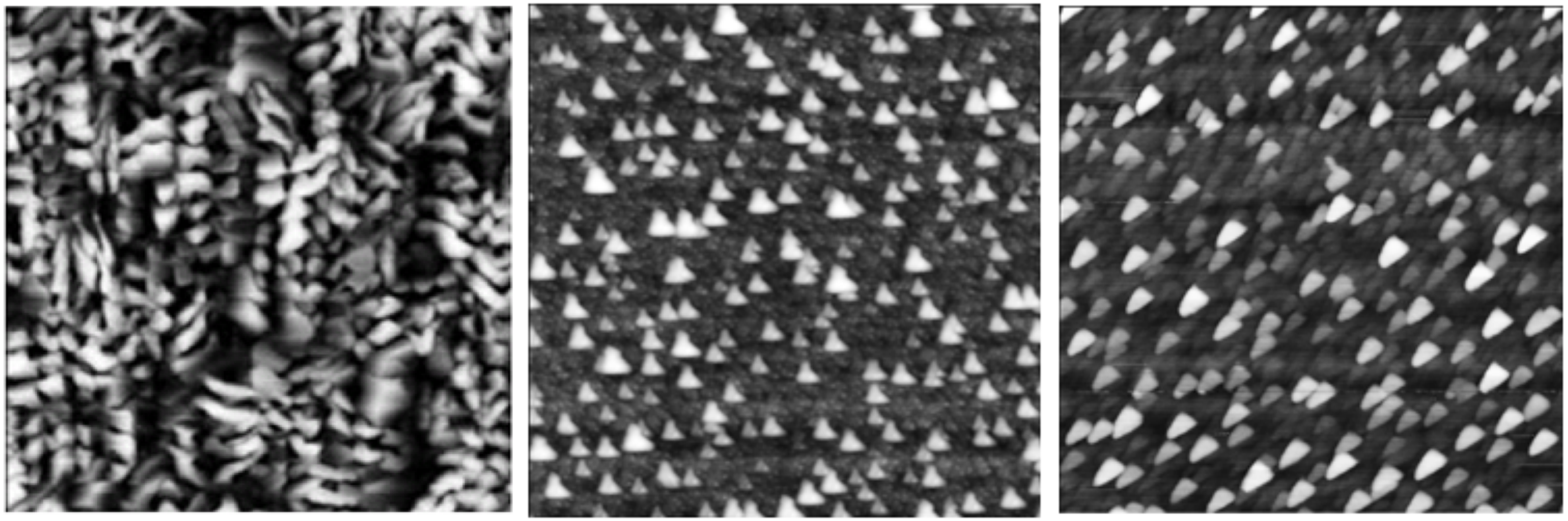
- Images dilation due to tip shape default, dirt or wear





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

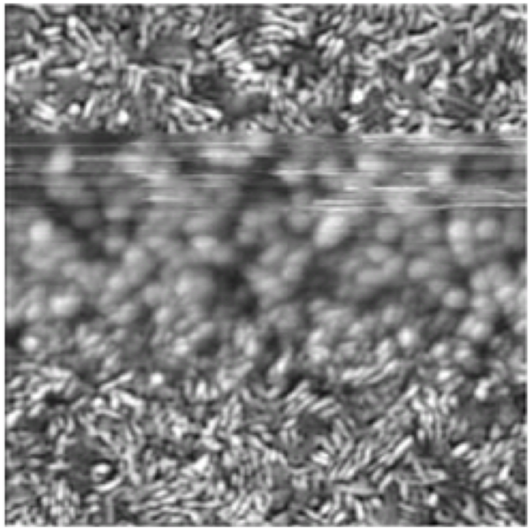
http://www.fc.up.pt/pessoas/peter.eaton/artifacts/artifacts.html

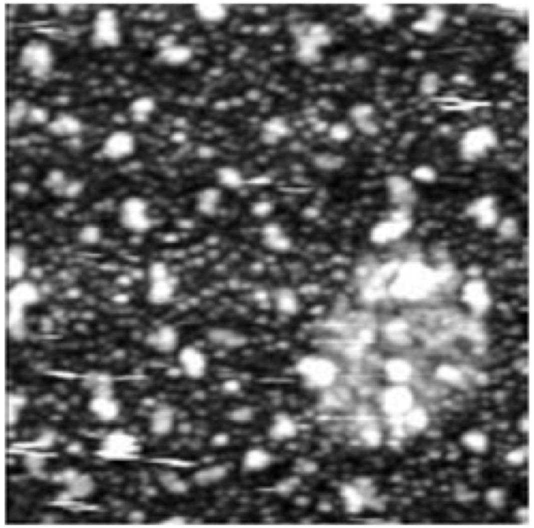


http://www.nanophys.kth.se/nanophys/facilities/nfl/afm/

AFM artifacts

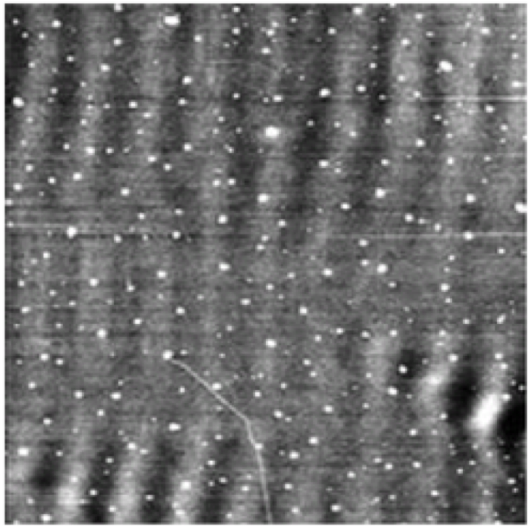
- Images dilation due to tip contamination

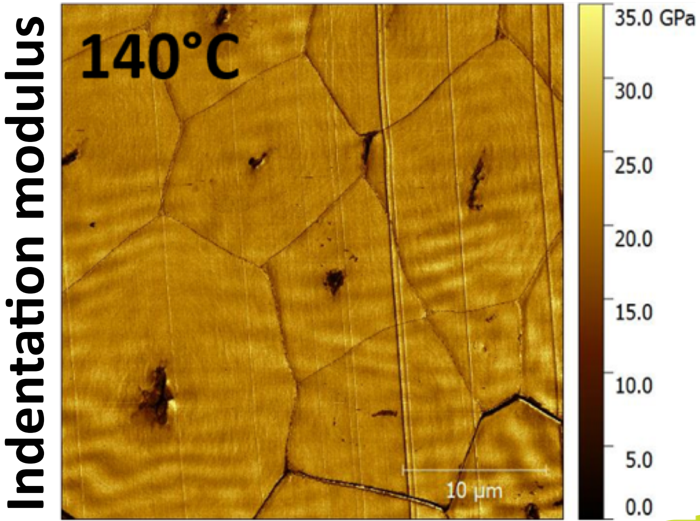




- Optical interference

[Sinsicalco et al, Polym. Test., 2018, 69:91-99]

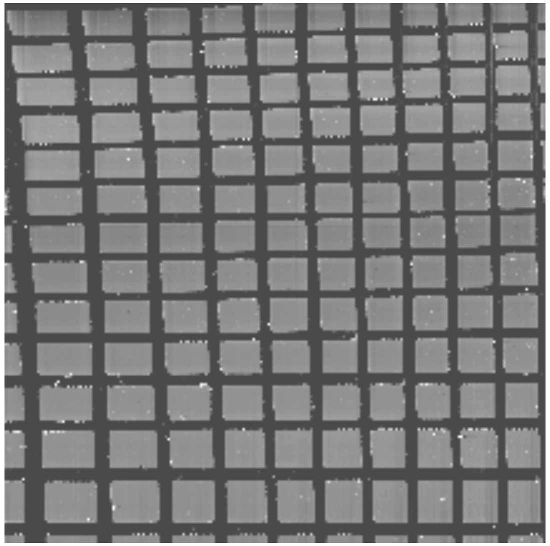




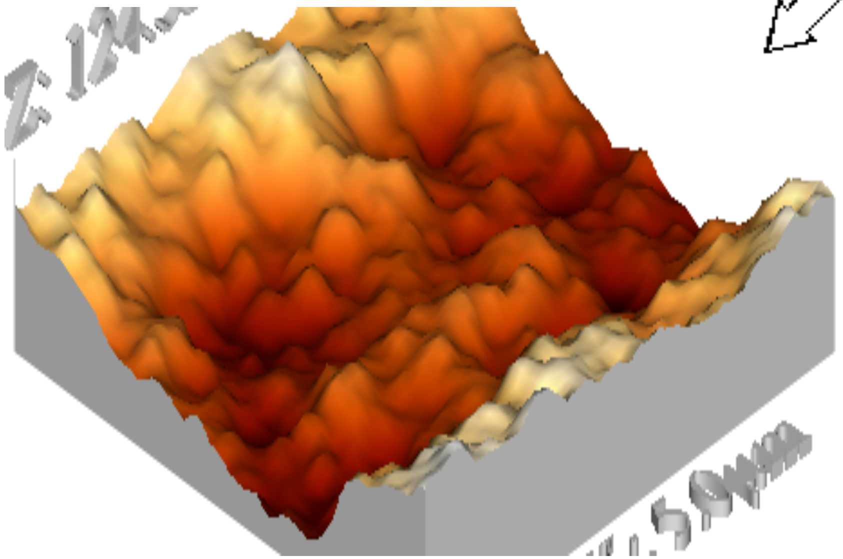
http://www.nanophys.kth.se/nanophys/facilities/nfl/afm/

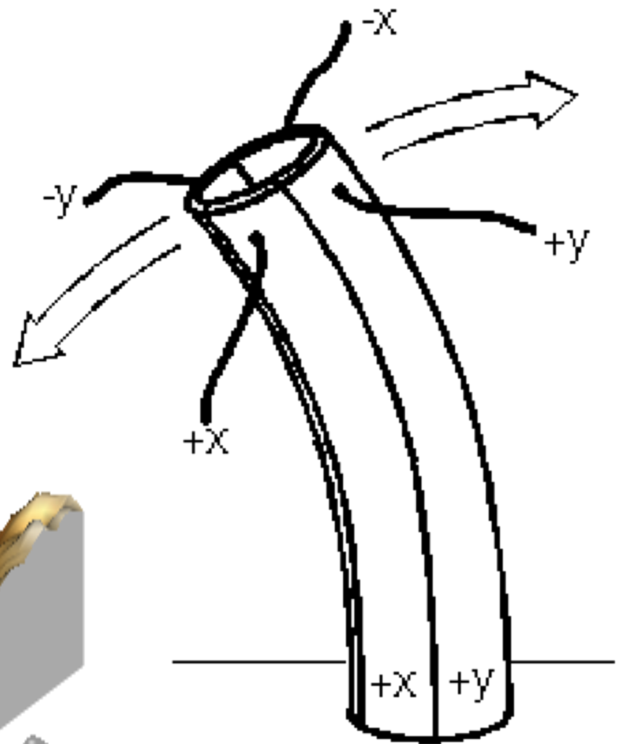
AFM artifacts

- Distortion due to non-linear piezo-scanner response + aging/creep



- Background bow/tilt





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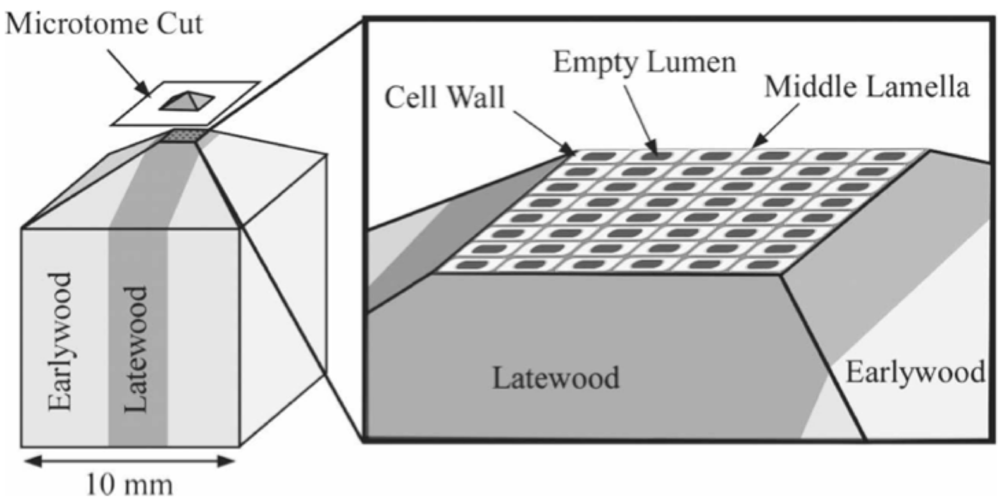
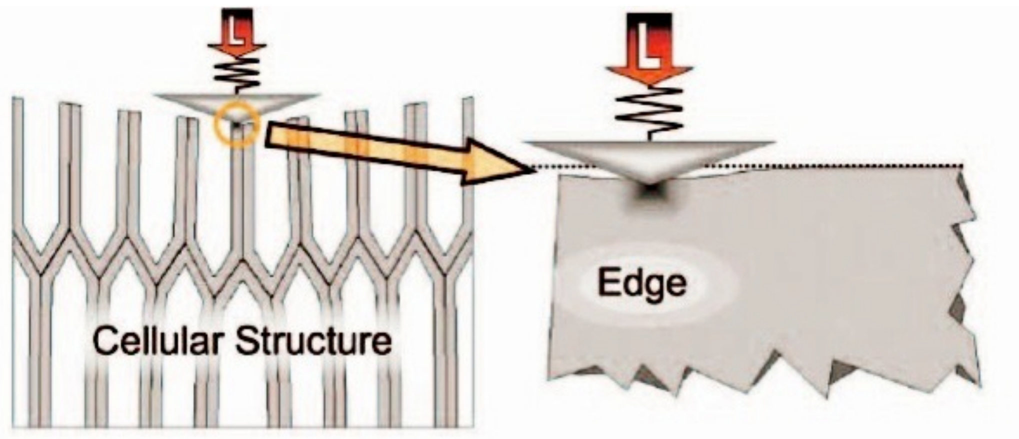
Sample preparation…

Without embbeding in a resin: no sample modifications but ±measurement articfacts

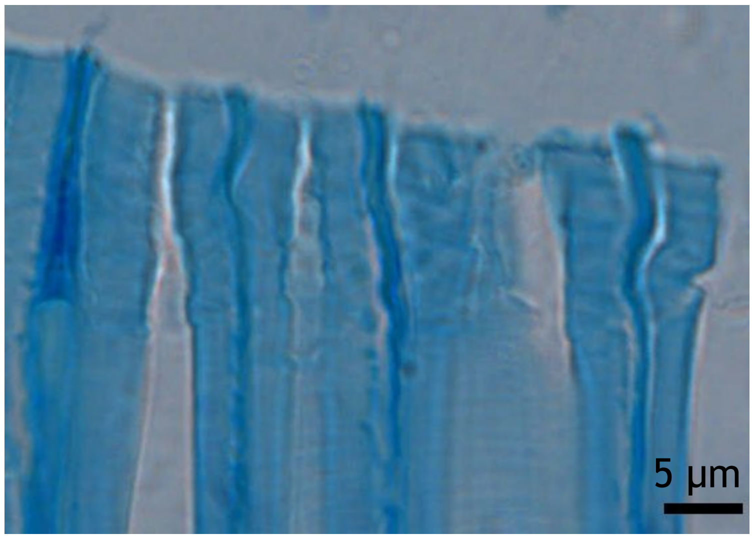




[Jakes et al, J. Mater. Res., 2008]

and topography…

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



[Clair, Gril et al, IAWA J., 2005]

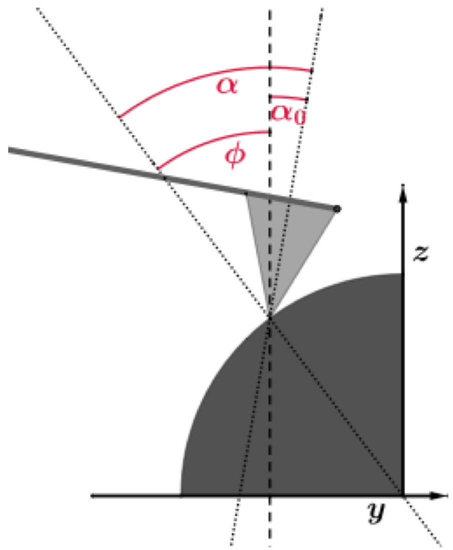
±no thermo-chemical modification of the cell wall (extractives…)?
Resin penetration / inclusion time?
Cell wall water content?

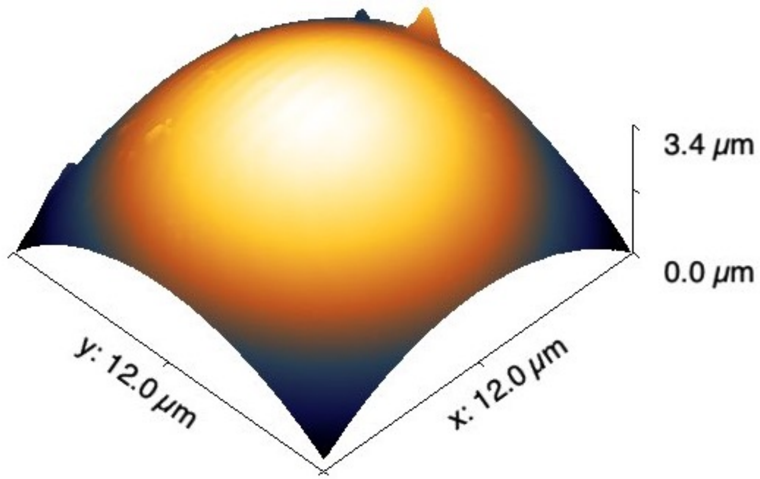
Microtome/Grinding/FIB…??!!

Sample surface effect

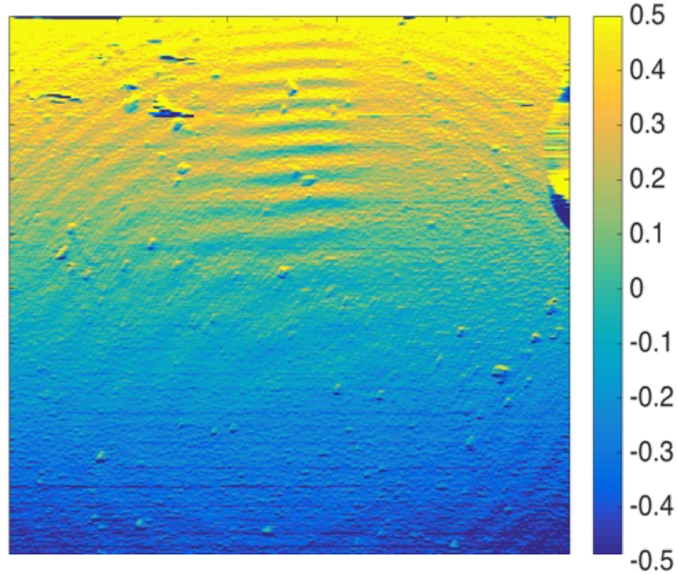
- Effect of sample local slope

[Heinze et al, Ultramicroscopy, 2018, to appear…]

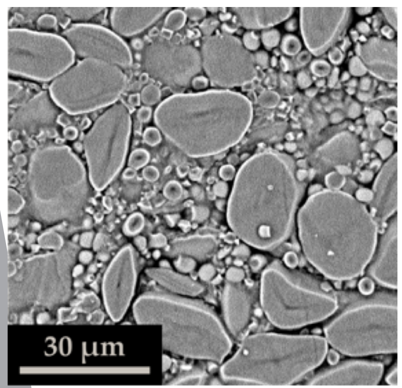




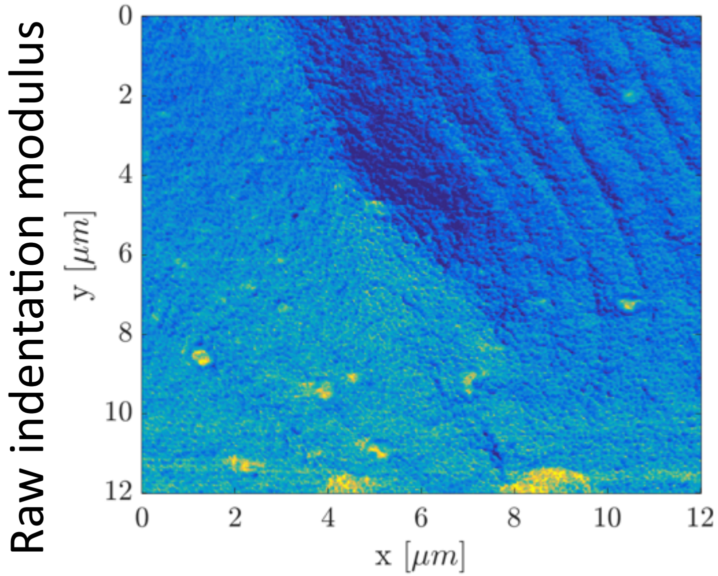
Topography

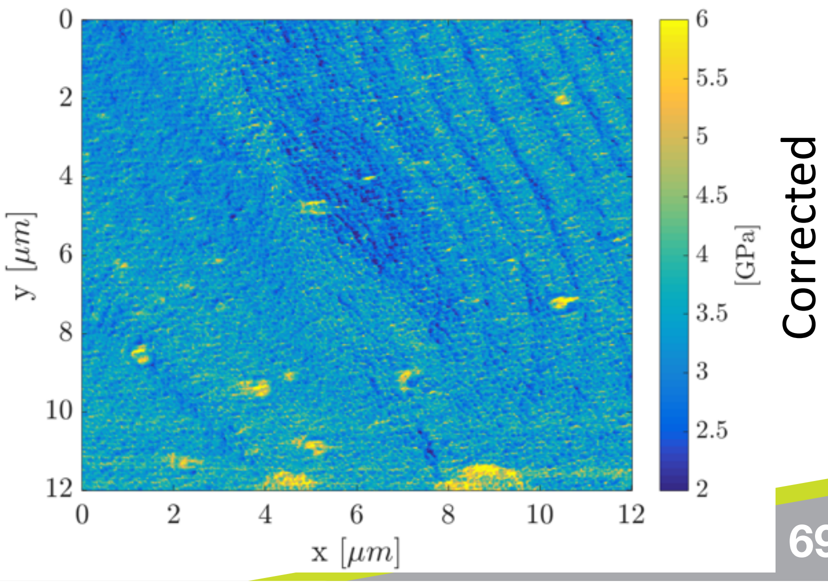


Local angle/cantilever ϕ (°)



Starch granules

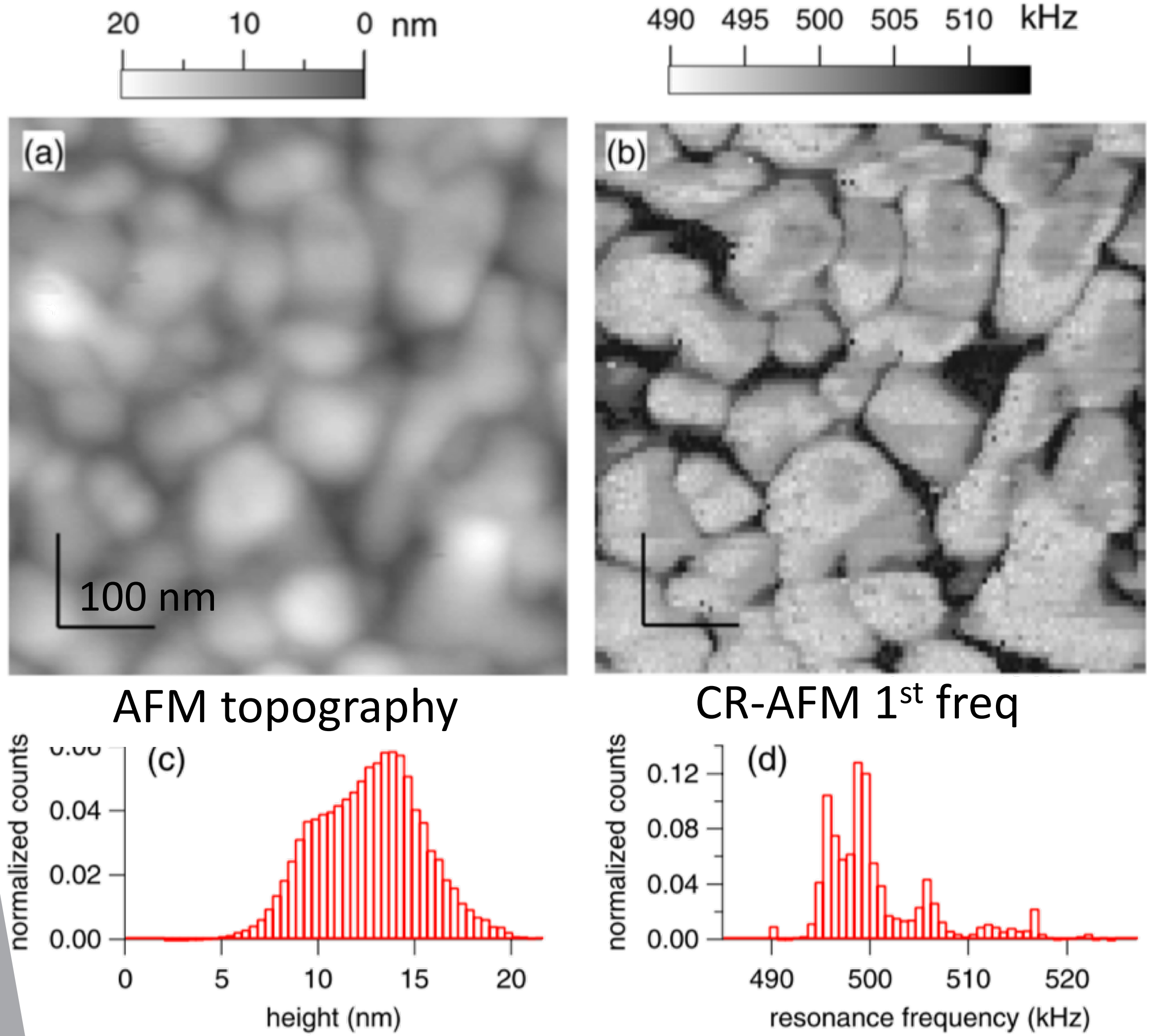




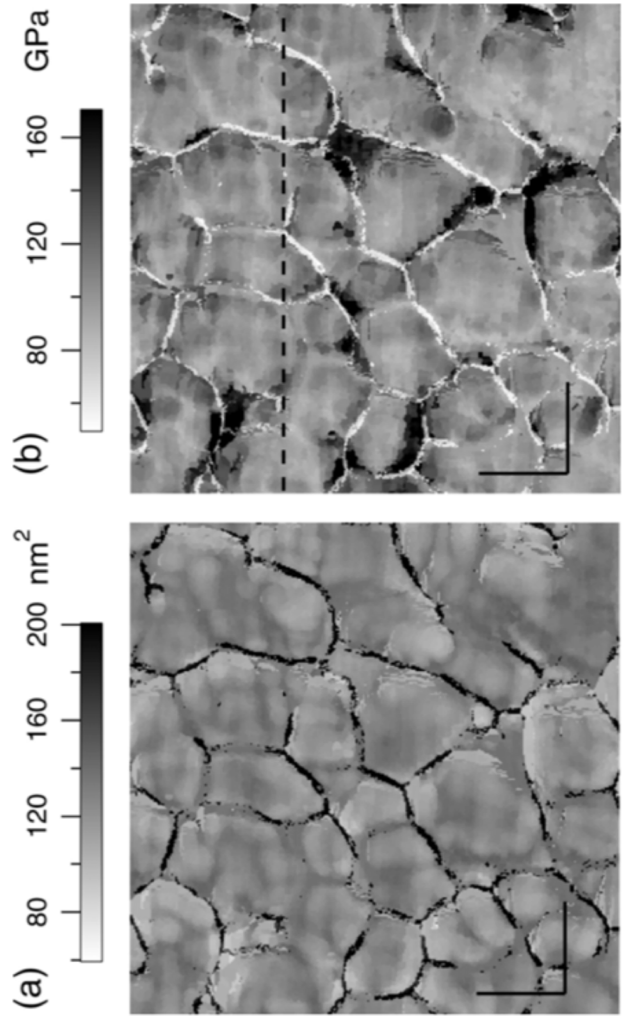
Sample surface effect

- Effect of sample roughness

Stan and Cook, Nanotechnology, 2008, 19, 235701



Corrected indentation modulus



Tip-sample contact area

Au film ≈ <110>, k_C = 14.8±0.5 N/m, 350 nN