

Scanning Force Microscopy

Roland Bennewitz

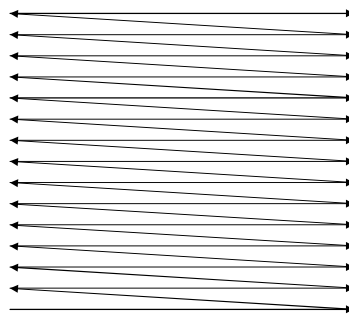
Rutherford Physics Building 405

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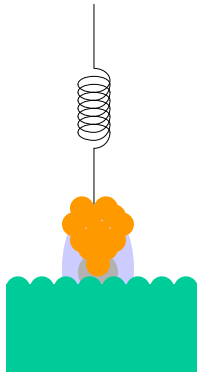
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Scanning ...

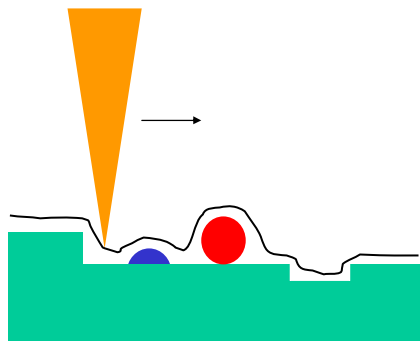
Probe is moved along scan lines over a sample surface



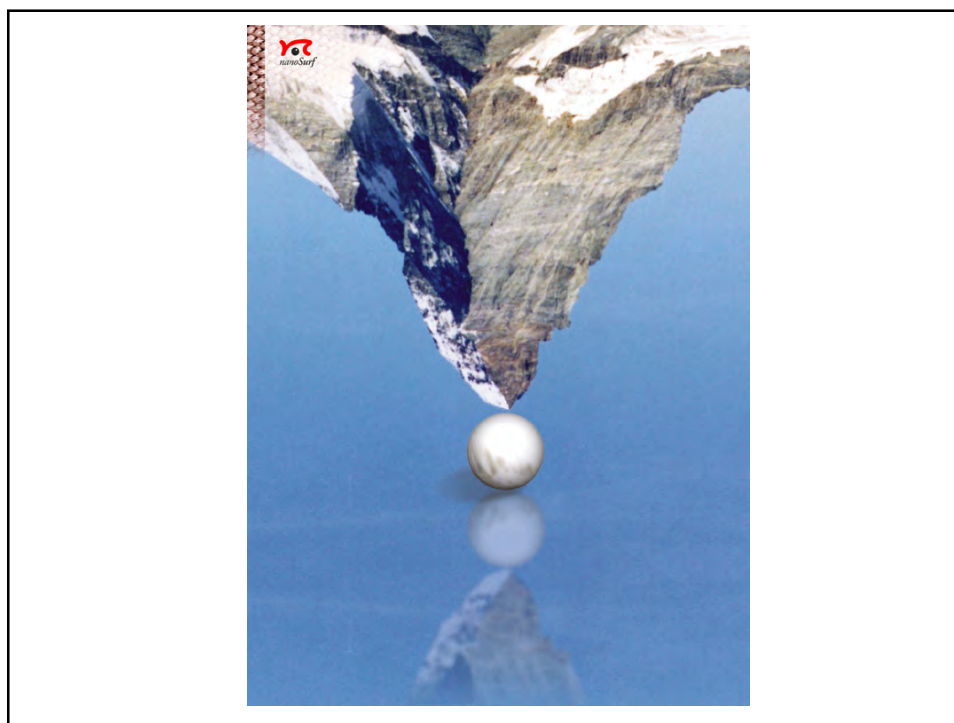
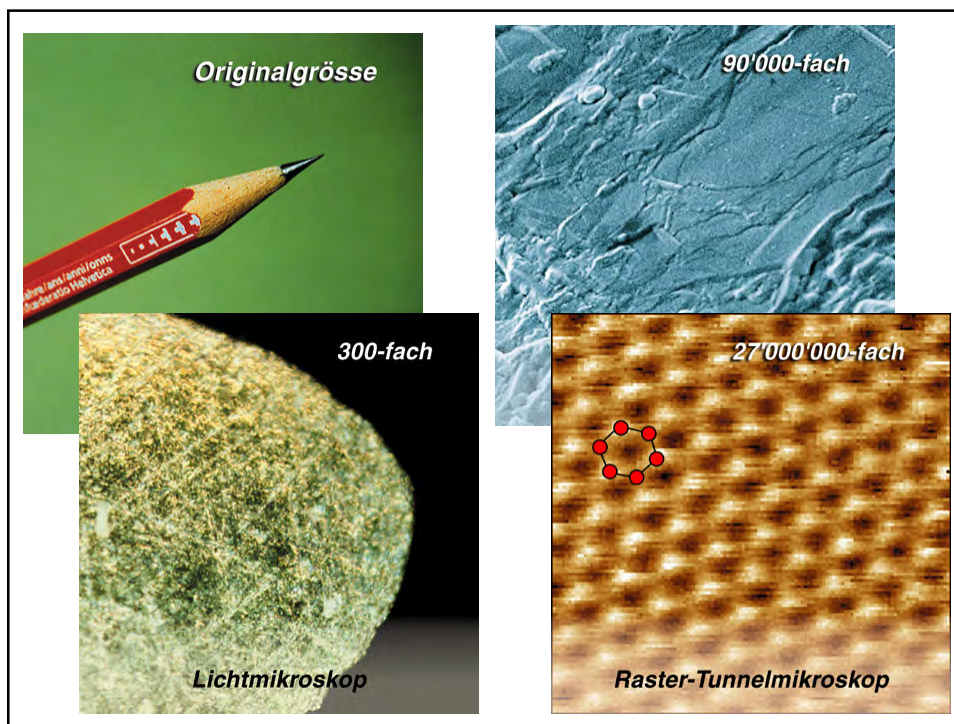
... Force ...



... Microscopy



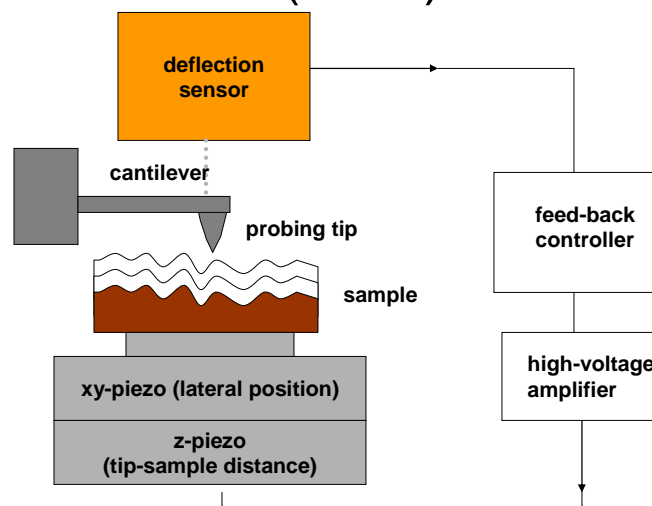
Data are recorded as a function of lateral position.
Display and analysis with help of computer.



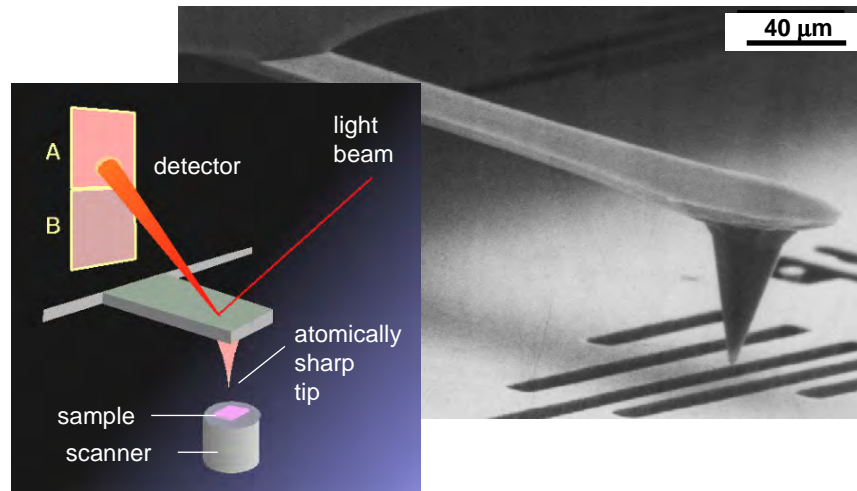
Parts of a Scanning Probe Microscope

- sharp tip probing near-field interactions
(tunneling current, chemical binding forces, optical near-field)
- scanner (positioning actuator) with atomic precision
(usually piezoelectric)
- coarse approach
(micrometer screws, optical microscope)
- vibration damping
(rubber stacks, air-damped tables)
- control electronics
(pre-amplifier, distance controller, high-voltage amplifier)
- data recording
(two screen computer, software)

Scanning Force Microscope (AFM)



Scanning Force Microscope (AFM)

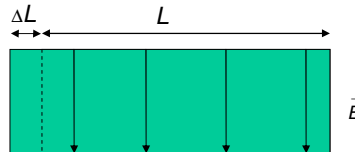


Piezoelectric effect

Piezoelectric scanners work with the transversal piezoelectric effect. The crystal is elongated perpendicular to the applied electric field.

$$\Delta L = d_{31} \cdot L \cdot \vec{E}$$

\vec{E} electric field, L length, ΔL elongation, d_{31} transversal piezoelectric coefficient



Typical material is PZT (Lead Zirkonium Titanat). The relation between Lead and Zirkonium determines the Curie-temperature and the piezoelectric coefficient.

Example: PZT-5H: $d_{31} = -2.62 \text{ \AA/V}$ i.e. $L = 1 \text{ cm}$, $\Delta L = 1 \text{ \mu m}$, $E = 380 \text{ V/mm}$

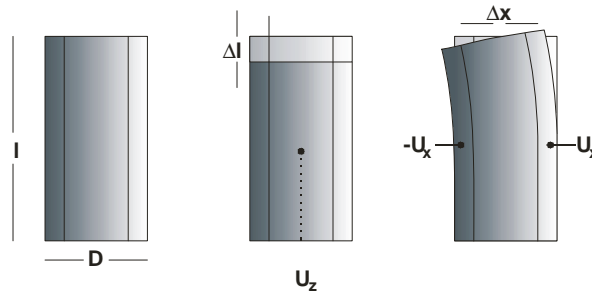
Piezoelectric tube scanner

Sensitivity of the tube scanner:

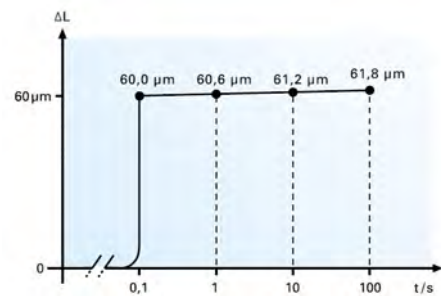
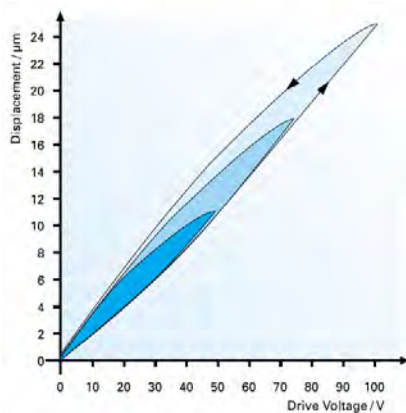
$$\Delta l = d_{31} \cdot U_z \cdot \frac{l}{H}$$

$$\Delta x = \frac{2\sqrt{2} d_{31} \cdot l^2 \cdot U_x}{\pi D H}$$

H thickness, D diameter, d_{31} transversal piezoelectric coefficient.

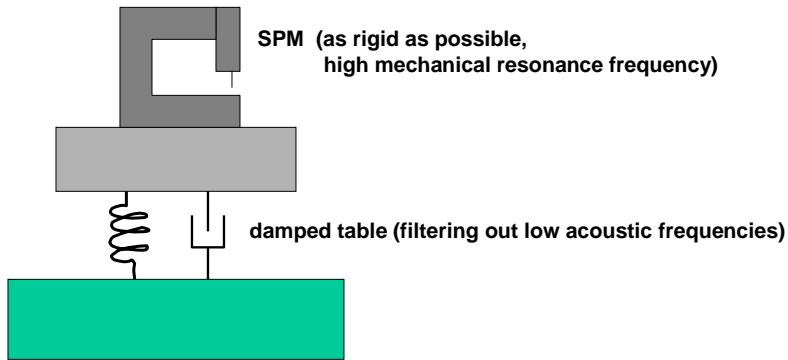


Hysteresis and creep of piezoelectric actuators

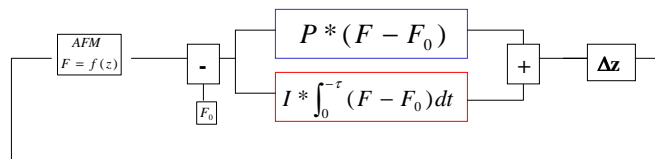


http://www.piceramic.com/pdf/PIC_Tutorial.pdf

Vibration damping

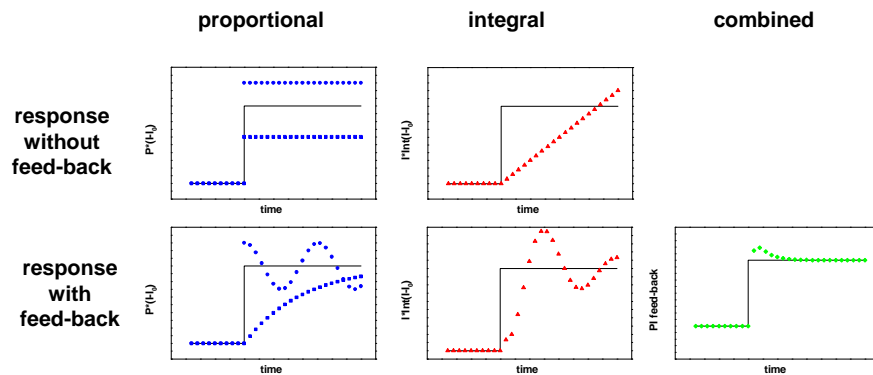


Feed-back controller



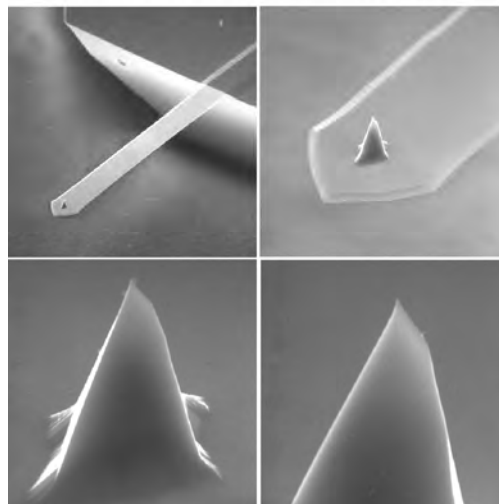
- Setpoint F_0 :** Force signal which should be maintained
- Error signal:** Difference between actual force signal and set point
- Proportional feedback:** Change in tip height proportional to error signal
- Integral feedback:** Change in tip height proportional to accumulated error signal
- Proportional constant P** too low – error will never be corrected
 too high – overshooting and oscillations
- Integral constant I** too low – error will be corrected too slowly
 too high – overshoot will be large

Feed-back answer to a step

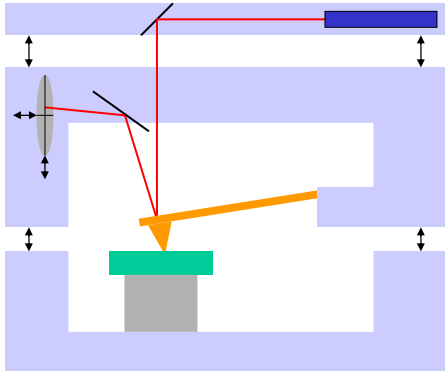


Microfabricated cantilever

Si-Cantilever ($1.5 \times 45 \times 450 \mu\text{m}$)

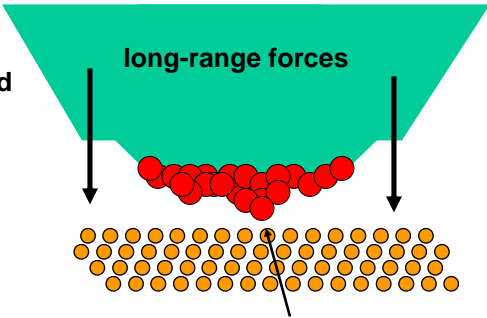


Example – DI microscope



Atomic Force Microscope: Measuring nN forces at surfaces

High resolution by near-field forces between tip apex and surface atoms
Long-range forces between tip body and sample



local chemical binding or repulsive forces

What is a NanoNewton?



What is 1 Newton?
A chocolate-oriented unit for force.

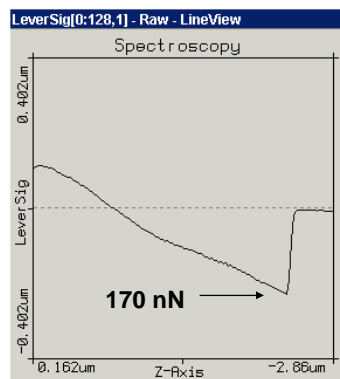
Nanoscience of chocolate?
1 bar are 100 cm^3 or 10^{23} nm^3
Weight of 1 nm^3 is 10^{-23} nN (=0)

Binding forces in chocolate
Cross-section 1 cm^2 or 10^{14} nm^2
Weight carried 4 kg
Rupture force $4 \times 10^{-13} \text{ N/nm}^2$

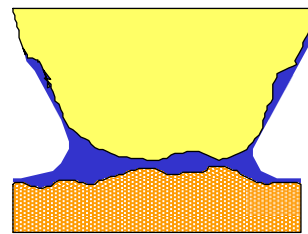
PicoNewton describe molecular forces in softer materials.
NanoNewton describe molecular forces in harder materials



Adhesion - how water disturbs

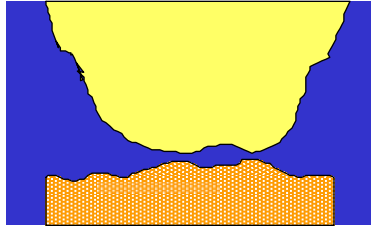


Force vs. distance curve

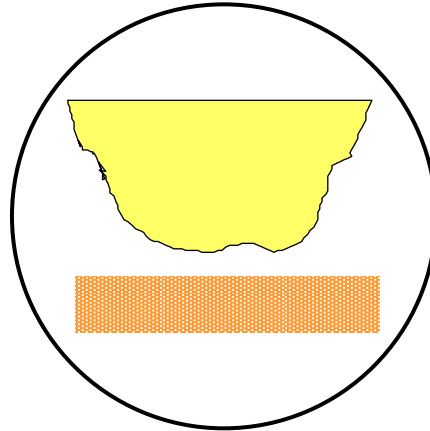


Capillary force

Way out of experimental physics



Measurements in water



... or in vacuum

Relevant forces

- short-range repulsive forces (Pauli exclusion) or ionic repulsion forces
- short-range chemical binding forces
- van der Waals forces (always present, retarded beyond 100 nm)
- electrostatic forces (long-ranged)
- magnetic forces

- interaction in liquids
 - hydrophobic / hydrophilic forces
 - steric forces
 - solvation forces

Literature:

J. Israelachvili

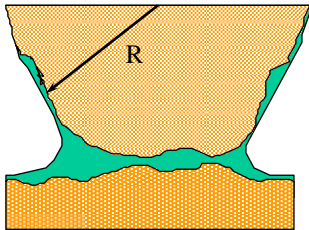
Intermolecular and Surface Forces with Applications to Colloidal and Biological Systems, Academic Press (1985)

Capillary forces

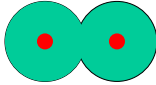
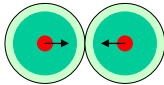
$$F_{\max} = 4 \pi R \gamma \cos(\theta)$$

$\gamma(\text{H}_2\text{O}) = 0.074\text{N/m}$ $R=100\text{nm}$
 Contact angle for hydrophilic surfaces $\theta \approx 0^\circ$

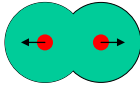
$$\Rightarrow F_{\max} = 90\text{nN}$$



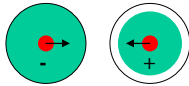
Atomic Forces



Chemical bond



Repulsion upon overlap of electrons

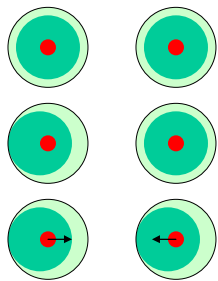


Ionic attraction



Dipole attraction

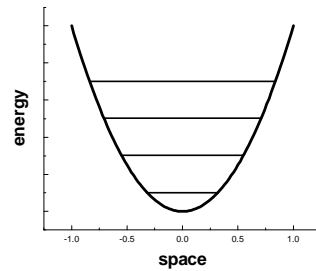
Van der Waals Force



Two neutral atoms

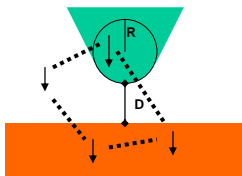
Dipole fluctuation:
Unavoidable in
quantum mechanics

Induced dipole



Range limited to 10 nm due to retardation effect.

Van der Waals forces between tip and sample



Integration of mutual
interactions

Force between the tip and surface can
be approximated as force between a
ball of radius R and a surface at
distance D:

$$F = -\frac{AR}{6D^2}$$

Hamaker constant A is material
dependent and of the order of 10^{-19} J.

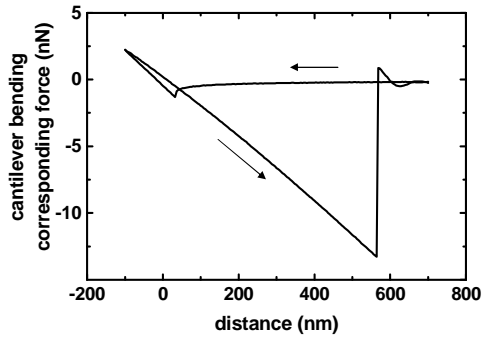
J. Israelachvili, *Intermolecular and surface forces*, chap. 11

Force vs. distance curves

Long-range contributions have force gradients like $dF/dz \gg 1-10 \text{ N/m}$.

Spring constants of $k \approx 0.01-1 \text{ N/m}$ provokes "jump-into-contact" when $k < dF/dz$.

Adhesion causes hysteresis in force vs. distance curves.



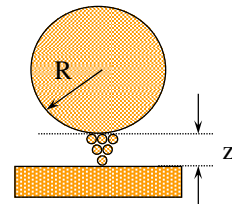
Van der Waals forces in vacuum

No capillary forces (no water).

Van der Waals and electrostatic forces dominate.

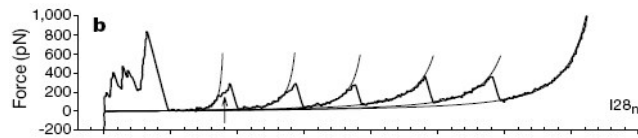
F.O. Goodman and N. Garcia,
Phys. Rev. B 43, 4728 (91)

$\Rightarrow R=100\text{nm}, z=1\text{nm}$



graphite-graphite	8 nN
diamond-diamond	17 nN
metal-graphite	10 nN
SiO ₂ -graphite	1.2 nN

Protein unfolding forces

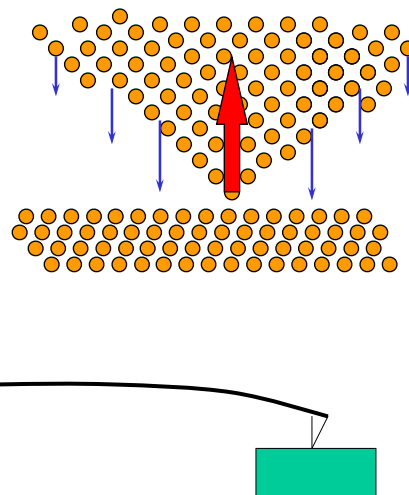


- **Breaking bonds within DNA and proteins: 60 pN to several hundreds of pN**

Fernandez group, Nature 402 (1999) 100

Estimation of forces

- **Typical long-range forces:**
 - in air: 10-100nN
 - in liquids: 1-100pN
 - in ultra-high vacuum: 0.1-10nN
- **Long-range forces are compensated by short-range repulsion. Bending of the cantilever can reduce the repulsive forces and the contact area.**

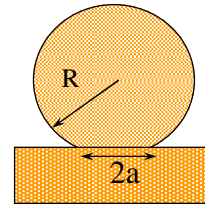


Contact area

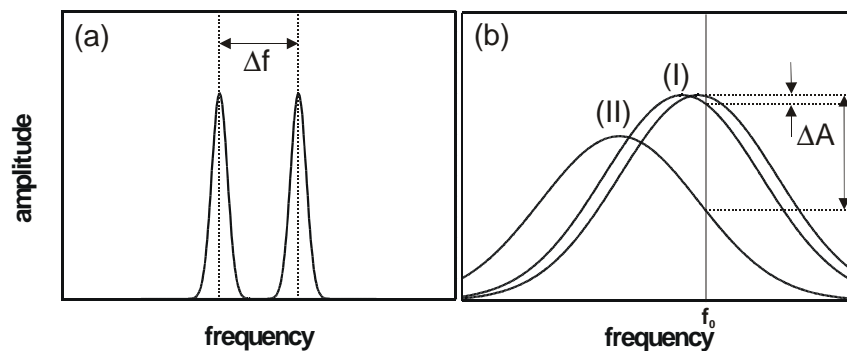
The contact area is given by

$$2a = 2E \sqrt[3]{F \cdot R} \quad (\text{Hertz theory})$$

- in air: 5-100nm
- in liquids: atomic resolution
- in ultra-high vacuum: 1-10 nm



Dynamic force detection



Cantilever is excited to oscillate, frequency shift and amplitude are measured for force detection

(a) high Q-factor (vacuum)

sharp resonance, detection of frequency shift: non-contact mode, Dynamic Force Microscopy

(b) low Q-factor (air, liquid)

fast amplitude response, detection of amplitude: intermittent contact or tapping mode

Advantages of Dynamic Force Microscopy

Avoid jump into contact.

Spring constants $k > 10 \text{ N/m}$.

Rather large amplitudes $kA > F_{adh}$ ($A=2-60\text{nm}$).

Reduced damage of the surface due to lateral surfaces.

Atomic resolution in non-contact modes.

Differentiation of force types in force vs. distance curves.

High force sensitivity.

Setup for tapping mode

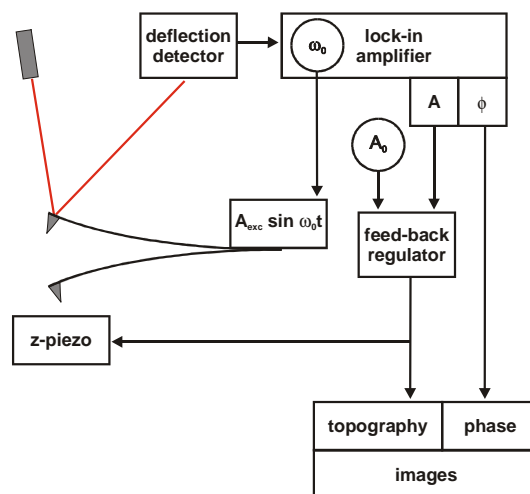
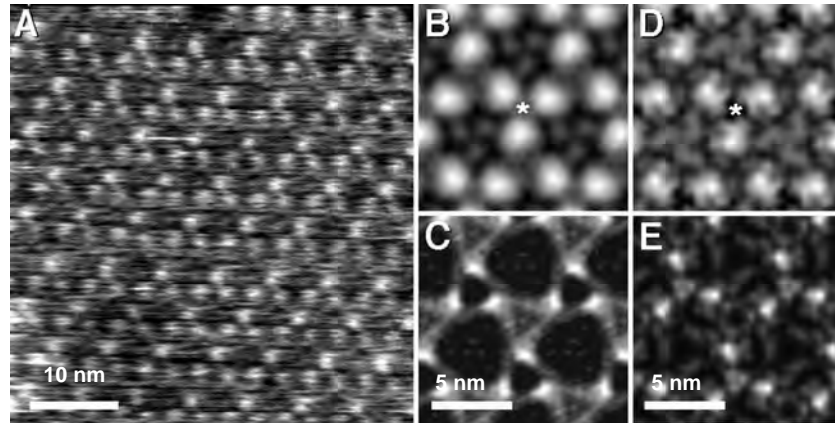


Image of a membrane surface



High-resolution tapping mode images (A). Correlation analysis (B) in comparison with contact mode images (D). C. Möller et al., *Biophys. J.* 77 (1999) 1150

Modelling of tapping mode

The motion of the tip can approximately be described as a damped harmonic oscillator:

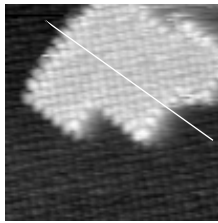
$$m\ddot{z} = -kz - \frac{m\omega_0}{Q}\dot{z} + F_{tip-sample} + F_0 \cos(\omega t)$$

The tip-sample and consequently the tip motion is non-linear, particularly in the intermittent contact mode.

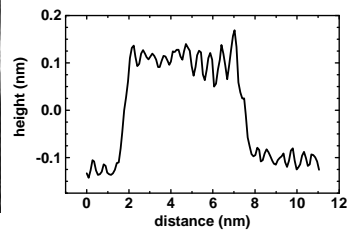
Quantitative analysis is difficult due to partly chaotic behaviour.

Garcia and San Paulo, *Phys. Rev. B* 61 (2000) R13381

Enhanced corrugation at steps



NaCl island



- **enhanced corrugation at low-coordinated sites**
- **instabilities at step edges**