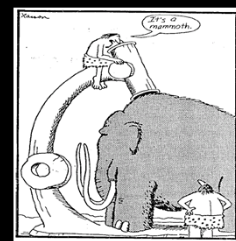
Introduction to Atomic Force Microscope

Not that kind of “atomic”

Tien-Ming Chuang (莊天明)
Institute of Physics, Academia Sinica

1

Introduction to Scanning Force Microscope



Early microscope

Tien-Ming Chuang (莊天明)
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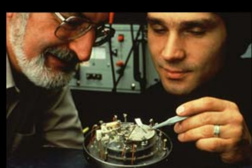
2

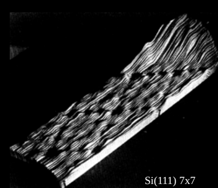
Scanning Tunneling Microscope (STM)

Applicable only to conducting samples.



Heinrich Rohrer & Gerd Binnig
Nobel Prize in 1986
©IBM

G. Binning *et al.*,Phys. Rev. Lett. 49, 57 (1982)



Si(111) 7x7

G. Binning *et al.*, Phys. Rev. Lett. 50, 120 (1983)

3

Invention of Atomic Force Microscope (AFM)

- One of the most versatile analytical tools to appear on the research scene in a century.
- No. 4 of Most Cited PRL papers as in November, 2002.

First AFM in Science Museum, London

Gerd Binnig
© Nobel Foundation

Calvin Quate
©Stanford

Christoph Gerber
©University of Basel

But no atomic resolution then!

G. Binnig *et al.*, Phys. Rev. Lett. 56, 930–933 (1986)

4

Invention of Atomic Force Microscope (AFM)

Seeing by feeling the force!

Hooke’s Law at atomic scale!

5

What force?

6

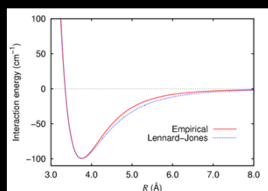
Tip-Sample Forces : Microscopic Description

- The Lennard-Jones potential:

$$V_{Lennard-Jones} = 4\varepsilon\left[\left(\frac{\sigma}{r}\right)^{12} - \left(\frac{\sigma}{r}\right)^{6}\right]$$

van der Waals, attractive

Short range, Pauli + chemical, repulsive



7

Long Range Tip-Sample Forces : Magnetic Force

- If the tip were coated with magnetic material, it would be sensitive to the stray field from the surface.
- Due to the extent of the tip geometry, the force acting on the tip is the integral over all the dipole moments on the tip:

$$F_{mag} = \int_{tip} \vec{m}_{tip}(\vec{r}) \cdot \nabla\vec{B}_{sample}(\vec{r})\mathrm{d}\vec{r}$$

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Long Range Tip-Sample Forces : Electrostatic Force

- The strength and distance dependence of electrostatic forces obey Coulombs law.
- Electrostatic forces :

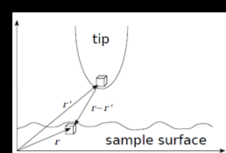
$$F_{el} = \pi\varepsilon_0 \frac{R}{z} U^2$$

- R : Tip radius ; U : Potential difference
- For R = 20 nm at a tip-sample distance of z = 0.5 nm and U= 1V, F_{el} =0.5nN
- Zero bias voltage normally does not correspond to a minimal electrostatic force. → The contact potential difference has to be compensated.

9

Tip-Sample Forces

Total force on the force sensor (cantilever)
= sum of all forces.



Reference: Intermolecular and surface forces (2011) by Jacob N. Israelachvili

10

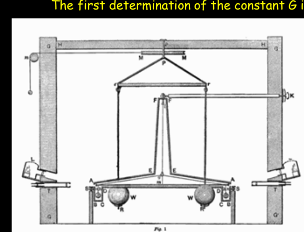
Force Sensors

You must feel the force around you.

11

Force Measurement

The first determination of the constant G in 1798 by Henry Cavendish



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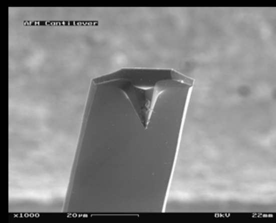
Force Sensors for AFM : Cantilevers

- Hooke's Law
  $F = k\Delta z$
- Spring constant k :
  $$k = \frac{E_Y w t^3}{4L^3}$$
  typical values: 0.01 - 100 N/m
  Young's modulus $E_Y \sim 10^{12}$ N/m^2
- Resonant frequency f_o:
  $$f_0 = \frac{1}{2\pi}\sqrt{\frac{k}{m_{eff}}}$$
  typical values: 7 - 500 kHz



©Wikipedia

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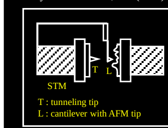
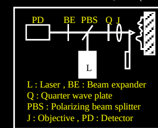
Deflection Sensors

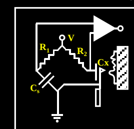
Tunneling current
Phys. Rev. Lett. **56**, 930 (1986).



Optical interferometer
J. Vac. Sci. Technol. A **6,** 266 (1987).



Capacitive detection
J. Vac. Sci. Technol. A **8,** 383 (1990).



Mostly obsolete from today's AFMs.

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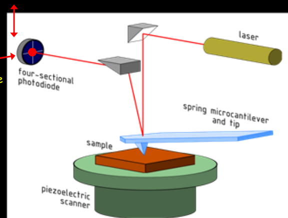
Deflection Sensors : Optical Beam Deflection

Most widely used in commercial AFM

AFM

Lateral Force Microscope



G. Meyer *et al.*, APL 53, 1045 (1988).

15

Deflection Sensors : Fiber Optic Interferometer

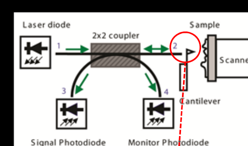
Fresnel Reflection

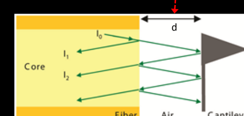
$$\frac{I_1}{I_0} = \left(\frac{n_{core} - n_{air}}{n_{core} + n_{air}}\right)^2 \sim 4\%$$

Interference :

$$I_{total} \sim I_1 + I_2 + 2\sqrt{I_1 I_2}\cos\left(\frac{4\pi d}{\lambda}\right)$$

Great for low temperature!

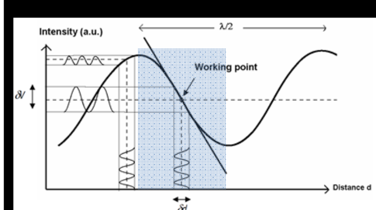


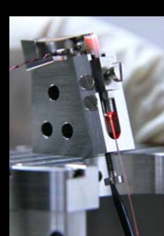


D. Rugar *et al.*, APL **55**, 2588 (1989)

16

Deflection Sensors : Fiber Optic Interferometer





Most sensitive! One should keep the tip-fiber distance here.
Practically, it will be fine-tuned by a piezo.

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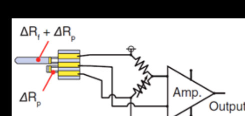
Deflection Sensors : Self-sensing Methods

Piezo-resistive detection

- Self-sensing
- Doped Si
- Low Q

$$V_{out} = \frac{\frac{\Delta R_f}{R}}{2\left(2 + \left(\frac{\Delta R_f + 2\Delta R_p}{R}\right)\right)} \cong \frac{\Delta R_f}{4R} V_{in}$$



M. Tortonese *et al.*, APL 62, 834 (1993)
Uijin G Jung *et al.*, J. Micromech. Microeng. 23, 045015 (2013)

Tuning fork detection

- Used in non-contact attractive force region.
- $f_0 = 2^{15}$Hz
- Frequency-modulated operation

Giessibl, Appl. Phys. Lett. 73, 3956 (1998)

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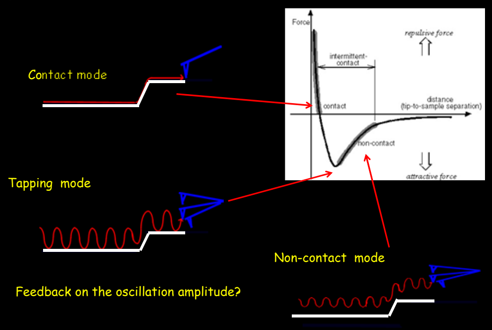
Operation Modes for Topography

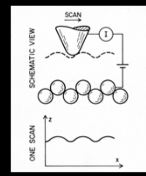


Feedback on the oscillation amplitude?

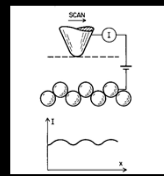
Contact Mode Operation

- Repulsive force region
- Slow scan rate.
- Local deformations or destructions of the sample surface.
- Tip wear issue

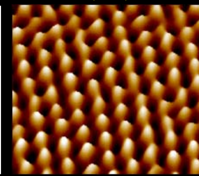
F = constant



z = constant



Mica in air, 5 nm, ©Veeco



Dynamic Operation of AFM

- Cantilever is driven at its resonance:

$$m\ddot{z} + \frac{m\omega_0}{Q}\dot{z} + m{\omega_0}^2 z = F\cos\omega_d t$$

- The particular solution is $z = z_c + z_p$
- Transient state:

$$z_c = [A_1 \exp\left(\sqrt{\frac{{\omega_0}^2}{4Q^2} - {\omega_0}^2 t}\right) + A_2 \exp\left(-\sqrt{\frac{{\omega_0}^2}{4Q^2} - {\omega_0}^2 t}\right) \cdot \exp(-\frac{\omega_0}{2Q} t)]$$

- Steady state:

$$z_p = A\cos(\omega_d t - \delta)$$

where $\delta = \tan^{-1}({}^{\omega_d\omega_0}/_{Q({\omega_0}^2 - {\omega_d}^2)})$

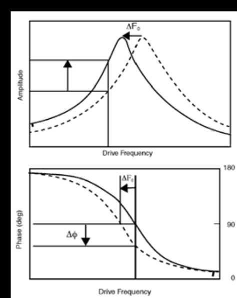
$$A = \frac{A_0(\omega_d/\omega_0)}{\sqrt{1 + Q^2(\frac{\omega_0}{\omega_d} - \frac{\omega_d}{\omega_0})^2}}$$

Dynamic Operation of AFM

- In a presence of force : $k_{eff} = k - \frac{\partial F}{\partial z}$
- The shift in resonance frequency :

$$\omega = \omega_0 \sqrt{1 - \frac{1}{k}\frac{\partial F}{\partial z}} \cong -\frac{\omega_0}{2k}\frac{\partial F}{\partial z}$$

- The phase shift : $\Delta\delta = -\frac{Q}{k}\frac{\partial F}{\partial z}$
- The settle time for amplitude : $\tau = \frac{2Q}{\omega_0}$
- High Q cantilever enhances the sensitivity but responds slower with amplitude detection (slope detection).
- Frequency modulation is used for high Q situation, especially in vacuum.

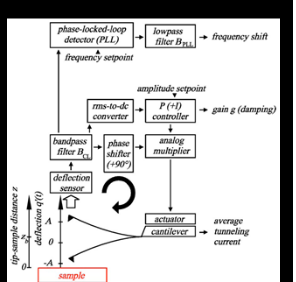


©Veeco

Dynamic Operation of AFM : Frequency Modulation

- Feedback loop 1:
The cantilever oscillation is controlled at the resonance with the same amplitude by tracking the phase shift.

- Feedback loop 2:
The tip-sample distance is controlled to keep the frequency shift constant and hence the same tip-surface force.



T. R. Albrecht *et al.*, J. Appl. Phys. 69, 668 (1991)
Franz J. Giessibl, Rev. Mod. Phys. 75, 949 (2003)

Limits of Force Sensitivity

- A cantilever in equilibrium with a bath of temperature, T has a energy expectation value for each mode equal to $k_B T/2$.

$$\frac{1}{2}k\langle A^2\rangle = \frac{1}{2}k_B T$$

- Minimum detectable force due to thermal fluctuation:

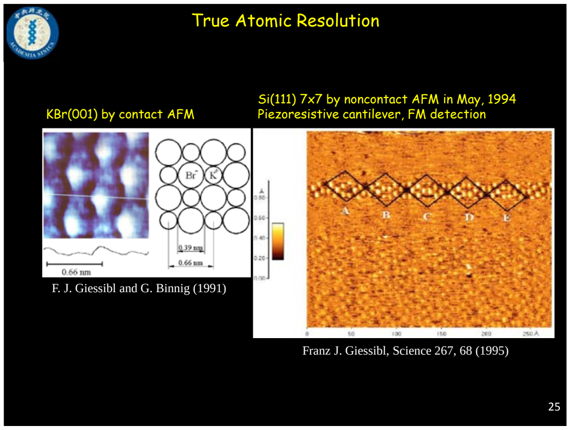
$$\frac{\partial F_z}{\partial z} = \frac{1}{A}\sqrt{\frac{4Bk_B Tk}{Q\omega_0}}$$, B=measurement bandwidth

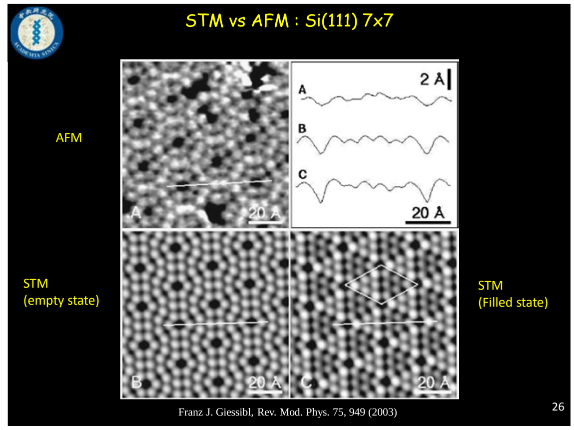
- Optimization:
1. Higher Q
2. Higher resonance frequency
3. Lower temperature
4. Amplitude?
5. Softer cantilever?

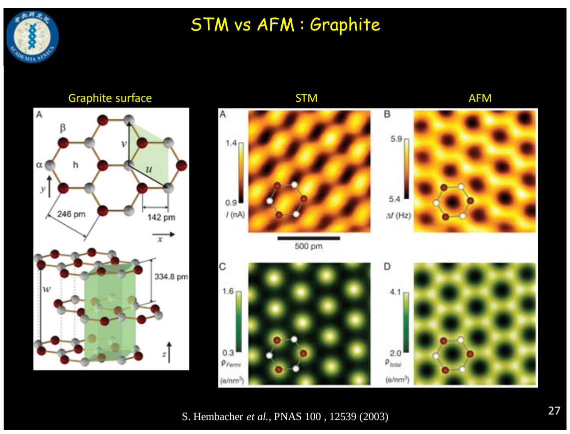
- Tuning fork !!!

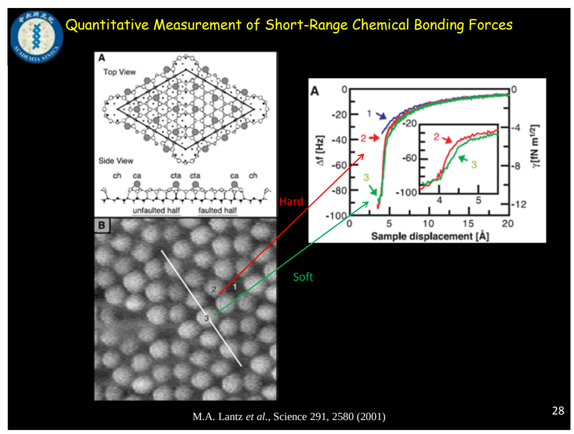
f_0~32kHz, k~1800N/m, Q~50000 in vacuum
Noncontact mode operation.

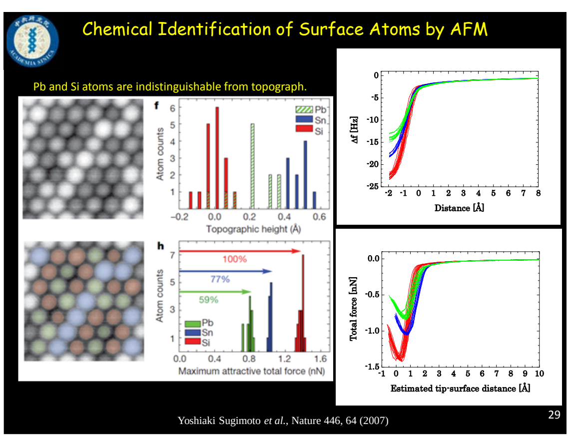
Y. Martin *et al.*, J. Appl. Phys. 61, 4723 (1987)
Franz J. Giessibl, Rev. Mod. Phys. 75, 949 (2003)

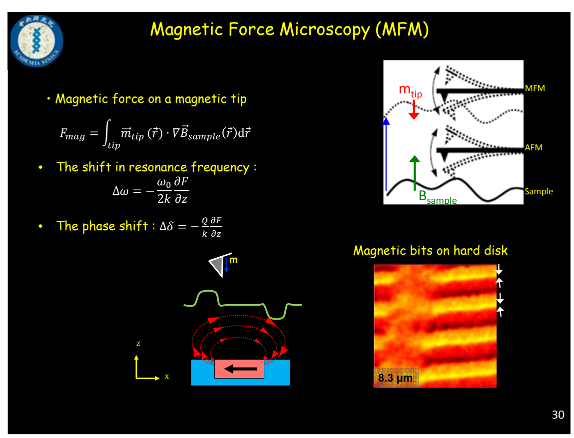

True Atomic Resolution
KBr(001) by contact AFM
Si(111) 7x7 by noncontact AFM in May, 1994
Piezoresistive cantilever, FM detection
F. J. Giessibl and G. Binnig (1991)
Franz J. Giessibl, Science 267, 68 (1995)
25


STM vs AFM : Si(111) 7x7
AFM
STM
(empty state)
STM
(Filled state)
2 Å
20 Å
Franz J. Giessibl, Rev. Mod. Phys. 75, 949 (2003)
26


STM vs AFM : Graphite
Graphite surface
STM
AFM
246 pm
142 pm
334.8 pm
500 pm
S. Hembacher et al., PNAS 100 , 12539 (2003)
27

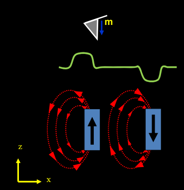

Quantitative Measurement of Short-Range Chemical Bonding Forces
Top View
Side View
unfaulted half
faulted half
Hard
Soft
Δf [Hz]
γ [fN m^1/2]
Sample displacement [Å]
M.A. Lantz et al., Science 291, 2580 (2001)
28


Chemical Identification of Surface Atoms by AFM
Pb and Si atoms are indistinguishable from topograph.
Atom counts
Topographic height (Å)
Maximum attractive total force (nN)
100%
77%
59%
Pb
Sn
Si
Δf [Hz]
Distance [Å]
Total force [nN]
Estimated tip-surface distance [Å]
Yoshiaki Sugimoto et al., Nature 446, 64 (2007)
29


Magnetic Force Microscopy (MFM)
• Magnetic force on a magnetic tip
$F_{mag} = \int_{tip} \vec{m}_{tip}(\vec{r}) \cdot \nabla \vec{B}_{sample}(\vec{r}) d\vec{r}$
• The shift in resonance frequency :
$\Delta\omega = -\frac{\omega_0}{2k}\frac{\partial F}{\partial z}$
• The phase shift : $\Delta\delta = -\frac{Q}{k}\frac{\partial F}{\partial z}$
MFM
AFM
Sample
Magnetic bits on hard disk
8.3 μm
30

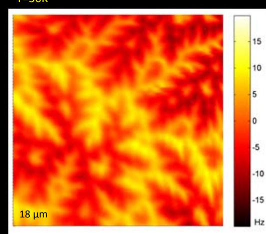
Magnetic Force Microscopy (MFM)

Out-of-plane magnetization



$La_{2-2x}Sr_{1+2x}Mn_2O_7$,x=0.32
Sample: Dr. J. Goodenough @ UT Austin

T=30K



T.-M. Chuang *et. al.*, RSI 78, 053710 (2007)
Junwei Huang *et al.*, PRB 77, 024405 (2008)

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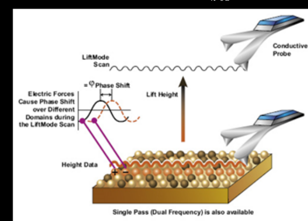
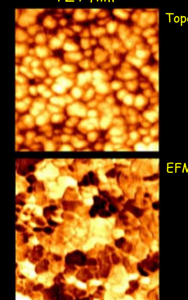
Electrostatic Force Microscopy (EFM)

- The shift in resonance frequency : $\Delta\omega = -\frac{\omega_0}{2k}\frac{\partial F}{\partial z}$
- The phase shift : $\Delta\delta = -\frac{Q}{k}\frac{\partial F}{\partial z}$



©Bruker

PZT film



©Park AFM

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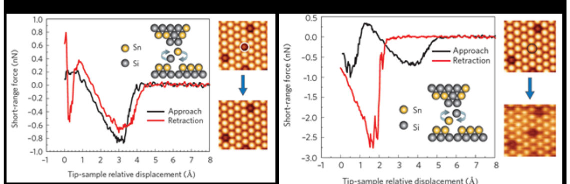
Atomic Manipulation by AFM

Sn on Si(111)



Review article:
Nature Nanotechnology 4, 803 - 810 (2009)

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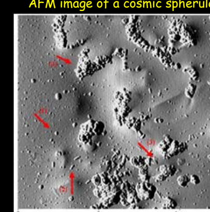
MIDAS (Micro Imaging Dust Analysis System)

Rosetta orbiter for Comet 67 P/Churyumov-Gerasimenko
Launched on March 2, 2004.
MIDAS is designed to capture and image dust particles in with AFM.

See also AFM on Mars:
http://phoenix.lpl.arizona.edu/index.php

©NASA

AFM image of a cosmic spherule



Space Science Reviews (2007) 128: 869–904

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The Need for High Speed AFM

- Mostly in tapping mode, scan size up to 500nm, >7 frames/sec.
- The scan rate is limited by the slowest component:

1. Smaller cantilever for higher resonance
2. Faster scanner
3. Faster feedback and control

Review article:
Toshio Ando, Nanotechnology 23, 062001 (2012)…Mostly bio-imaging

AFM manufacturers:
http://www.bruker.com/fastscan
http://www.asylumresearch.com/Gallery/CypherFastScanning/CypherFastScanning.shtml

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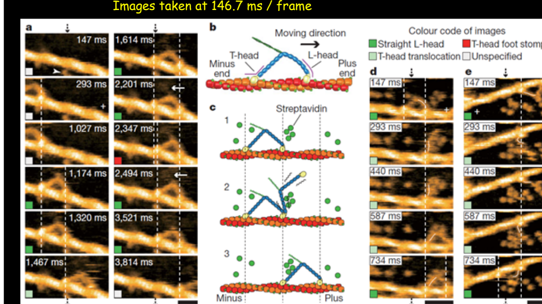
Imaging of Molecular Dynamics by High-speed AFM

Directly visualized walking Myosin V molecules .
Images taken at 146.7 ms / frame



N. Kodera *et al.*, Nature 468, 72 (2010)

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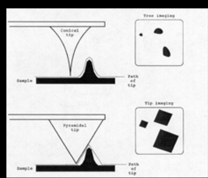
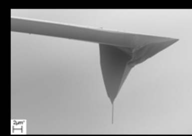
Improving AFM Cantilevers



AFM Cantilever with AgGa nanoneedle



©NaugaNeedles

Young's modules ~1T Pa for Carbon nanotube!

Review:
Carbon nanotube tips for AFM
Nature Nanotechnology 4, 483 (2009)

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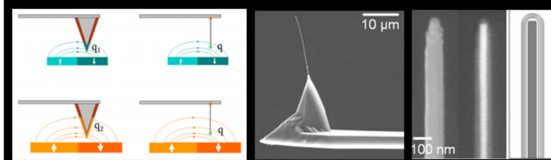
Improving MFM Cantilevers

Fe-filled Carbon nanotube for MFM



F. Wolny *et al.*, J. Appl. Phys. 104, 064908 (2008)
F. Wolny *et al.*, Nanotechnology 21 435501 (2010)

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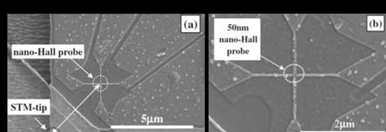
Other Magnetic Force Imaging Techniques

- Scanning Hall Probe Microscopy

Direct B measurement
Limited spatial resolution due to senor size
Other means for feedback for topography is required.



Jpn. J. Appl. Phys., 43,777 (2004)

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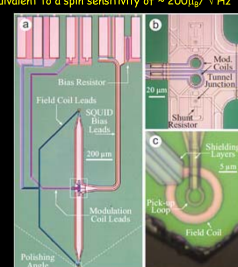
Other Magnetic Force Imaging Techniques

Scanning SQUID Microscopy

The spectral density of the flux noise in the frequency-independent region is $0.25\mu\Phi_0/\sqrt{Hz}$ at T<0.2K, equivalent to a spin sensitivity of ~ $200\mu_B/\sqrt{Hz}$



RSI **79**, 053704 (2008)

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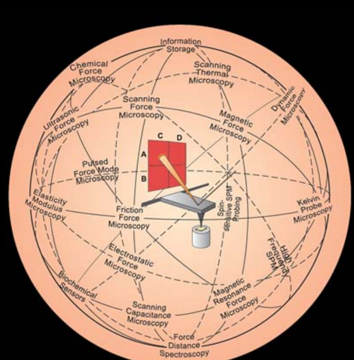
AFM Universe



Ch. Gerber, H.P. Lang, Nature Nanotechnology **1**, 3 (2006)

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