Scanning Force Microscopy

“Nanostructure characterization techniques”

UT-Austin
PHYS 392 T, unique # 59770
ME 397 unique # 19079
CHE 384, unique # 15100

Instructor:
Professor C.K. Shih

Lecture Note: 9/22/09 and 9/24/09
Schematic of an AFM. The tip is fastened to a cantilever spring that has a lower spring constant than the effective spring between two atoms. With sufficient sensitivity in the spring deflection sensor, the tip can reveal surface profiles with atomic resolution.

Realization of the possibility

[Idea – 1985 by Binnig & Quate; see PRL 56, 93 (1986)]

\[ \omega \sim 10^{13} \text{ sec}^{-1}; \ m \sim 10^{-25} \text{ kg} \rightarrow \text{Inter-atomic spring constant} \sim 10 \text{ N/m} \]

A typical household Al foil of a size 4 mm x 1 mm \( \rightarrow \) 1 N/m

If one can detect 1Å cantilever deflection \( \rightarrow \) detecting a force of \( 10^{-10} \) N

\[ \rightarrow \text{Design of Cantilever \& Force Sensor} \]
Fundamental Forces

1. Gravitation – too small to be important
2. E.M. (responsible for most of solid-solid, atom-atom interactions)
3. Weak
4. Strong \( \text{Range} < 10^{-15} \text{ m.} \)

Forces between Atoms and Molecules

- Ionic Bonds
- Covalent Bonds
- Metallic Bonds
- Repulsive Forces
- Van der Waals Forces
  - Dipole – Dipole (Hydrogen Bonds)
  - Dipole – Induced dipole
  - Induced dipole – Induced dipole

Valence electrons play the dominant role
MAGNETIC 10 nm Fe PARTICLES

COULOMB 2 ELECTRONS

MAGNETIC TWO SPINS

very small, almost impossible for AFM.
# Forces relevant to SFM

<table>
<thead>
<tr>
<th>Short-range</th>
<th>Long range</th>
</tr>
</thead>
<tbody>
<tr>
<td>• All the bonding forces</td>
<td>• Van der walls</td>
</tr>
<tr>
<td>mentioned earlier</td>
<td>• Magnetic</td>
</tr>
<tr>
<td>- ionic, covalent, metallic</td>
<td>• Electrostatic</td>
</tr>
<tr>
<td>bonds</td>
<td>Capillary (Meniscus): important</td>
</tr>
<tr>
<td>• Repulsive</td>
<td>when it is not “clean” solid-</td>
</tr>
<tr>
<td>- Coulomb repulsion</td>
<td>solid interaction.</td>
</tr>
<tr>
<td>- Pauli exclusion principle</td>
<td>- Can be reduced under water</td>
</tr>
<tr>
<td>• Friction</td>
<td>or other liquid</td>
</tr>
<tr>
<td>• Elastic, plastic deformation</td>
<td>- Atomically clean tip, sample</td>
</tr>
<tr>
<td>• Others (e.g. exchange force)...</td>
<td>under UHV → No capillary</td>
</tr>
<tr>
<td></td>
<td>force</td>
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</table>
Components of AFM

• Cantilever + Tip (low k,* high $\omega_n$)
  * for non-contact AFM, this is no-longer true)
• Force sensing ($10^{-14} – 10^{-4}$ N)
• DC + AC modes operations
• Feedback circuits
Force sensing

**Fig. 11.8** Schematics of the four more commonly used detection systems for measurement of cantilever deflection. In each setup, the sample mounted on piezoelectric body is shown on the right, the cantilever in the middle, and the corresponding deflection sensor on the left [11.118]

(figures copied from Springer Handbook of Nanotechnology)
optical deflection

L: optical path from cantilever to detector

l: cantilever length

S: deflection of cantilever

⇒ 2S: deflection of reflected beam

\[ \frac{\Delta X}{L} = 2S \]

\[ \frac{\Delta \delta}{\delta} = S \quad \text{(more precisely)} \]

\[ \Rightarrow \frac{\Delta X}{\Delta \delta} = \frac{3L}{\delta} \quad \text{geometric gain} \geq 100 \]
Different Modes of Operation

- Contact Mode
- Non-contact Mode
- Tapping Mode (or Intermittent contact mode)
Tip-sample interaction induced resonance frequency shift

\[ k_{\text{total}} = k + k_{ts} = k - \frac{\partial F_{ts}}{\partial z}. \]

\[ \omega^2 = (\omega_0 + \Delta \omega)^2 \]
\[ = k_{\text{total}}/m^* = \left( k - \frac{\partial F_{ts}}{\partial z} \right)/m^*. \]

\[ \frac{\Delta \omega}{\omega_0} \approx -\frac{1}{2k} \frac{\partial F_{ts}}{\partial z}. \]

Idea: measuring \( \Delta \omega (z) \to \partial F_{ts}/\partial z (z) \to F_{ts} (z) \)

This is possible only if \( \partial F_{ts}/\partial z (z) \) is well-defined within the oscillation cycle \( \to \) very small oscillation amplitude \( \to \) dilemma (large amplitude is needed to avoid jump to contact)
Vibration of a cantilever near its resonance

\[ \frac{\omega_0}{Q} = \omega \]

\[ \omega_0 = \sqrt{\frac{K}{m}} \]

Force interaction between sample & tip

\[ k = -\frac{\partial F}{\partial z} \]

Effective spring

\[ K' = K - k \]

Shift in the resonance frequency

AM operation (amplitude modulation)
By choosing the operation point near the highest \( \frac{\partial A}{\partial w} \Rightarrow \) highest sensitivity in detecting force gradient.

(Also, the higher the \( Q \), the higher \( \frac{\partial A}{\partial w} \))

Feedback control!

\( \Rightarrow \) Constant force gradient.

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Note:
If the operation point is chosen on the opposite of the resonance curve.

\( \Rightarrow \) Tapping Mode.
In Early Days, only limited samples (e.g. Graphite) can exhibit atomic resolution.

**Fig. 11.13a, b** Typical AFM images of freshly-cleaved (a) highly-oriented pyrolytic graphite and (b) mica surfaces taken using a square pyramidal Si$_3$N$_4$ tip.
Typical tip shapes

Fig. 11.16a–c SEM micrographs of a square-pyramidal PECVD Si₃N₄ tip (a), a square pyramidal etched singlecrystal silicon tip (b), and a three-sided pyramidal natural diamond tip (c)
Schematics of (a) triangular cantilever beam with square pyramidal tip made of PECVD Si3N4,
Si$_3$N$_4$ tip fabrication

Apply mask  
\[ \text{Si} \]

Anisotropic etch

Remove mask

Deposit Si$_3$N$_4$

Remove Si

Si tip fabrication

Si cantilever  
\[ \text{Si} \]

Apply mask

Under etch

Remove mask
(b) Rectangular cantilever beams with square pyramidal tips made of etched single-crystal silicon,
(c) rectangular cantilever stainless steel beam with three-sided pyramidal natural diamond tip
Image = convolution of surface topography and the tip shape

If the probe tip shape is known → deconvolution is possible (but not 100% recovery)
By using a sample with known sharp spikes → probe shape can be determined
Fig. 12.10 A schematic drawing of the setup for manual assembly of carbon nanotube tips (*top*) and optical microscopy images of the assembly process (the cantilever was drawn in for clarity)
Fig. 11.18 SEM micrograph of a multi-walled carbon nanotube (MWNT) tip physically attached on the single-crystal silicon, square-pyramidal tip (Courtesy Piezomax Technologies, Inc.)
Fig. 12.11 Nanotube tip buckling. Top diagrams correspond to labeled regions of the force curves. As the nanotube tip buckles, the deflection remains constant and the amplitude increases, from [12.16]
Fig. 11.17a,b Schematics of (a) HART Si3N4 probe, and (b) FIB milled Si3N4 probe
Fig. 11.19 (a) Schematic defining the $x$- and $y$-directions relative to the cantilever, and showing the sample traveling direction in two different measurement methods discussed in the text, (b) schematic of deformation of the tip and cantilever shown as a result of sliding in the $x$- and $y$-directions. A twist is introduced to the cantilever if the scanning is in the $x$-direction (b), lower part) [11.136]
Fig. 11.20 (a) Schematic showing an additional bending of the cantilever – due to friction force when the sample is scanned in the +y (right) or −y direction (left). (b) This effect will be canceled by adjusting the piezo height by a feedback circuit (right) [11.136]
Fig. 11.21 Schematic illustration of the height difference of the piezoelectric tube scanner as the sample is scanned in $+y$ and $-y$ directions.
Fig. 11.22 The trajectory of the laser beam on the photodetectors in as the cantilever is vertically deflected (with no torsional motion) for a misaligned photodetector with respect to the laser beam. For a change of normal force (vertical deflection of the cantilever), the laser beam is projected at a different position on the detector. Due to a misalignment, the projected trajectory of the laser beam on the detector is not parallel with the detector vertical axis (the line joint T-B) [11.136]
Fig. 11.23a,b Illustration showing the deflection of cantilever as it is pushed by (a) a rigid sample or by (b) a flexible spring sheet [11.136]
Subject: Non-contact (or Dynamic) AFM

References:

Lecture Note on 9/24/09
A typical F-Z curve

- Tip-to-sample separation, $z$
- Position of the z-piezo actuator, $z_a$
- Deflection of the cantilever, $\delta$
- Force constant of the cantilever, $k_o$
- Tip-sample force, $F_{ts}$

Note, $z = z_a + \delta$ and $\delta = F_{ts}/k_o$

Analysis of DC (static) mode

- Jump to contact during approach
- Snap back during retraction

Instability occurs when

$$\left. \frac{\partial F_{ts}}{\partial z} \right|_z > k$$

Dilemma:
Soft cantilever $\rightarrow$ better force sensitivity but can not avoid jump-to-contact
Hard cantilever $\rightarrow$ avoiding jump-to-contact but low force sensitivity (?! counter argument made in RMP 75, 949 which favors stiff cantilever)
Option 1: Hard cantilever
How hard do we need?
Inter-atomic force $\sim 10 \text{ N/m}$, but the total force $\sim \# \text{ of atoms}$
$\Rightarrow k >> 100 \text{ N/m}$

[Images of atomic force microscope images]

Option 2: Dynamic AFM
Preload the cantilever with a large restoring force (oscillating cantilever). Note this works even for soft cantilever (if the oscillation amplitude is large enough)
Driven Damped Harmonic Oscillator

\[ z_d(t) = A_d \cos(\omega t) . \]  
\[ m\ddot{z}(t) = -\alpha \dot{z}(t) - k z(t) - k z_d(t) . \]

Zd: piezo drive  
Ad: driving amplitude  
Z: tip position  
\( \alpha \): damping  
Q: quality factor

\[ Q = m^* \omega_0 / \alpha . \]

\[ \ddot{z}(t) + \frac{\omega_0^2}{Q} \dot{z}(t) + \omega_0^2 z(t) = A_d \omega_0^2 \cos(\omega t) . \]

Steady state solution

\[ z_1(t) = A_0 \cos(\omega t + \varphi) . \]

Transient solution (at the first 2Q cycle)

\[ z_2(t) = A_t e^{-\omega_0 t / 2Q} \sin(\omega_0 t + \varphi_t) \]

Q = 4
Transient response of a damped harmonic oscillator

Time constant in response to the transient change

\[ \tau = \frac{2Q}{\omega_0} \]
Amplitude and Phase

\[ A_0 = \frac{A_d Q \omega_0^2}{\sqrt{\omega^2 \omega_0^2 + Q^2 (\omega_0^2 - \omega^2)^2}} \]

\[ \varphi = \arctan \left( \frac{\omega \omega_0}{Q (\omega_0^2 - \omega^2)} \right). \]

Also, damping induced shift of resonance frequency

\[ \omega_0^* = \omega_0 \sqrt{1 - \frac{1}{2Q^2}}. \]

Approximation \( \omega_0^* = \omega_0 \)

\( \Rightarrow A_0 = QA_d. \)

Note, this is different from the frequency shift due to the tip-to-sample force interaction
Different operation modes of dynamic AFM

- Amplitude modulation (AM) \( \tau = 2Q/\omega_0 \)
- Frequency modulation (FM) \( \tau = 1/\omega_0 \)

Ultimate goal \( \rightarrow \) determine a complete curve of \( F_{ts} \) vs \( z \)

Internal Parameters vs. External Parameters
- Amplitude
- Frequency
- Phase
- Driving Amplitude
- Driving Frequency
AM – Tapping Mode
Hypothetical model

Note:

Attractive interaction  →  lower $\omega_o$

Repulsive interaction  →  raise $\omega_o$
Theoretical Modeling

Attractive interaction → lower $\omega_0$

Repulsive interaction → raise $\omega_0$
Experimental measurements (Si cantilever on Si wafer in Air)
Dynamic AFM operated in the self-excitation mode, where the oscillation signal is directly fed back to the excitation piezo. The detector signal is amplified with the variable gain $G$ and phase shifted by phase $\varphi$. The frequency demodulator detects the frequency shift due to tip-sample interactions, which serves as the control signal for the probe-sample distance.
When all external noise sources are eliminated, the minimum detectable force gradient

$$\left. \frac{\partial F}{\partial z} \right|_{\text{MIN}} = \sqrt{\frac{4k_B TB \cdot k}{\omega_0 Q \langle z_{osc}^2 \rangle}}.$$  

High Q, low T, large oscillation amplitude are desired. But large amplitude faces other problems.

$\text{Large oscillation amplitude}$

$$k_{\text{eff}}(z) = \frac{2}{\pi A^2} \int_{z}^{z+2A} F(x) g \left( \frac{x-z}{A} - 1 \right) \, dx$$

with $g(u) = -\frac{u}{\sqrt{1-u^2}}$.  

Sample
Small oscillation amplitude

\[ k_{ts}(z) = \left. \frac{\partial F}{\partial z} \right|_{z_0} \]

Large oscillation amplitude

\[ k_{\text{eff}}(z) = \frac{2}{\pi A^2} \int_{z}^{z + 2A} F(x) g \left( \frac{x - z}{A} - 1 \right) \, dx \]

with \( g(u) = -\frac{u}{\sqrt{1-u^2}} \).
Weight function versus oscillation amplitude

![Graph showing weight function versus oscillation amplitude](image)

**FIG. 27.** Calculation of the frequency shift \( \Delta f \): \( \Delta f \) is a convolution of a semispherical weight function with the tip-sample force gradient. The radius \( A \) of the weight function is equal to the oscillation amplitude of the cantilever. The weight function \( w \) is plotted in arbitrary units in this scheme—\( w \) has to be divided by \( \pi A^2/2 \) for normalization (see Fig. 28).
small amplitudes increase the sensitivity to short-range forces
When oscillation amplitude is larger than the force interaction range

\[ k_{\text{eff}}(z) = \frac{\sqrt{2}}{\pi} A^{3/2} \int_{z}^{\infty} \frac{F(x)}{\sqrt{x-z}} \, dx. \]

\[ \Delta f = \frac{f_0}{\sqrt{2\pi k A^{3/2}}} \int_{z}^{\infty} \frac{F(x)}{\sqrt{x-z}} \, dx. \]

\[ \Delta f = \frac{f_0}{k A^{3/2}} \gamma(z) \]

with \( \gamma(z) = \frac{1}{\sqrt{2\pi}} \int_{z}^{\infty} \frac{F(x)}{\sqrt{x-z}} \, dx. \)

\( \gamma(z) \) depends only on the F(z) curve and is independent of external parameters.
Implementation of FM-AFM:
True atomic imaging

Most recent example: Pentacene on Cu(111)
Science 325, 1110 (2009)

See also http://www.youtube.com/watch?v=jnLRI_74BZs
Unlike the tunneling current, which has a very strong distance dependence, $F_{ts}$ has long- and short-range contributions.

\[
V_{\text{vdW}} = -\frac{A_H}{6z} \quad V_{\text{Morse}} = -E_{\text{bond}} \left( 2e^{-\kappa(z-\sigma)} - e^{-2\kappa(z-\sigma)} \right)
\]

\[
F_{\text{electrostatic}} = -\frac{\pi \varepsilon_0 RU^2}{z} \quad V_{\text{Lennard-Jones}} = -E_{\text{bond}} \left( 2\frac{\sigma^6}{z^6} - \frac{\sigma^{12}}{z^{12}} \right)
\]
Key challenges for non-contact AFM

- Jump-to-Contact and Other Instabilities
- Contribution of Long-Range Forces
- Noise in the Imaging Signal
- Non-monotonic Imaging Signal
- Stable feedback is only possible on a monotonic subbranch of the force curve.
Jump to contact can be avoided if

$$kA > \max(-F_{ts}) = F_{ts}^{\max}$$

Typical $kA$ used in non-contact AFM: 100 – 1000 nN

Also, in order to maintain stable oscillations

$$\frac{k}{2}A^2 \geq \Delta E_{ts} \frac{Q}{2\pi}.$$ 

where $\Delta E_{ts}$ is the hysteresis energy loss due to the tip-sample interaction in one oscillation cycle
For a spherical tip with radius $R$ next to a flat surface the van der Waals potential is

$$V_{vdW} = -\frac{A_H R}{6z}.$$  \hspace{1cm} A_H \sim 1 \text{ eV}

Estimation of the van der Waals (vdW) interaction energy and vdW force

$R = 100 \text{ nm}, z = 0.5 \text{ nm} \rightarrow E_{vdW} = -30 \text{ eV} ; F_{vdW} = -10 \text{ nN}$

Electrostatic interaction

$$F_{electrostatic}(z) = -\frac{\pi \epsilon_0 RU^2}{d}.$$  

Estimation of the electrostatic interaction

$R = 100 \text{ nm}, d = 0.5 \text{ nm}, U = 1 \text{ V} \rightarrow F_e = -5.5 \text{ nN}$
Force Sensor

Micro-cantilever
qPlus sensor derived from tuning fork

Vertical force sensor

Lateral force sensor

FIG. 11. (Color in online edition) Micrograph of a “qPlus” sensor—a cantilever made from a quartz tuning fork. One of the prongs is fixed to a large substrate and a tip is mounted to the free prong. Because the fixed prong is attached to a heavy mass, the device is mechanically equivalent to a traditional cantilever. The dimensions of the free prong: length, 2400 μm; width, 130 μm; thickness, 214 μm.

FIG. 12. (Color in online edition) Micrograph of a “qPlus” lateral force sensor. The lateral force sensor is similar to the normal force sensor in Fig. 11. It is rotated 90° with respect to the normal force sensor and its tip is aligned parallel to the free prong.
Choice of Cantilever Materials

Frequency variation as a function of temperature for silicon [110]-oriented cantilevers and quartz tuning forks in X + 5° cut (see Momosaki, 1997).

Typical Δf sensitivity needed for atomic imaging → better than 10^{-5}
Si-tip

FIG. 13. Transmission electron micrograph of an extremely sharp silicon tip. The native oxide has been etched away with hydrofluoric acid before imaging. The 15–20-Å-thick coating of the tip is mostly due to hydrocarbons which have been polymerized by the electron beam. Interestingly, the crystal structure appears to remain bulklike up to the apex of the tip. From Marcus et al., 1990.

FIG. 15. Scanning electron micrograph of a cleaved single-crystal silicon tip attached to the free prong of a qPlus sensor. The rectangular section is the end of the free prong with a width of 130 μm and a thickness of 214 μm. The tip is pointed in the [111] direction and bounded by (111), (111), and (111) planes according to the method of Giessibl et al. (2001b). Figure courtesy of Christian Schiller taken from Schiller, 2003.
Not only the sharpness of a tip is important for atomic force microscopy, but also the coordination of the front atom.

FIG. 14. (Color in online edition) Model of atomic arrangements for bulklike terminated silicon tips, (a) pointing in a [001] direction and (b) in a [111] direction.
FIG. 16. Noise spectrum of a typical cantilever deflection detector (schematic), characterized by $1/f$ noise for low frequencies and white noise for intermediate frequencies. For very high frequencies, the deflection noise density of typical cantilever deflection sensors goes up again ("blue noise," not shown here).
TABLE I. Operating parameters of various FM-AFM experiments: *, early experiments with nearly atomic resolution, experiments with standard parameters (classic NC-AFM) on semiconductors, metals, and insulators; ***, small-amplitude experiments; ***, internal cantilever damping calculated from $\Delta E = 2\pi E/Q$. When $Q$ is not quoted in the original publication, a $Q$ value of 50,000 is used as an estimate.

<table>
<thead>
<tr>
<th>Year</th>
<th>$k$ N/m</th>
<th>$f_0$ kHz</th>
<th>$\Delta f$ Hz</th>
<th>$A$ nm</th>
<th>$\gamma$ fN/\sqrt{m}</th>
<th>$kA$ nN</th>
<th>$E$ keV</th>
<th>$\Delta E_{CL}$ eV***</th>
<th>Sample</th>
<th>Ref.</th>
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<td>Al$_2$O$_3$(0001)</td>
<td>Barth and Reichling (2001)</td>
</tr>
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<td>24.0</td>
<td>164.7</td>
<td>-8</td>
<td>12.0</td>
<td>-1.5</td>
<td>288</td>
<td>2.2</td>
<td>1.4</td>
<td>KCl$<em>{0.8}$Br$</em>{0.4}$</td>
<td>Bennewitz, Pfeiffer, et al. (2002)</td>
</tr>
<tr>
<td>2002</td>
<td>46.0</td>
<td>298.0</td>
<td>-20</td>
<td>2.8</td>
<td>-0.46</td>
<td>129</td>
<td>1.1</td>
<td>0.13</td>
<td>Si(111)</td>
<td>Eguchi and Hasegawa (2002)</td>
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<td>2000**</td>
<td>1800</td>
<td>16.86</td>
<td>-160</td>
<td>0.8</td>
<td>-387</td>
<td>1440</td>
<td>3.6</td>
<td>11</td>
<td>Si(111)</td>
<td>Giessibl et al. (2000)</td>
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<td>2001**</td>
<td>1800</td>
<td>20.53</td>
<td>85</td>
<td>0.25</td>
<td>+29.5</td>
<td>450</td>
<td>0.4</td>
<td>1</td>
<td>Si(111)</td>
<td>Giessibl, Bielefeldt, et al. (2001)</td>
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$k$ values exceeding hundreds of N/m help to reduce noise and increase stability
FIG. 22. (Color in online edition) First AFM image of the silicon 7×7 reconstruction with true atomic resolution. Parameters: $k=17$ N/m, $f_0=114$ kHz, $A=34$ nm, $\Delta f= -70$ Hz, and $Q=28,000$. Reprinted from Giessibl, 1995. Copyright (1995) American Association for the Advancement of Science.
AFM image of the silicon 7x7 reconstruction with amplitude-modulation (AM) mode. Image size 100x100 Å². A comparison between (A) an AFM image and (B) empty and (C) filled-state STM images. The gray scales in the images correspond to a height difference of 1 Å. The STM images were recorded with tip voltages of -2 and +2.2 V, respectively, and a constant current of 0.1 nA. The AFM image was low-pass filtered using a 3x3 convolution filter, while the STM images show unfiltered data. The cross sections through the four inequivalent adatoms are obtained from raw data. The 7x7 unit cell is outlined in the filled-state STM image. The faulted and unfaulted halves correspond to the left-hand and right-hand sides, respectively. From Erlandsson et al., 1997.
Five operating parameters:
• The spring constant of the cantilever $k$.
• The eigen frequency of the cantilever $f_0$.
• The quality factor value of the cantilever $Q$.
• The oscillation amplitude $A$.
• The frequency shift of the cantilever $\Delta f$.

Physical observables: $\Delta f$, $A$, $\phi$
FIG. 29. (Color in online edition) Experimental normalized frequency-shift versus distance data, acquired with a low-temperature ultrahigh-vacuum AFM with a graphite sample surface and a silicon cantilever. The average distance between the center of the tip’s front atom and the plane defined by the centers of the surface atom layer is \( d \), thus the minimal tip-sample distance is \( d - A \). Five experimental frequency-shift versus distance data sets with amplitudes from 54 to 180 Å are expressed in the normalized frequency shift \( \gamma = kA^{3/2}\Delta f/f_0 \).
Normalization of Frequency Shift measured at different oscillation amplitudes

PRB 61, 12678 (2000)
First true atomic resolution of AFM in UHV
[Si(111) 7x7 surface; Science 267, 68 (1995)]

FIG. 22. (Color in online edition) First AFM image of the silicon 7×7 reconstruction with true atomic resolution. Parameters: \(k=17\ \text{N/m},\ f_0=114\ \text{kHz},\ A=34\ \text{nm},\ \Delta f=-70\ \text{Hz},\ \text{and}\ Q=28\ 000.\ \text{Reprinted from Giessibl, 1995. Copyright (1995) American Association for the Advancement of Science.}
FIG. 35. (Color in online edition) First frequency-modulated atomic force microscopy (FM-AFM) image of an insulator (KCl) with true atomic resolution. Instrument and parameters similar to Fig. 22. From Patrin, 1995.
FIG. 37. Noncontact ultrahigh-vacuum AFM image of the cleaved InP(110) surface. The scan area was 100 Å by 100 Å. Experimental conditions: spring constant of the cantilever \( k =34 \text{ N/m} \), mechanical resonant frequency \( \nu_0 = 151 \text{ kHz} \), vibration amplitude \( A = 20 \text{ nm} \), and frequency shift \( \Delta \nu = -6 \text{ Hz} \). Atomic defects (a) and adsorbates [(b) and (c)] are visible. Reprinted from Sugawara et al., 1995. Copyright (1995) American Association for the Advancement of Science.
FIG. 36. FM-AFM image of a xenon thin film. Image size 70 × 70 Å². The maxima correspond to individual Xe atoms. Sputtered Si-tip, $f_0 = 160$ kHz, $\Delta f = -92$ Hz, $A = 9.4$ nm, $T = 22$ K, approximately 20 pm corrugation. From Allers et al., 1999b.
FIG. 38. (Color in online edition) FM-AFM image of an Al$_2$O$_3$ surface. Si-tip, $f_0 = 75$ kHz, $\Delta f = -92$ Hz, $k = 3$ N/m, $A = 76$ nm, ambient temperature. From Barth and Reichling, 2001. Reprinted with permission by Nature (London).
M.A. Lantz et al., Science 291, 2580 (2001)
(A) Frequency shift $\Delta f$ and normalized frequency shift $g$ versus distance, as measured above the positions labeled 1, 2, and 3 in Fig. 1B. The inset adjusts the scales for $\Delta f$ and distance to give a better picture of the data acquired above the two inequivalent adatoms. (B) Force-distance relation determined above the corner hole (blue symbols) and a fit to the data using a sphere-plane model for the vdW force (black line). (C) Total force (red line with symbols) and short-range force (yellow line) determined above the adatom site labeled 2 in Fig. 1. In the inset, the measured short-range force is compared with a First-principles calculation (black line with symbols).
Fig. 3. (A) Frequency shift measured above the corner hole (symbols) and extrapolated from the model fit to the data of Fig. 2B (blue line). For comparison, the data acquired above adatom site 2 from Fig. 2A are also plotted (red line). (B) Short-range force and interaction energy (inset) measured above the sites labeled 2 and 3 in Fig. 1.
K = 1800 N/m,
f_o = 17 KHz, \Delta f = -160 Hz
A = 0.8 nm
Fig. 2. Series of topographic images of Si(111)-(7×7) observed by FM-AFM. The fast scanning direction was from left to right for the left side and from right to left for the right side. All images are raw data, except for the background plane subtraction. All images were recorded with a scanning speed of four lines per second in the horizontal direction, and the image size is 256 by 256 pixels; thus, the acquisition time for one frame is 64 s. The tip bias was maintained at ±1.6 V with respect to the sample. The direction of the slow scan was reversed after an image was completed; in (A), (C), and (E), the slow scan direction was from top to bottom, and in (B), (D), and (F), the direction was from bottom to top. (A) Frequency shift $\Delta f = -130$ Hz; weak contrast. (B) $\Delta f = -160$ Hz, optimal contrast. A defect is seen in the left upper section of the image, and there are dual maxima for each adatom. (C) $\Delta f = -160$ Hz, image similar to (B). (D) Before the start of this scan, the tip was displaced to the right by a distance of 2 nm; consequently, the defect appears shifted to the left by a distance of 2 nm. After 102 scan lines were completed, the set point of the frequency shift was set to $\Delta f = -140$ Hz. From then on, each adatom showed only one maximum, the contrast decreased, and the overall noise increased. Because the lateral positions of the atoms are not shifted, the same tip atom is active in the lower and upper sections of the image. (E) $\Delta f = -140$ Hz, image comparable to lower section of (D). (F) $\Delta f = -160$ Hz. The noise in the upper 35 scan lines was very low again. However, at scan line 35, the tip has apparently collapsed. This is evident because of the increased noise and a shift in the image pattern by ~1 nm to the lower right. After this tip change, excellent imaging conditions could not be retained with this tip.
Fig. 3. Enlargement of a single adatom image

Fig. 4. (A) Schematic drawing of the geometry used for the calculation, sketching the $3sp^3$ hybrid orbitals of the tip atom (green sphere) and sample adatoms (black spheres). Simulated 2.8 Å by 2.8 Å constant height images (normalized frequency shift) of a single adatom for (B) $z = 310$ pm, (C) $z = 285$ pm, and (D) $z = 285$ pm with the tip tilted $3^\circ$ around the y axis.
FIG. 44. (Color in online edition) Lateral force microscopy data on a single adatom of Si(111) imaged with a qPlus lateral sensor: (A) Simulated constant average current topographic image; (B) experimental topographic image of a single adatom; (C) experimental data of frequency shift; (D) experimental data of dissipation energy.