Nanoscale imaging

2.674

Jeehwan Kim

Slides courtesy Profs. Sang-Gook Kim, Rohit Karnik & Mathias Kolle

Let’s look at the human eye

The human eye is a very useful organ … but it has several shortcomings

- Limited field of view, limited light gathering power, limited resolution (0.1mm)
- Has limited frequency response (visible wavelengths): Blue 435.8 nm, Green 546 nm, Red 700 nm
- Distinguishes a new image multiple times a second. No accumulation of light.
- Cannot store an image.

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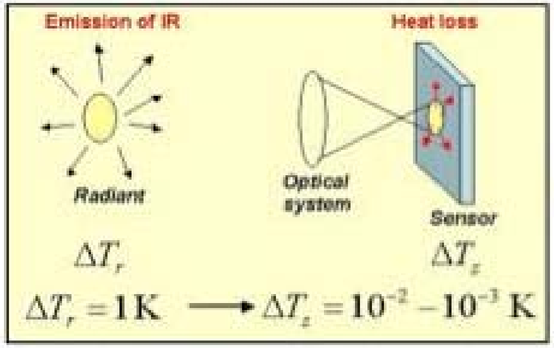
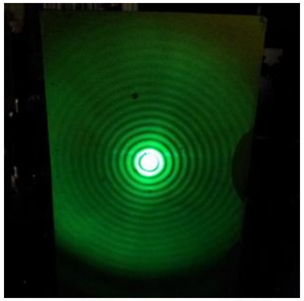
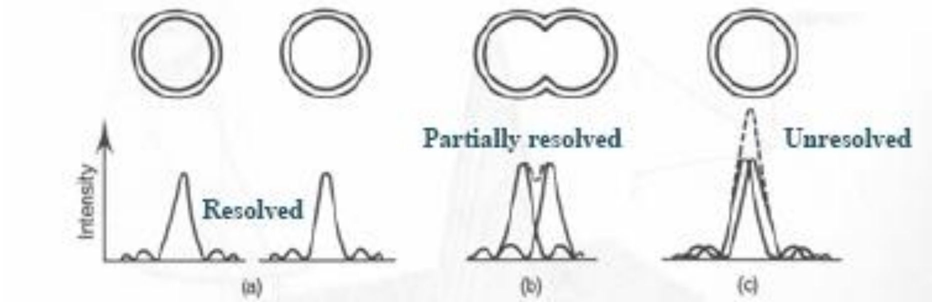


Resolutions of image

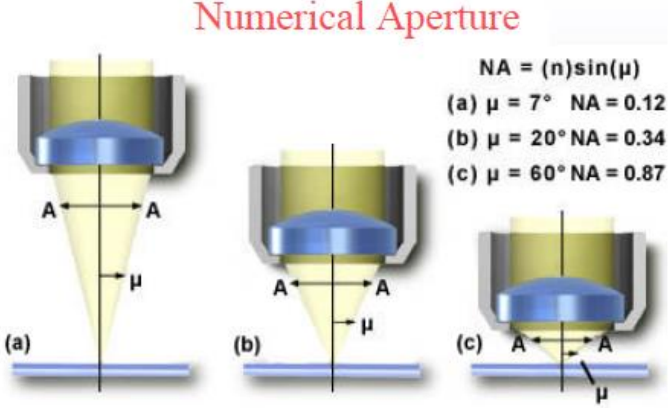


Airy rings, Diffraction

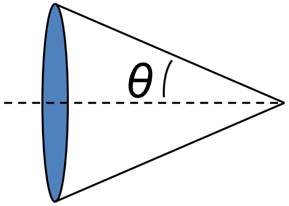
: best focused spot of light that a perfect lens with a circular aperture can make



Best resolution we can see through Optical microscope?



Rayleigh Criterion



$$\Delta x = 0.61\frac{\lambda}{n\sin\theta} \cong 0.61\frac{\lambda}{NA} \cong \frac{\lambda}{2}$$

$$NA = n\sin\theta$$

- Δx: Distance separating two objects
- λ: Wavelength of imaging light
- n: Index of refraction
- θ: Acceptance angle
- NA: Numerical aperture, n sin θ : Range of angles over which the system can accept or emit light → Lens to object distance & Refractive index

Imaging *via* light vs electron

Rayleigh Resolution $$\Delta x = 0.61\frac{\lambda}{NA}$$

Energy of photon in visible light ~ 2 eV

Wavelength of visible light ~ 600nm

NA of optical microscope ~ 1.2

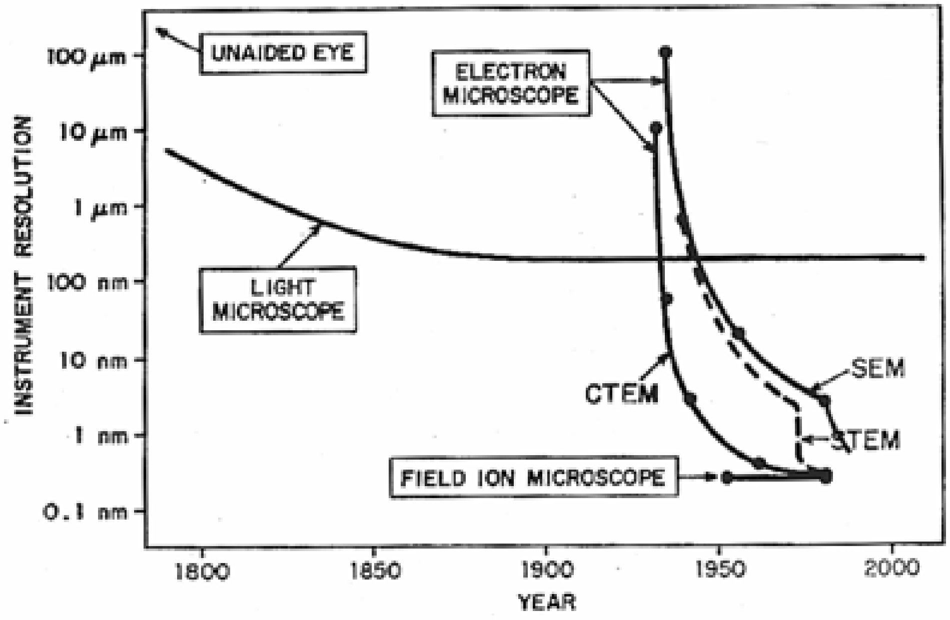
Resolution ~ 300 nm

Energy of electron beam ~ 10 keV

Wavelength of electron beam~ 0.012 nm

NA of electron microscope = 0.01

Resolution ~ few nm



de Broglie *wavelength(Wavelength of electron)*

$$\lambda = \frac{h}{\sqrt{2meV}}$$

λ: wavelength associated with the particle

h: Plank’s constant 6.63×10^{-34} Js

V: accelerating voltage

m= 9.1×10^{-31} kg;

e = 1.6×10^{-19} coulomb

The wavelength of the electron can be tuned by changing the accelerating voltage.

Electron microscopy

	SEM	TEM
Acceleration V	1-40 keV	1-1000 keV
Magnification	up to 200,000x	up to 2,000,000x
Resolution	Few nm	Few Å
Imaging method	Scattered e-beam	Transmitted e-beam

Power of the human eyes: 1×, 0.2 mm
Optical microscope: 1000×, 0.2 µm
Scanning electron microscope: 200,000×, few nm
Transmission electron microscope: 2,000,000×, few A

Scanning Electron Microscope

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Please see: http://www.hit.ac.il/staff/lapsker/sem/column_schematic.jpg.

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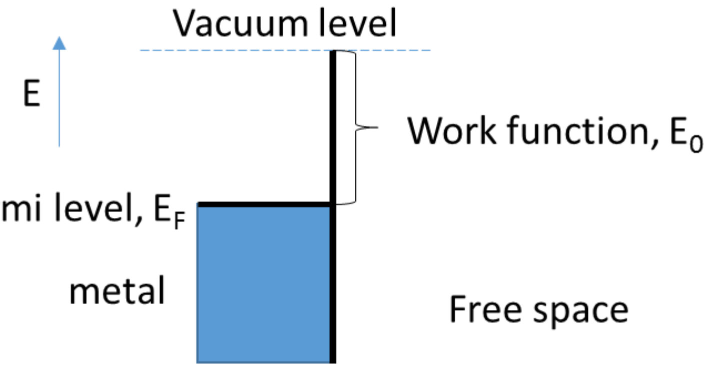
E-beam source

Electron Gun: Emits electron clouds that are accelerated and focused into collimated beams

Thermionic Emission

Tungsten (W)



Vacuum level

E

Work function, E_0

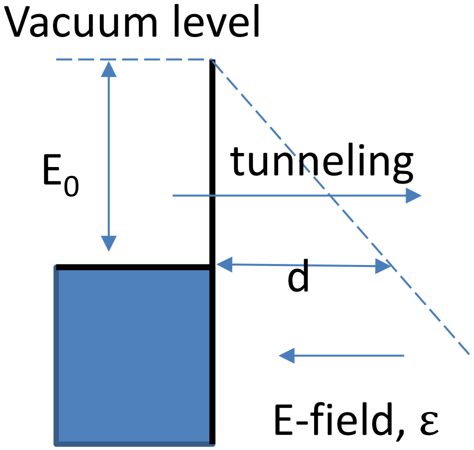
Fermi level, E_F

metal

Free space

Electrons overcome the work function of metal through thermal excitation

Field Emission



Vacuum level

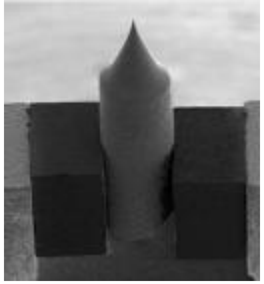
E_0

tunneling

d

E-field, ε

Etched Carbide

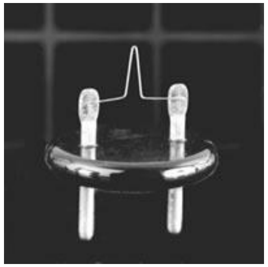


Slope is related to ε

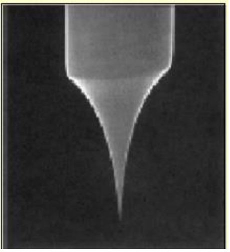
High electric field counter acts the energy needed to overcome work function.

Sharp tips are needed to enhance e-field

Electrons emission



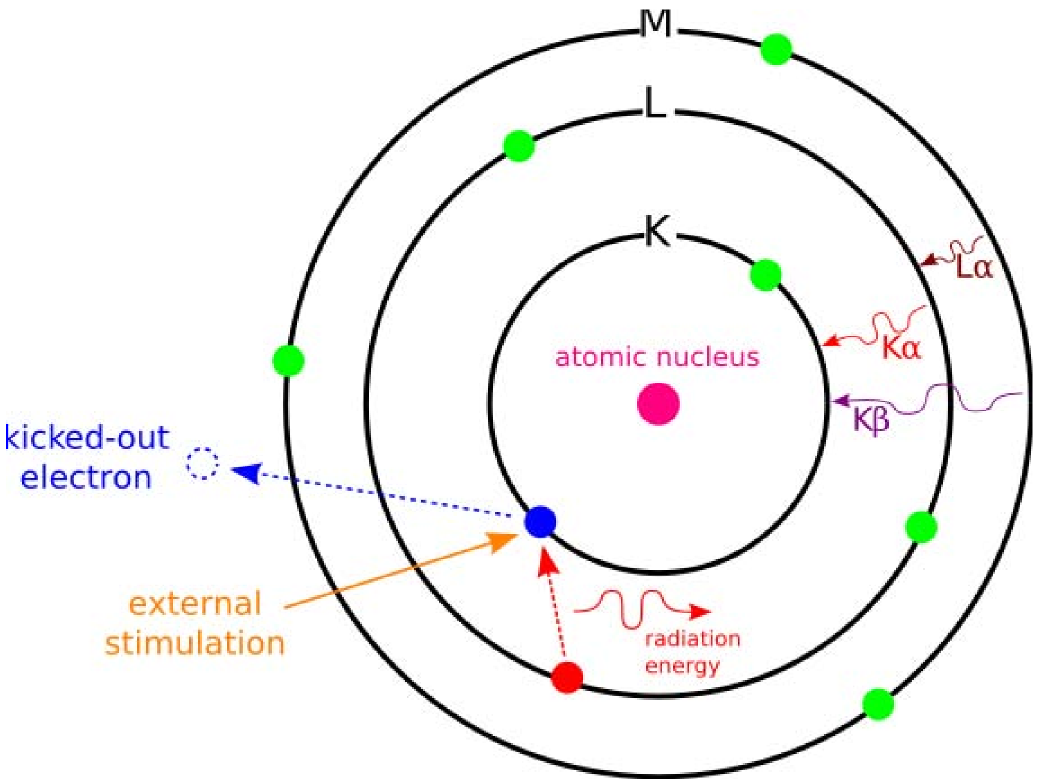
- Thermionic emission: Tungsten filament
- Field emission: 0.5-2 nm resolution, 3-6 times better than thermal emission.



	Tungsten Filament	**Field Emission**
Source size	100 μ	<100 A
brightness	1 A/cm^3 steradian	100-1000 A/cm^3 steradian
vacuum	10^{-6} Torr	10^{-9} Torr

Electron-Specimen Interactions

Inelastic scattering: Energy and momentum transfer to electrons / atoms



Electron-Specimen Interactions

Elastic scattering: Interaction of electrons with the nucleus

Electrons don't change energy, only direction of momentum

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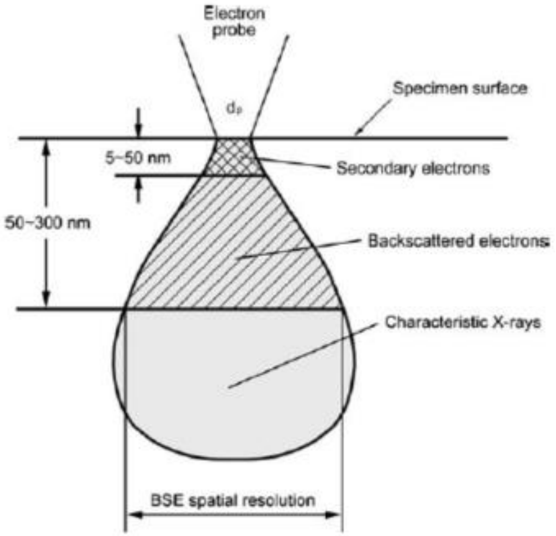
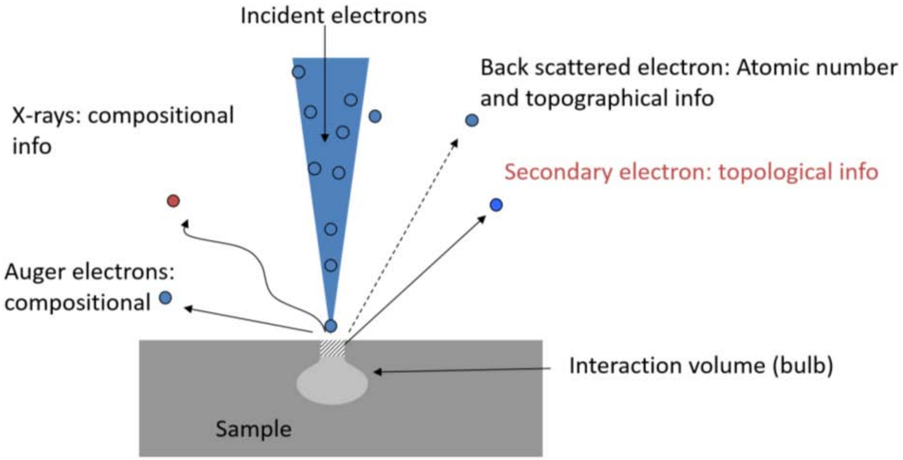
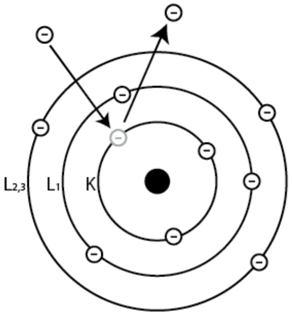
Please see: http://www.mse.iastate.edu/files/2013/06/bse_draw.gif.

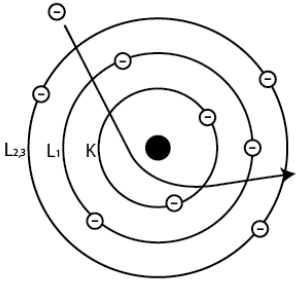
Information collected by electron-matter interaction



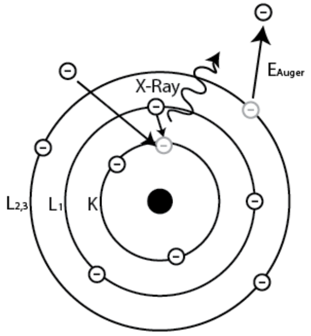




Secondary Electrons



Backscattered Electrons



Auger Electrons or
X-Ray Fluorescence

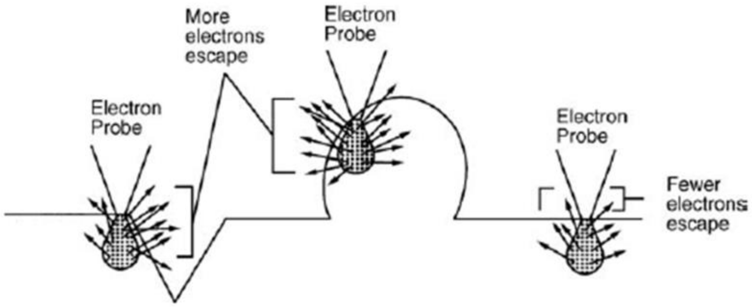
Secondary Electrons:
Electron excited by incident E-beam (Inelastic)

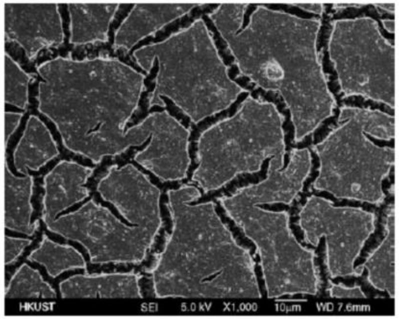
Backscattered Electrons:
Original electrons that are scattered back out (Elastic)

Xray:
Photoexcitation by incident E-beam

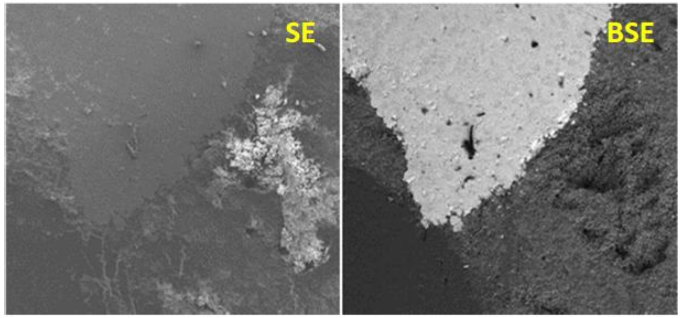
Images by collecting scattered electrons

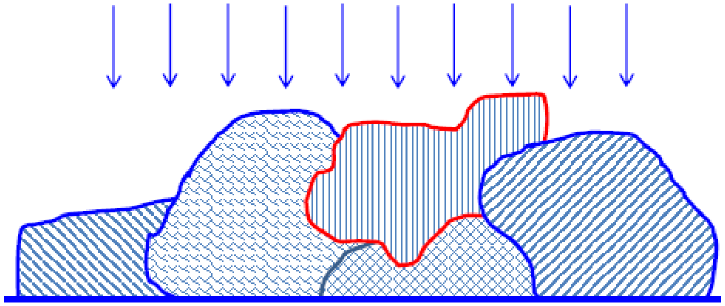
Topological information from secondary electron



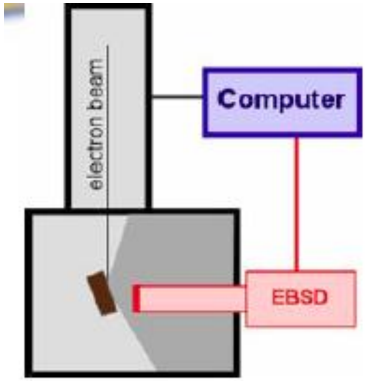


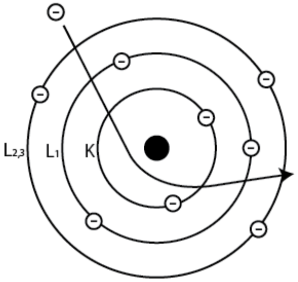
Compositional contrast from backscattered electron



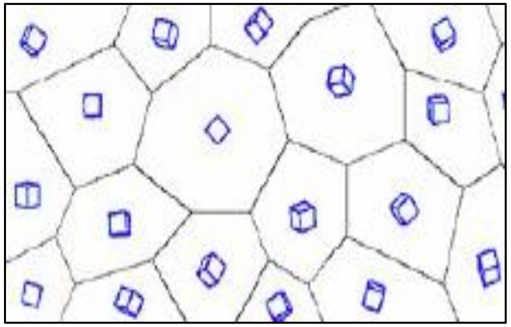


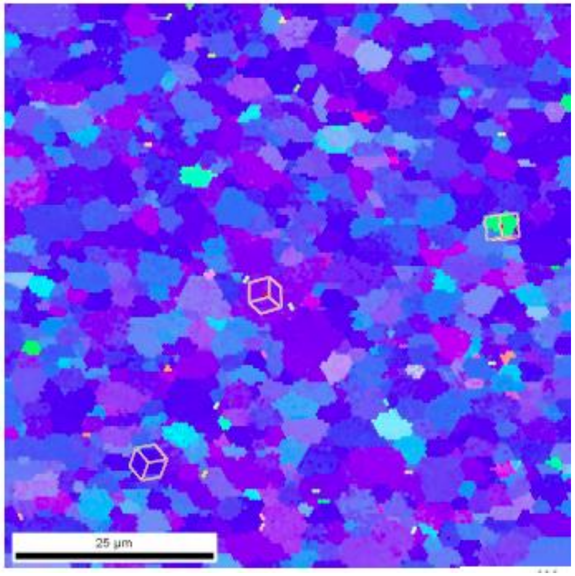
Electron backscatter diffraction (EBSD)





Backscattered Electrons





EBSD used to determine the orientation of polycrystalline

High acceleration good for high resolution?

Recall diffraction limit:

$$d_0 = \frac{0.61\lambda}{NA} \longleftarrow \lambda \sim 1.23\,nm/\sqrt{E(eV)} \longleftarrow E = qV$$

Increasing Accelerating Voltage (V) increases brightness and decreases diffraction limit, but interaction volume also increases thus resolution falls.

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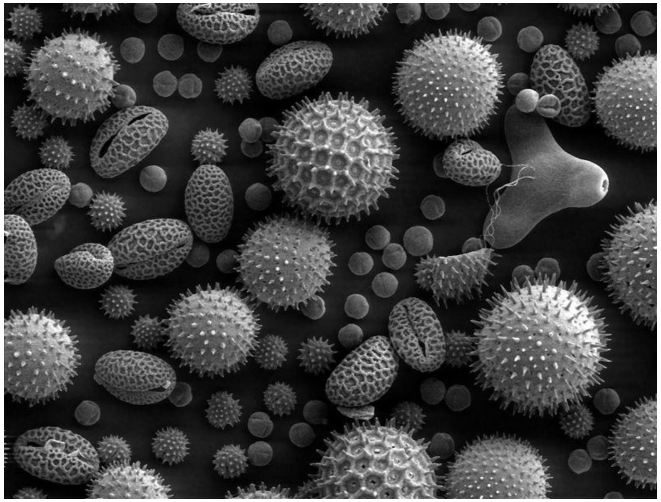
Images of things around us…



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Sample Prep

- Conductive samples
- Non-conductive sample should be coated with ultra thin metal such as gold.
- Biological samples?
  - Dehydrated
  - Fixation
  - Cryofixation
  - Environmental SEM

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Please see: https://commons.wikimedia.org/wiki/File:Gold_Spider_SEM_sample.jpg.

Optical/SEM images of lotus leaf

20 µm

Optical image

SEM image

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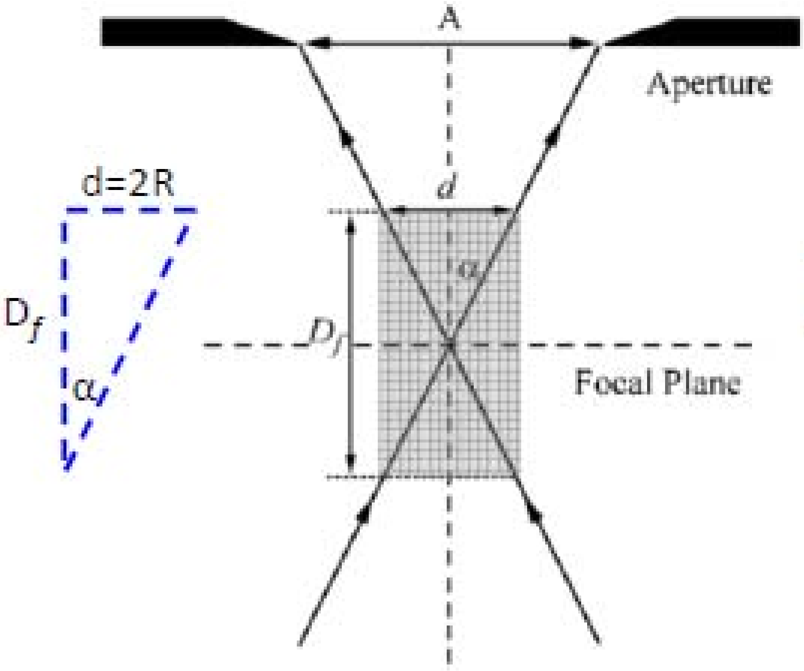
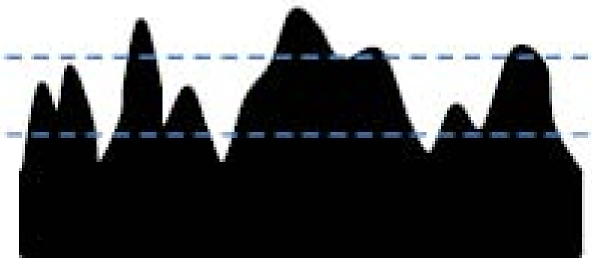
Depth of field





$$D_f = \frac{2R}{tan\alpha} = \frac{2R}{\alpha}$$ (α is small for SEM)

For 10 nm resolution SEM, α~0.01 → *D_f=200R*

For 200 nm resolution Optical, α~1.2 → *D_f=0.8R*

Electron microscope images of transistors

45 nm Intel Technology (SEM)
2007

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Please see: http://m.eet.com/media/1041489/NEWS1502_UTH_3.gif.

14 nm Intel Technology (TEM)
2016

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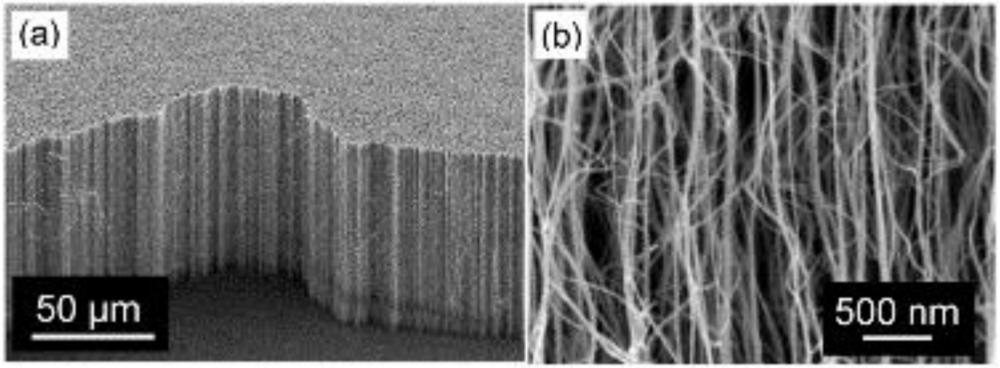
Please see: http://www.overclockers.ua/news/cpu/114557-intel-14-nm.jpg.

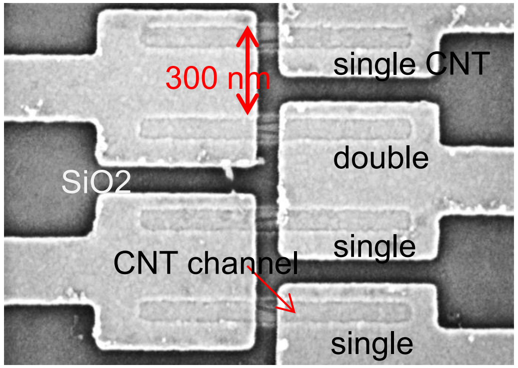
Electron microscope images of CNT

CNT Forest (SEM)



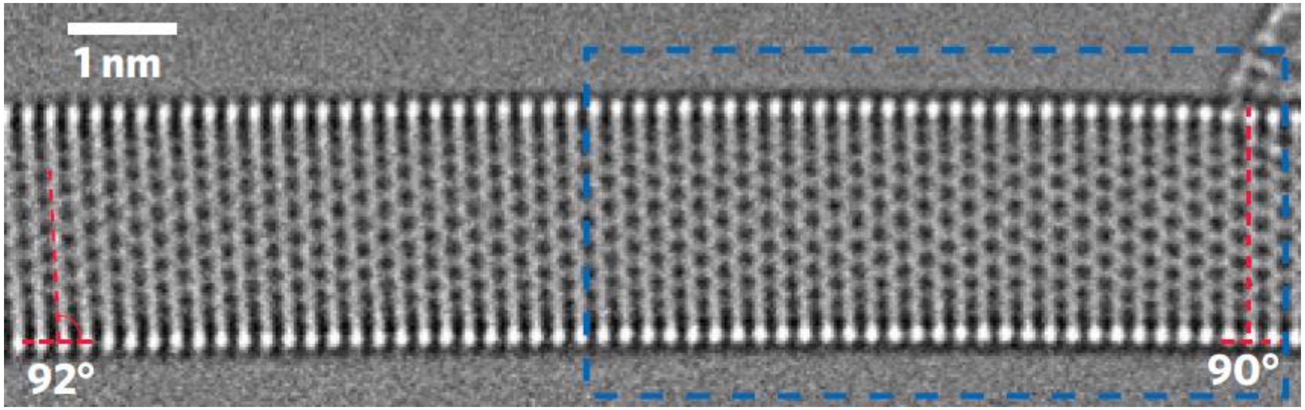
Materials 2010, 3(1), 127-149; doi:10.3390/ma3010127

CNT Transistor (SEM)



H. Park *et al.*, *Nature Nanotechnology* 7, 787(2012)

Zig Zag SWCNT (TEM)

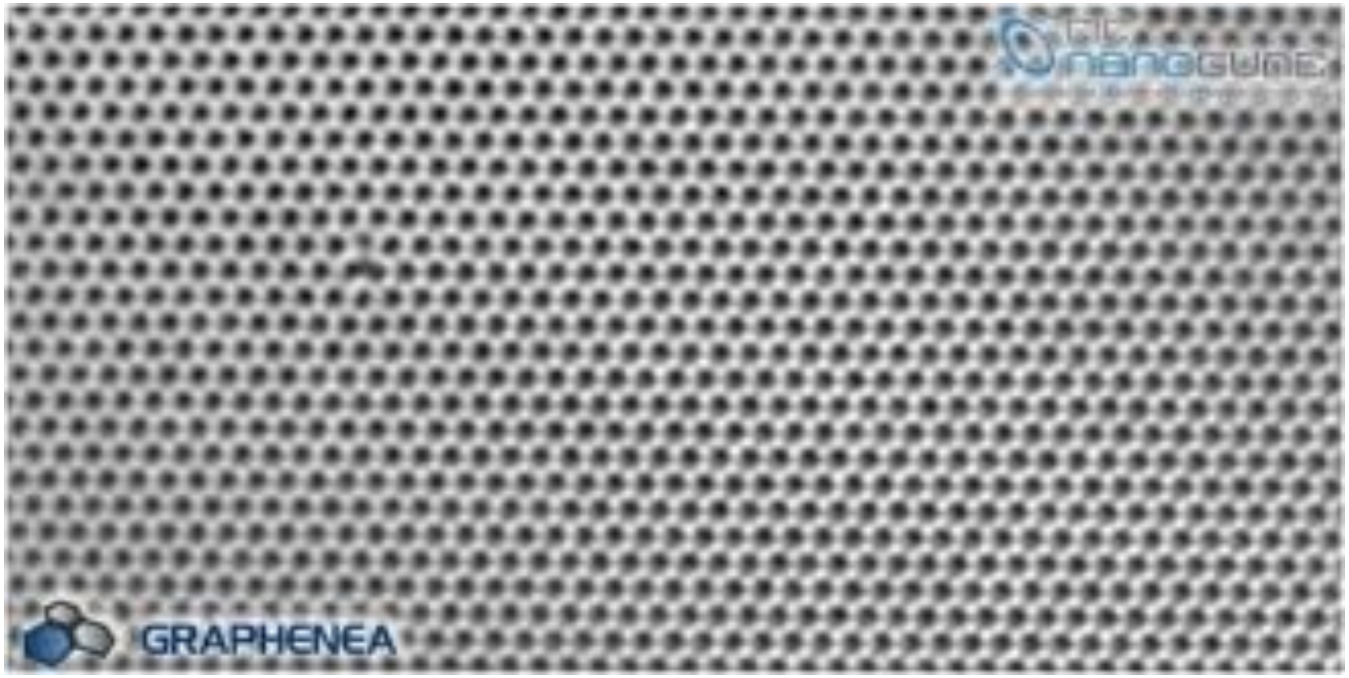


Warner et al, NATURE MATERIALS | VOL 10 | DECEMBER 2011

Electron microscope images of graphene

1 nm

TEM image

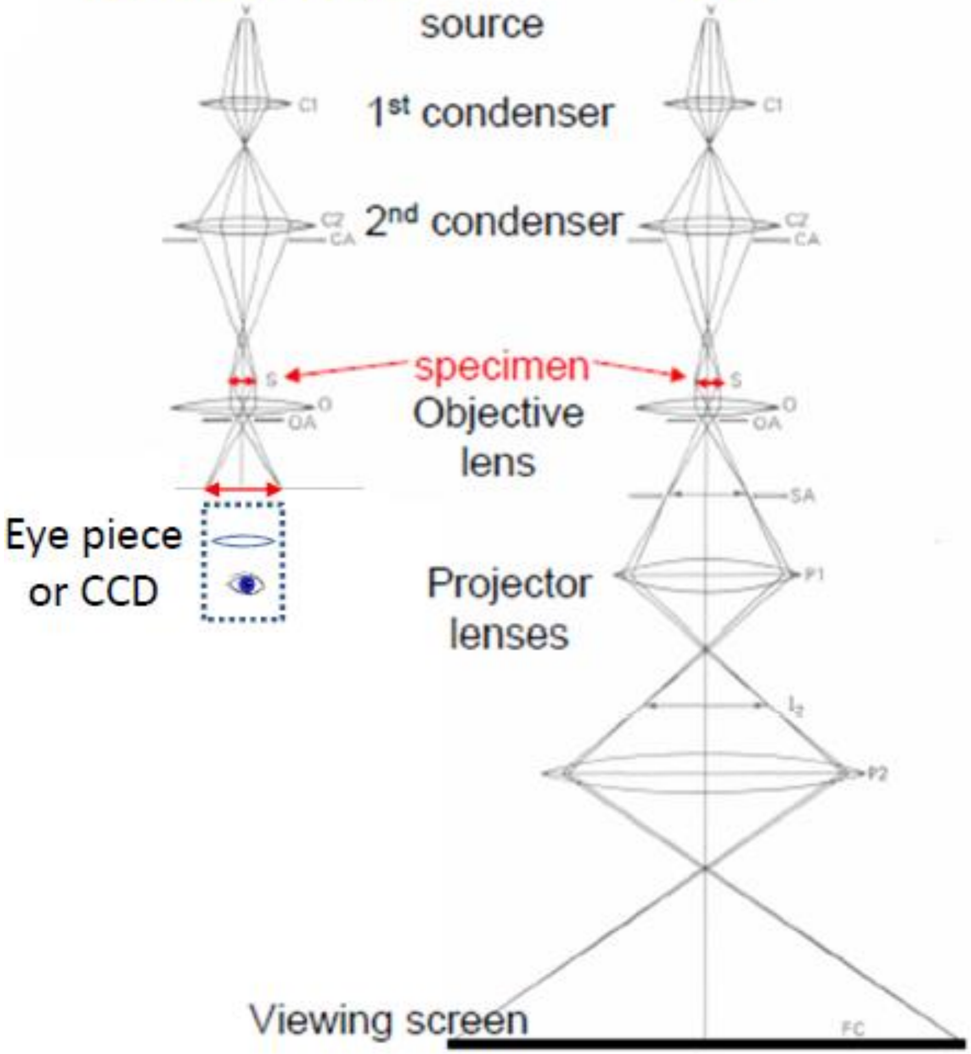


For having high resolution image: TEM, AFM, STM

	Scanning Electron Microscope	Transmission Electron Microscope	Scanning Tunneling Microscope	Atomic Force Microscope
	SEM	TEM	STM	AFM
Lateral Resolution	5 nm	0.2 nm	0.1 nm	30 nm
Vertical Resolution	None	None	0.01 nm	0.1 nm
Magnification	2D	2D	3D	3D
Sample preparation	No	Difficult (FIB, Milling)	Extremely clean surface	Clean surface
Environment	Vacuum	Vacuum	Vacuum	Vac/Air/Liquid
Cross-section image	Yes	Yes	No	No

TEM vs SEM

Light microscope **TEM**



source

1st condenser

2nd condenser

specimen

Objective lens

Eye piece or CCD

Projector lenses

Viewing screen

SEM

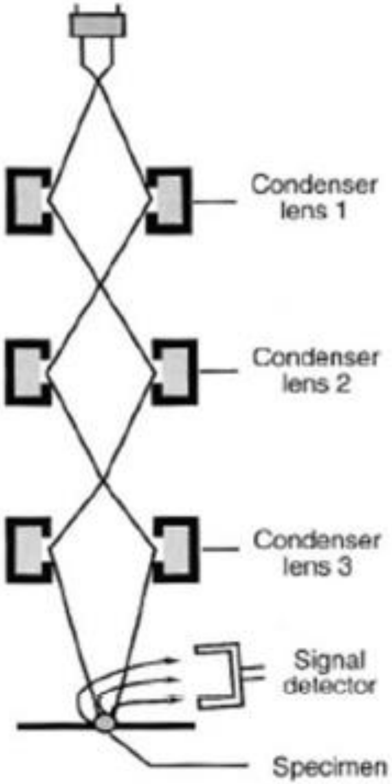
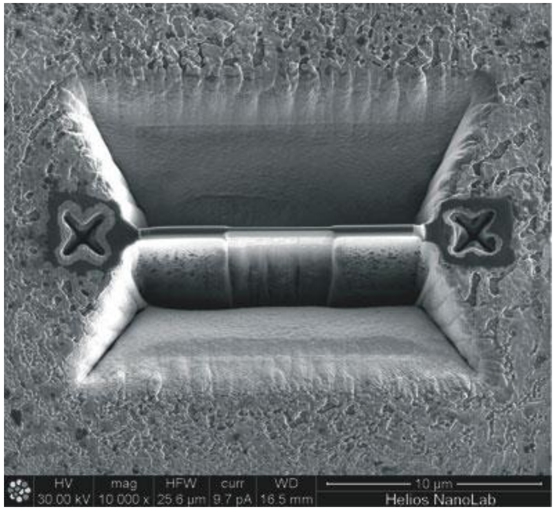
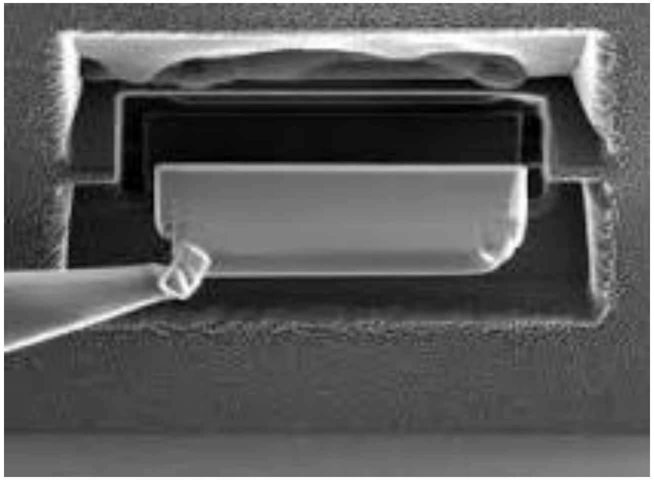


Image formed by scanning a small spot

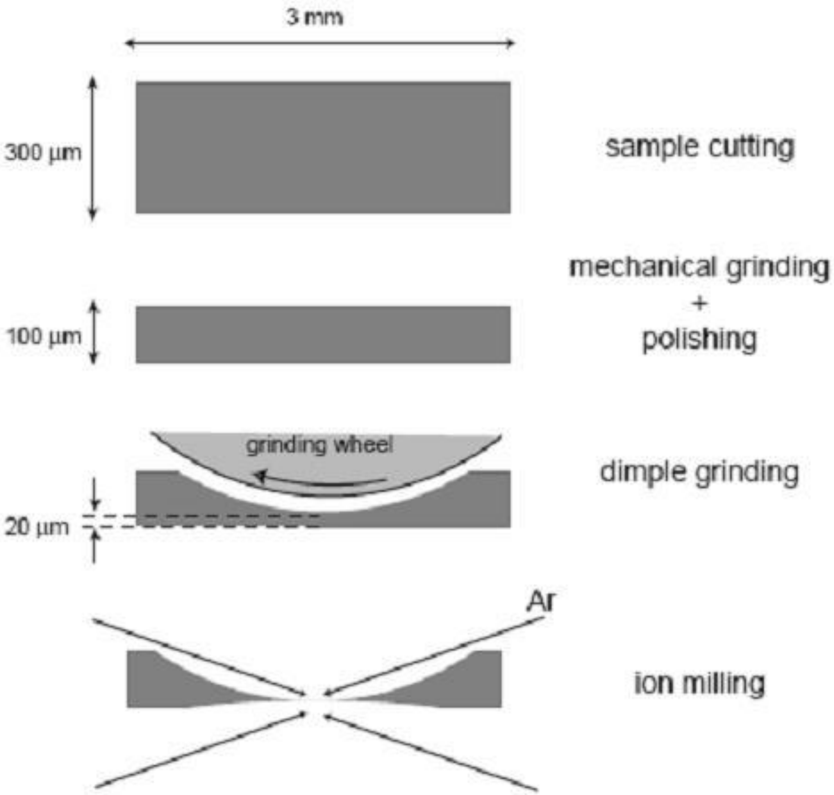
TEM sample preparation
(maximum sample thickness: ~100 nm)

Focused Ion beam (FIB) for cross-section





Ion milling for plan-view



STM

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Please see: Figure 1-2 in http://public.wsu.edu/~hipps/pdf_files/spmguide.pdf.

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Please see: http://www.fhi-berlin.mpg.de/pc/IMAGE/resmethodpict_STM.jpg.

- STMs use a sharpened, conducting tip with a bias voltage applied between the tip and the sample.
- When tip is brought within a short distance of a surface (≈ 1nm), electrons begin to tunnel and a current flows
- The tunneling current varies with distance of tip from surface and forms the basis for the imaging.
- STMs can not image non-conducting materials.
- Imaging with sub-Angstrom-precision (0.01nm) vertically and atomic resolution laterally (0.1nm).
- Scanning of samples in constant height (current is the data set) or constant current (vertical motion of scanner is the dataset) mode.

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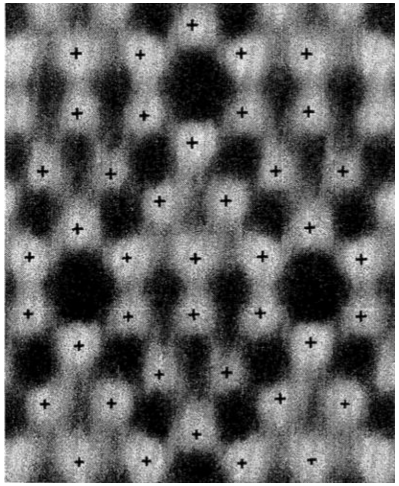
Please see: http://www.chem.ucla.edu/~kodohert/0f1b3060.



Binnig et al. PRL 50, 120-123 (1983)

Imaging of the 7 x 7 reconstruction

of the Si(111)surface

The inventors and their STM

Rohrer and Binnig with their STM

Invented: 1981

Nobel Prize: 1986

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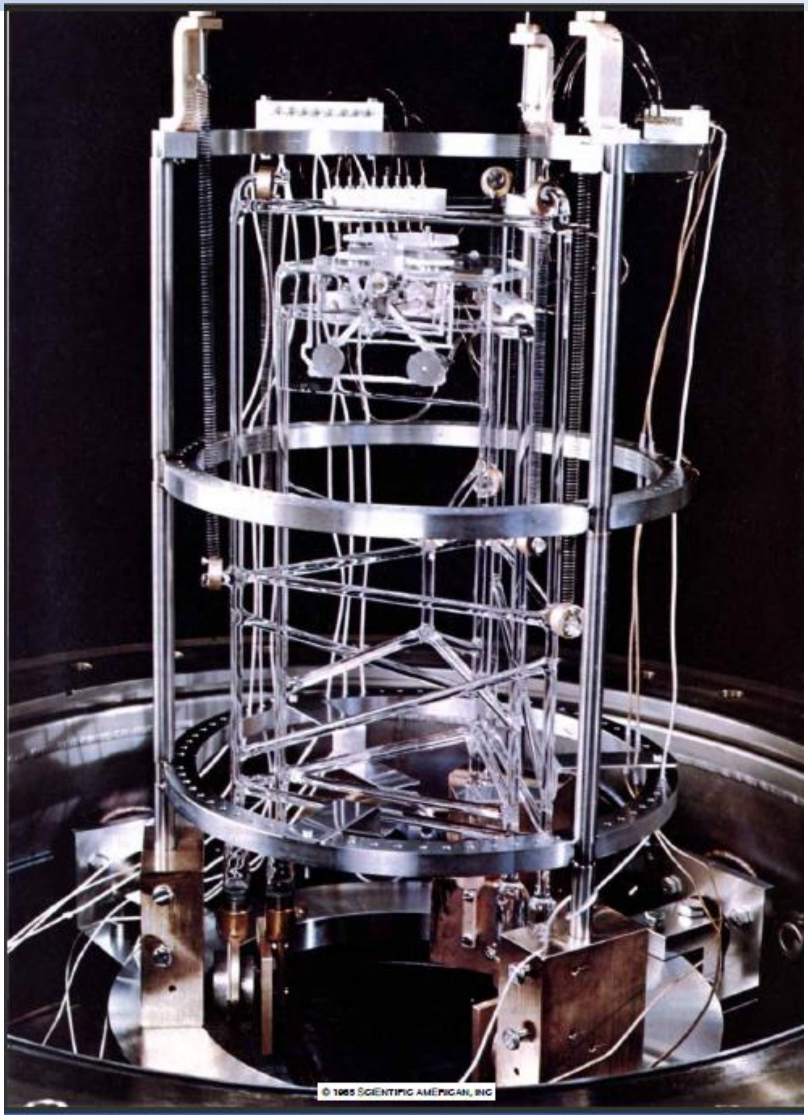
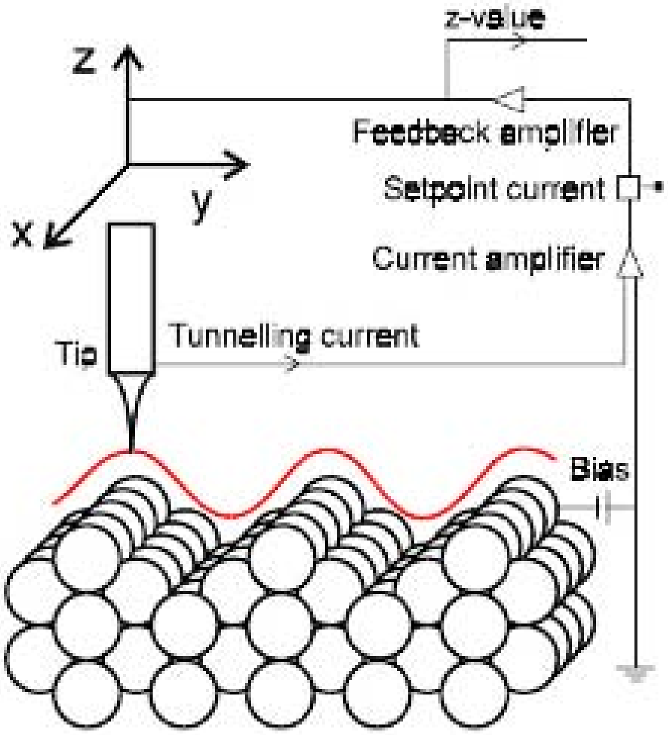
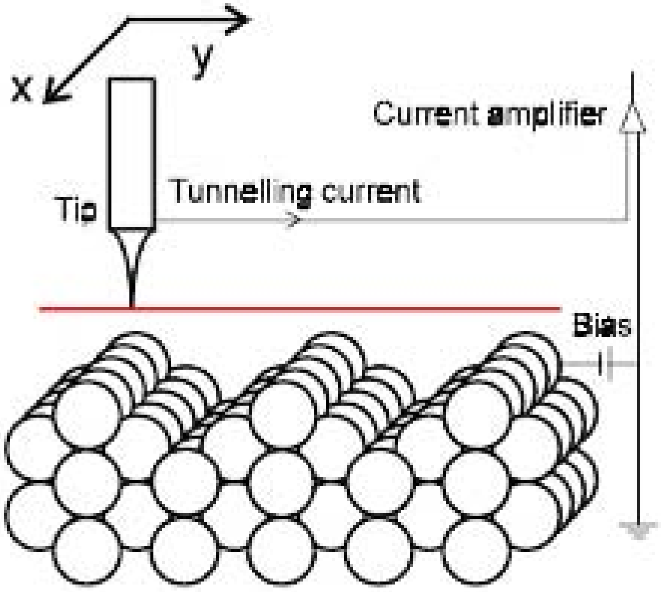


Modes of scanning



Constant Current Mode

- Typical mode of operation
- Slow: z-stage must respond!
- Can tolerate rougher surface

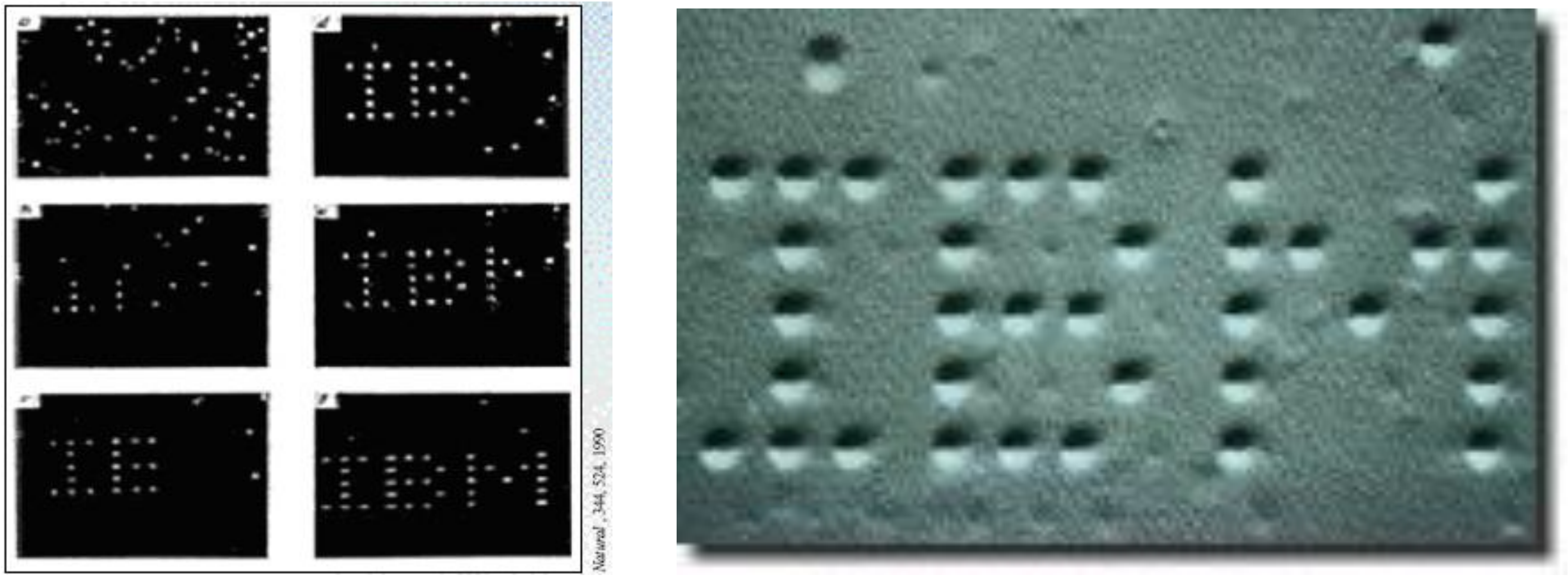


Constant Height Mode

- Fast: z-stage need not respond
- Tilt sensitive
- Minimal drift
- Cannot tolerate rough surface

STM as a fabrication tool

- Can be used to move individual atoms!
  - Higher current creates a temporary "bond" between the tip and atom
  - "Bond" atom, move tip to new position, "release" atom!

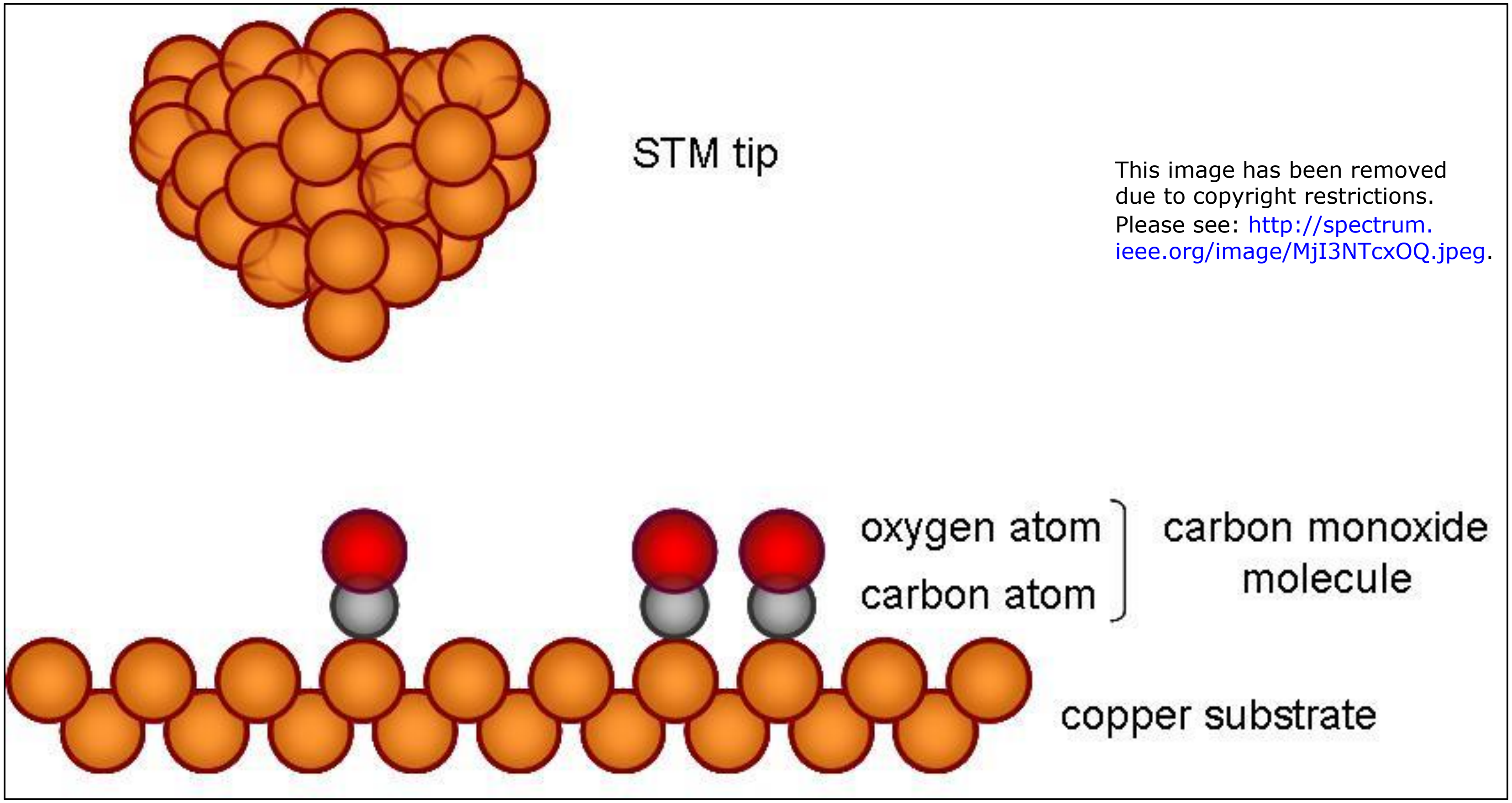


Writing "IBM" with 35 xenon atoms on nickel (IBM Almaden, 1989)

The atoms of (and in) Adam

To be precise – it's really molecules

Carbon monoxide on a copper (111) surface
T around 5 K (-268ºC)



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http://ibmresearchnews.blogspot.com/2013/05/atom-atom-molecule.html?cm_mc_uid=18927854944214476364271&cm_mc_sid_50200000=1447636427

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Please watch the video at https://www.youtube.com/watch?v=oSCX78-8-q0.

Quantum corral

Arranging a circle of iron atoms on copper.

The iron atoms affect electronic structure and create "standing waves" that can be imaged by the STM!

Courtesy of IBM

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Development of scanning probe methods

- STM was the first scanning probe microscope
- Led to many other scanning probe techniques:
  - AFM (Binning, Gerber, and Quate, PRL 56, 930-933, 1986)
  - Near-field scanning optical microscopy (1984)
  - Scanning thermal microscopy (Williams &Wickramasinghe, 1986)
  - Chemical force microscopy (Lieber, 1994)

Robert Pool "The children of the STM," Science 1990: **247**, 634-636.

- Revolution in our ability to sense and manipulate matter at the nanoscale!

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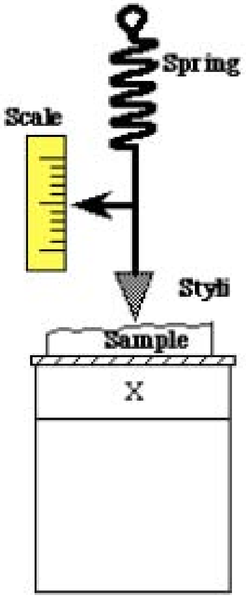
We now have feeling(s)
for surfaces!

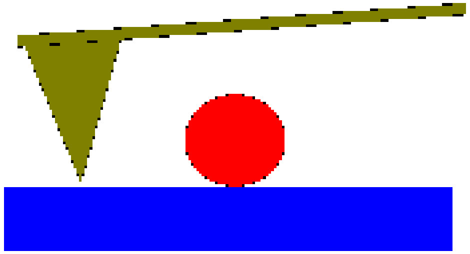
Atomic force microscopy

- AFM was invented by Binning, Quate, and Gerber in 1986 as an extension of the STM to measure forces and non-conducting samples
- AFM follows the concept of a stylus profiler

Reading Topography

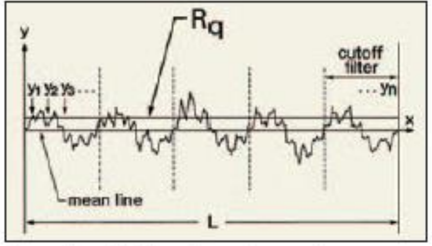


Atomic Force Microscopy



http://www.weizmann.ac.il/Chemical_Research_Support/surflab/peter/afmworks/

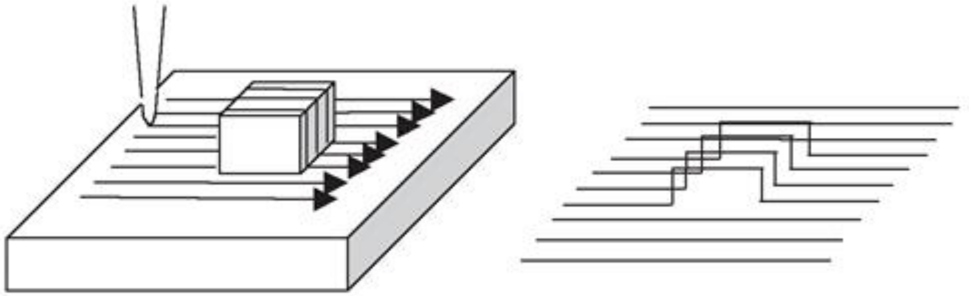
Stylus Profilometer

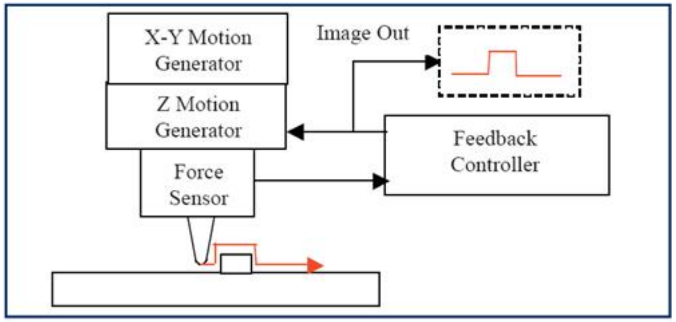


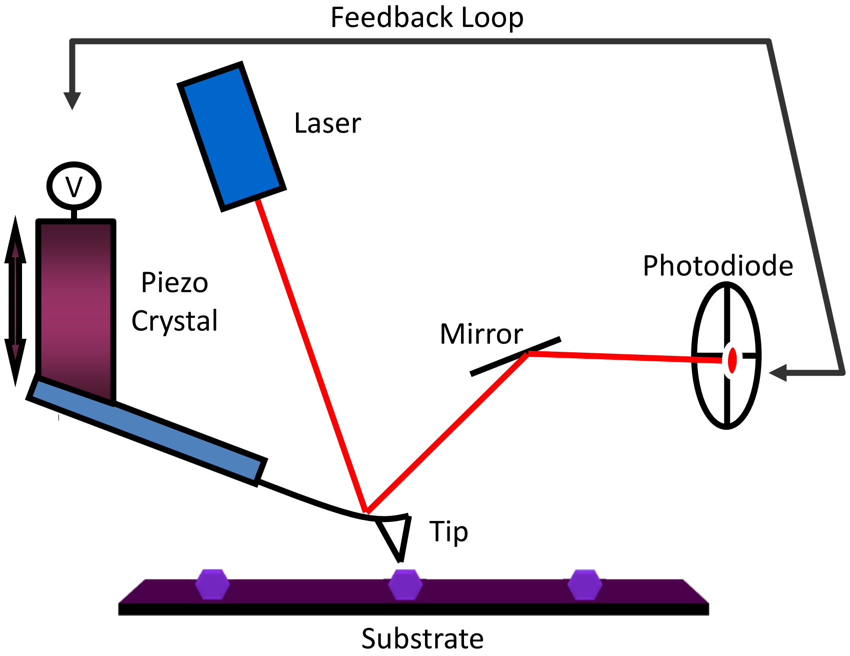
R_q is the RMS value of roughness.

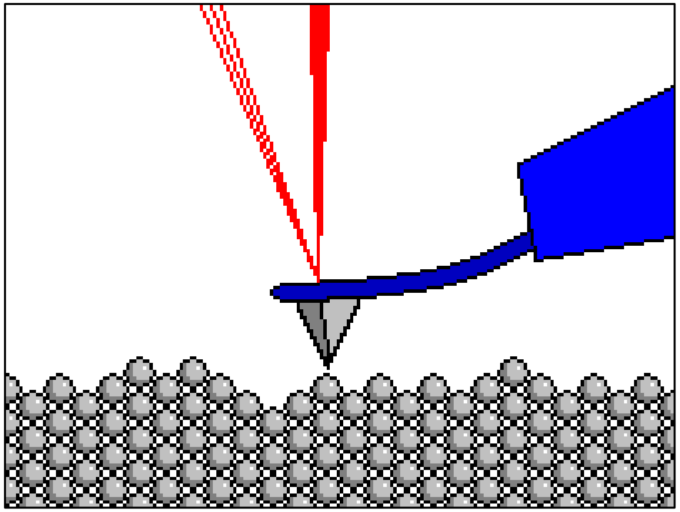
$$R_q = \sqrt{\frac{1}{L}\int_0^L y^2(x)\,dx}$$

Atomic Force Microscope

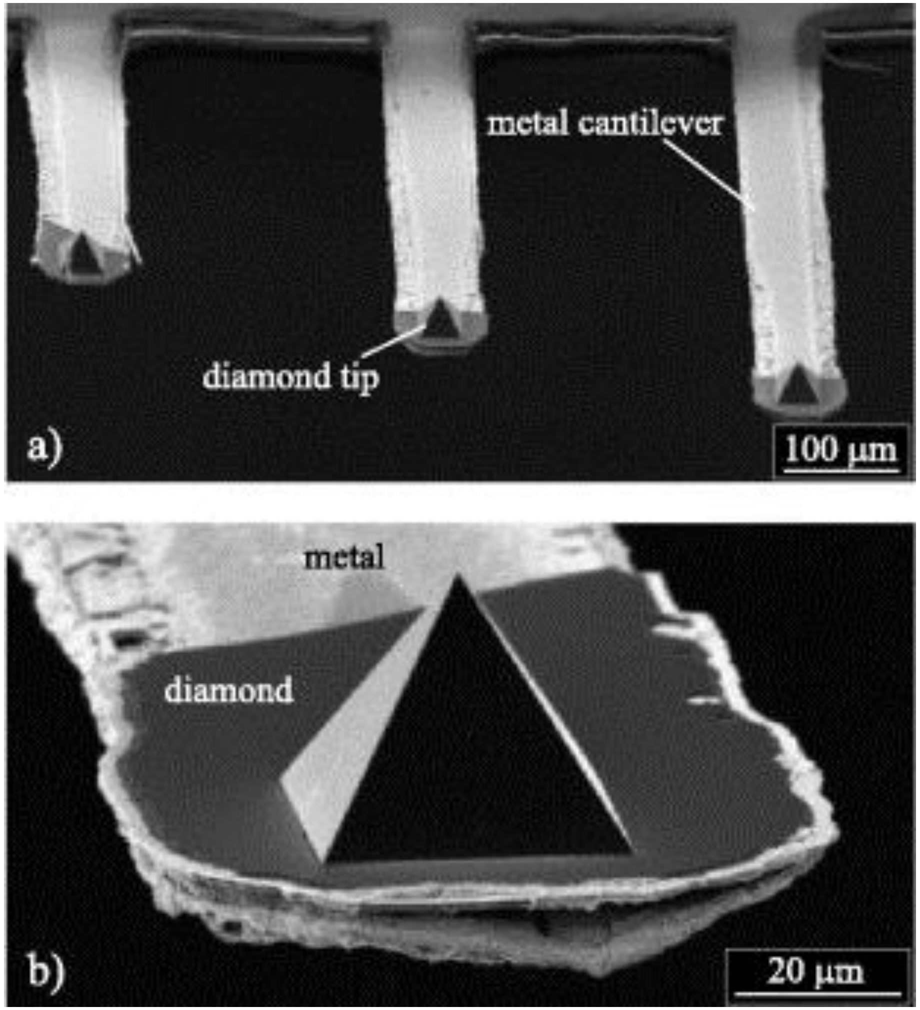








SEM image of AFM tip

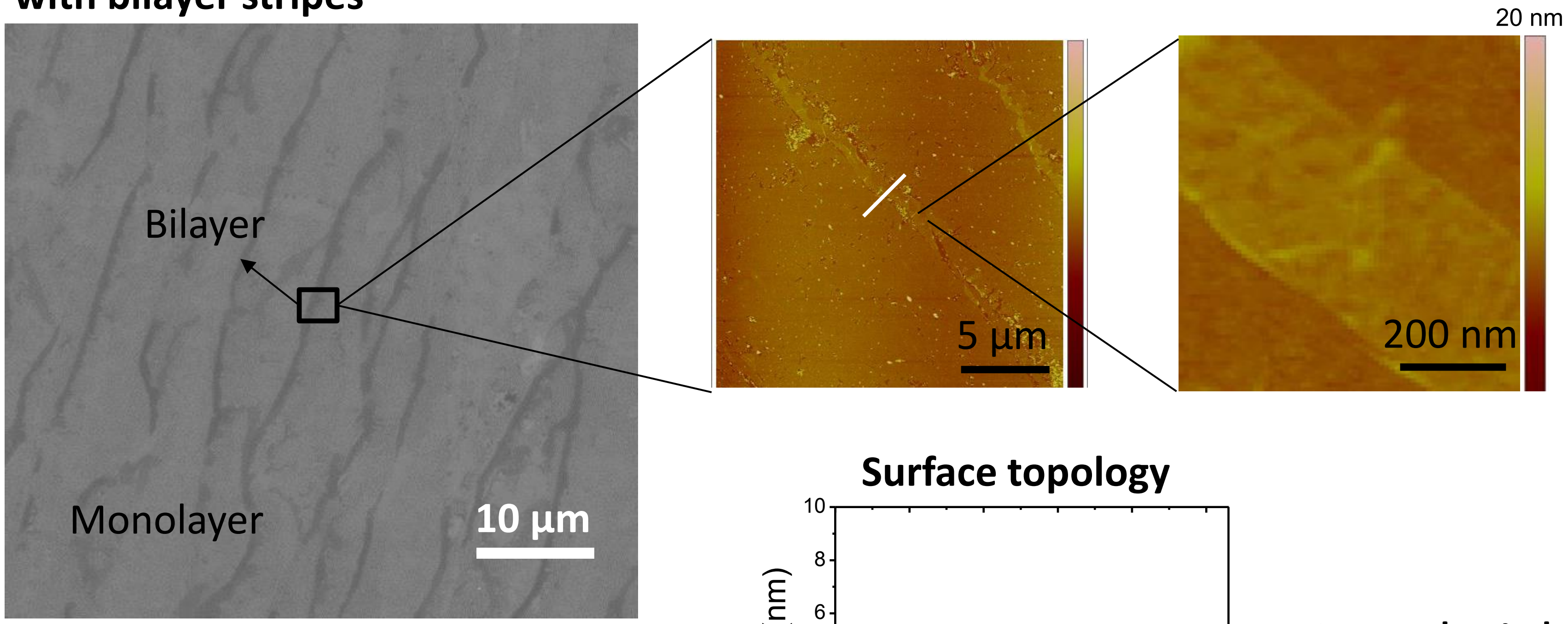


Example of using nanoscale imaging

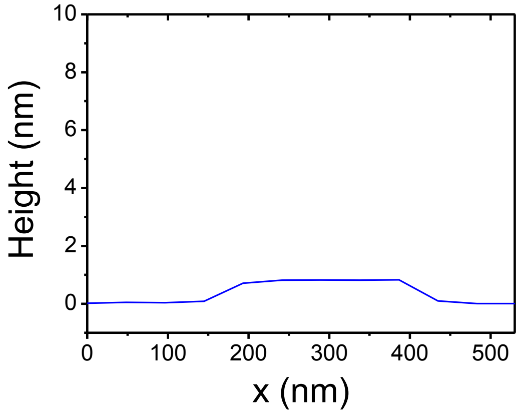
SEM/AFM image of graphene

SEM image of monolayer graphene with bilayer stripes

AFM



Surface topology

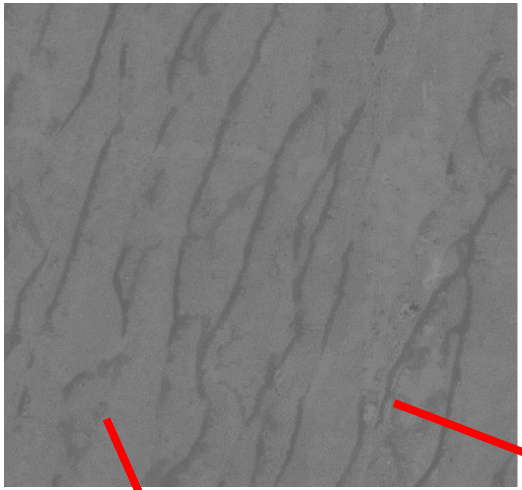


<1 nm height

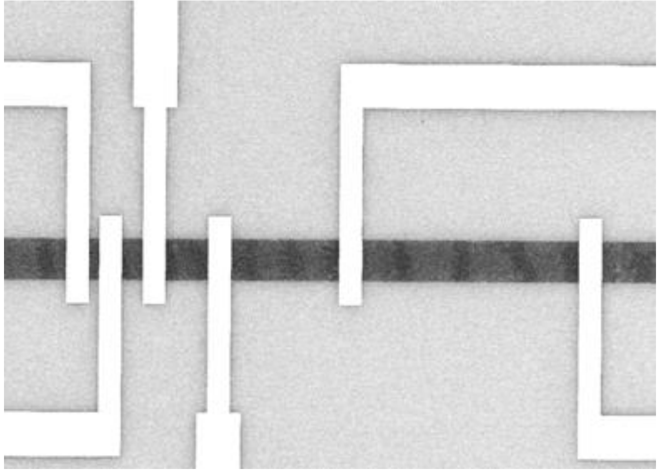
→ 1 ML

SEM/STM images of graphene

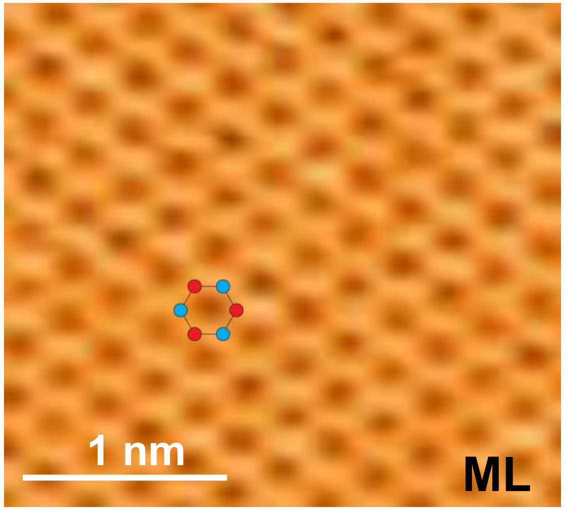
SEM image



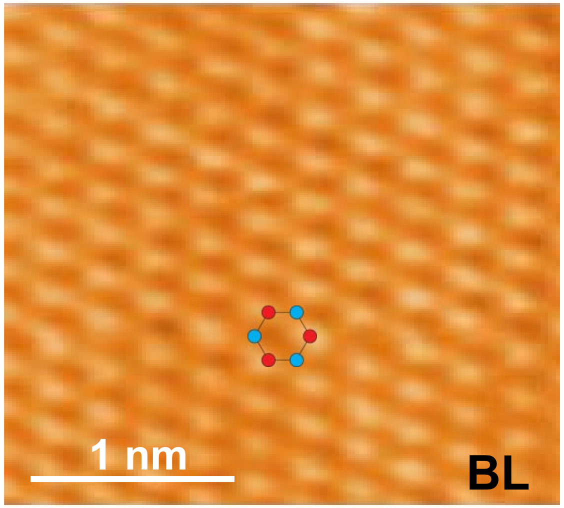
SEM image of graphene transistor



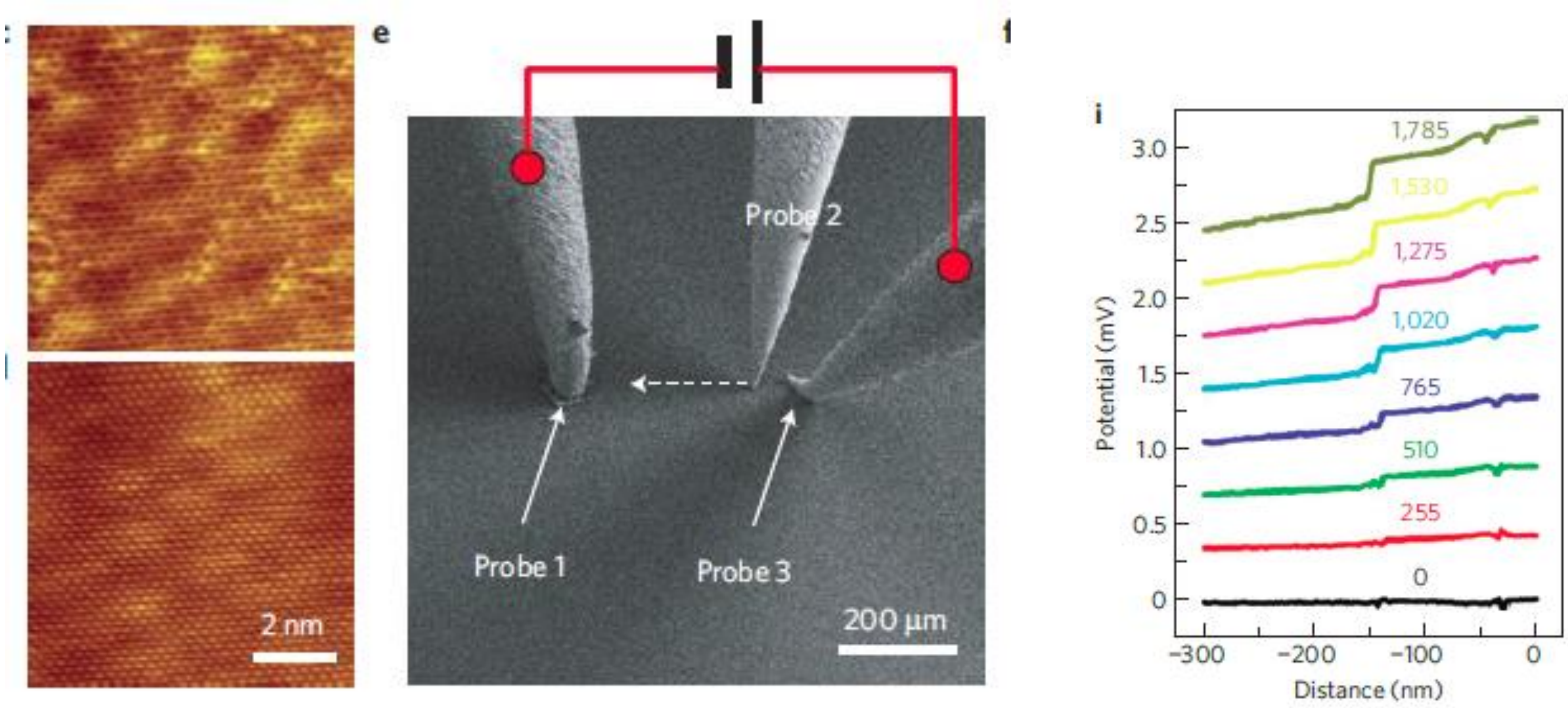
STM image monolayer graphene



STM image bilayer graphene



STM scan of graphene



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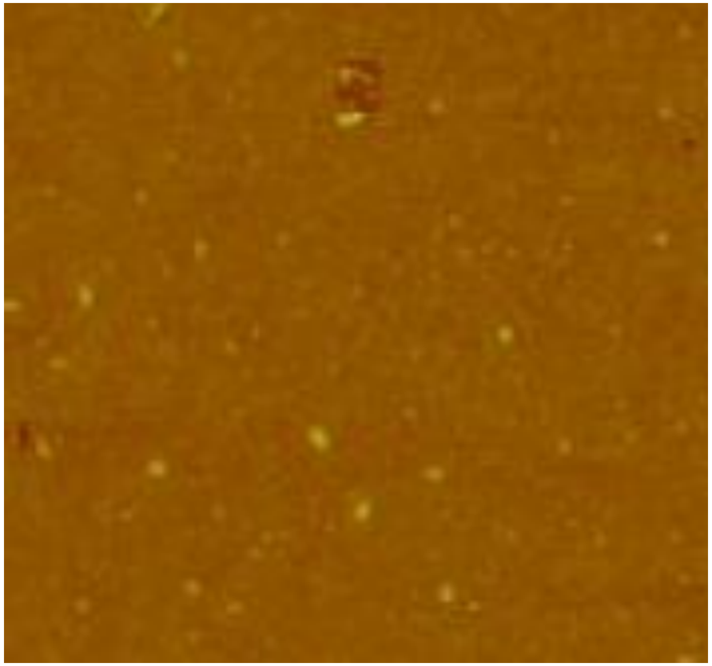
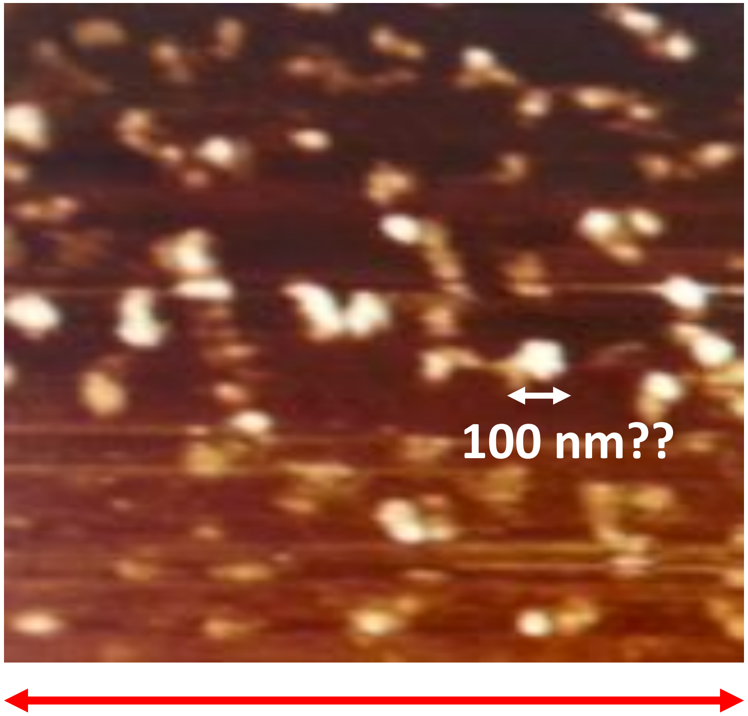
AFM image of monlayer graphene

Without process residue



With process residue



2 µm

2.674 / 2.675 Micro/Nano Engineering Laboratory
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