

# Characterization and Manipulation at Nanometer Scale

## Syllabus (2009)

Week 1 (2/17)	Overview
Week 2 (2/24)	STM: structure and working principles
Week 3 (3/03)	AFM: structure and working principles
Week 4 (3/10)	SPM: structure and working principles
Week 5 (3/17)	EM: structure and working principles (Prof. Hsu, NTHU)
Week 6 (3/24)	EM: operations and examples (Prof. Hsu, NTHU)
Week 7 (3/31)	X-ray and other microscopies (Prof. Hwu, AS)
Week 8 (4/07)	Spectroscopy: optical, electronic, vibrational
Week 9 (4/14)	Midterm Written Exam (50%)
Week10 (4/21)	Optical imaging (Prof. Kao, NYMU)
Week11 (4/28)	Lithography: optical, e-beam (Prof. Chen, AS)
Week12 (5/05)	Atom manipulation
Week13 (5/12)	Papers study ()
Week14 (5/19)	Papers study ()
Week15 (5/26)	Papers study ()
Week16 (6/02)	Papers study ()
Week17 (6/09)	Papers study ()
Week18 (6/16)	Presentation and responses (50%)



# **Nanoscale measurements and manipulation**

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[http://www.phys.sinica.edu.tw/TIGP-NANO/Course/2008\\_Spring.htm](http://www.phys.sinica.edu.tw/TIGP-NANO/Course/2008_Spring.htm)

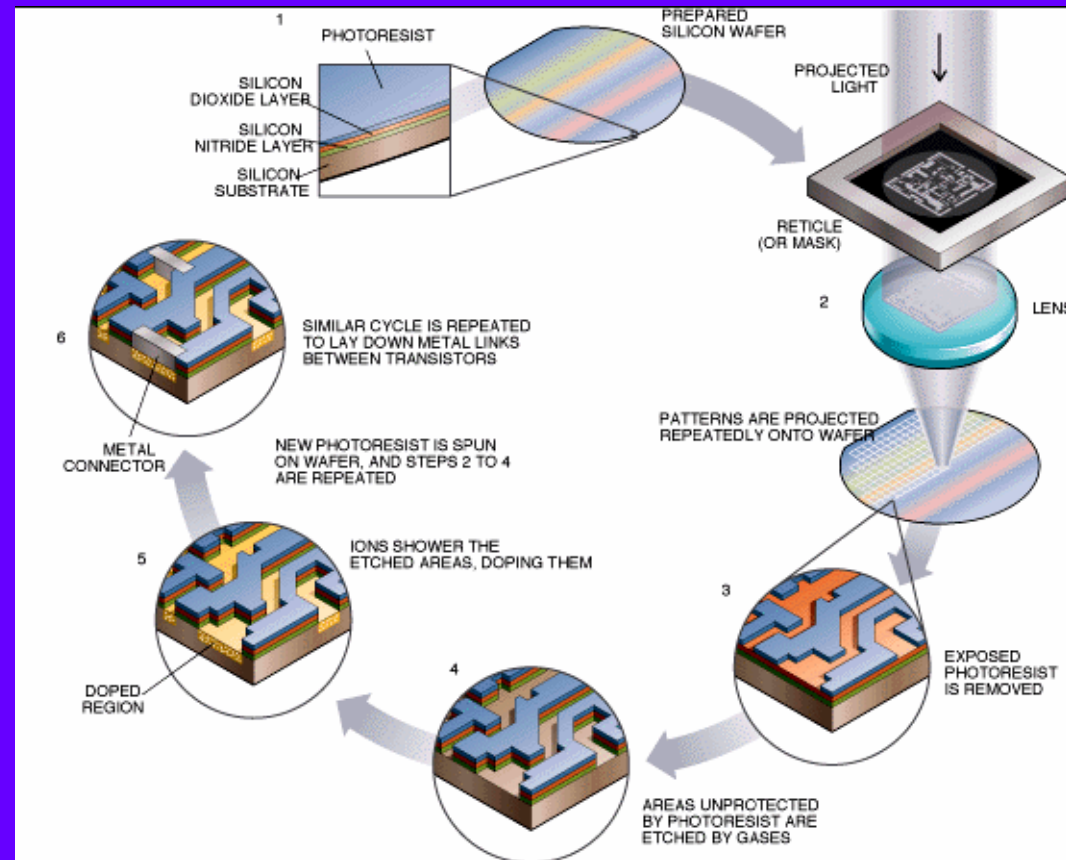
<http://www.phys.sinica.edu.tw/~nano/>



# Outline

- A. Introduction
- B. Operational Principles:
  - a. Electron b. X-ray c. Scanning Probe
- C. From measuring to sensing
- D. From measuring to manipulating
- E. From measuring to fabricating
- F. Considerations for making nanoscale tools
- G. Future development of nanoscale measurements
- H. Conclusions

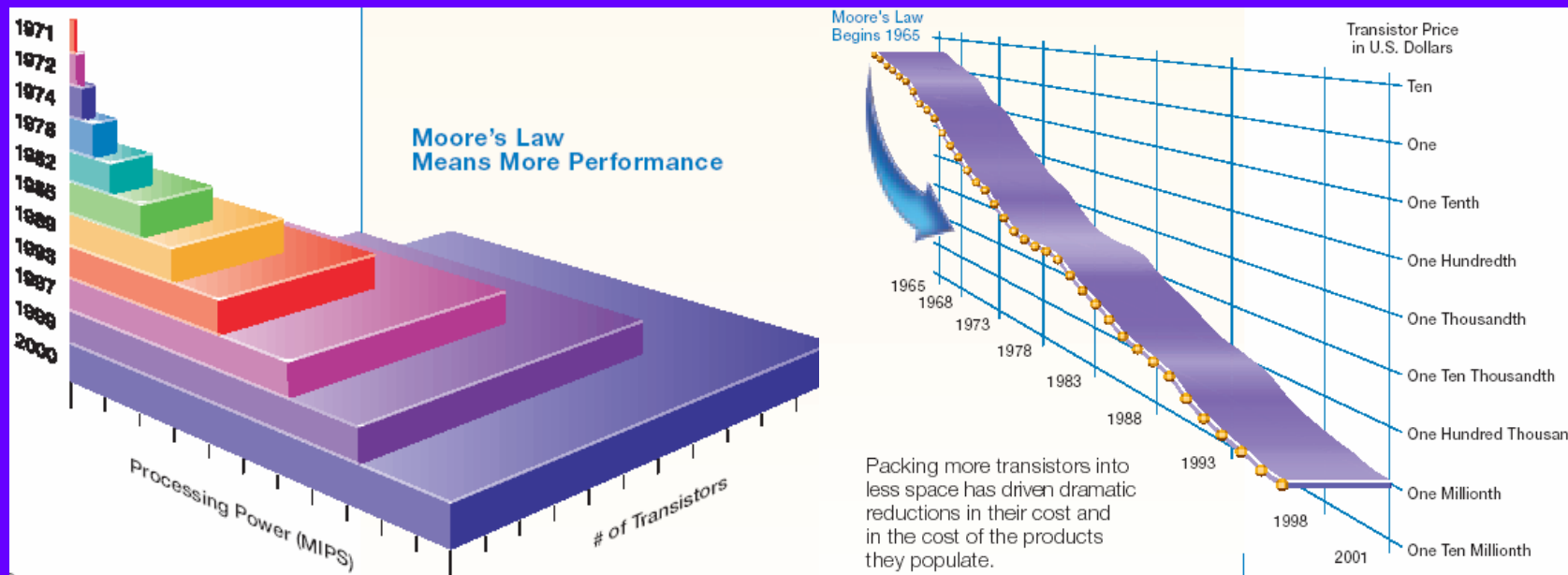
# Optical lithography and planar fabrication of VLIC



Intel Corp.



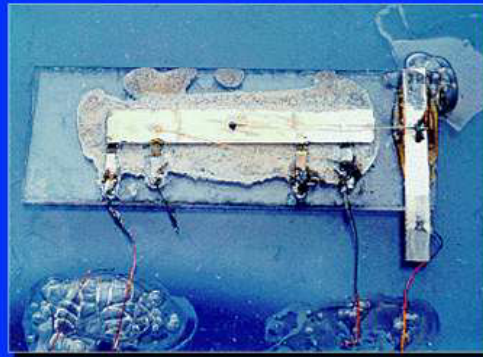
# Numbers of transistors in an IC



Intel Corp.

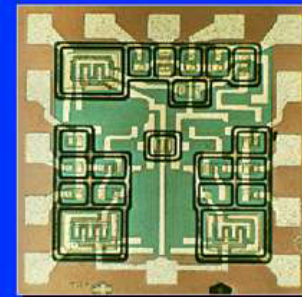
# Evolution of IC industry

## Kilby's First IC, 1958

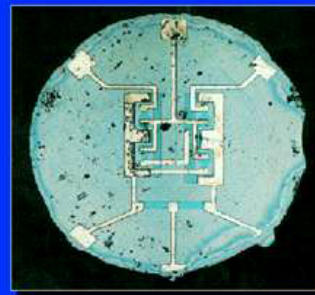


## 1965

- This DTL triple-gate device was the first radiation hardened product made with dielectric isolation and thin-film resistors.



## The First Planar Integrated Circuit, 1961



Jean Hoerni

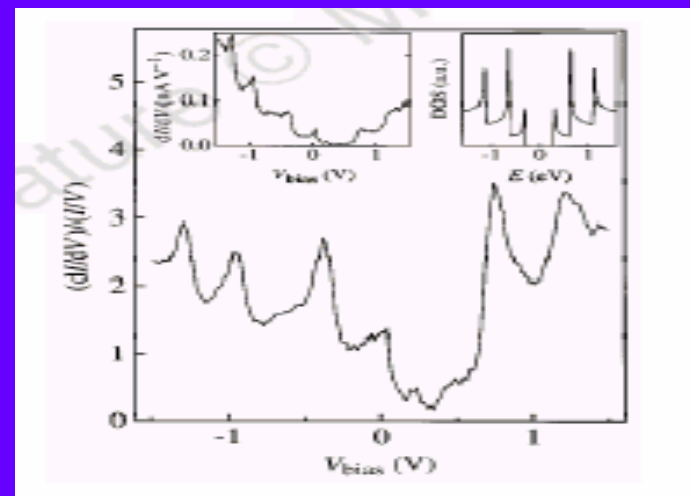
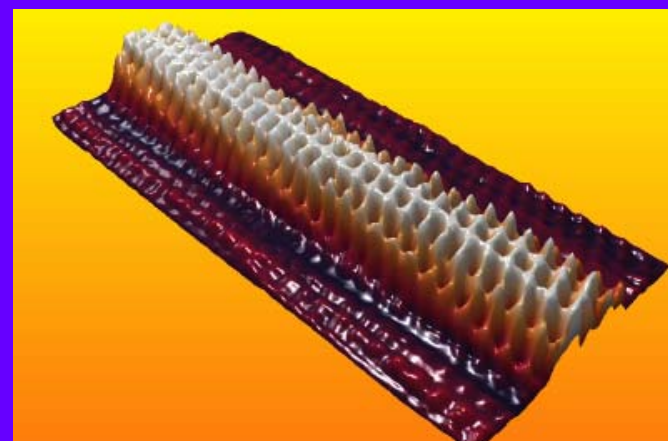
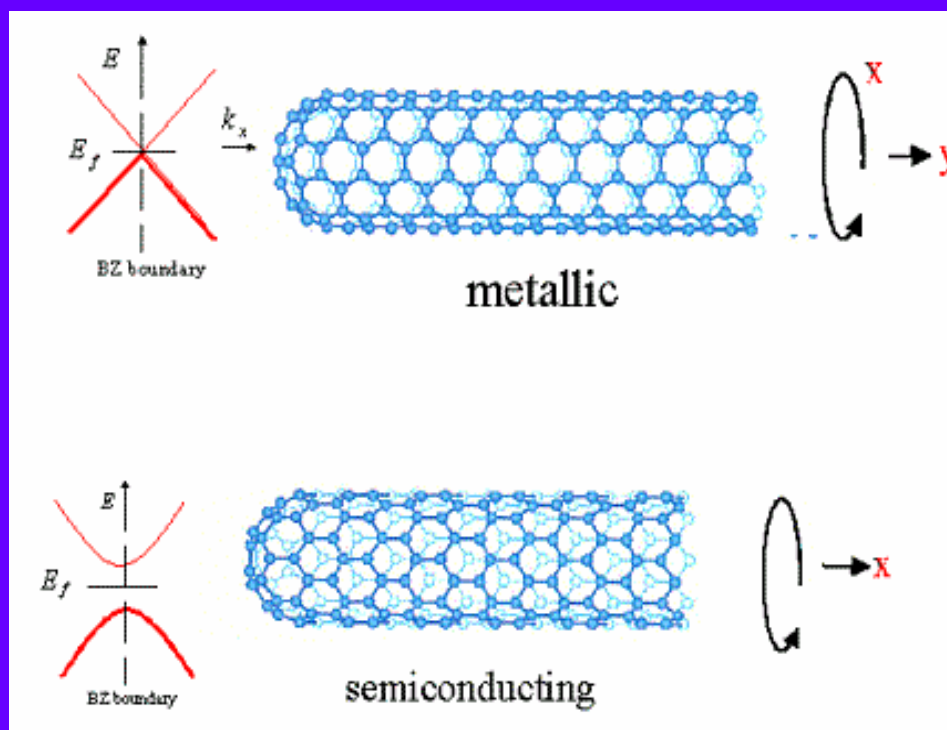
## Modern IC

### Pentium® III Processor

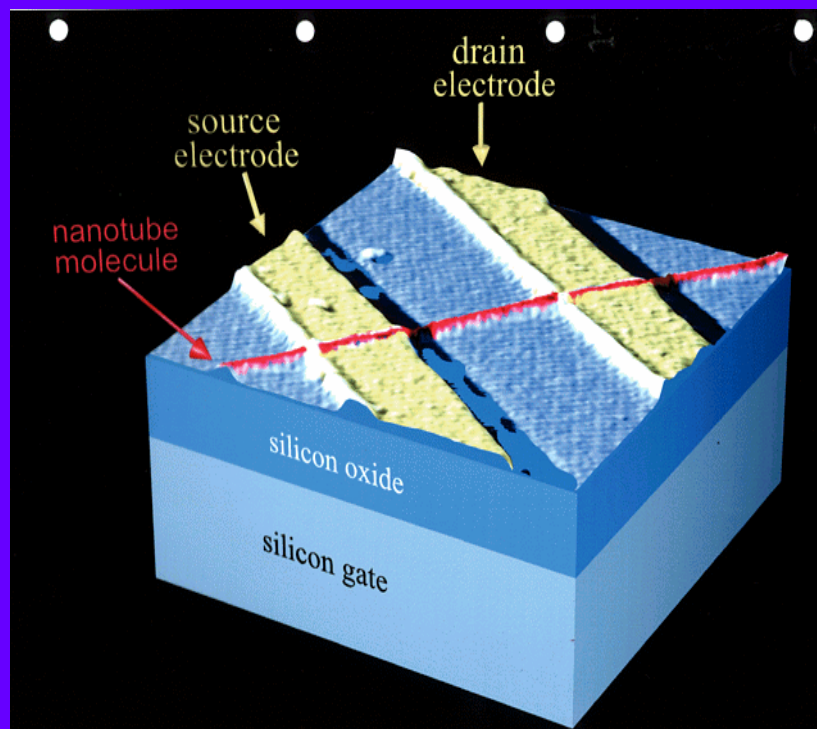
Transistors: 9,503,153  
Die Size: 408.9 mils x 484.8 mils  
Area: 127.9 mm<sup>2</sup>



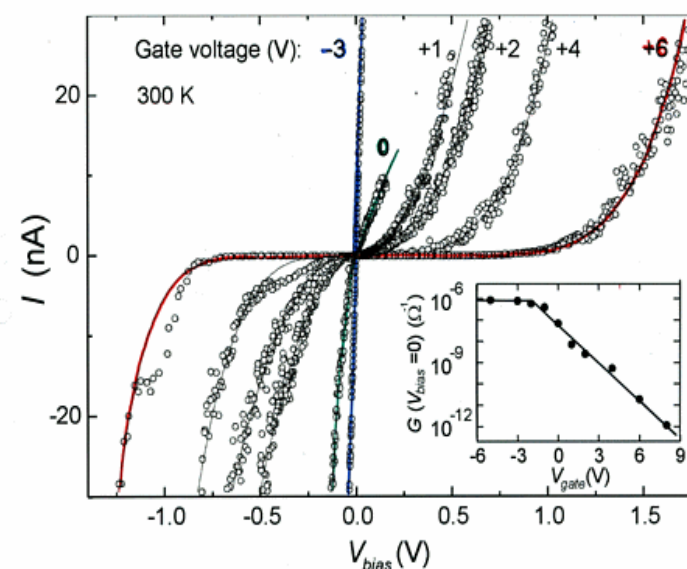
# Electronic properties of a carbon nanotube



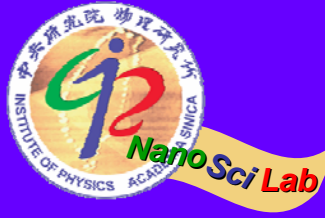
# Single-walled CNT transistor



single-molecule transistor at room temperature based on a semiconducting nanotube



S.J. Tans *et al.*, *Nature* 393, 49 (1998).



# Definitions of nanoscience and nanotechnologies

***Nanoscience is the study of phenomena and manipulation of materials at atomic, molecular and macromolecular scales, where properties differ significantly from those at a larger scale.***

***Nanotechnologies are the design, characterization, production and application of structures, devices and systems by controlling shape and size at nanometer scale.***



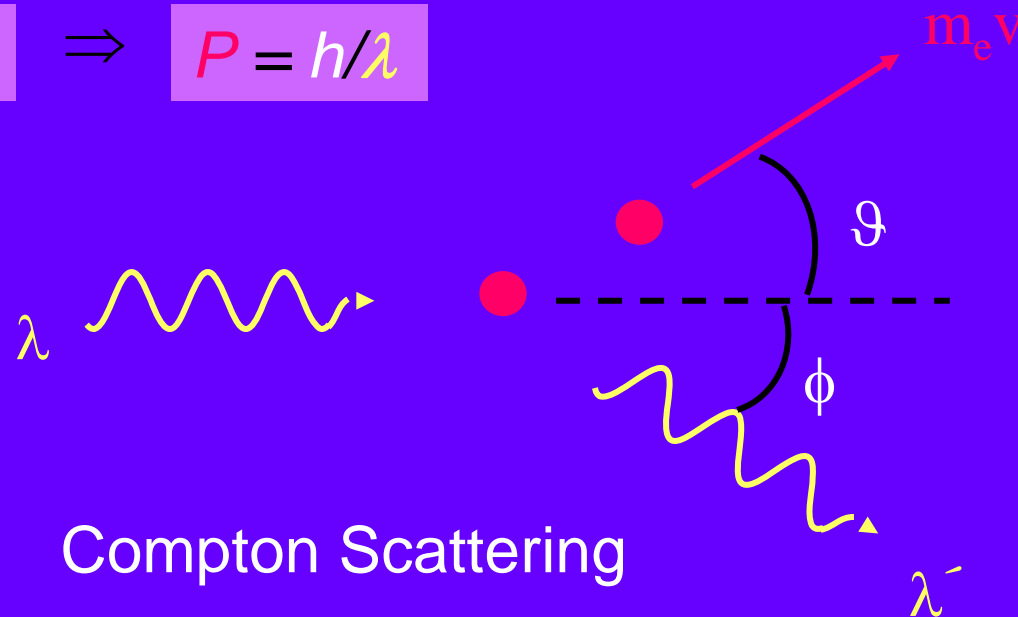
# Photon as a particle

Einstein's proposal:

$$E = h\nu$$

$\Rightarrow$

$$P = h/\lambda$$



Compton Scattering



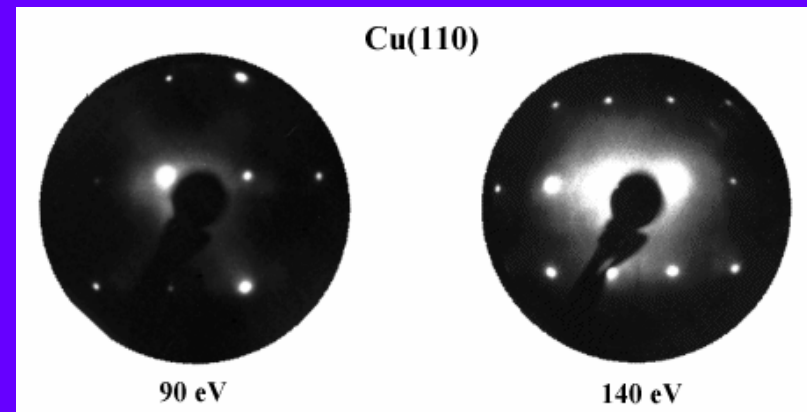
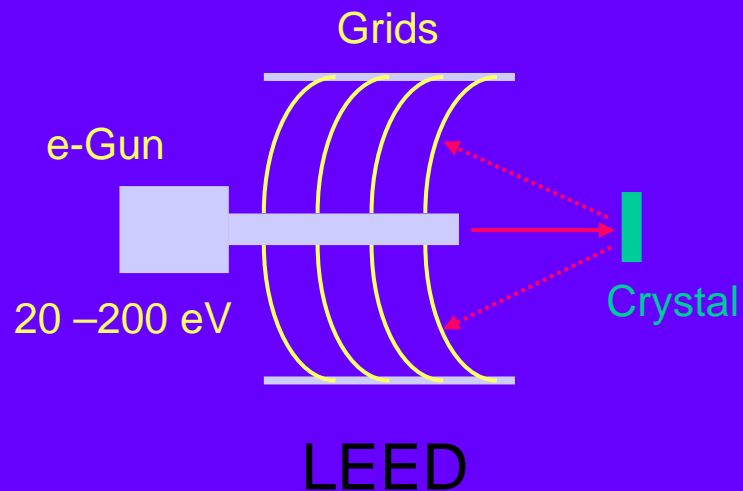
# Electron as a wave

de Broglie's proposal:

$$\lambda = h/P \Rightarrow \nu = h/E$$

*For electrons:*

$$\lambda \text{ (nm)} = 1.22/E^{1/2} \text{ (eV)}$$



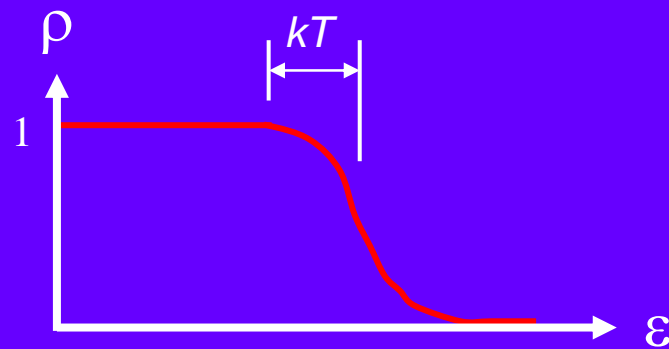
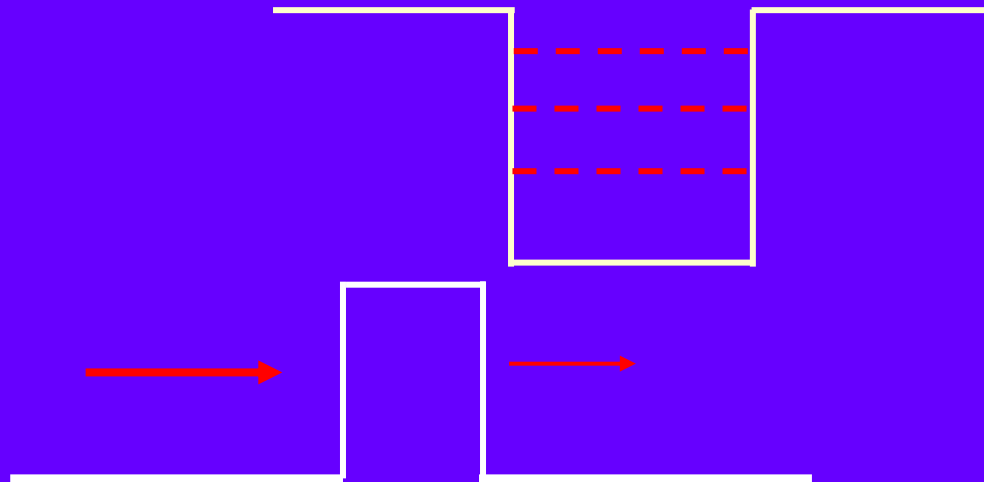


# Fundamentals of Quantum Mechanics

1. Quantization

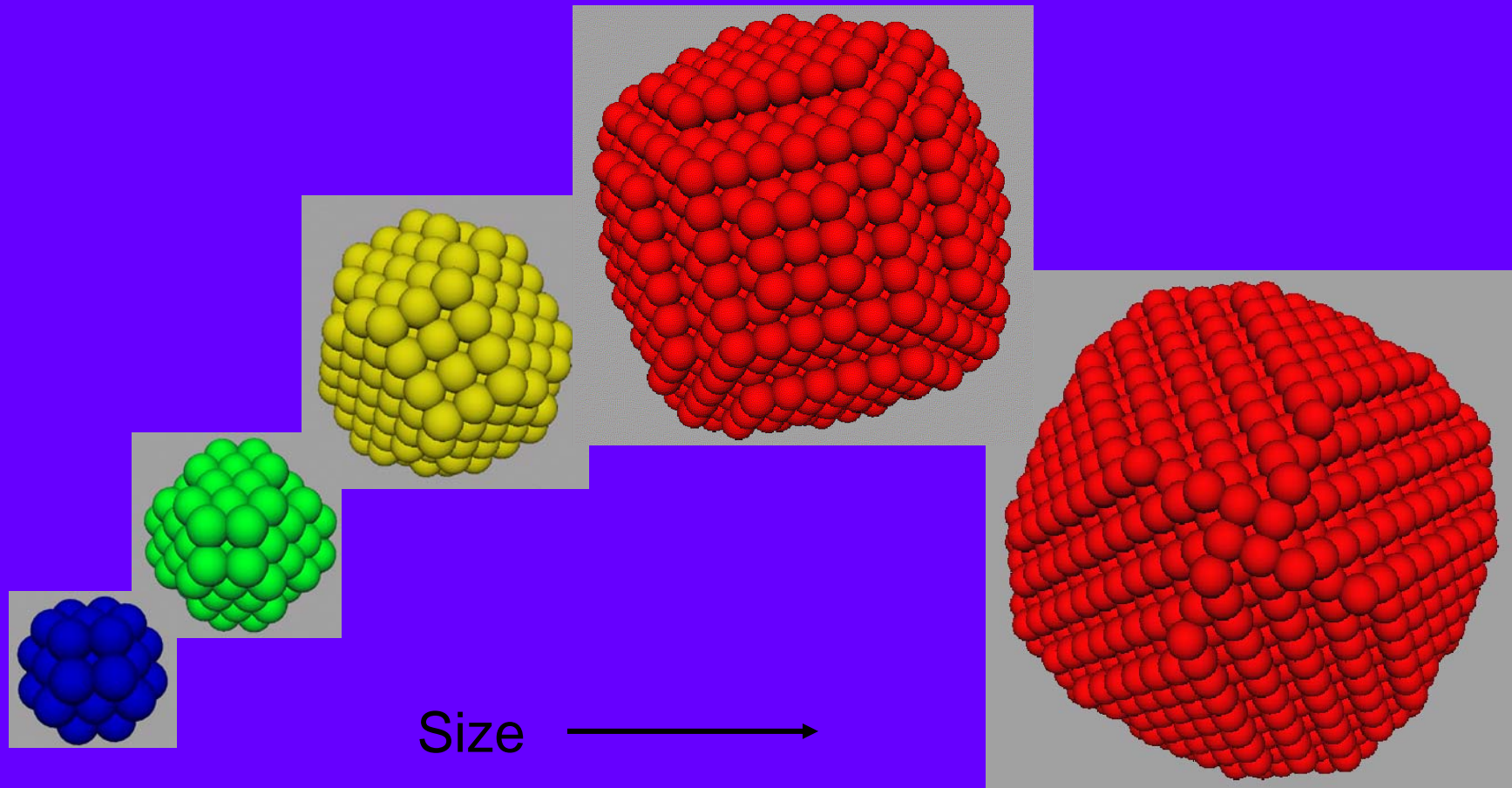
2. Tunneling

3. Statistics



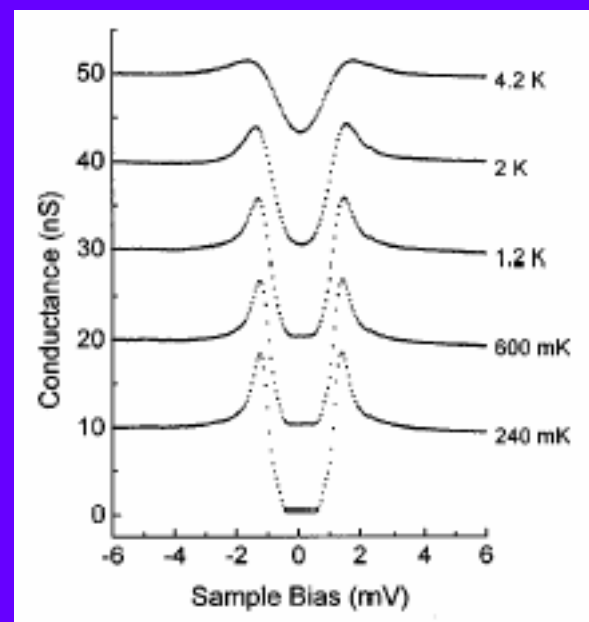
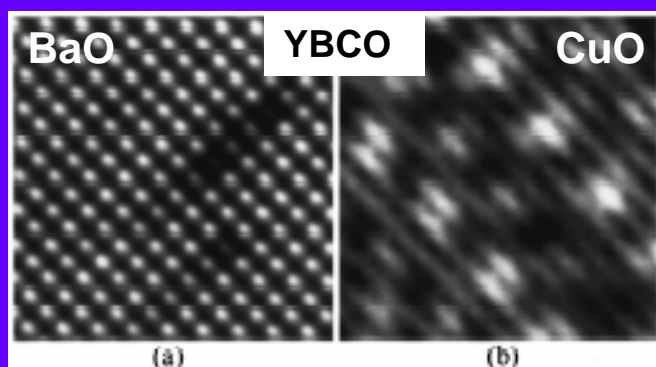


# Size effect





# Measurement of electronic states



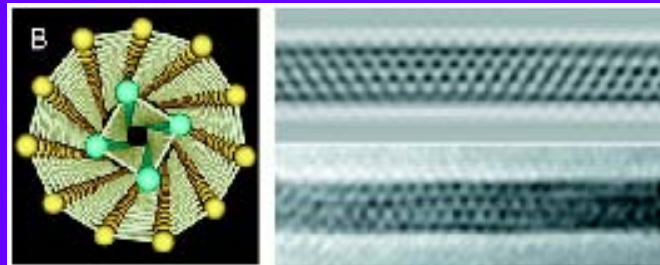
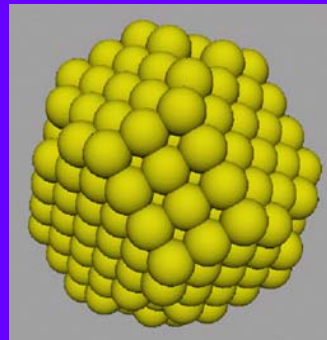
S. H. Pan *et al.*, *Rev. Sci. Instrum.* 40, 1459 (1999).



# Properties of a nanostructure

## Microscopic

Size  
Structure  
Shape  
Symmetry  
Domains  
Defects



.  
. .  
. .

## Analytical

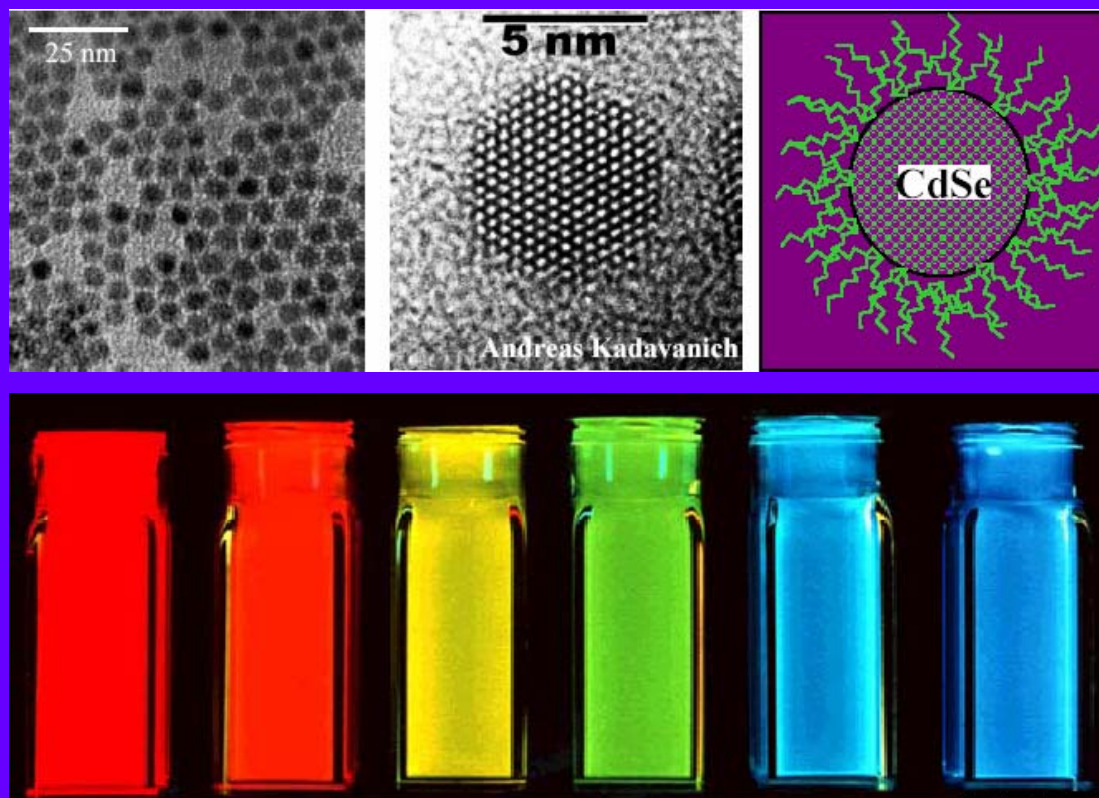
Composition  
Stoichiometry  
Electronic  
Magnetic  
Thermal  
Mechanical

.  
. .  
. .

photons, electron, ion, neutron, proximal nanoobjects ...

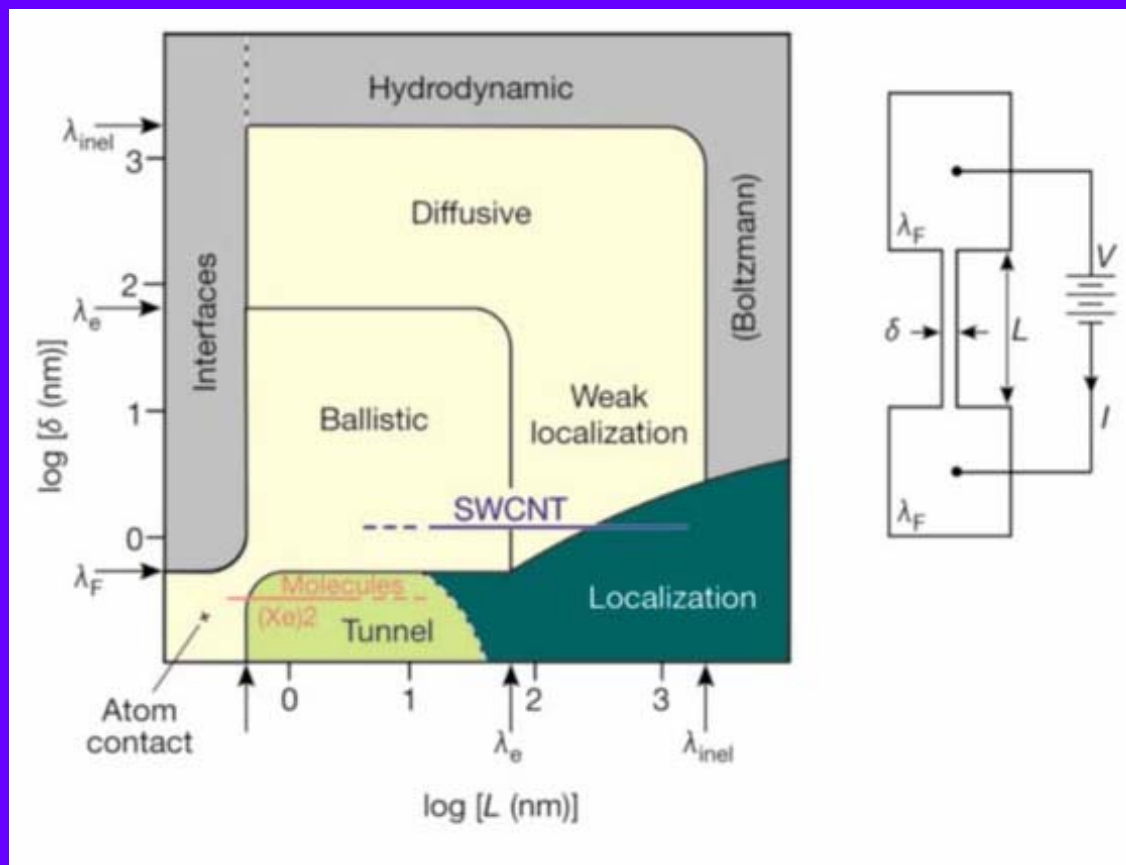


# Semiconducting quantum dots



(Reproduced from Quantum Dot Co.)

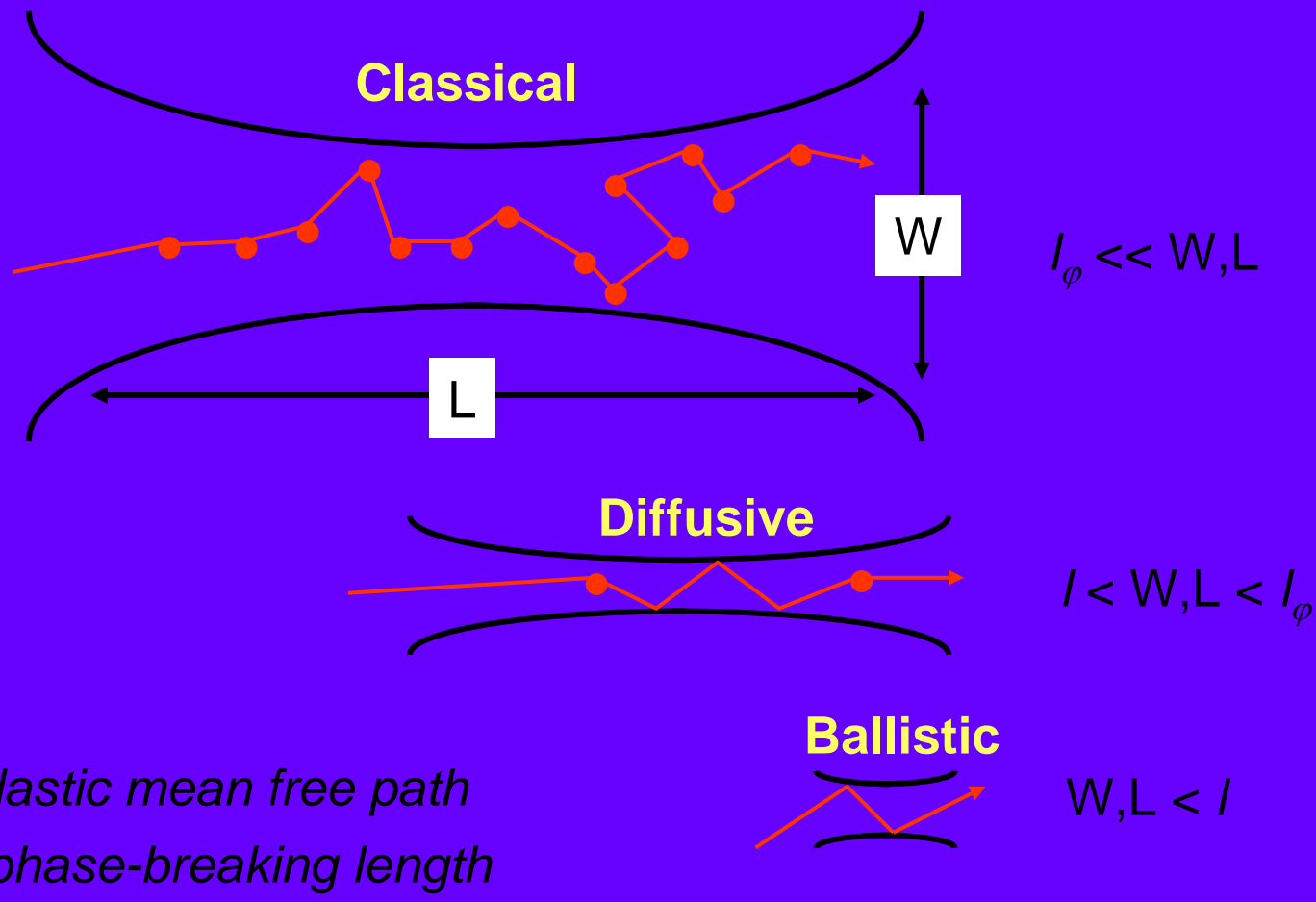
# Critical lengths



C. Joachim *et al.*, *Nature* 408, 541 (2000).

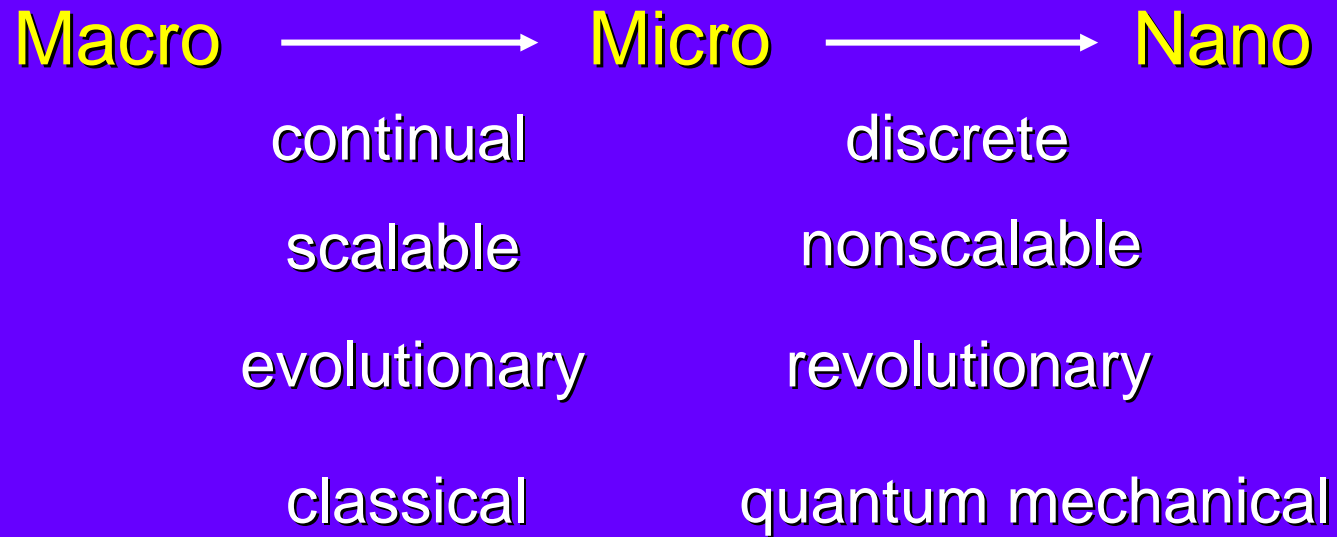


# Characteristics of electron transport

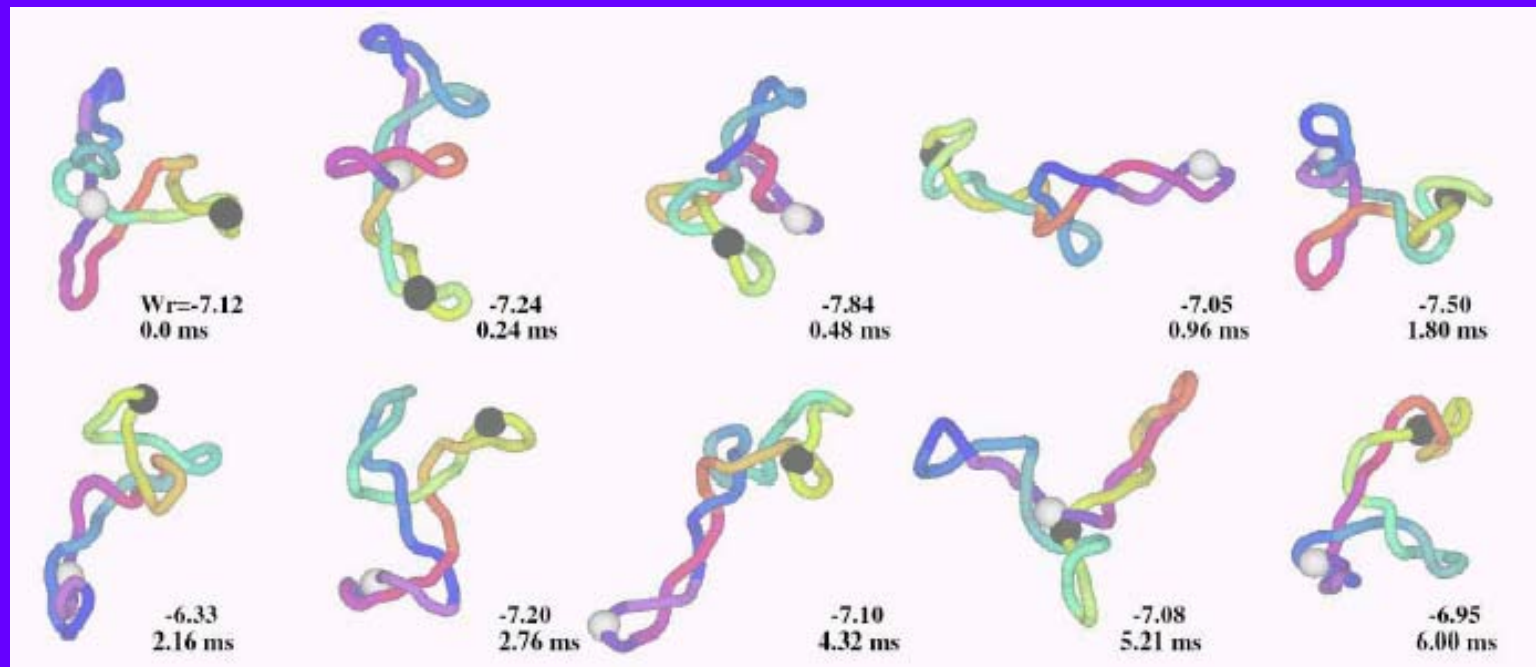




# Minituralization of length scale

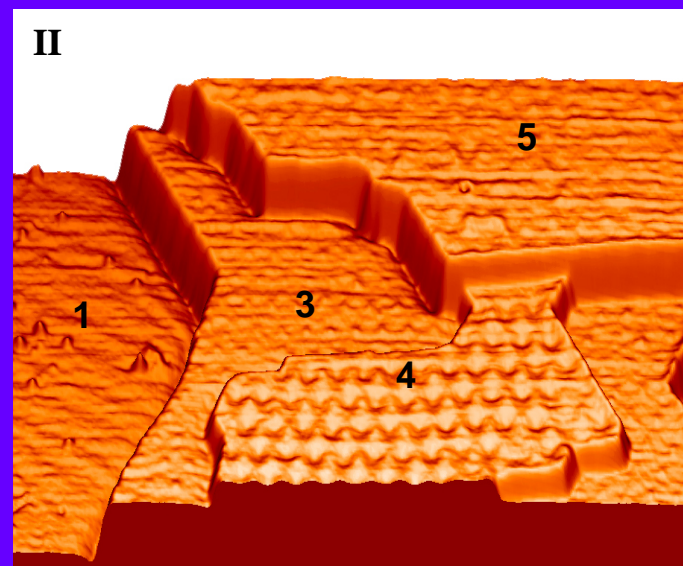
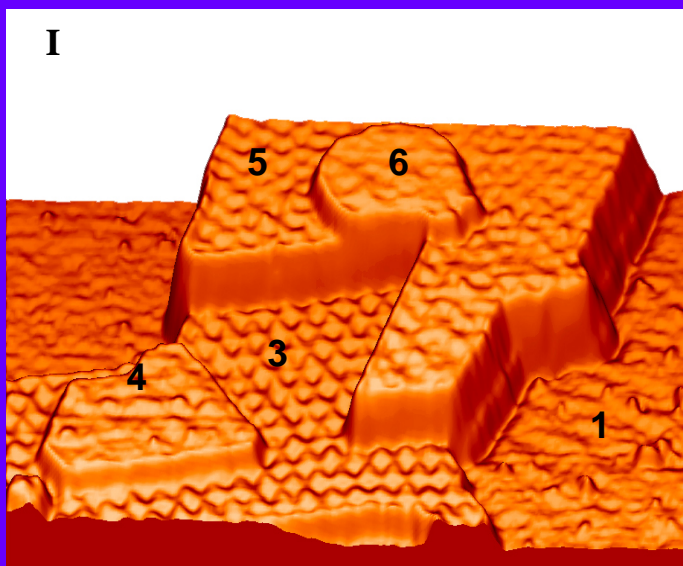


# Simulation of DNA dynamics



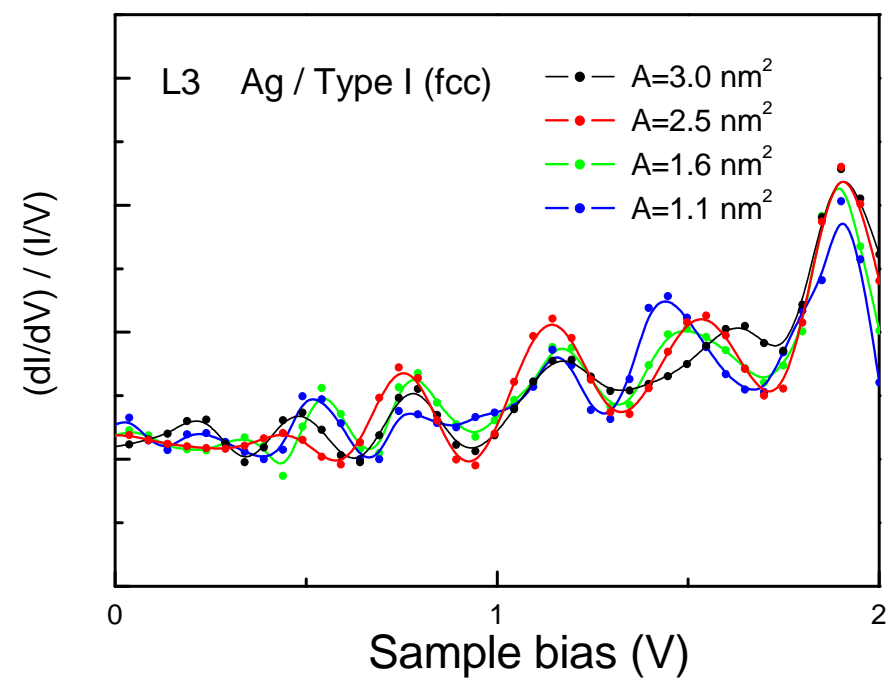
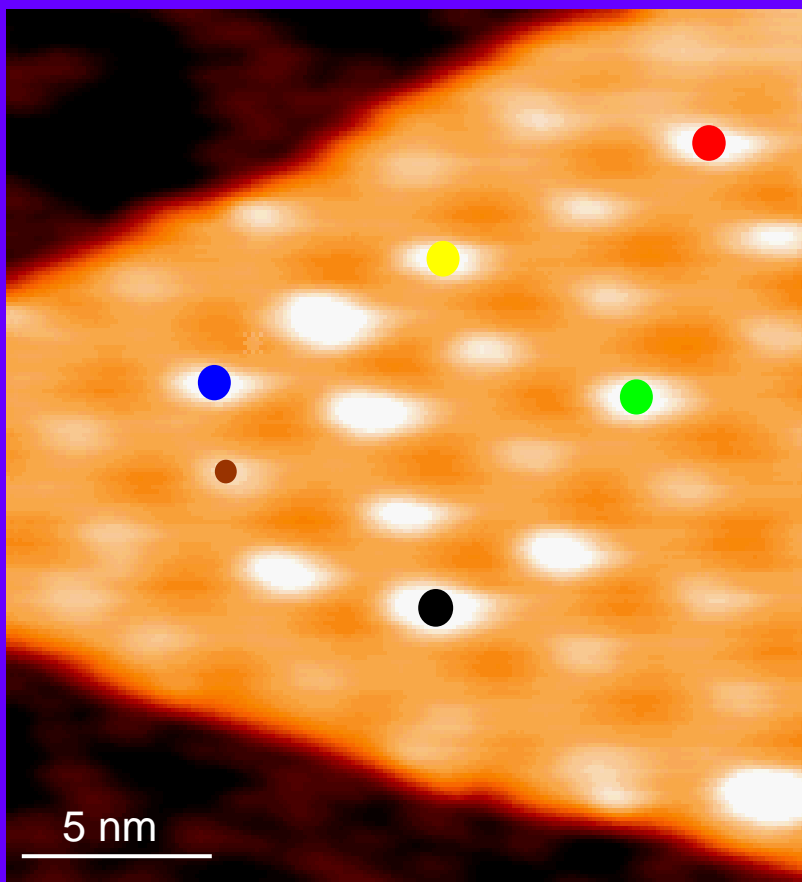


## Alternating and complementary contrast

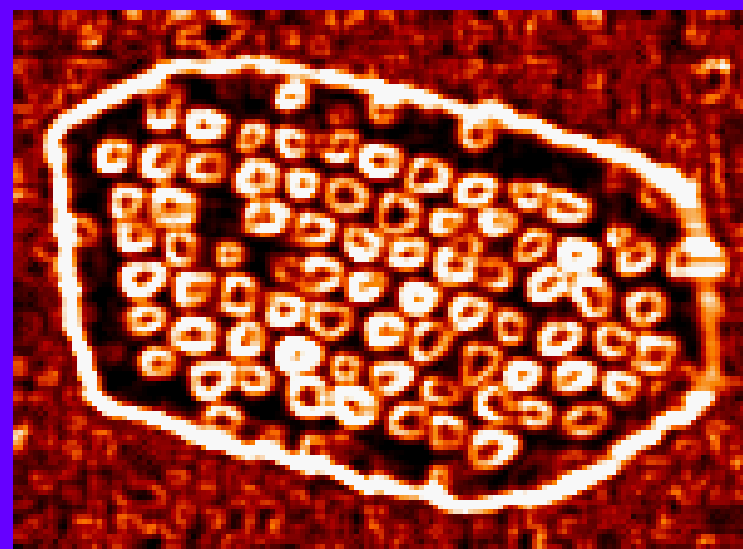
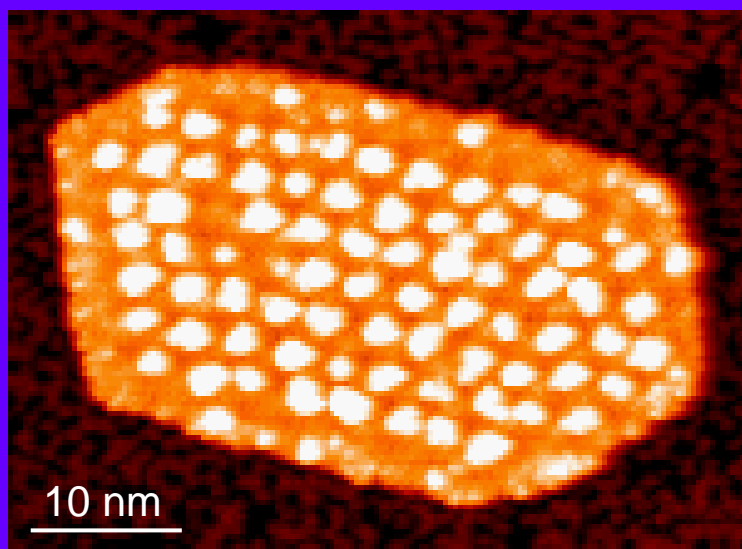


W.B. Jian *et al.* PRL **90**, 196603 (2003)

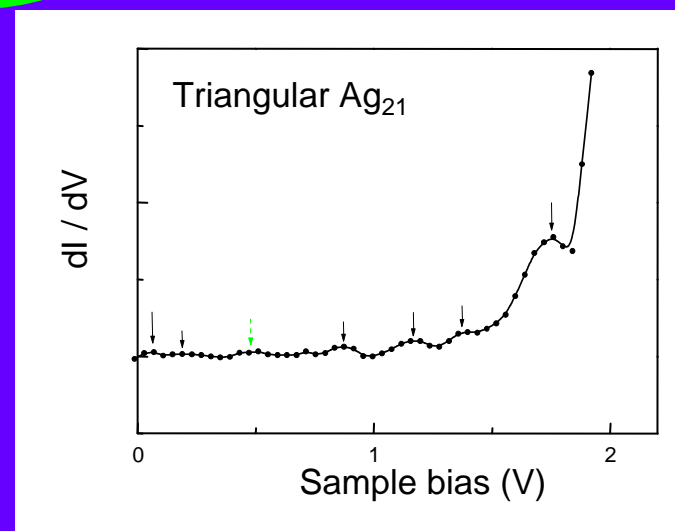
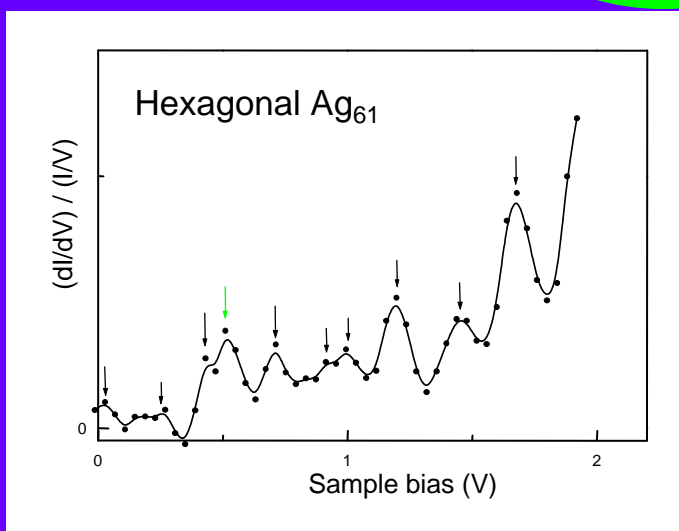
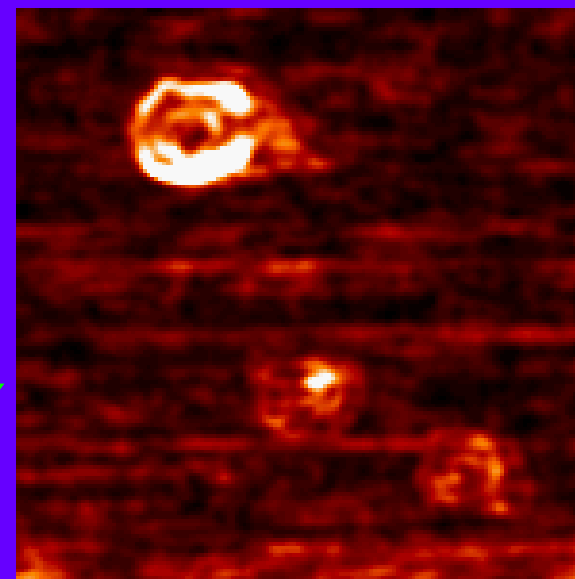
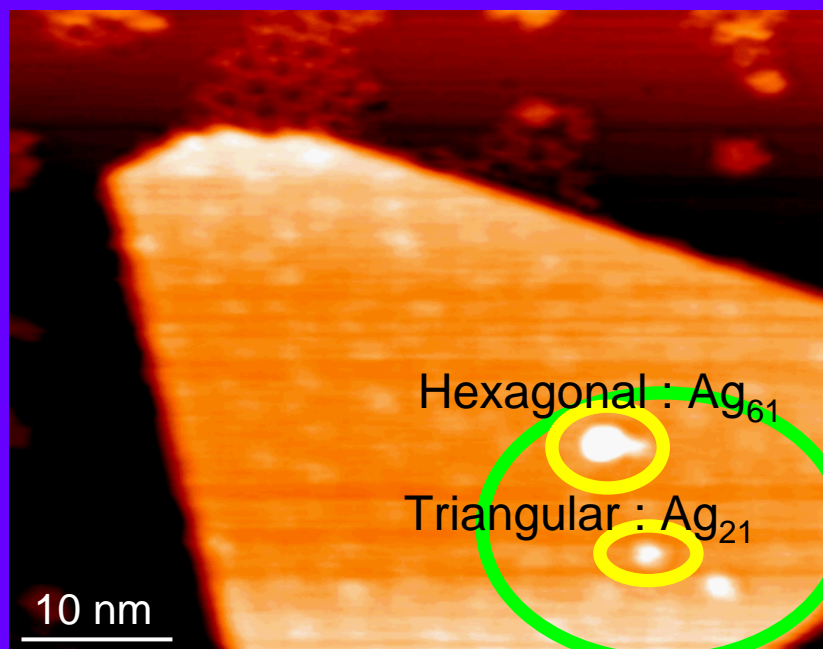
# Size-dependent I/V spectra by STS



## Shapes of Ag nanopucks



# Size- and shape-dependent I/V spectra



# Characterization of single nanostructure

## Specs

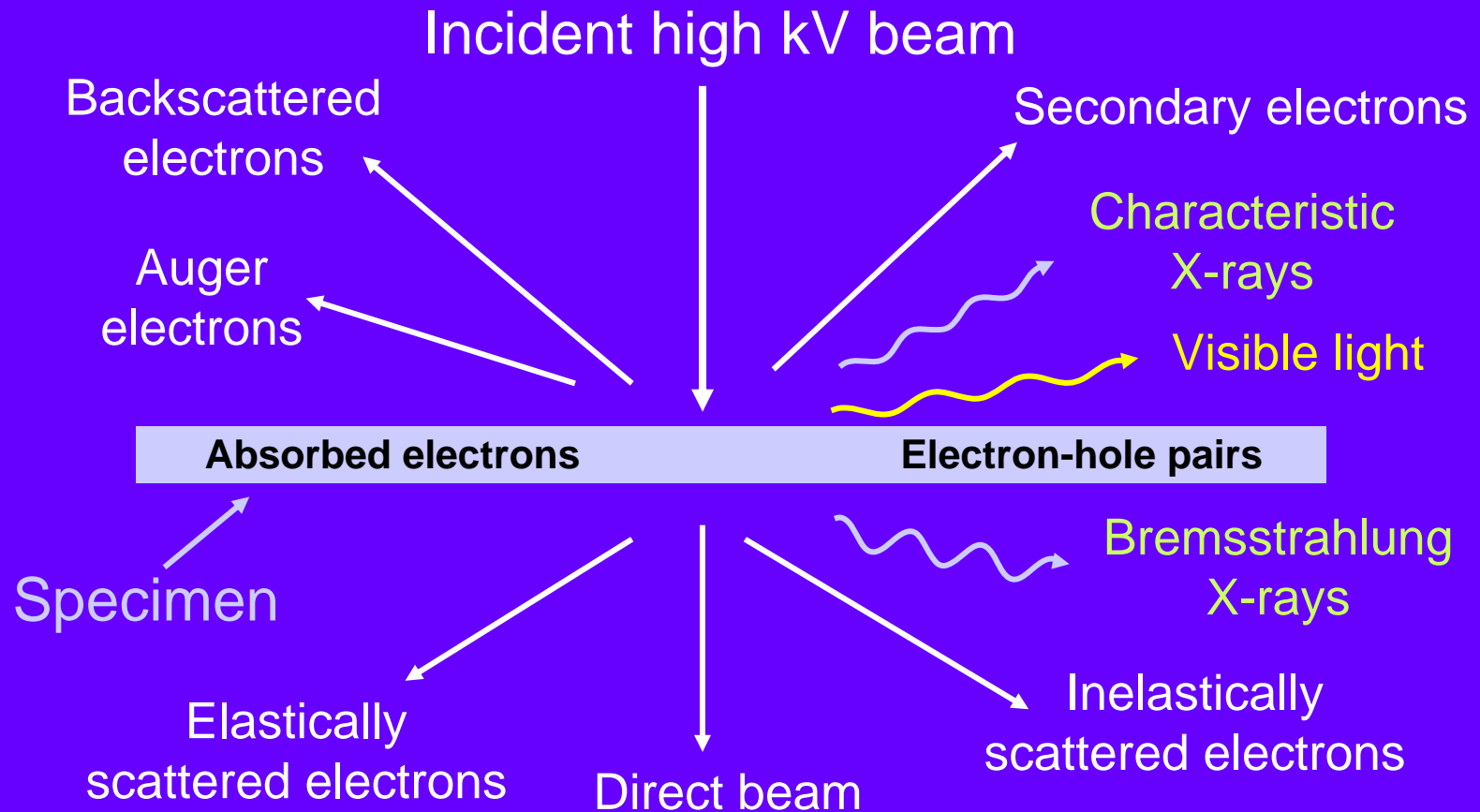
- Small size
- Weak signal
- High reactivity
- Flexible  
functionality

## Demands

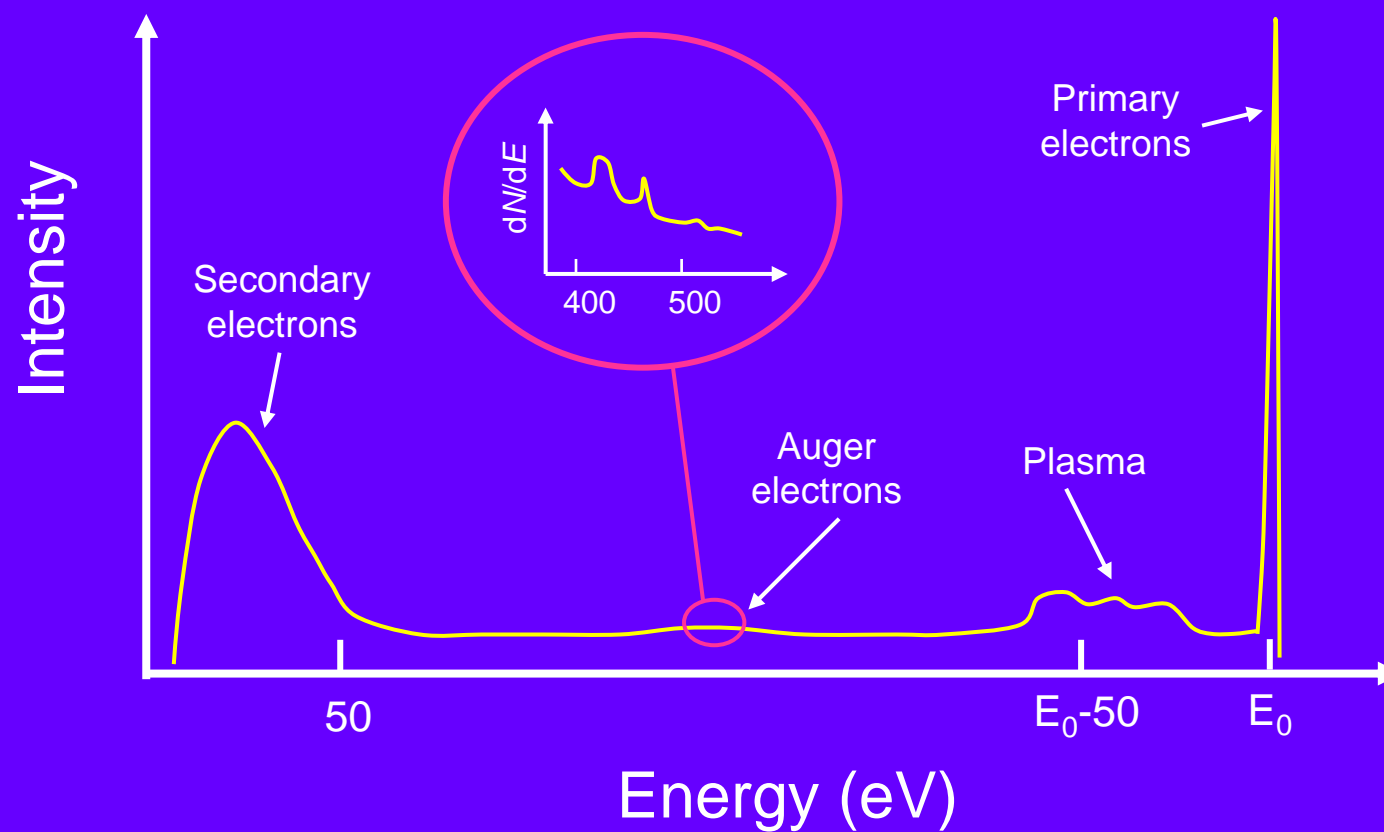
high brightness  
small probe  
high sensitivity  
low interference  
controlled environment  
low temperature  
high speed  
*in situ* measurement



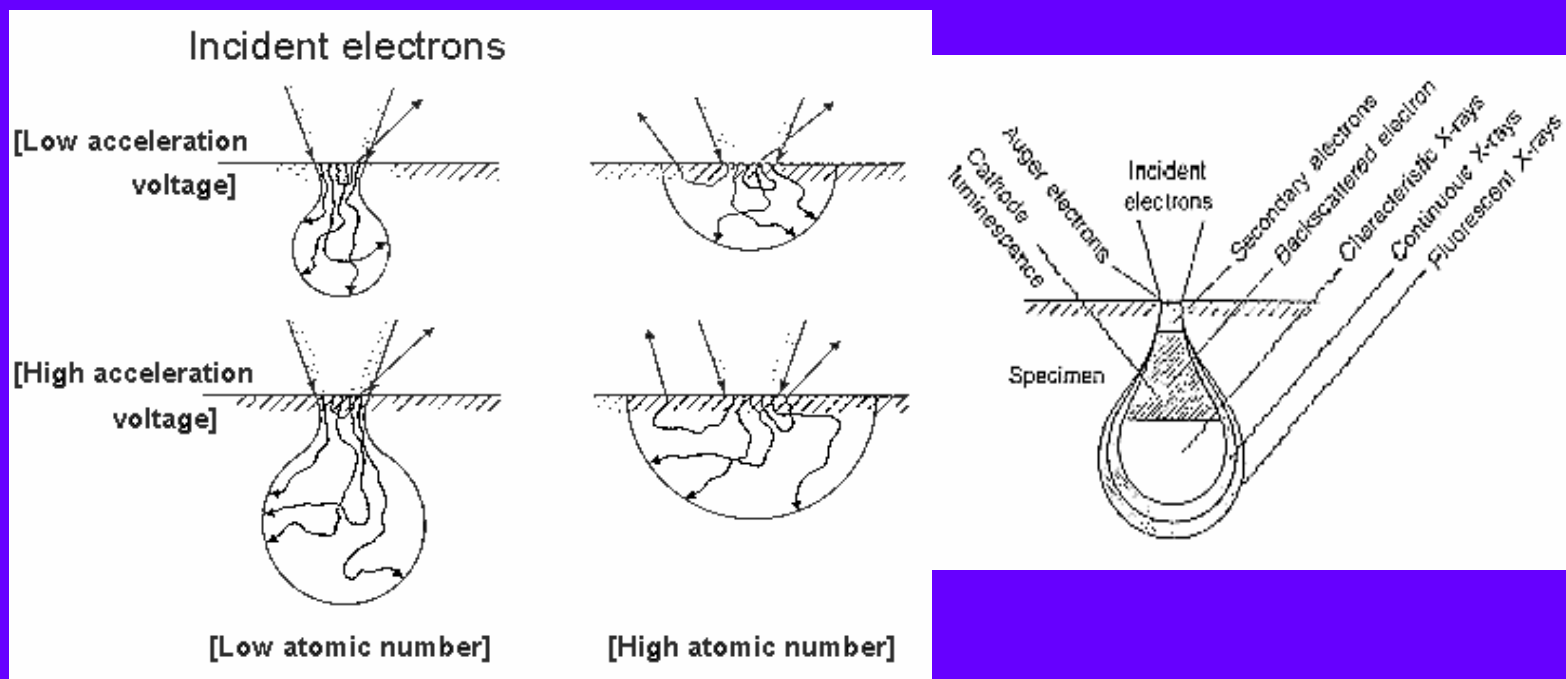
# Interaction between e-beam and sample



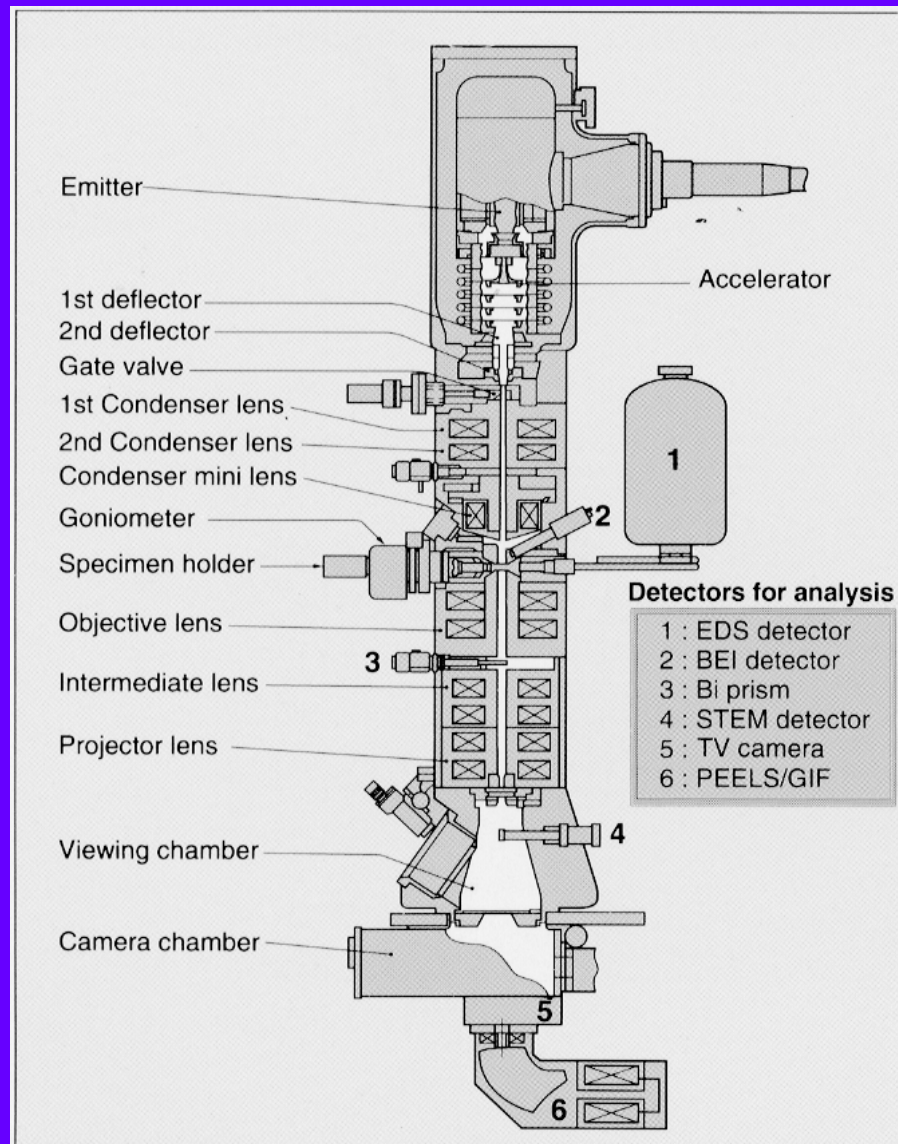
# Energy distribution after interaction



# Penetration power of energetic e beam

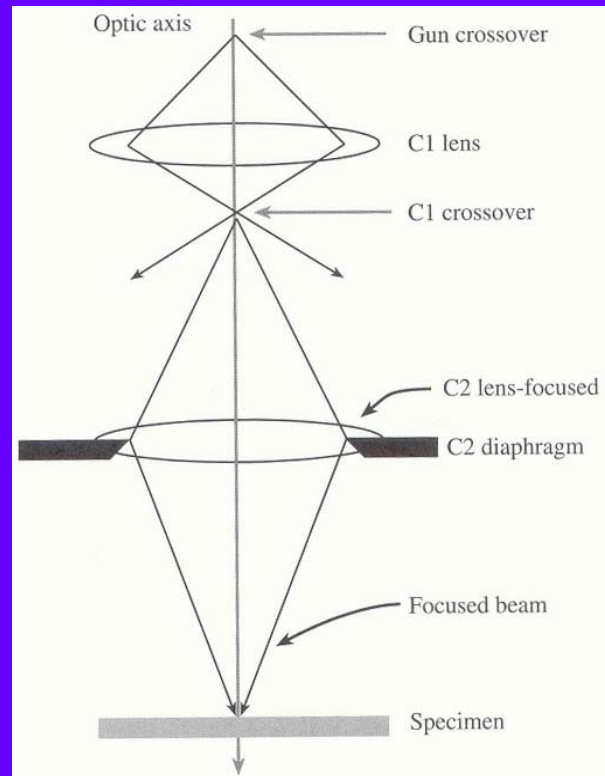
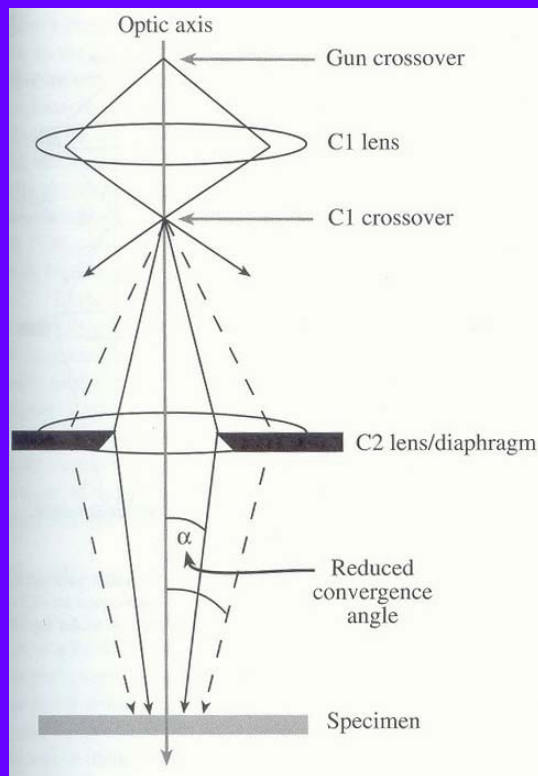


# Cross section view of HRTEM

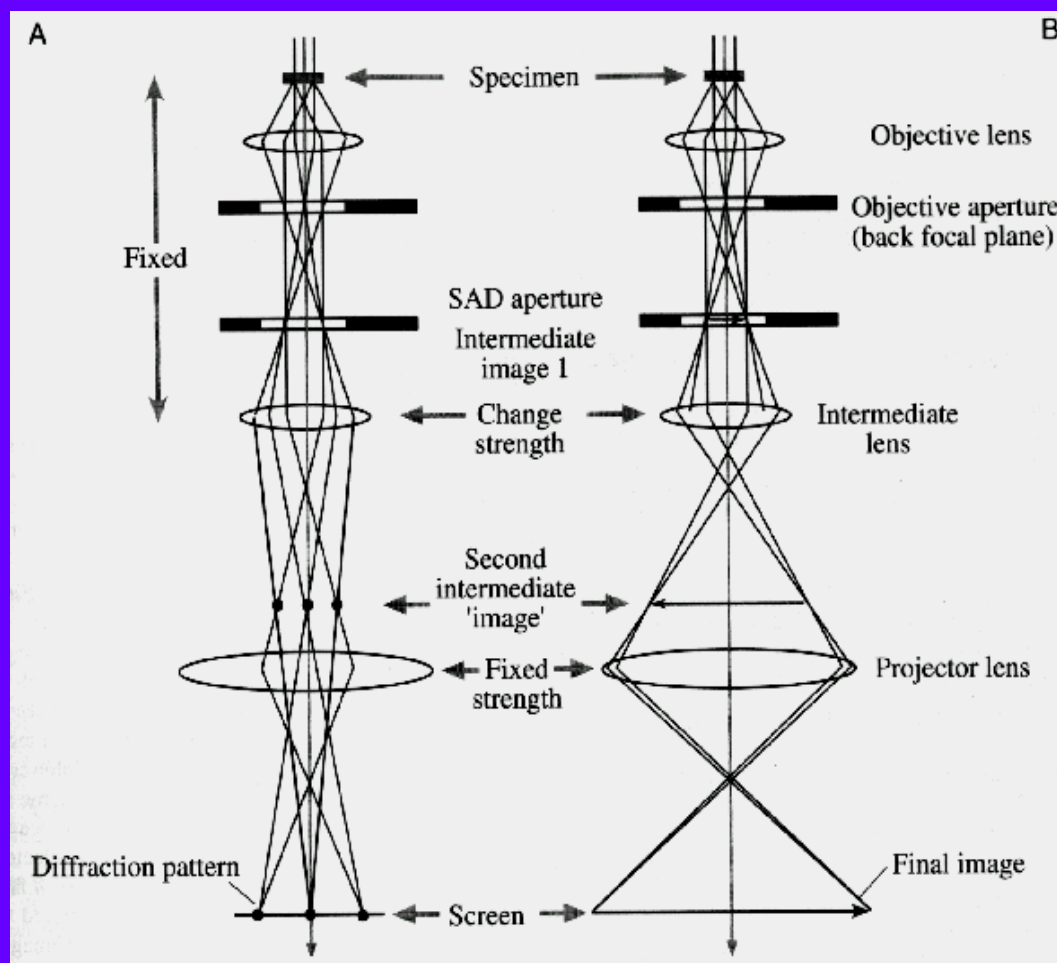


Cross section of JEM-2010F and assignrment of detectors

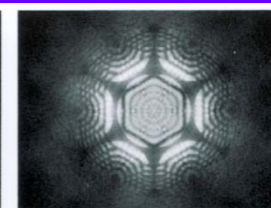
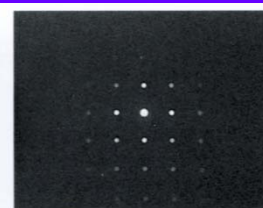
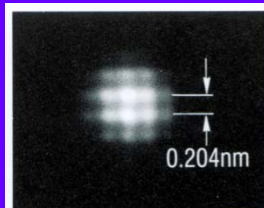
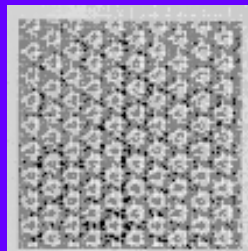
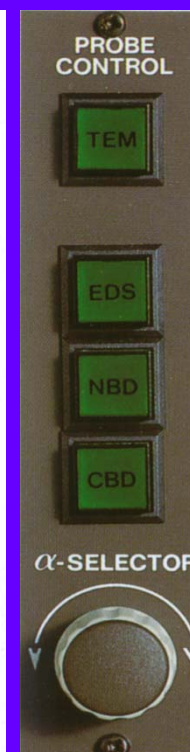
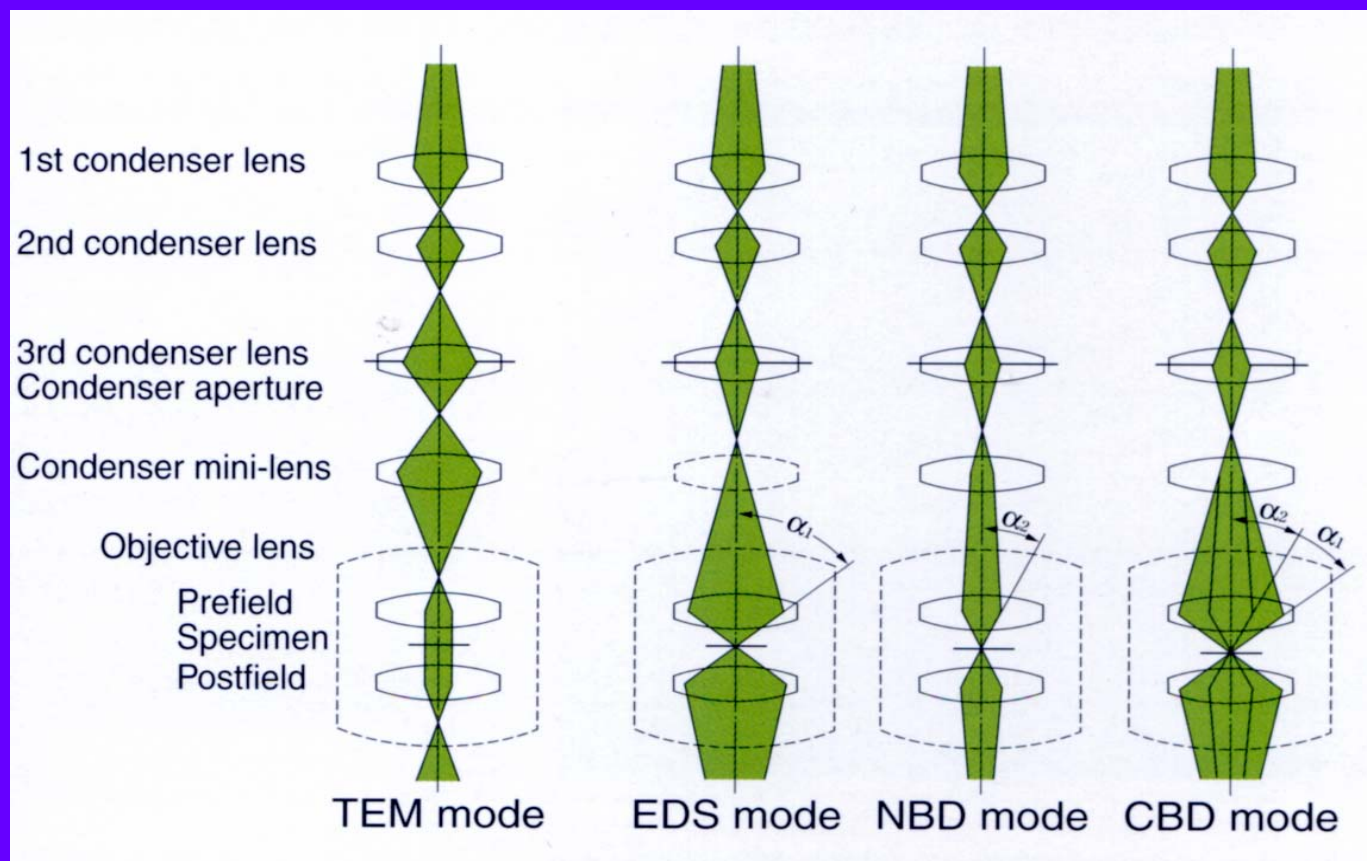
# E-beam source of TEM



# Imaging system



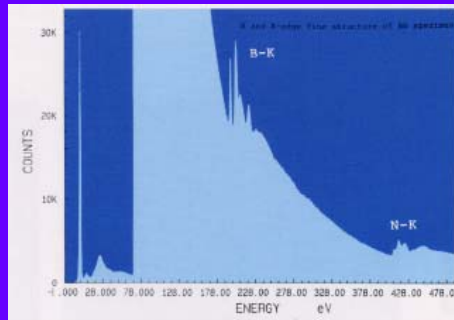
# Quick Beam Select System



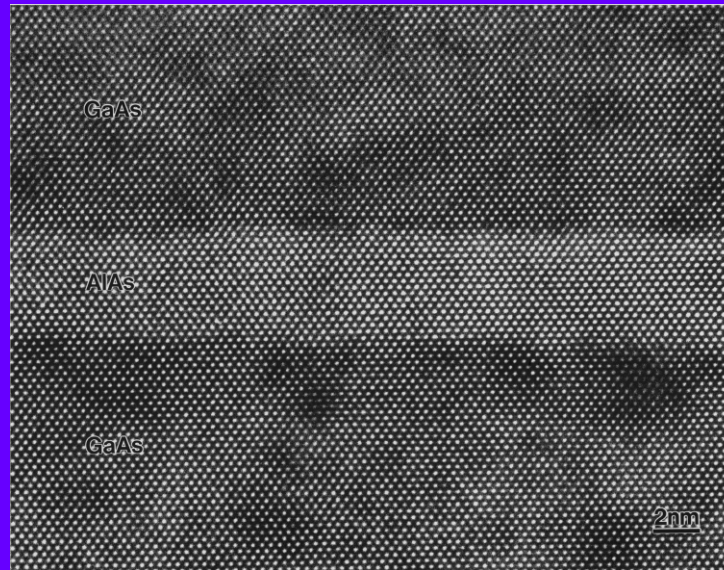
# Information from TEM



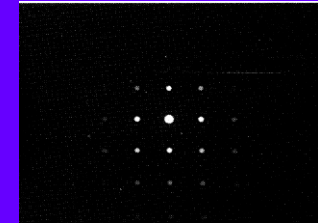
EDS



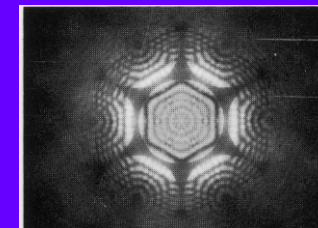
EELS or GIF



Lattice image  
GaAs/AlAs



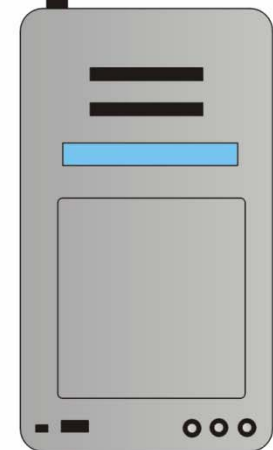
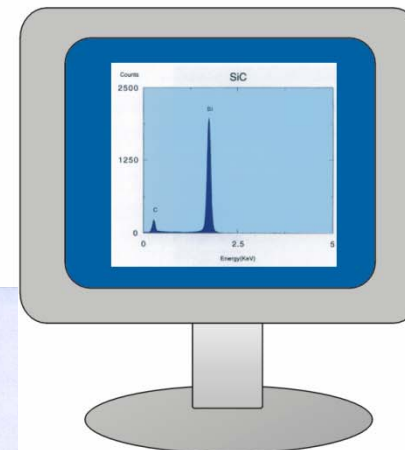
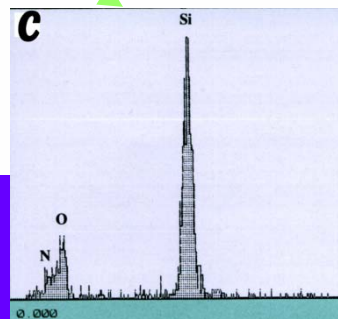
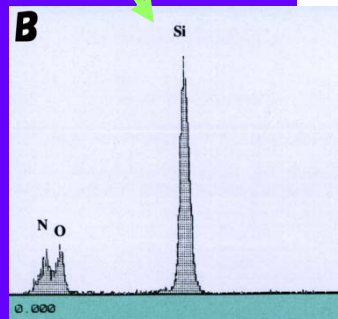
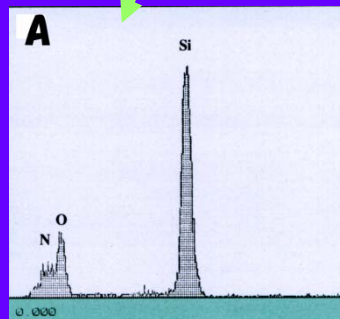
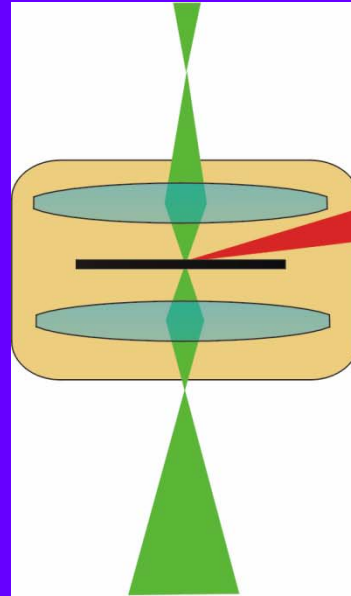
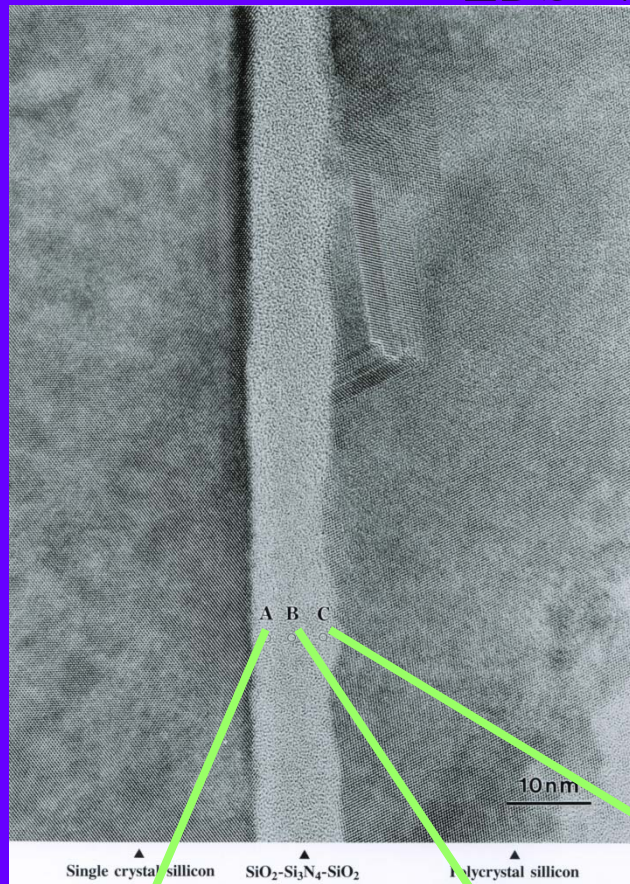
Electron Diffraction



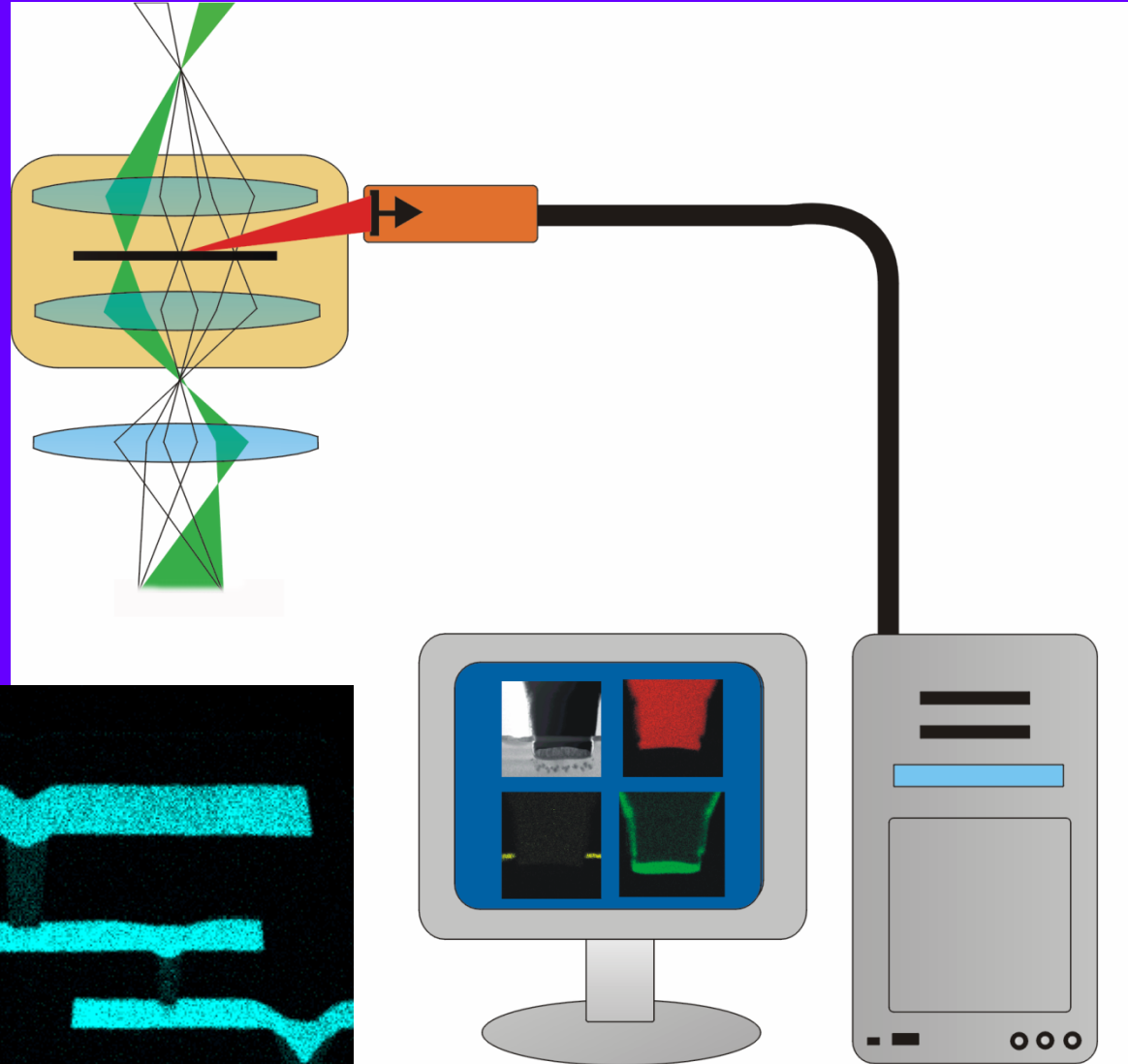
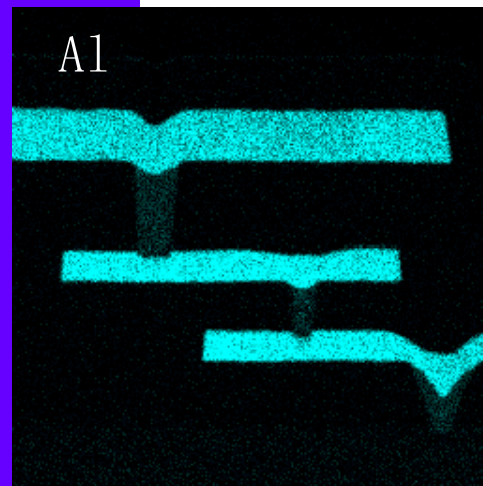
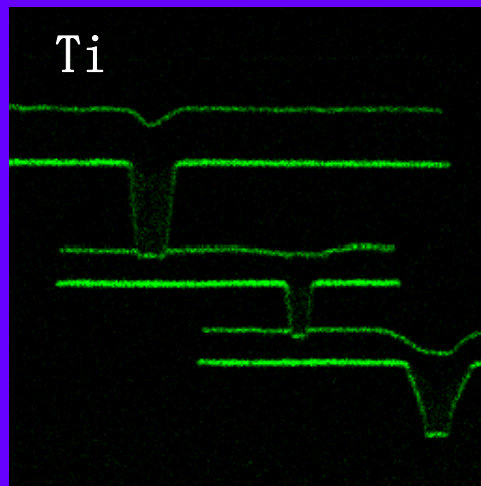
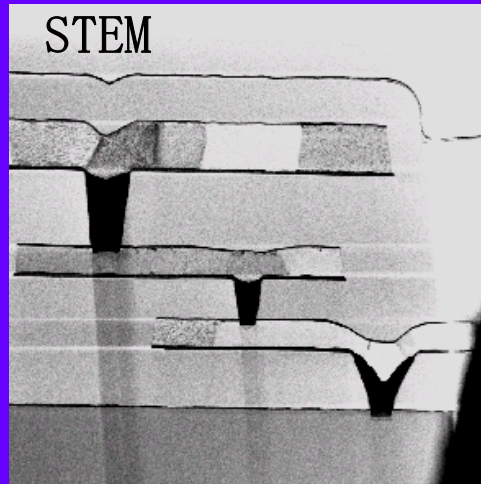
CBED

STEM+BF, HAADF → Mapping and Z-contrast image

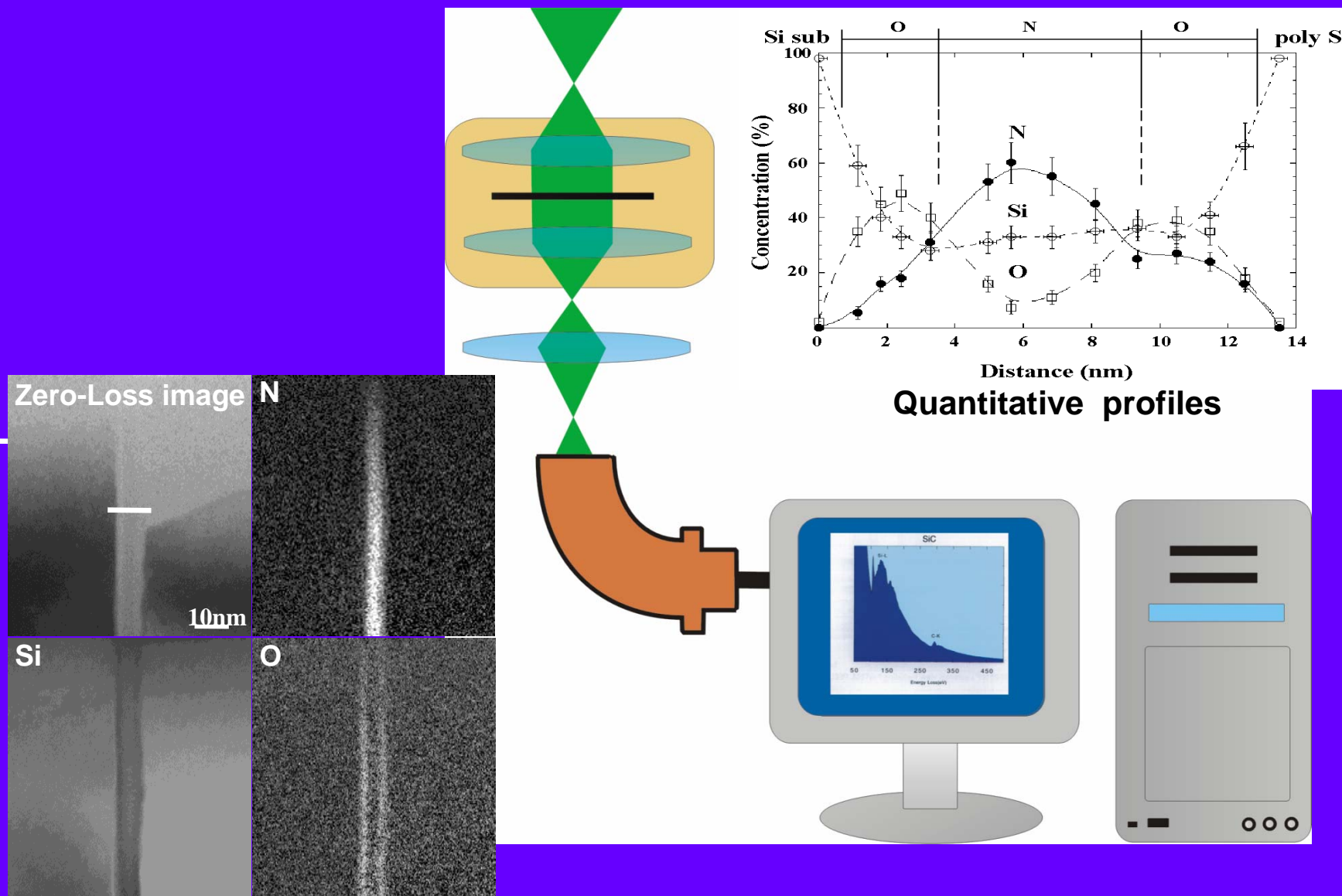
# *EDS with spatial resolution*



# *EDS mapping*



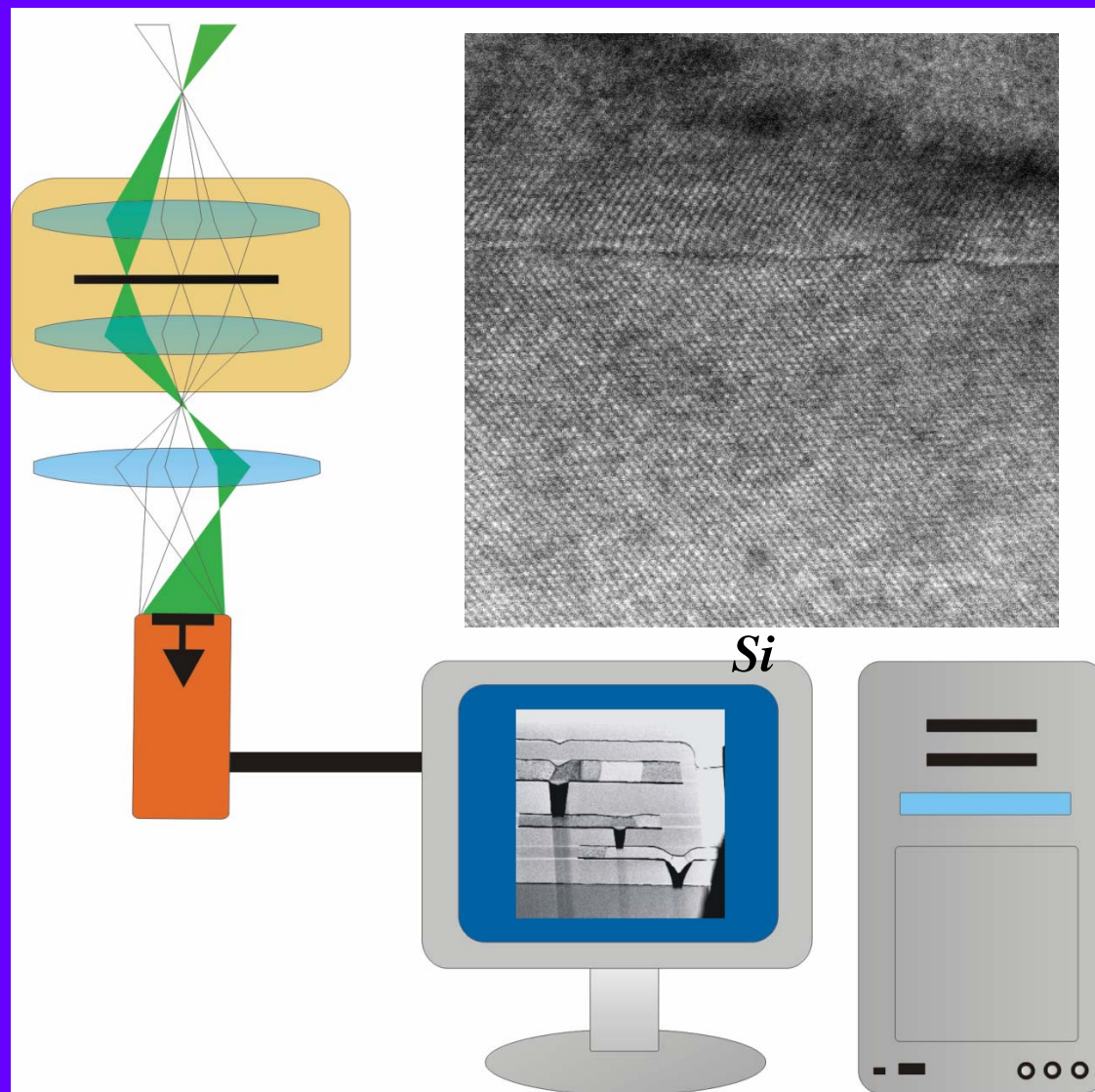
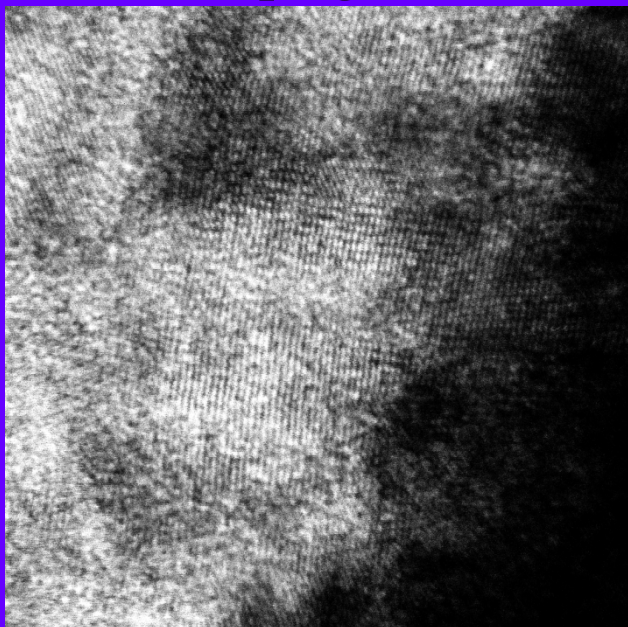
# High resolution EELS



Energy Filtered images of insulating layer

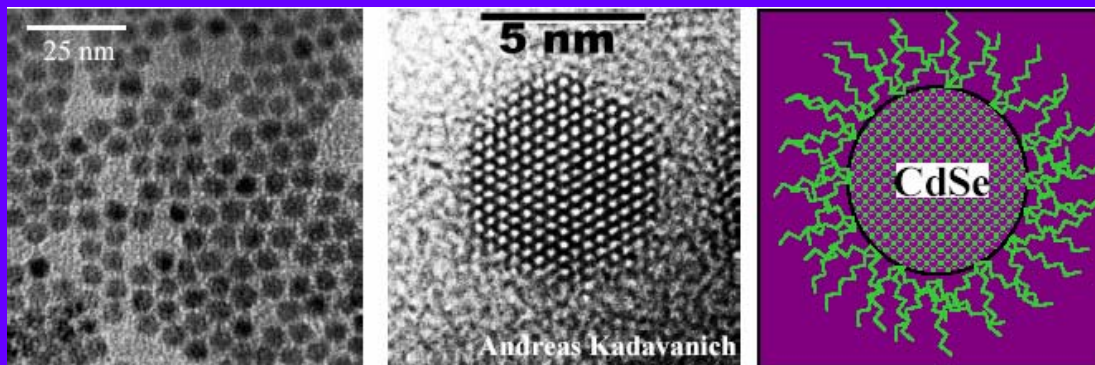
# *High resolution STEM image*

W-plug.

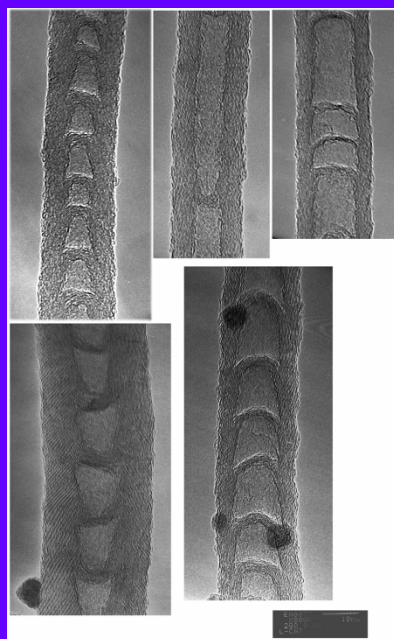




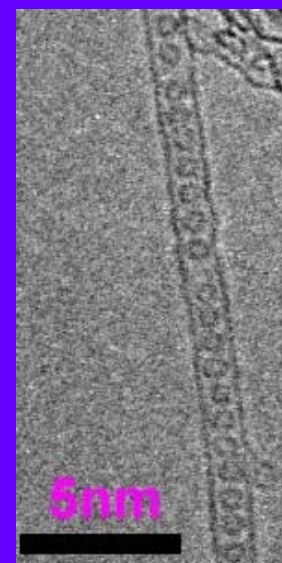
# Semiconducting quantum dots and carbon nanotubes



(Reproduced from Quantum Dot Co.)



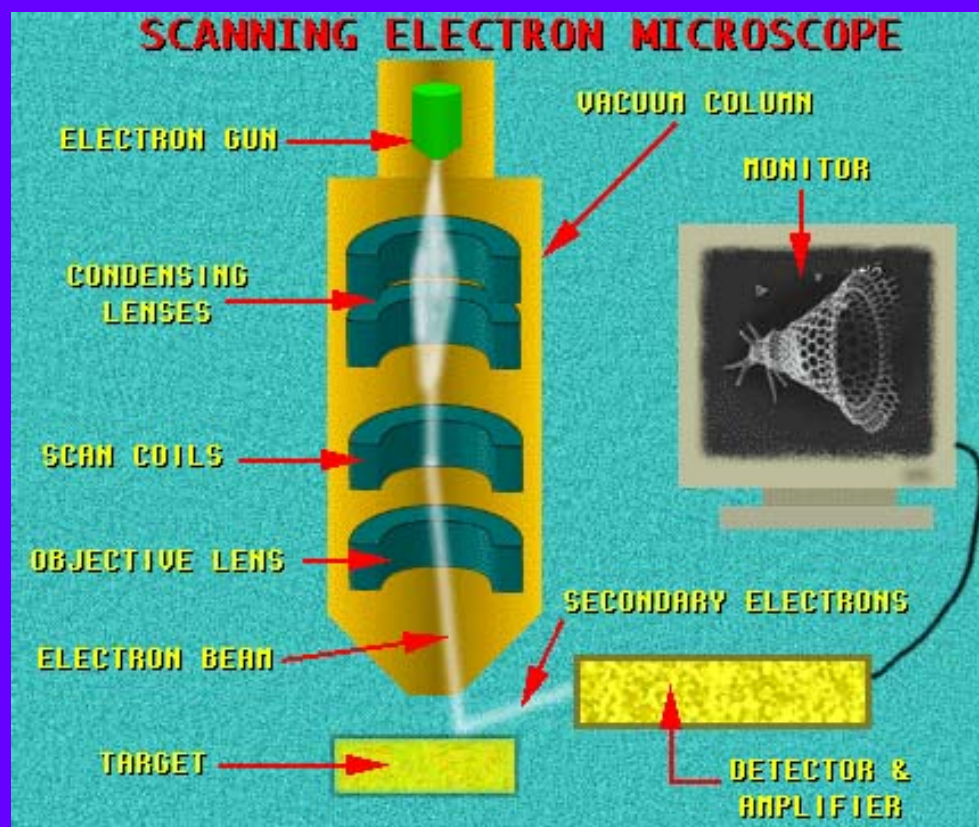
Multiwall CNT



Peapod CNT

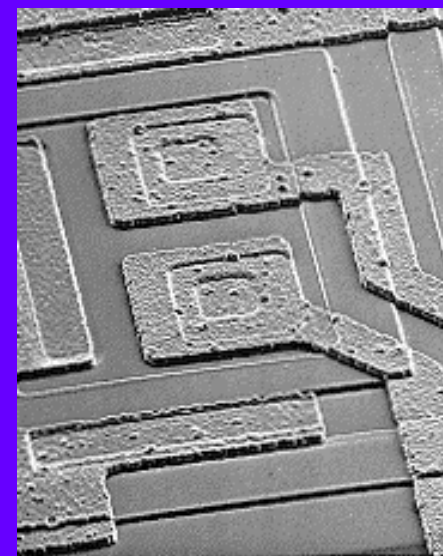
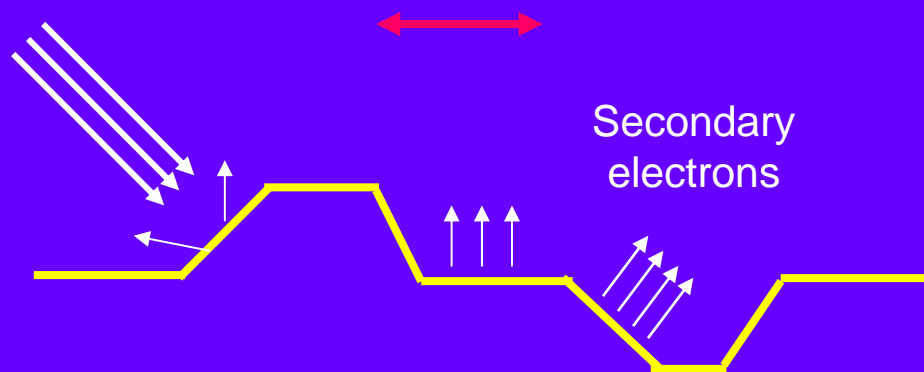


# Scanning electron microscopy (SEM)



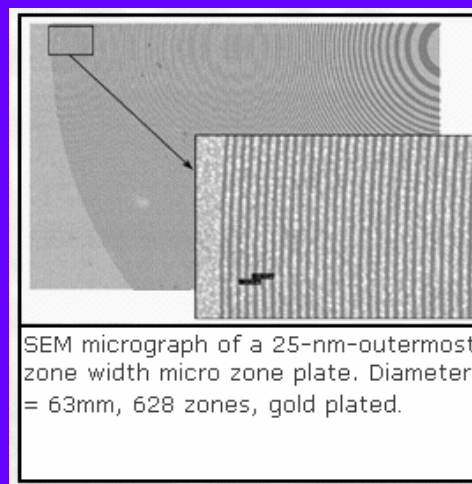
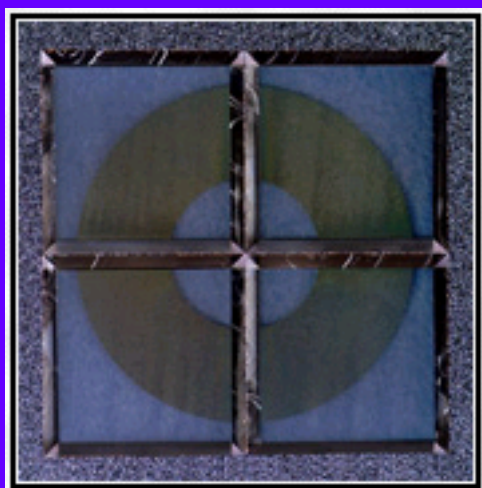
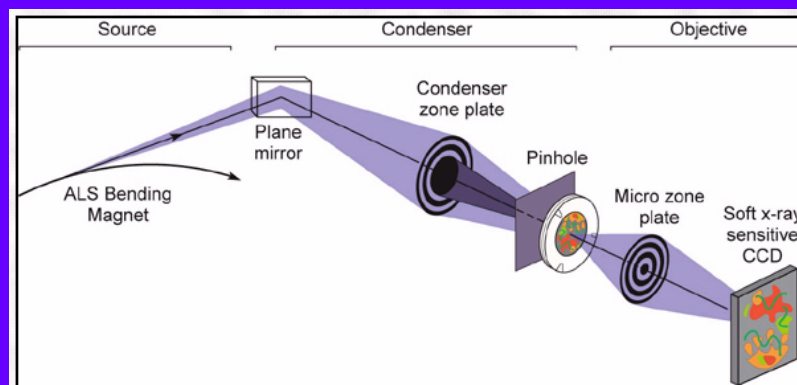
# SEM contrast

Scanning e beam



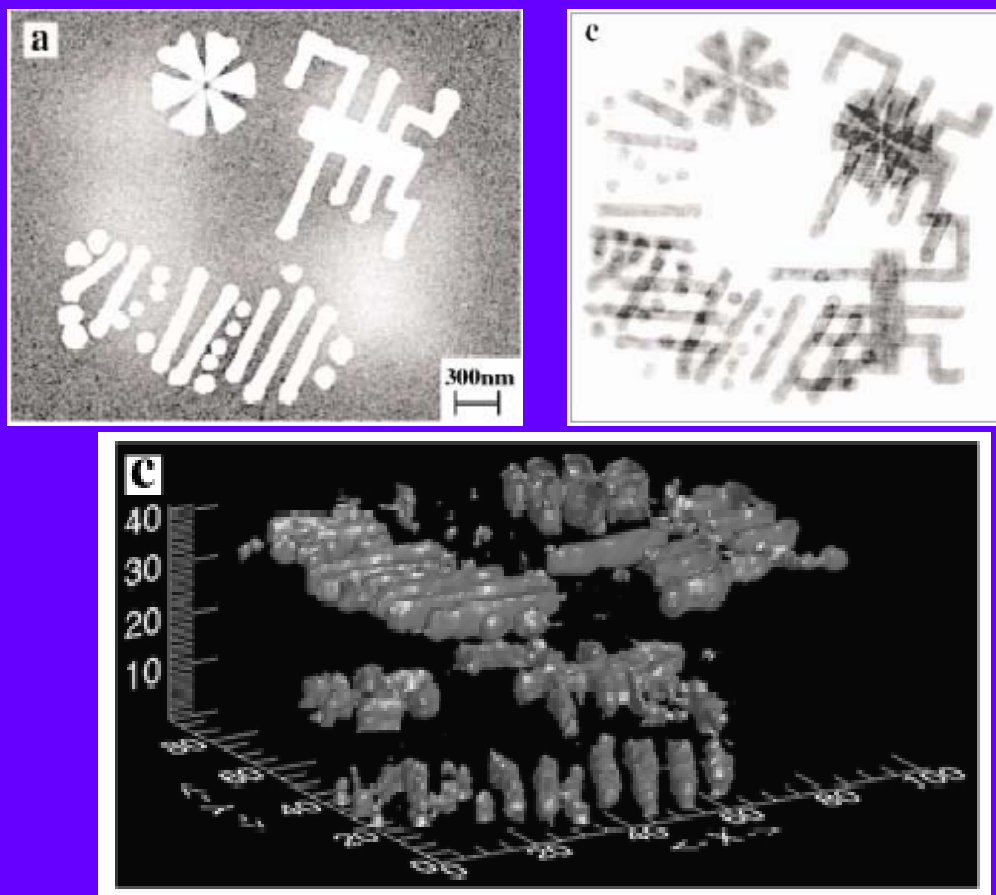
(a) 5 kV x720  
Tilt angle: 50°

# X-Ray Microscopy





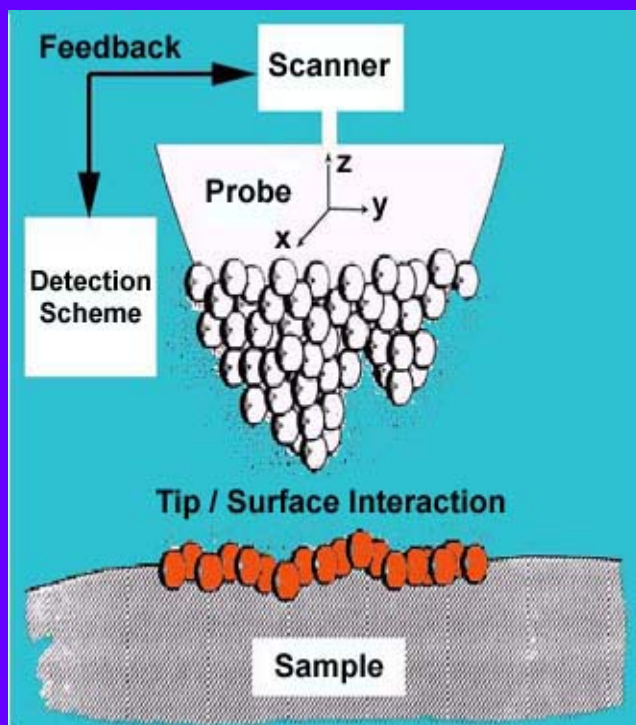
## 3D X-Ray Image



J. Miao et al., PRL 89, 088303 (2002).



# Historical development of SPMs



## Scanning Tunneling Microscopy (STM)

--- G. Binnig, H. Rohrer et al, (1982)

## Near-Field Scanning Optical Microscopy (NSOM)

--- D. W. Pohl (1982)

## Atomic Force Microscopy (AFM)

--- G. Binnig, C. F. Quate, C. Gerber (1986)

## Scanning Thermal Microscopy (SThM)

--- C. C. Williams, H. Wickramasinghe (1986))

## Magnetic Force Microscopy (MFM)

--- Y. Martin, H. K. Wickramasinghe (1987)

## Friction Force Microscopy (FFM or LFM)

--- C. M. Mate et al (1987)

## Electrostatic Force Microscopy (EFM)

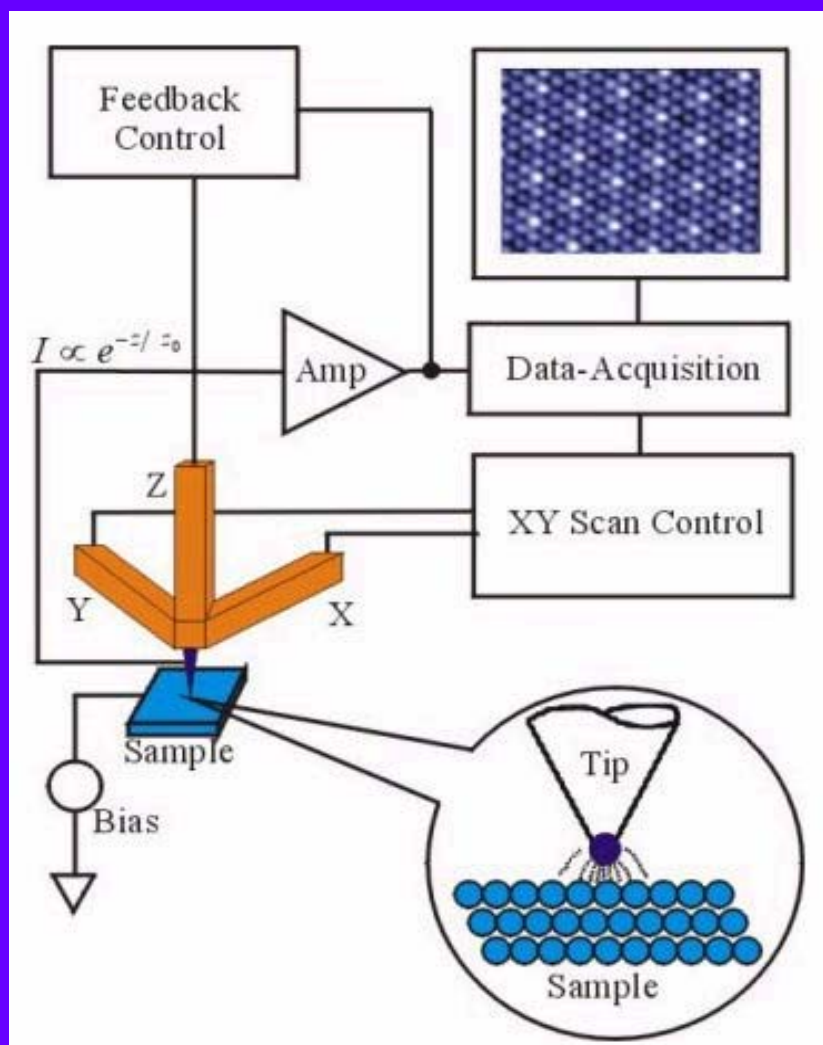
--- Y. Martin, D. W. Abraham et al (1988)

## Scanning Capacitance Microscopy (SCM)

--- C. C. Williams, J. Slinkman et al (1989)

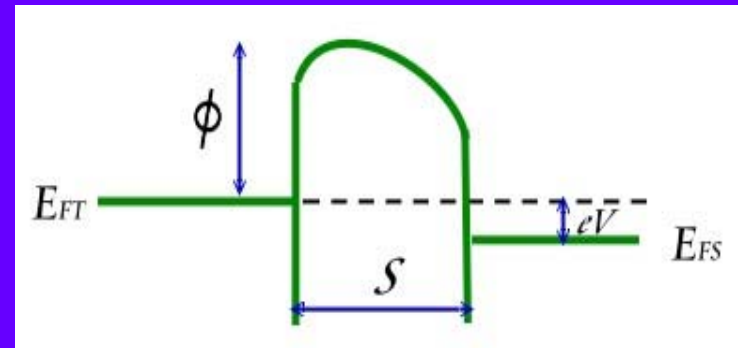
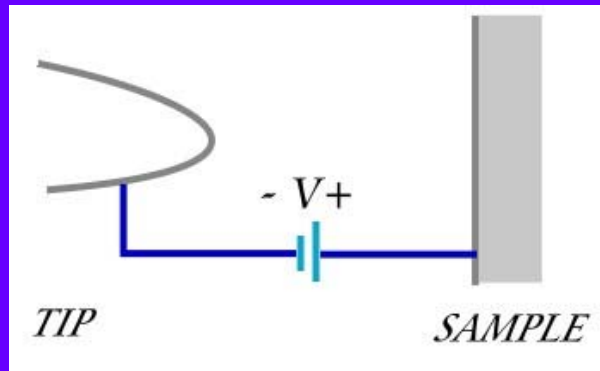
## Force Modulation Microscopy (FMM)

--- P. Maivald et al (1991)



# Scanning Tunneling Microscopy

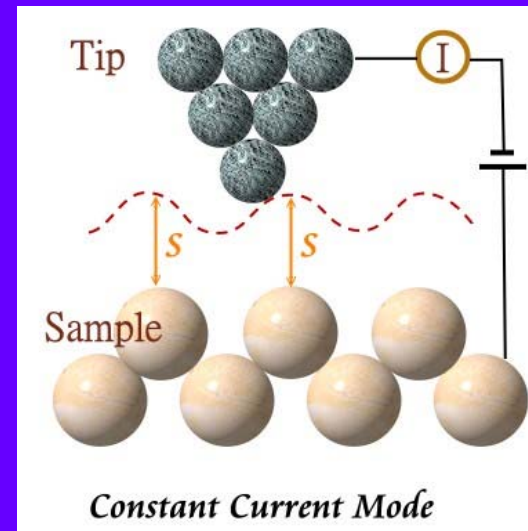
# Theory of STM



From one-dimensional tunneling problem tunneling current ( $eV \ll \phi$ )

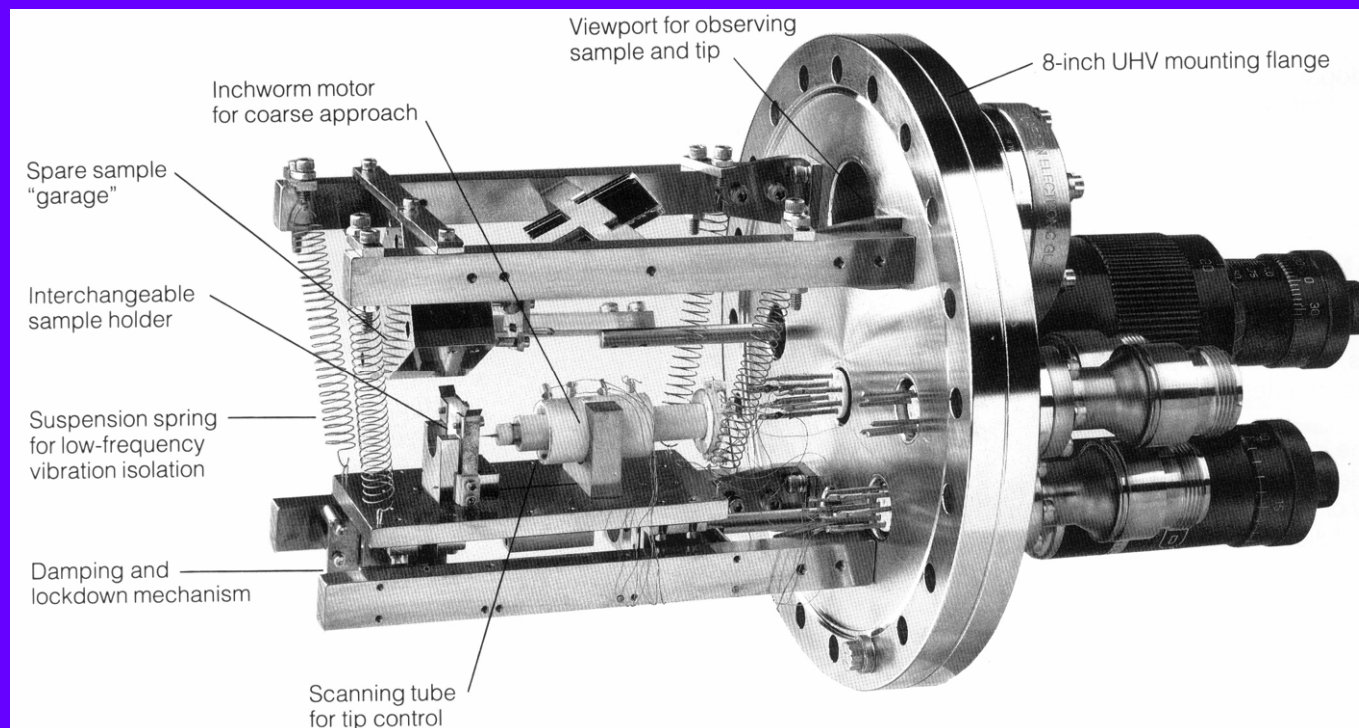
$$I \propto \frac{V}{S} \exp\left(-A\phi^{\frac{1}{2}}S\right)$$

$$A = 1.025 (eV)^{-0.5} \text{ \AA}^{-1}$$



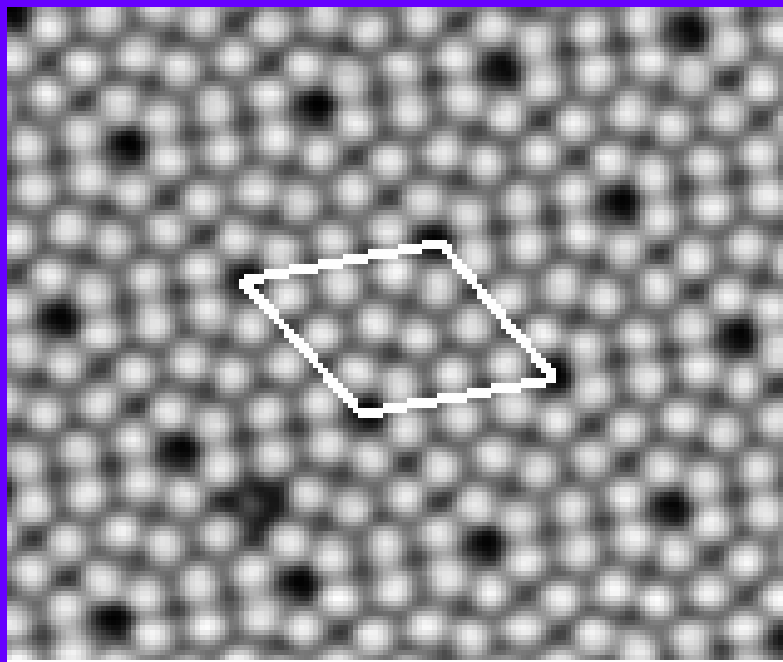


# UHV STM

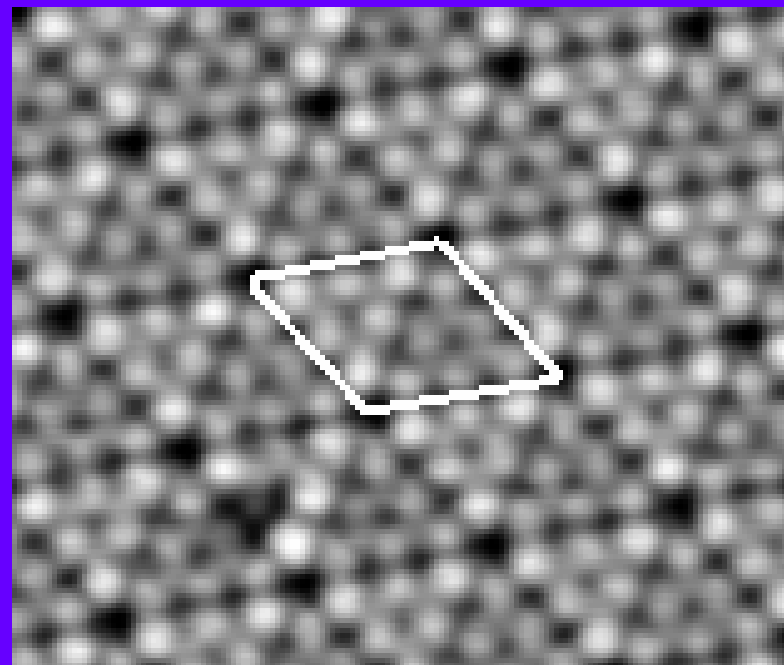




## *STM Images of Si(111)-(7×7)*



Empty-state image

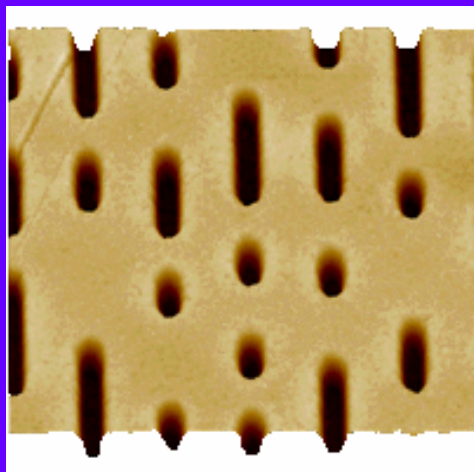


Filled-state image

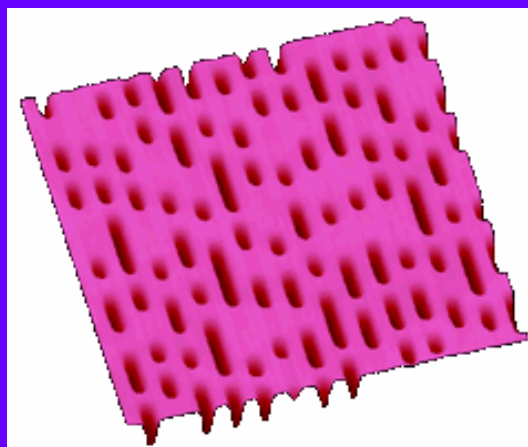


## *AFM images*

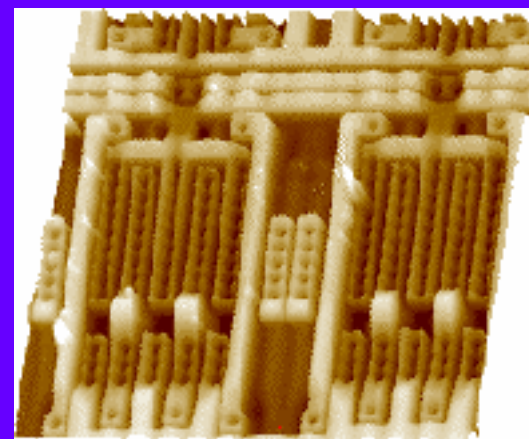
CD pits



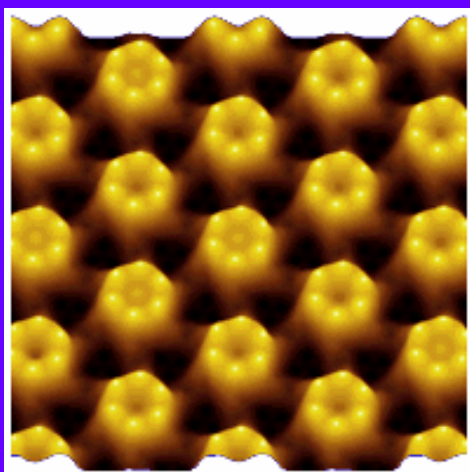
DVD pits



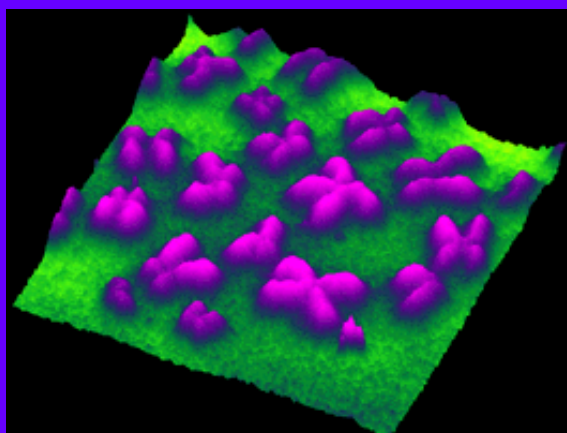
Integrated Circuit



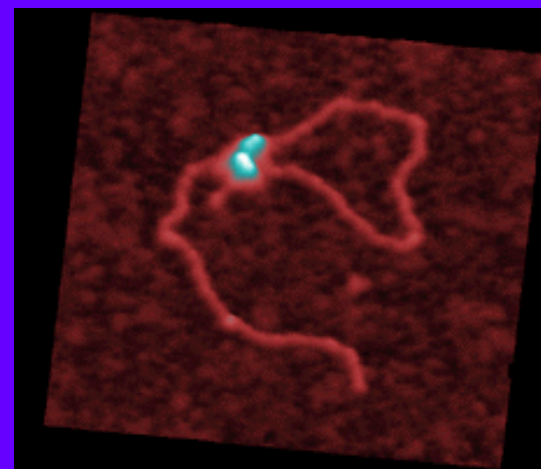
Bacteria



Chromosomes



DNA

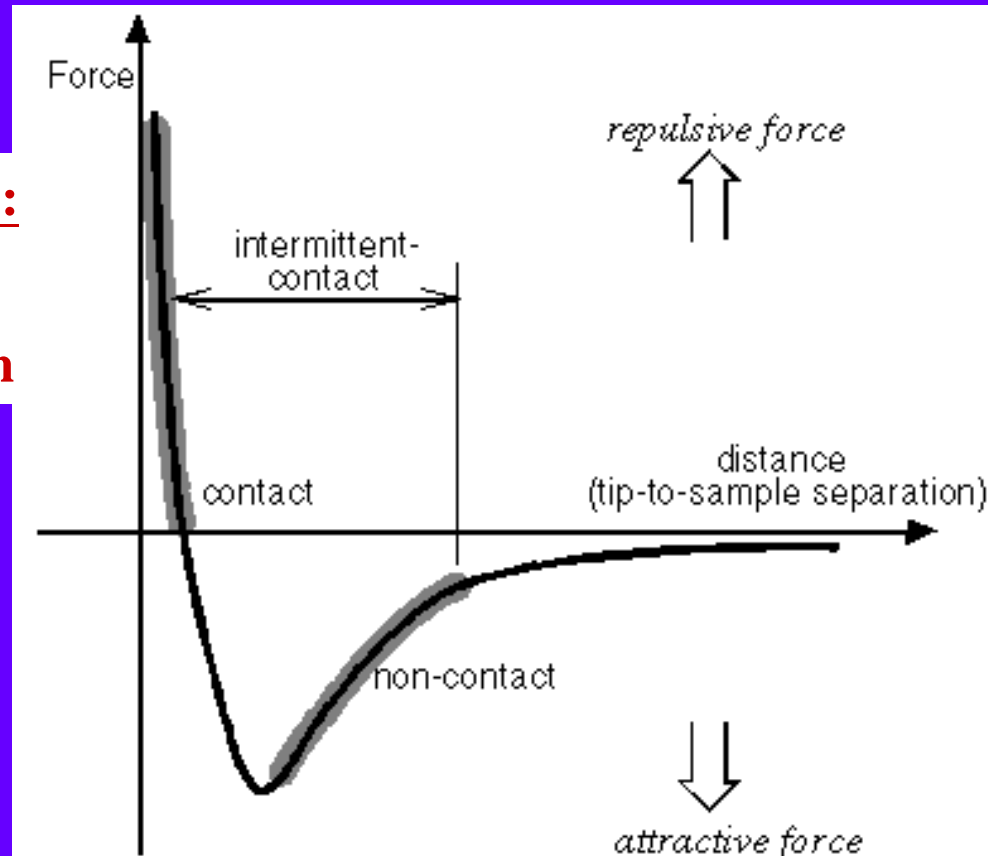




# Interaction between the probe and sample

## Short-range:

- 1) Bonding
- 2) Repulsion



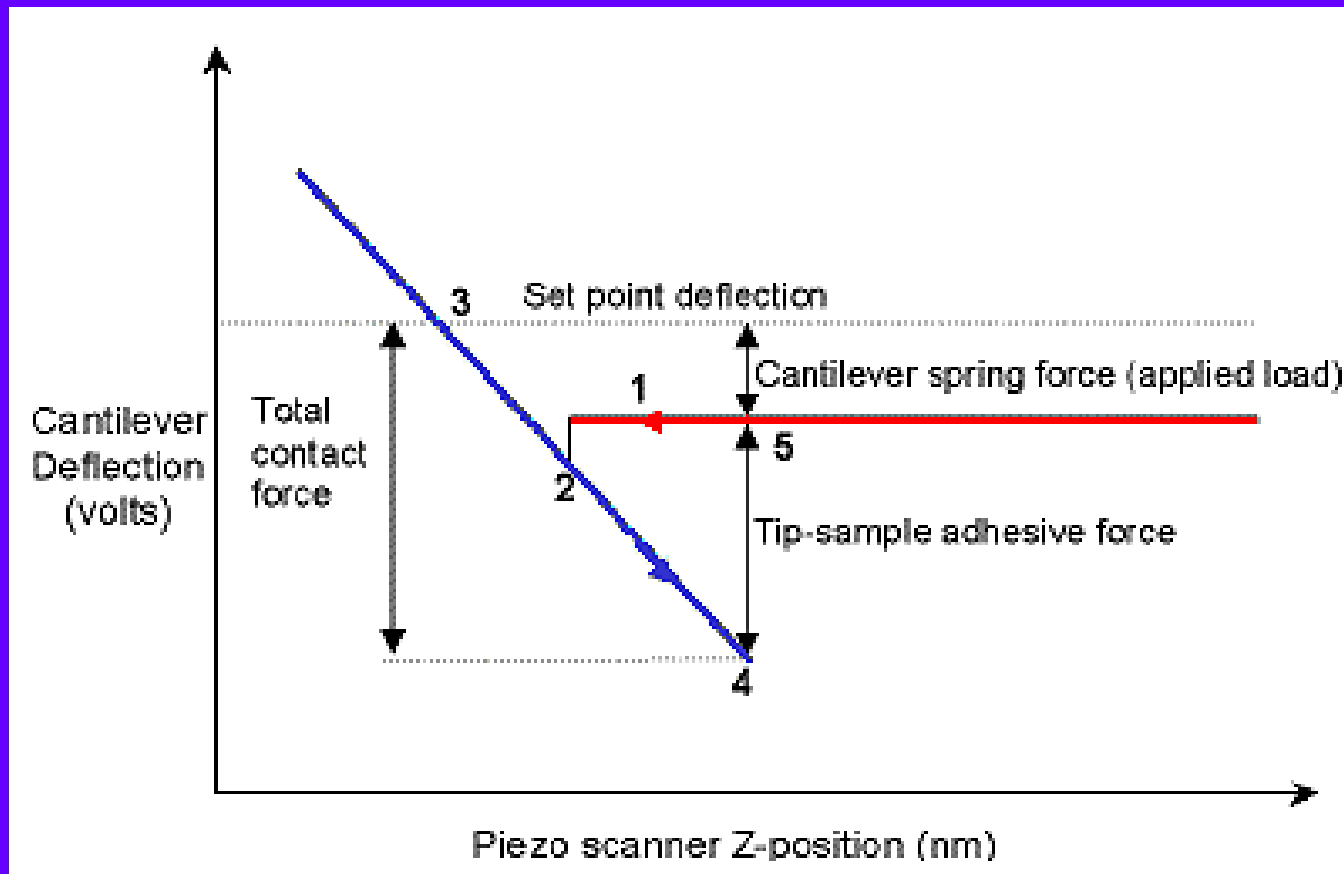
## Long-range:

- 1) Van der Waal
- 2) Capillary
- 3) Magnetic
- 4) Electrostatic

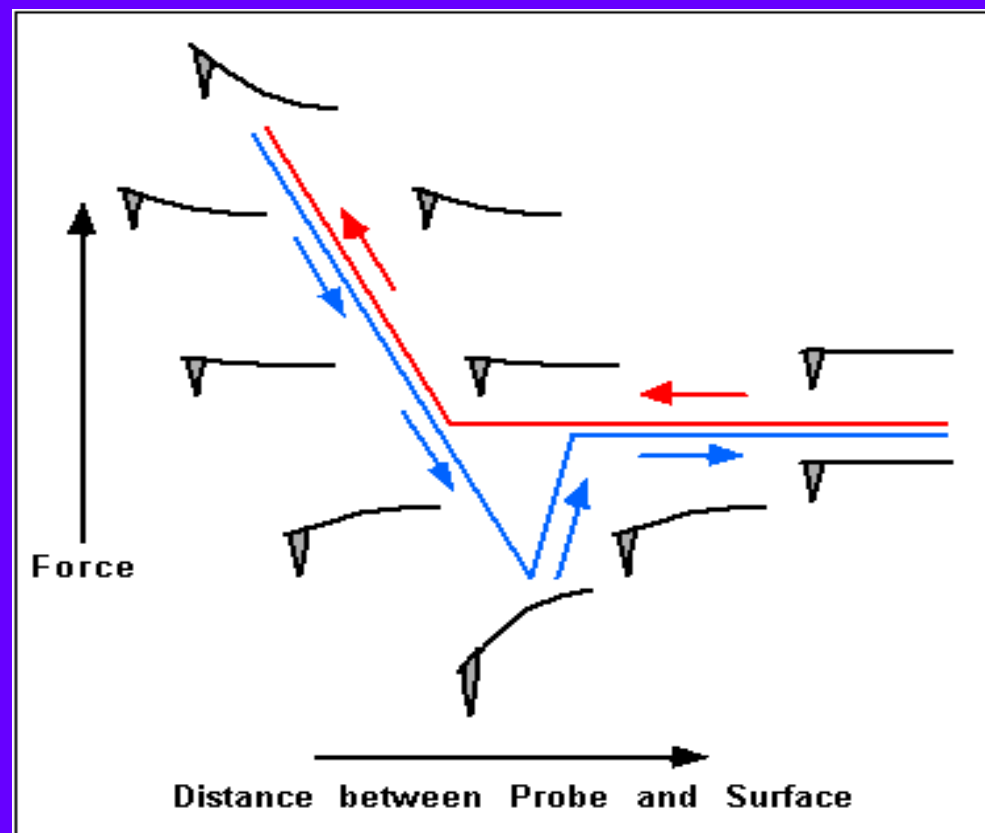
Lennard-Jones potential  $\phi(r) = - A/r^6 + B/r^{12}$



# Deflection of Cantilever vs Piezo displacement

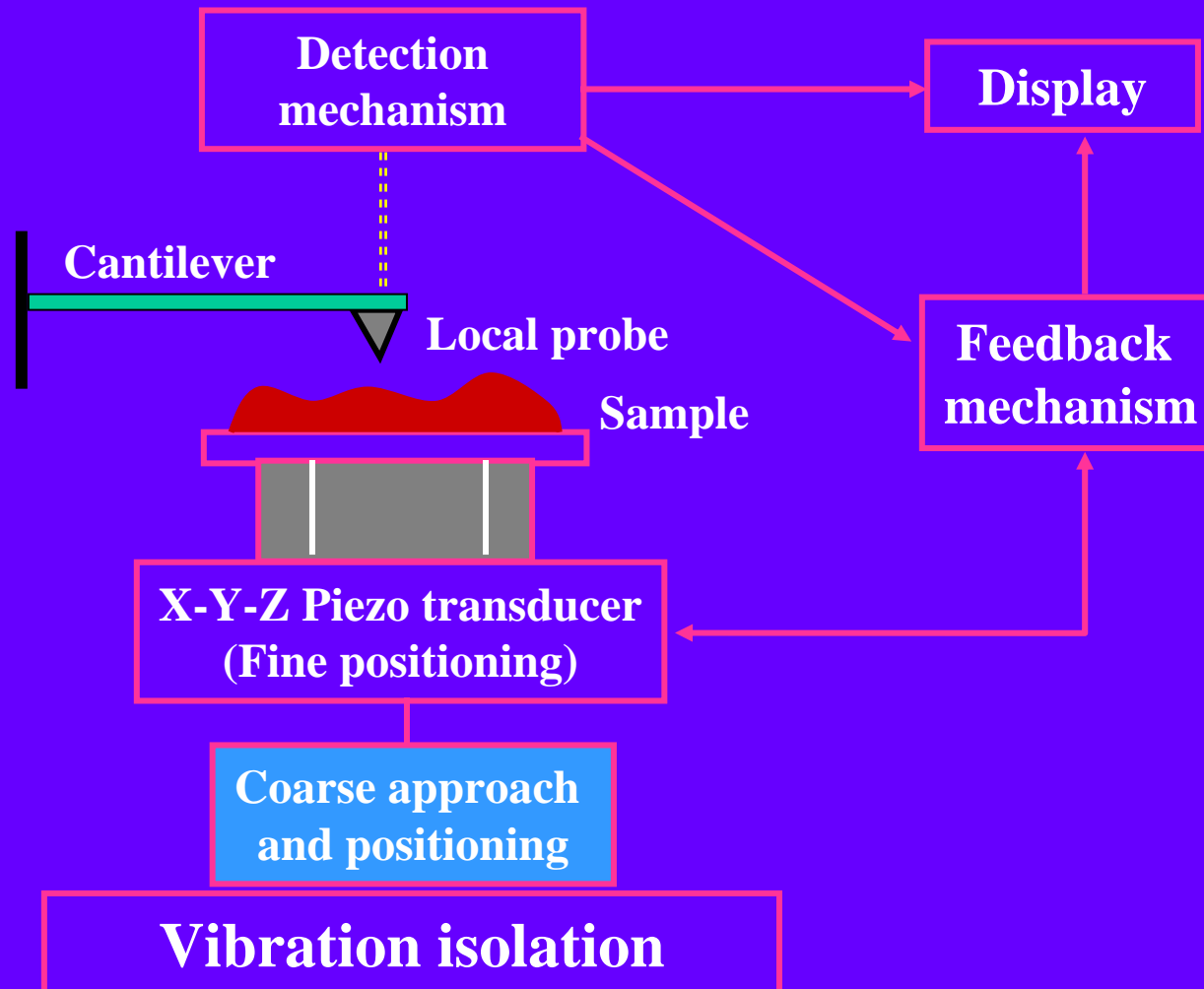


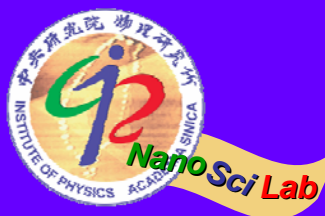
## Reaction of probe to the force



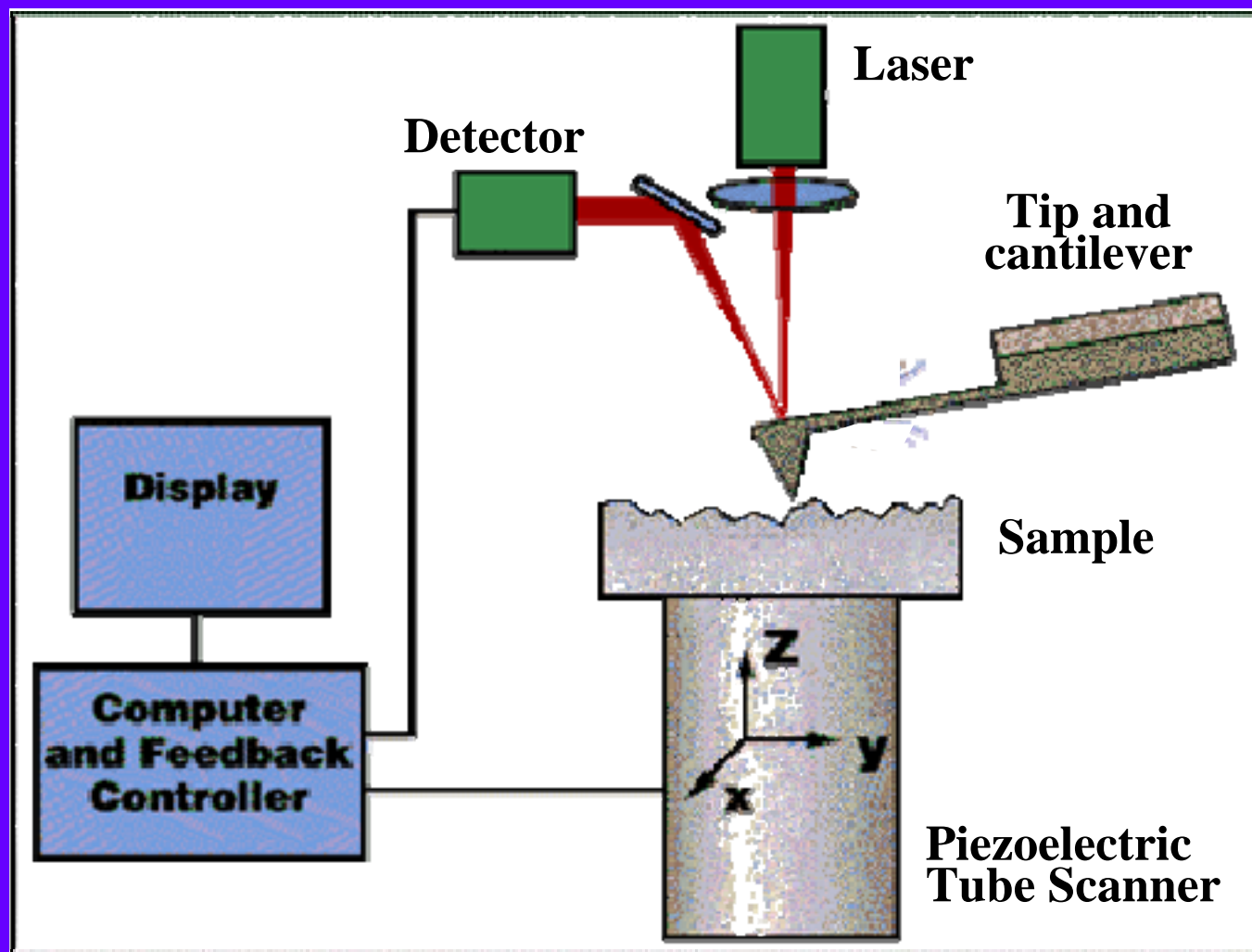


# Basic configuration of AFM



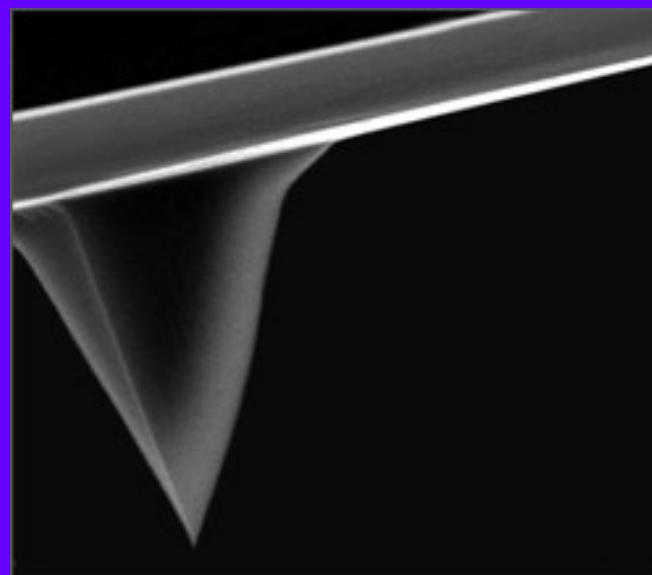
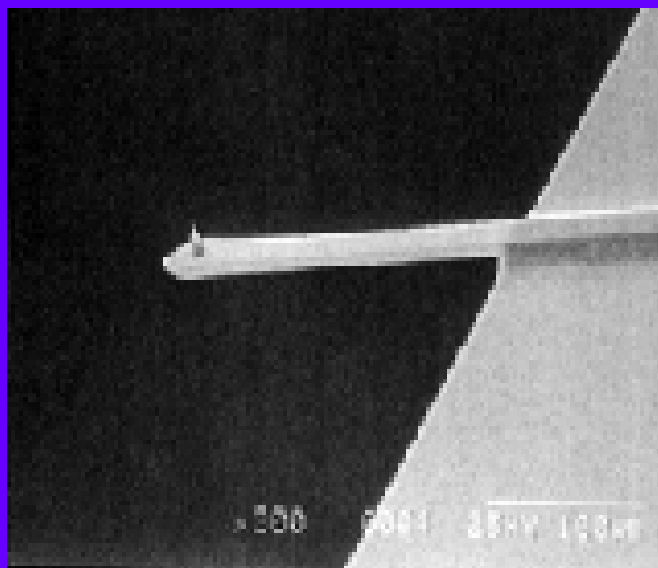


# Core components of an AFM

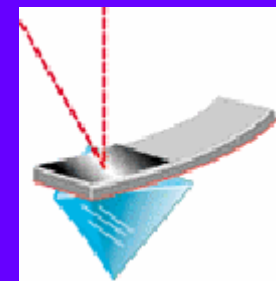




## Probes for the tapping mode



Typical Tip Dimension:  
 $150\mu\text{m} \times 30\mu\text{m} \times 3\mu\text{m}$   
 $f_r \sim 100 \text{ kHz}$   
Materials: Si





# Comparisons among various microscopies

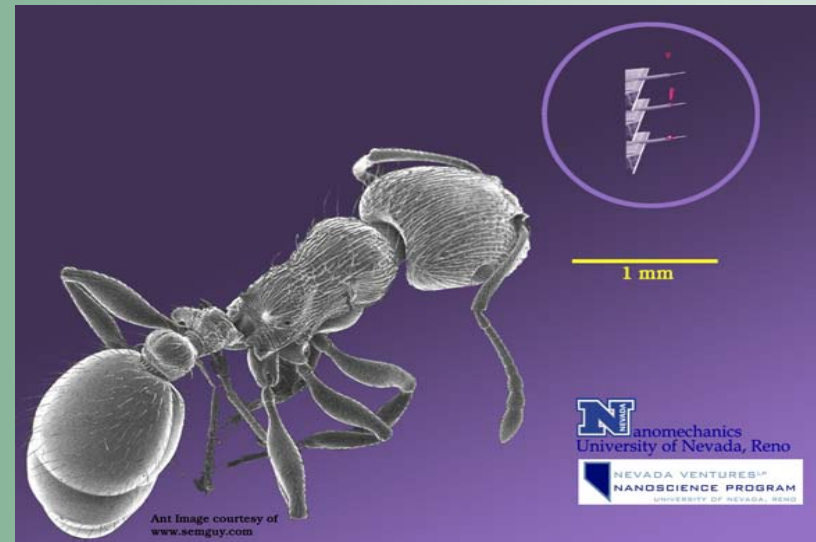
	光學顯微術 (OM)	掃描電子顯微術 (SEM)	穿透電子顯微術 (TEM)	掃描探針顯微術 (SPM)
lateral	300nm	1nm	0.1nm	0.1nm
vertical	20nm	10nm		0.01nm
Imaging area	1mm	1mm	0.1mm	0.1mm
Imaging environ	none	vacuum	vacuum	none
Sample prep	no	yes	yes	no
Elemental anal	yes	yes	yes	no

# Molecular Scale Nanomechanics

## *Driving Force in Bio-systems*

At the fundamental level, all interactions in biology and chemistry are mechanical in nature

### Mechanical Sensing in Nature



Hair bundle:  
frog's inner ear



# Micro and Nano cantilever arrays - *Emulating nature*

- Ideal displacement sensor
  - Sub nm sensitivity
- Displacement  $\sim$  force
- Surface stress, temperature
- Mass loading

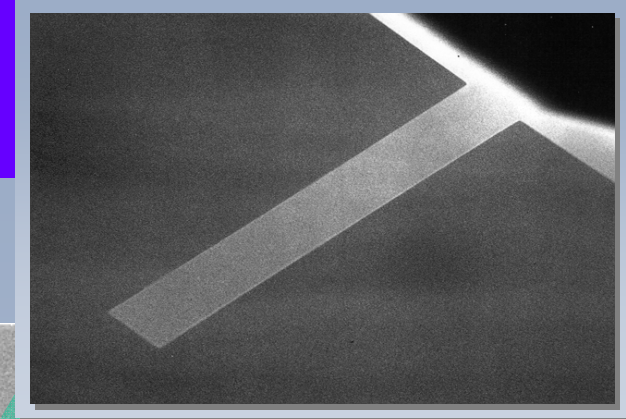
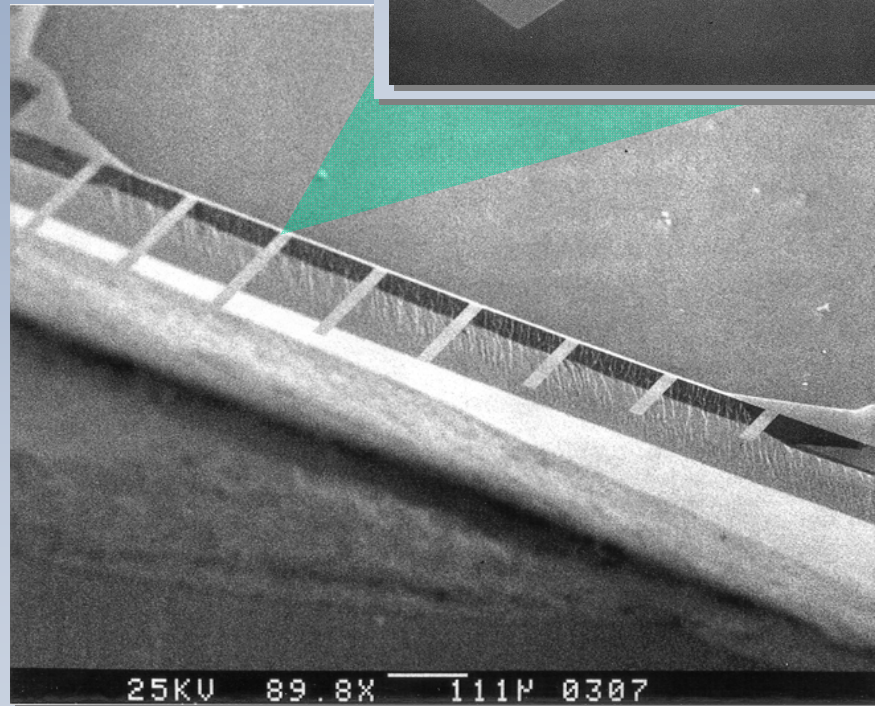
Sensitivity:

Function of dimensions

Selectivity:

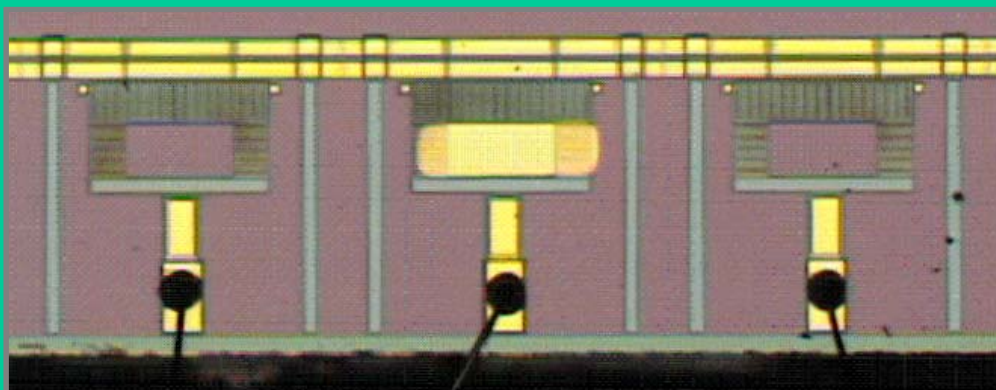
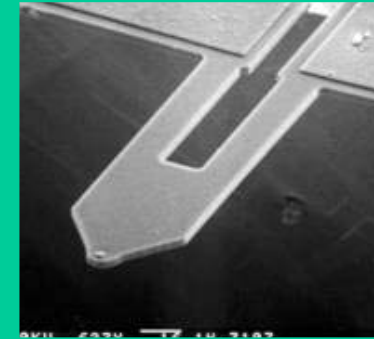
Function of coatings

- Mass production



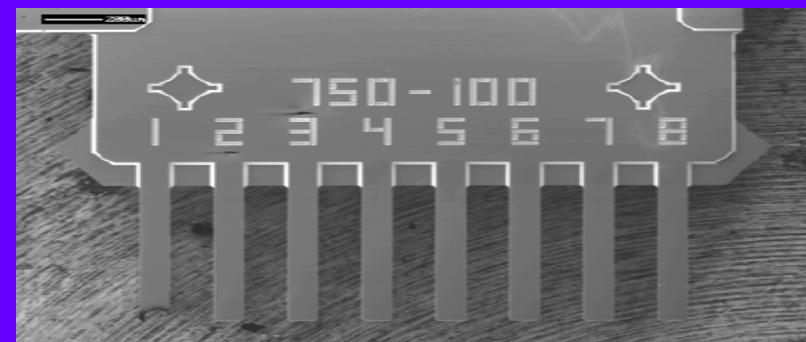
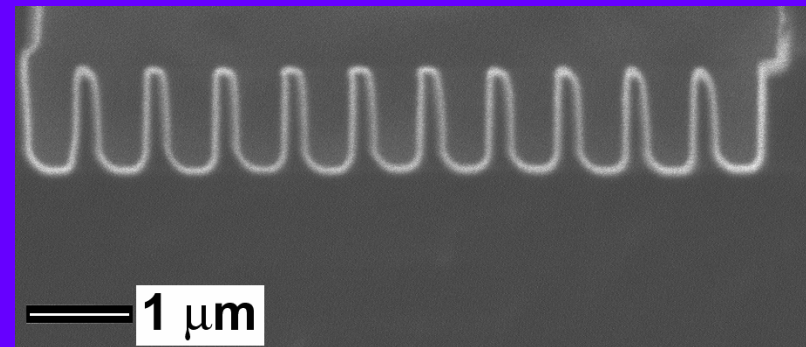
# ***Microcantilevers:*** **Getting the signal out**

- Optical
- Piezoresistivity
- Piezoelectricity
- Capacitance



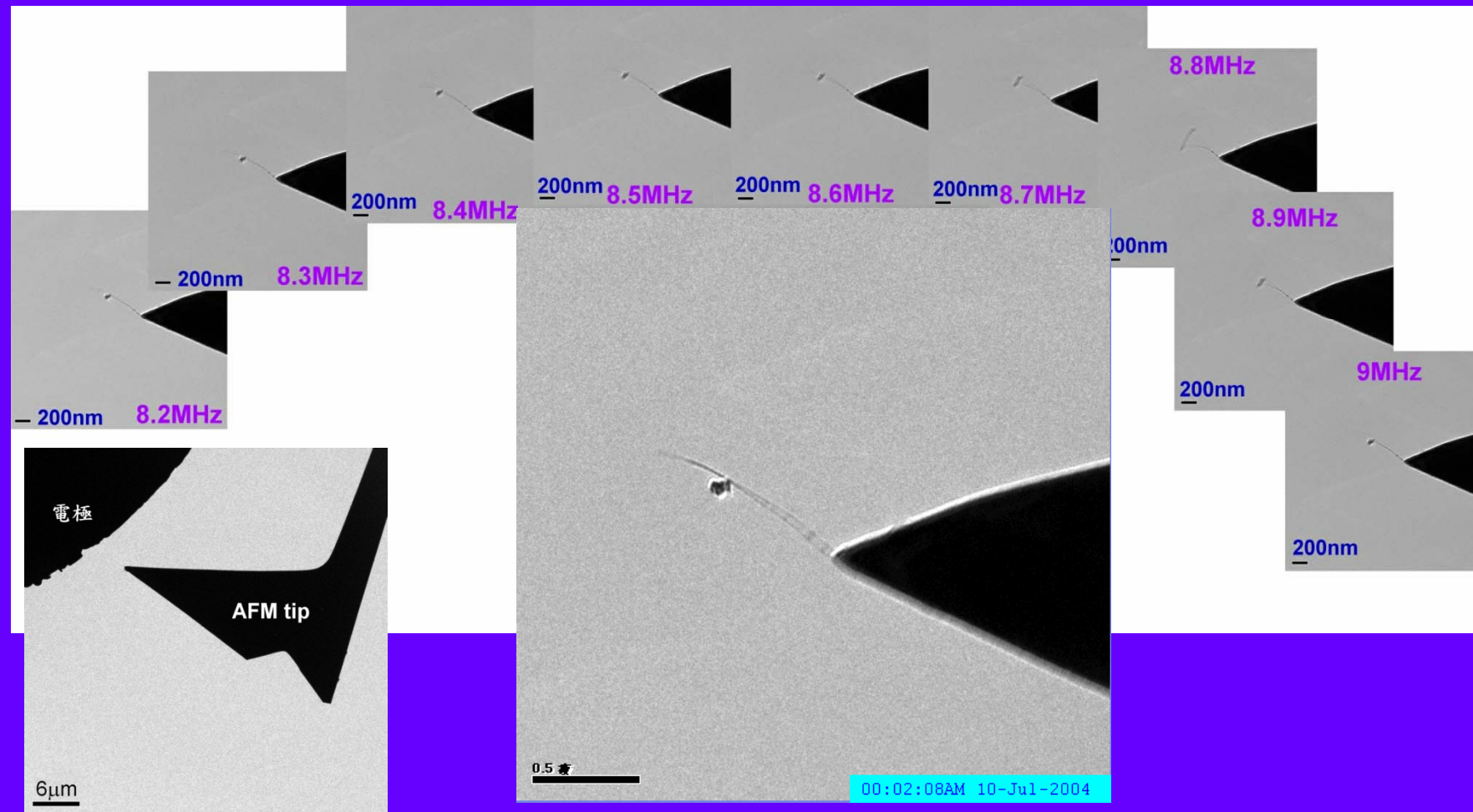
# Microcantilevers To Nanocantilevers

- Increased Sensitivity
- High Resonance Frequency
- Small Spring Constants
- Single Molecule Detection
- Challenges:
  - Signal Transduction
  - Mass production

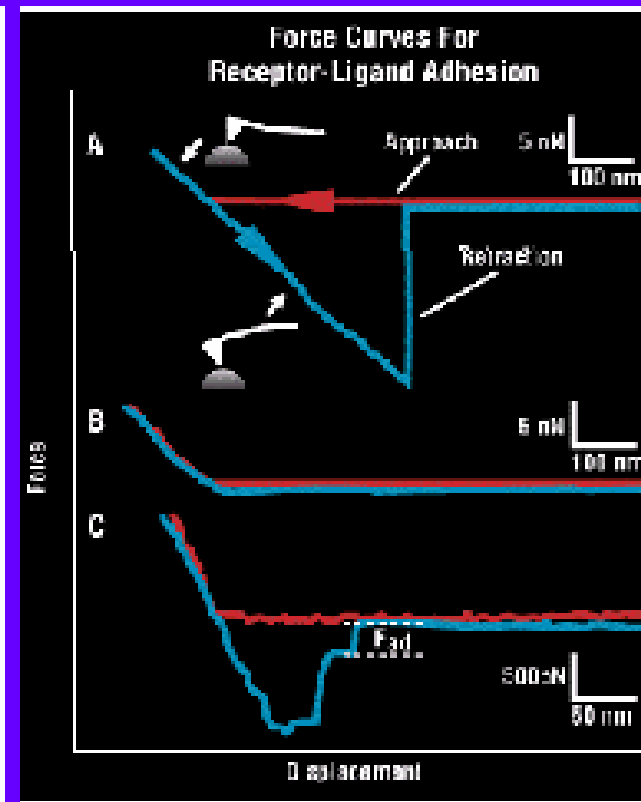
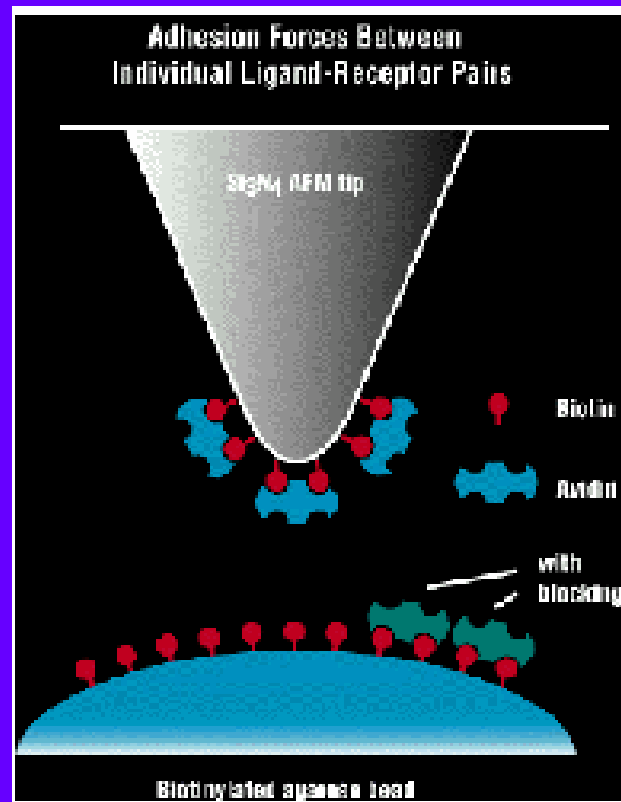


(Craig Prater, DI)

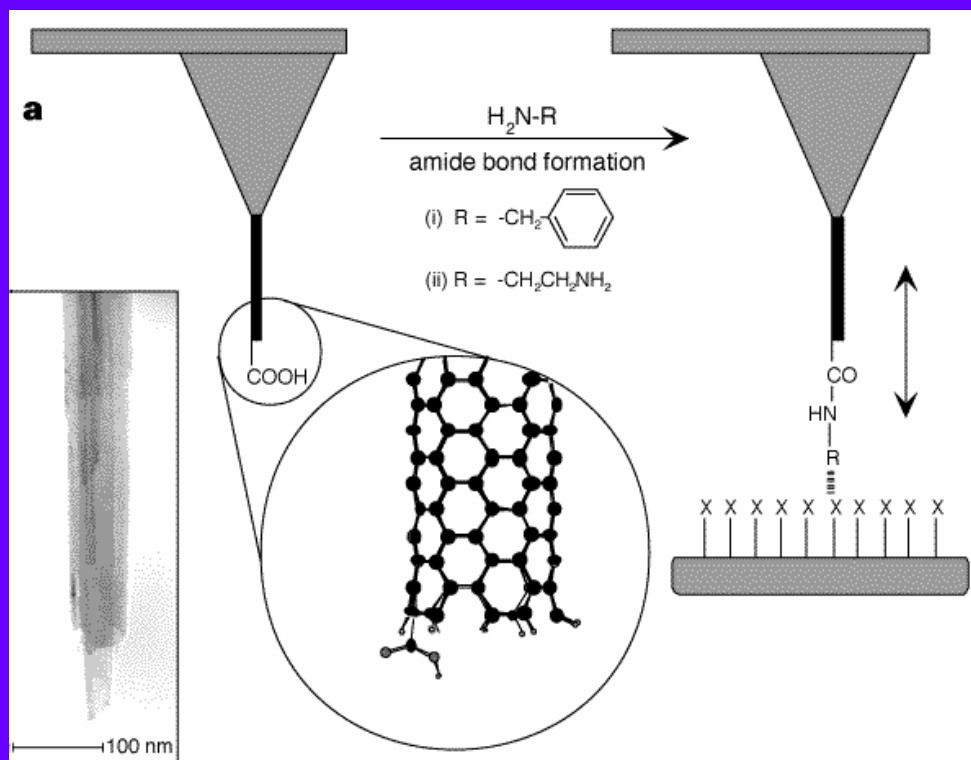
# Nanoelectromechanical oscillator



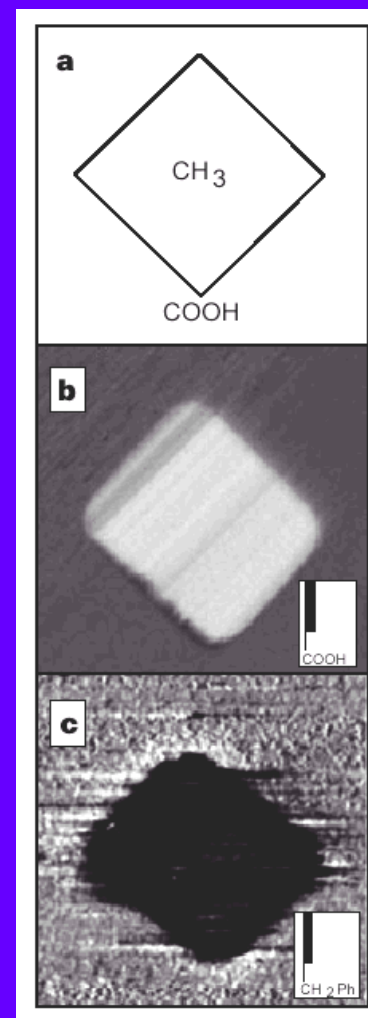
# Molecular sensing



# Chemical nanoprobe



S. S. Wong *et al.*, *Nature* 394, 52 (1998).

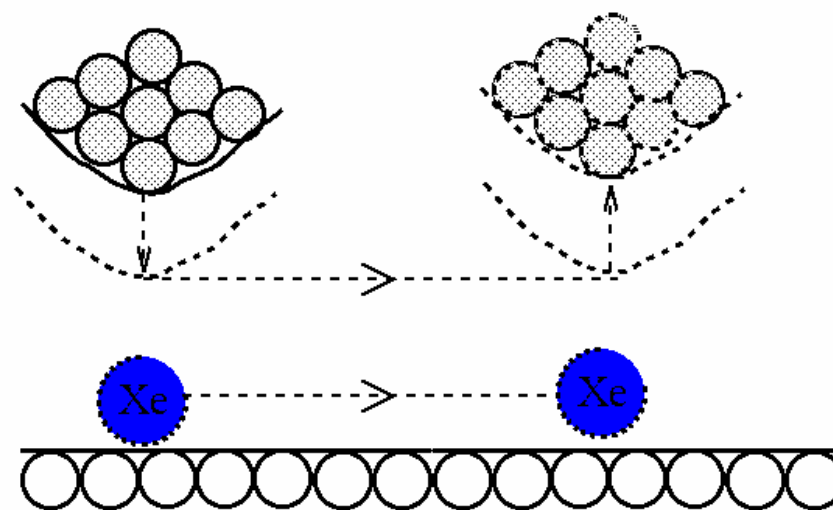




# Manipulation method

## Positioning Atoms with an STM

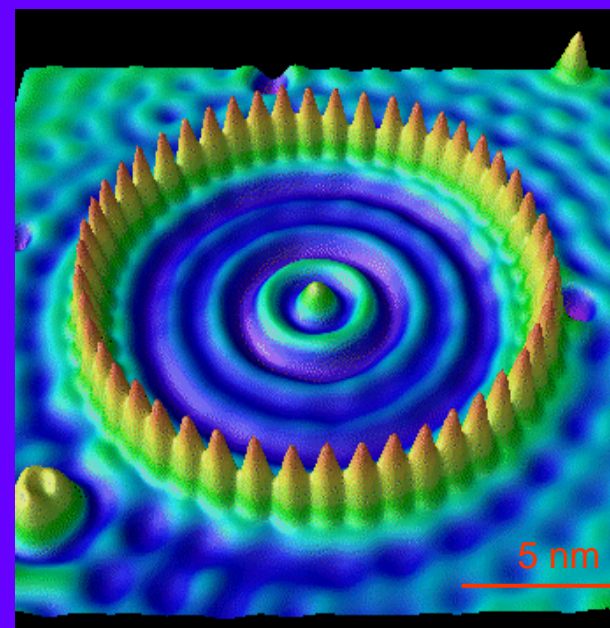
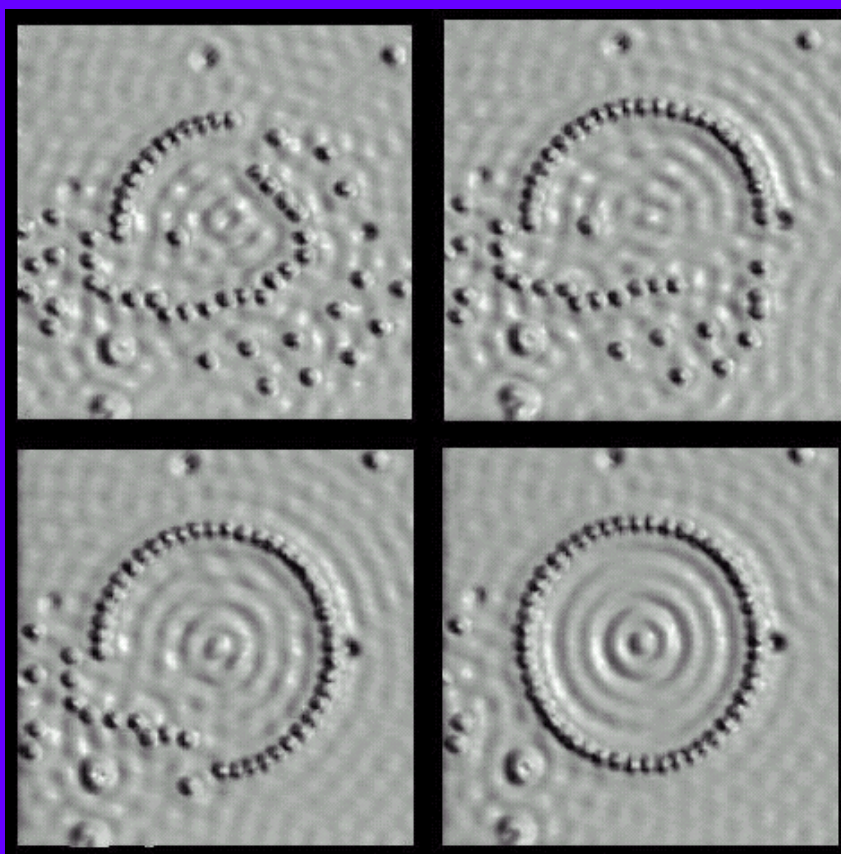
D.M. Eigler & E.K. Schweizer Nature **344** 524 (1990)



The STM tip is brought down near the atom, until the attraction is enough to hold it as the atom is dragged across the surface to a new position.

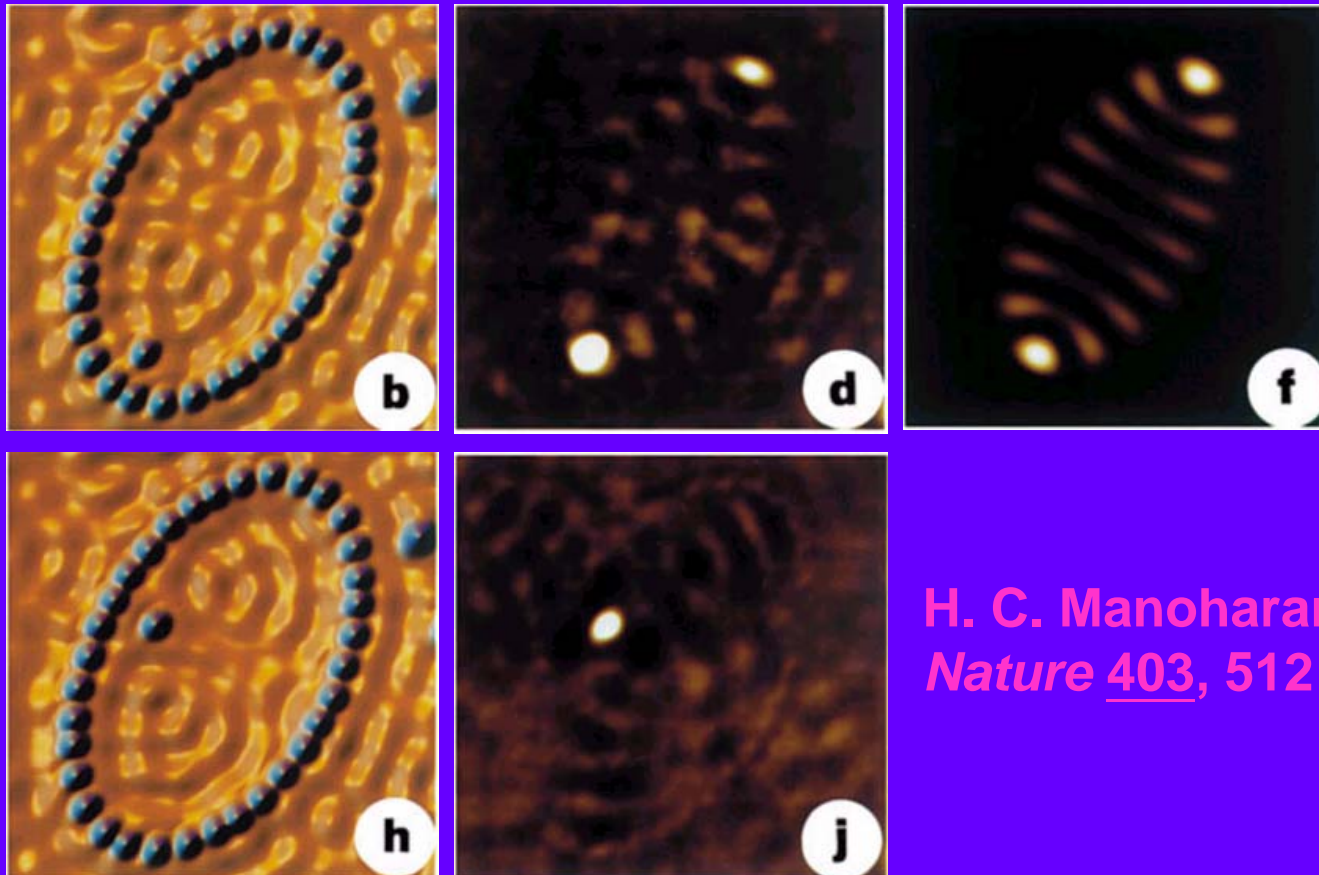


# Ultimate goal of nanotechnology



M.F. Crommie *et al.*,  
*Science* 262, 218 (1993).

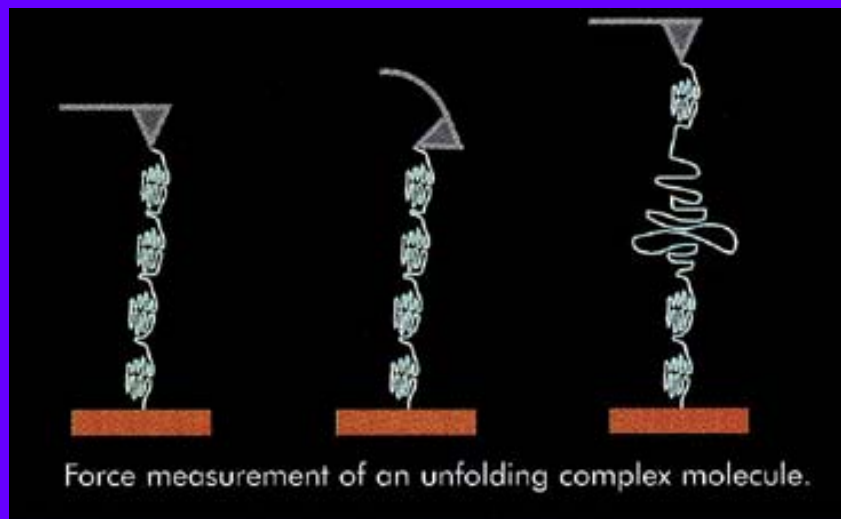
# Quantum Mirage



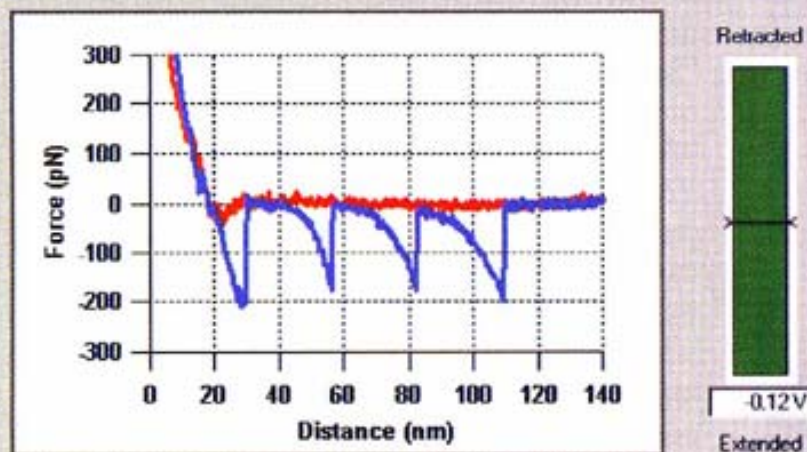
H. C. Manoharan *et al.*,  
*Nature* 403, 512 (2000).



# Molecular unfolding

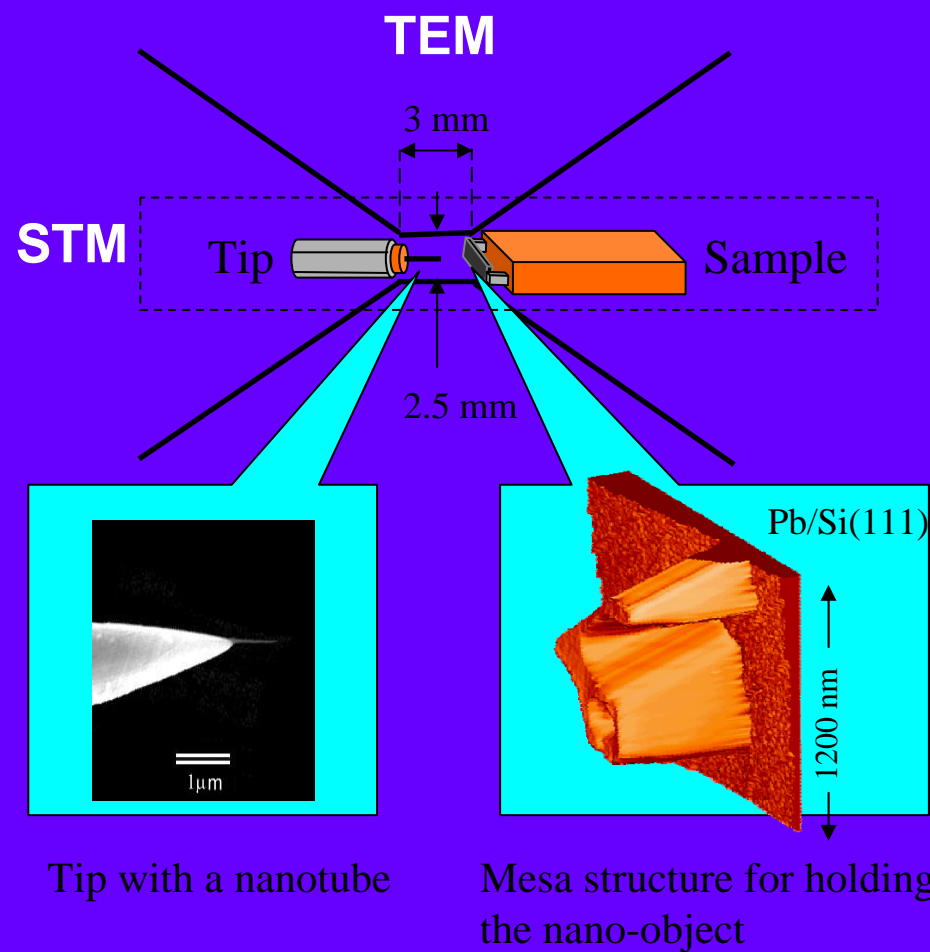
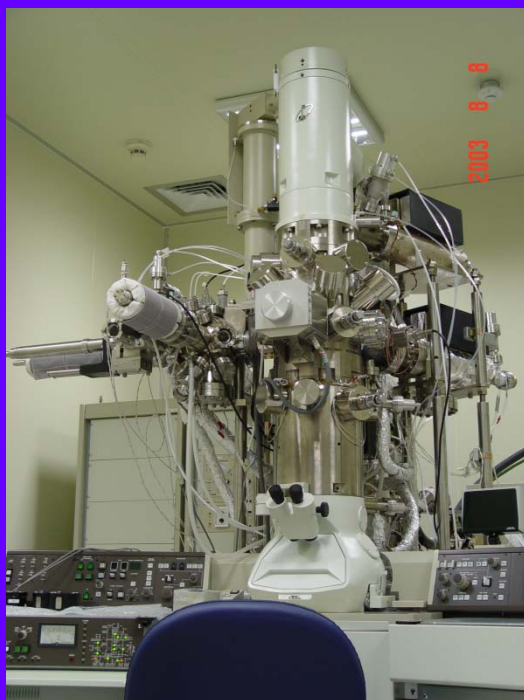


Force measurement of an unfolding complex molecule.

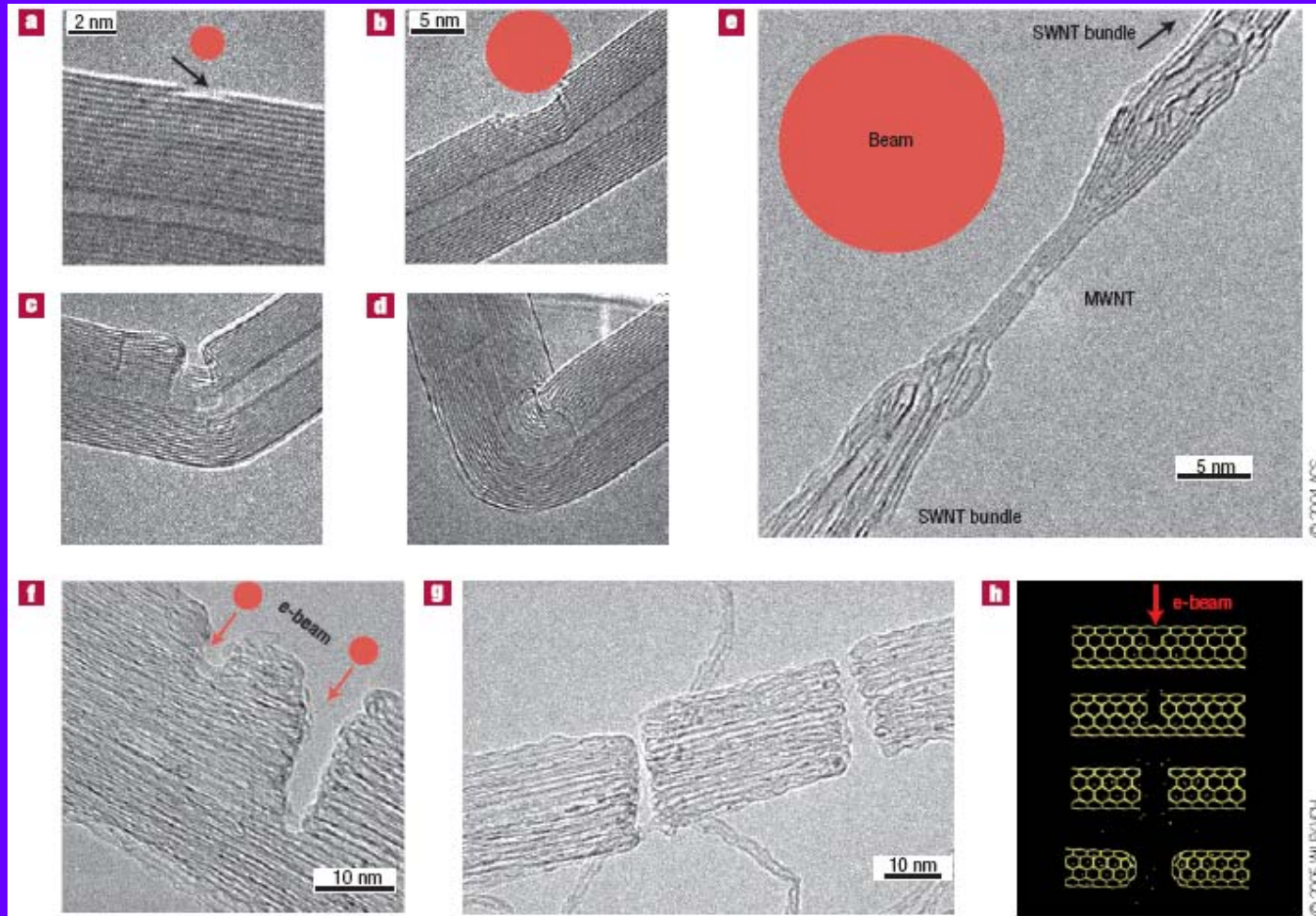


Advanced graphical user interface shows titin muscle molecule force curve.

# UHV HRTEM/STM



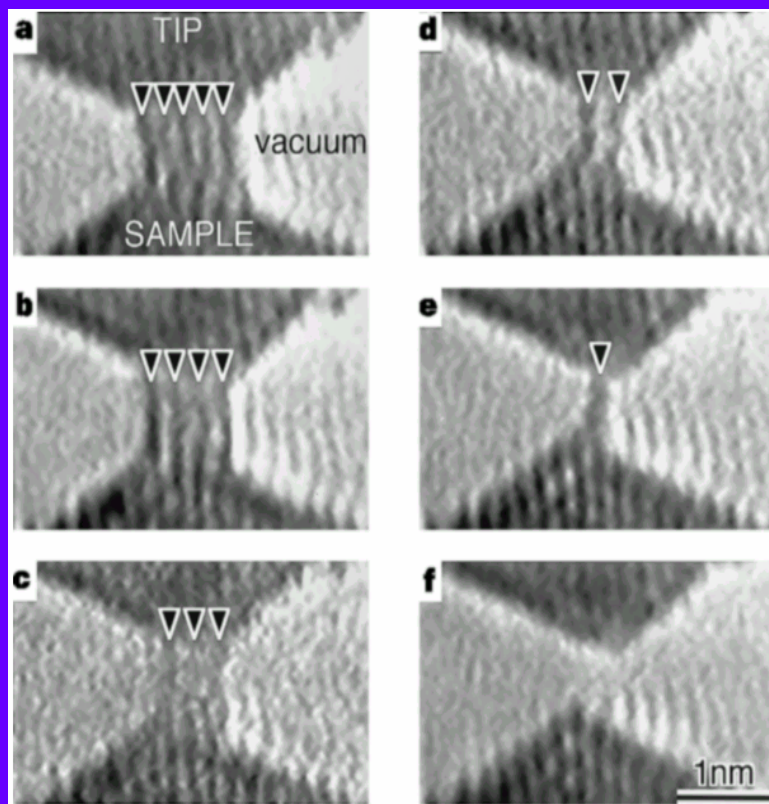
# Engineering CNT with energetic e-beam



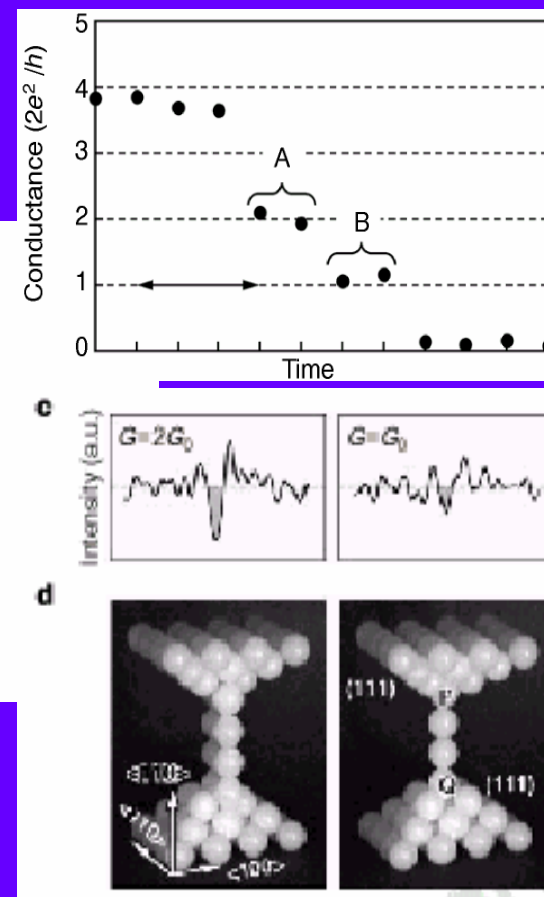
A.V. Krasheninnikov and F. Banhart  
*Nature Materials* **6**, 723 (2007)



# *Point contact of Au wire*

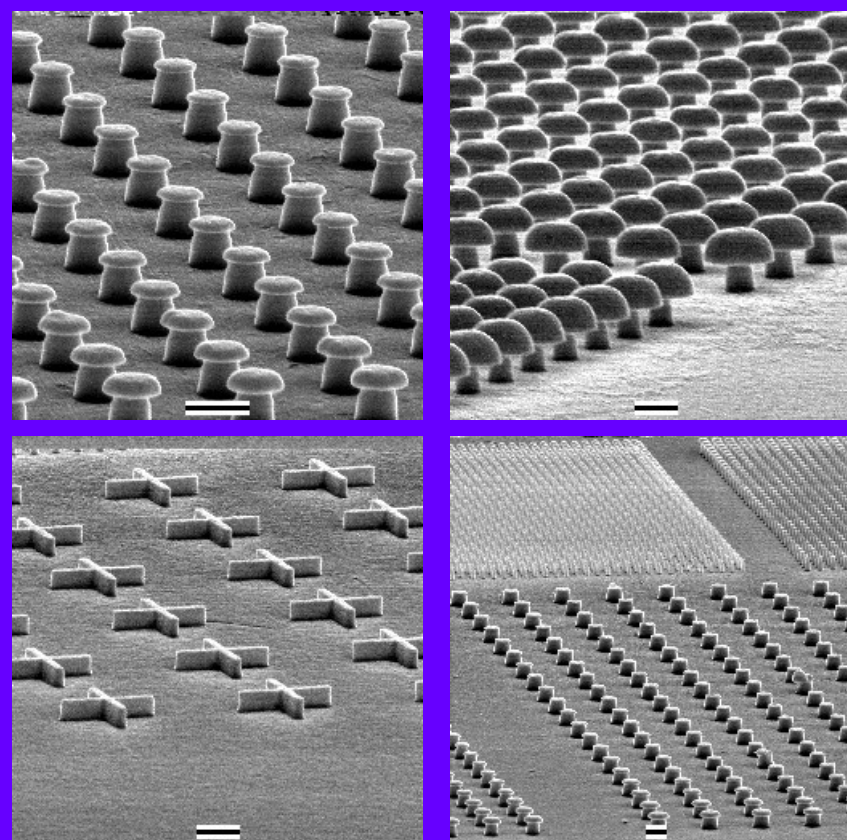
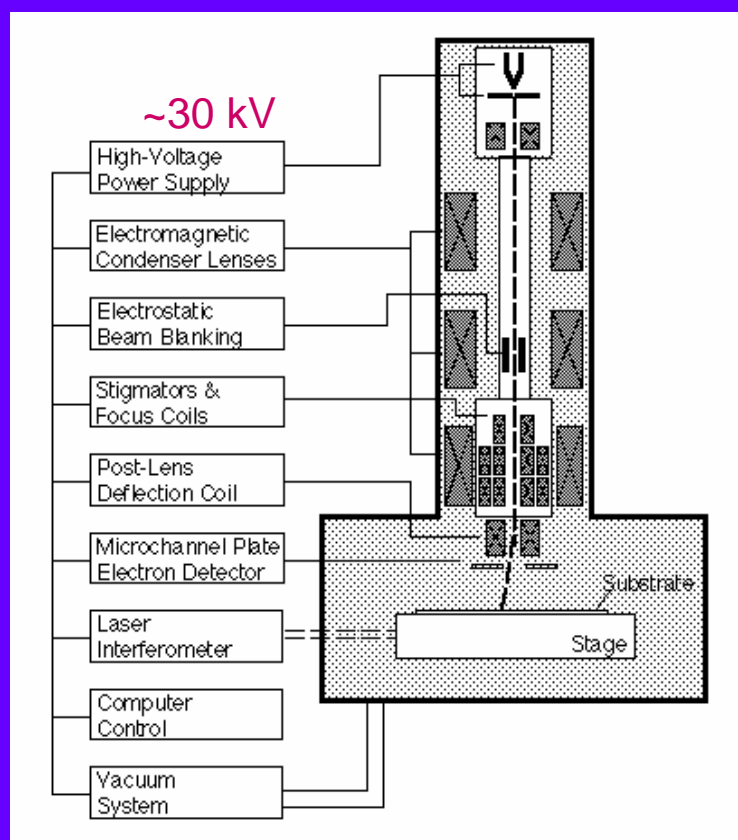


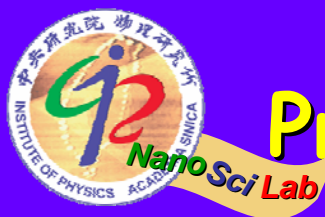
H. Ohnishi *et al.*  
 Nature 395, 780 (1998).



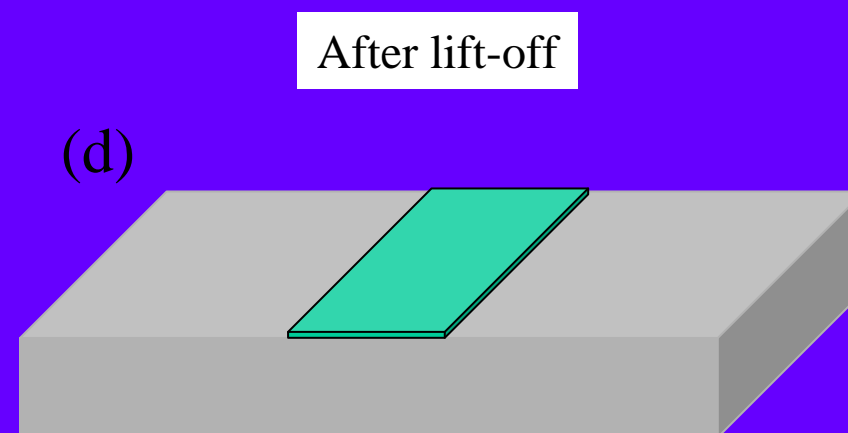
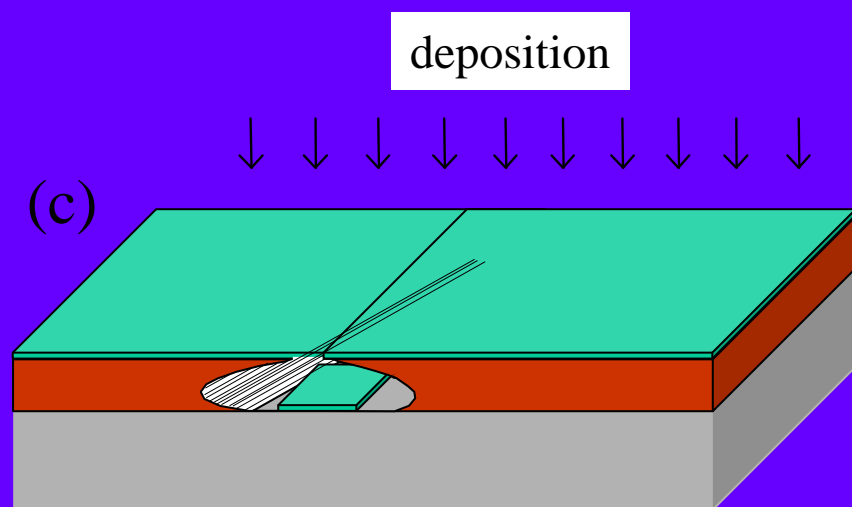
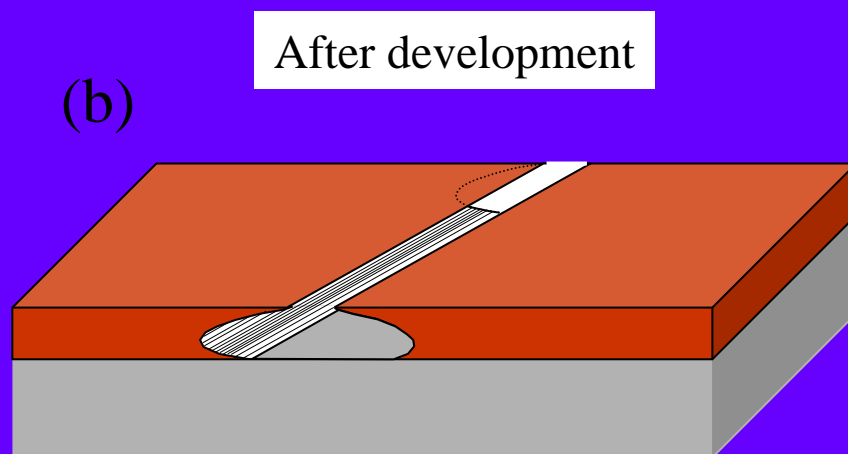
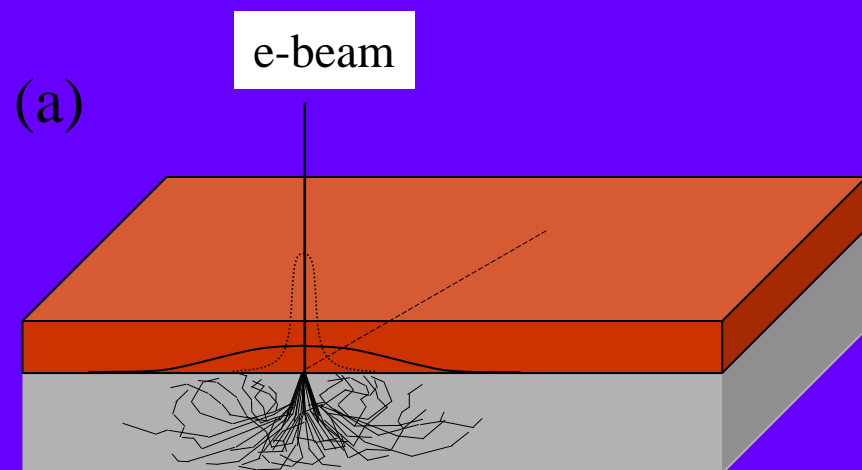


# Periodic nanostructures by e-beam lithography

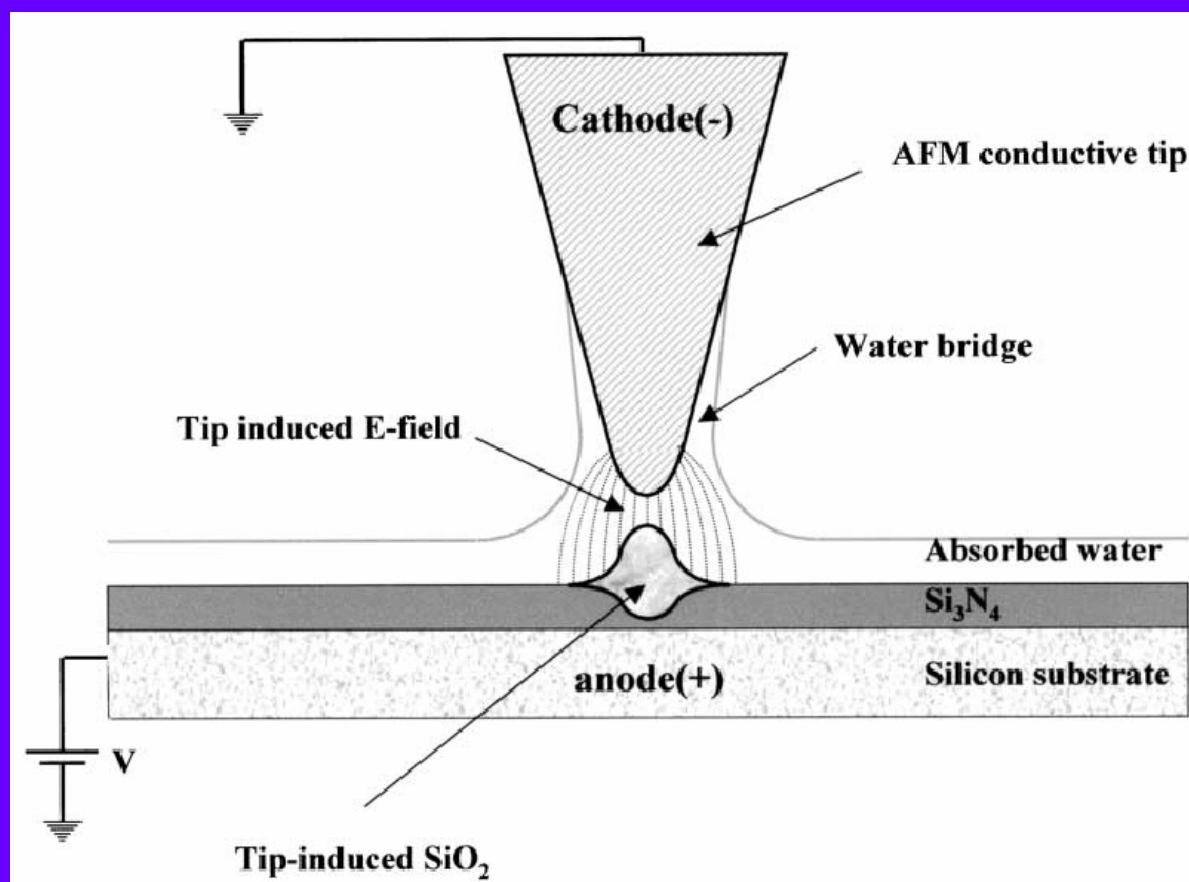




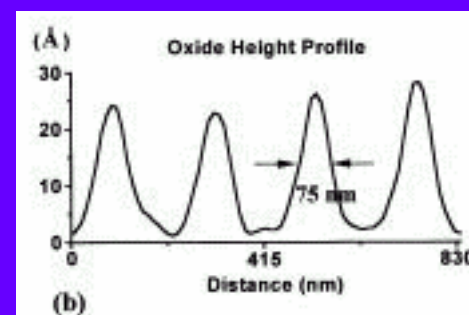
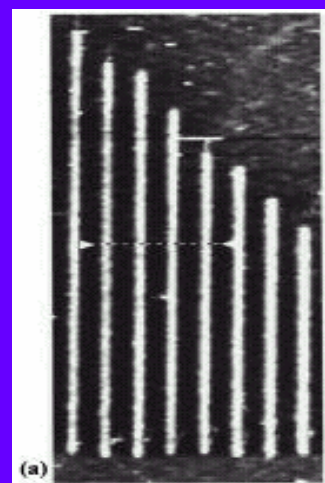
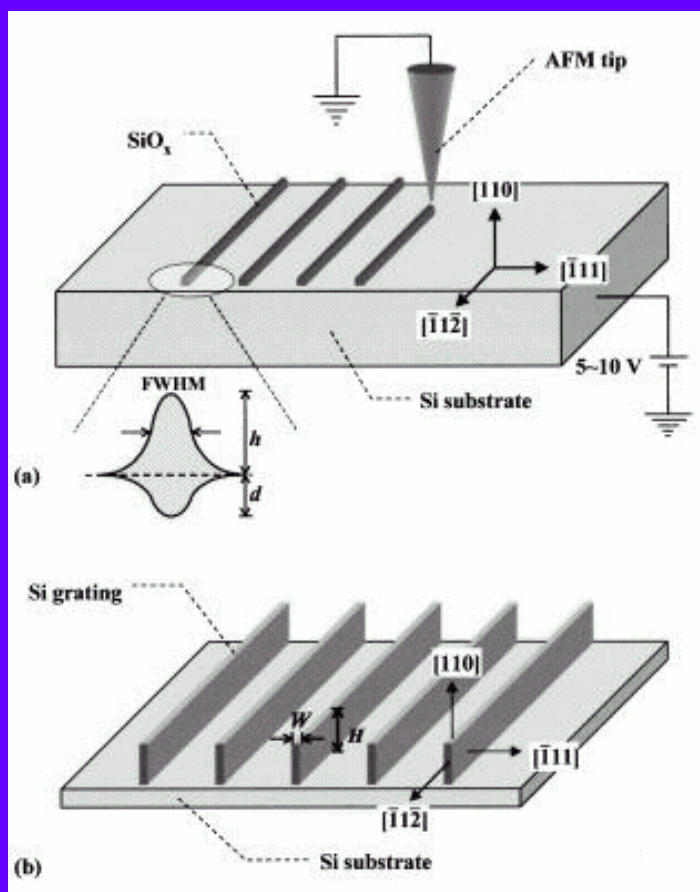
# Procedures of e-beam lithography



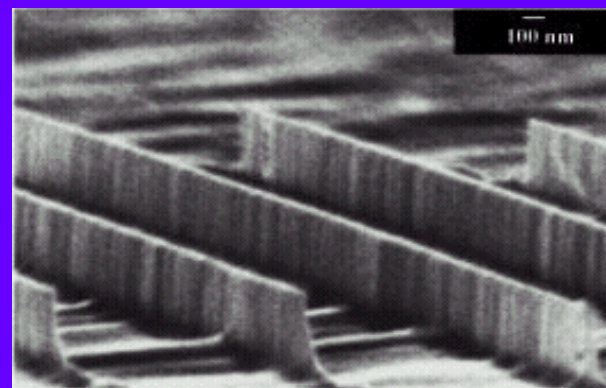
## *Nano-Lithography with an AFM tip*



# Electrical Lithography

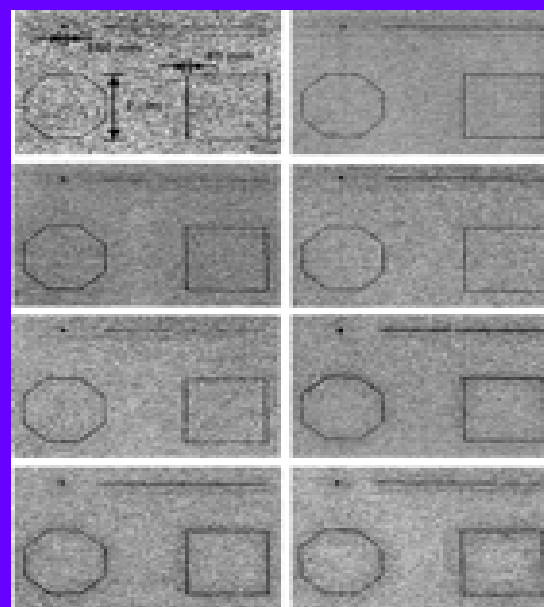
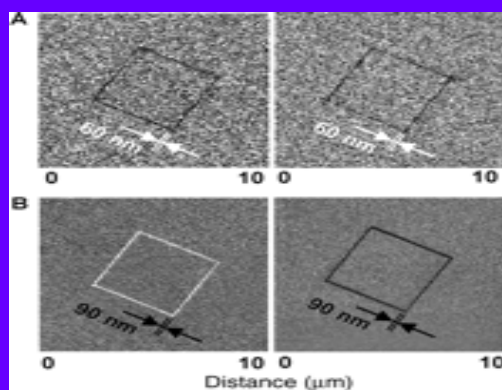
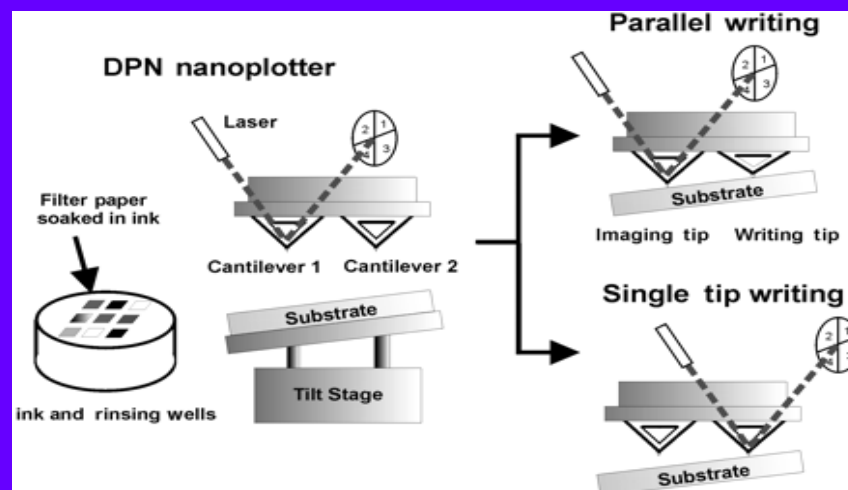


F.S.-S. Chien *et al.*  
 APL 75, 2429 (1999)





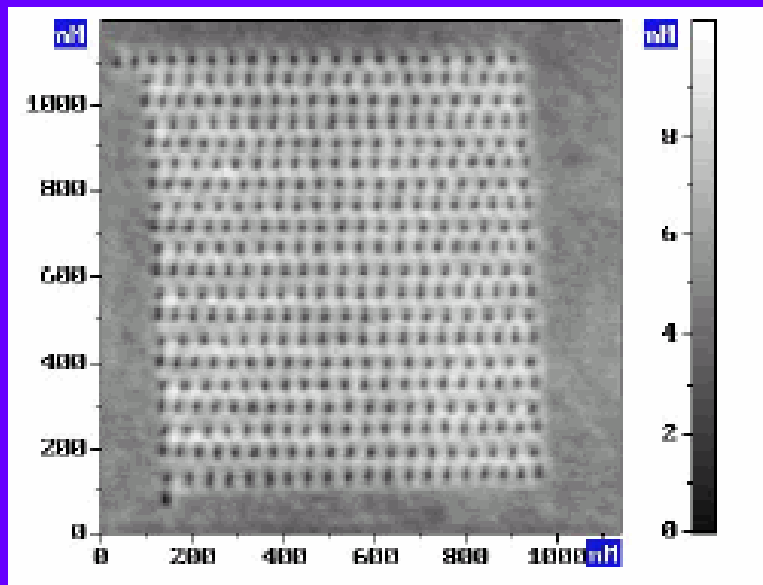
# Nanoplotter



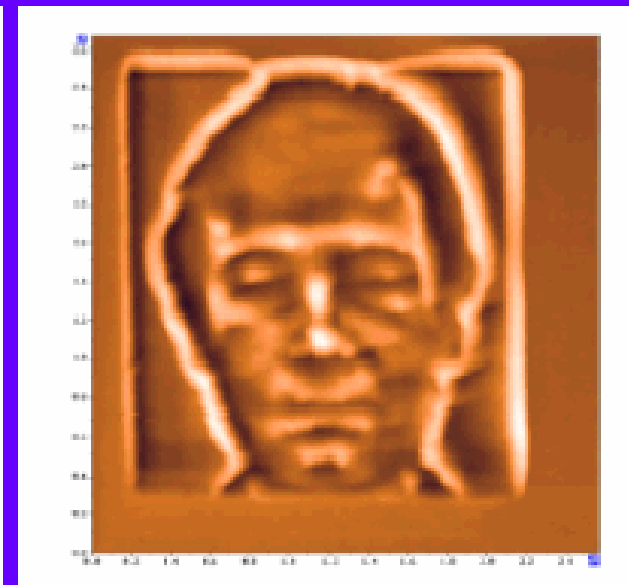
C.A. Mirkin *et al.*  
 Science 288, 1808 (2000)



## *Nanolithography of Tapping-Mode AFM*



$(1.2 \mu\text{m} \times 1.2 \mu\text{m})$

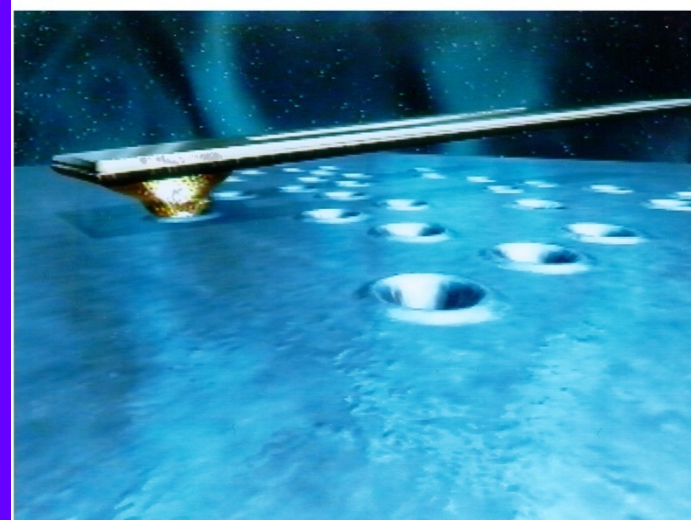
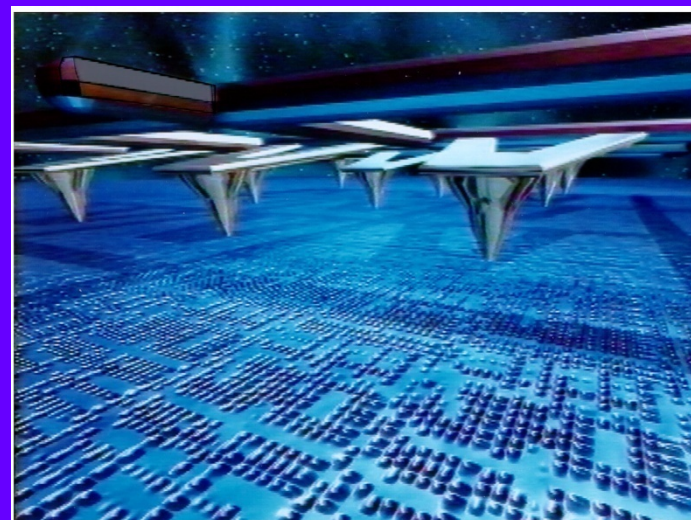


$(2.5 \mu\text{m} \times 2.5 \mu\text{m})$

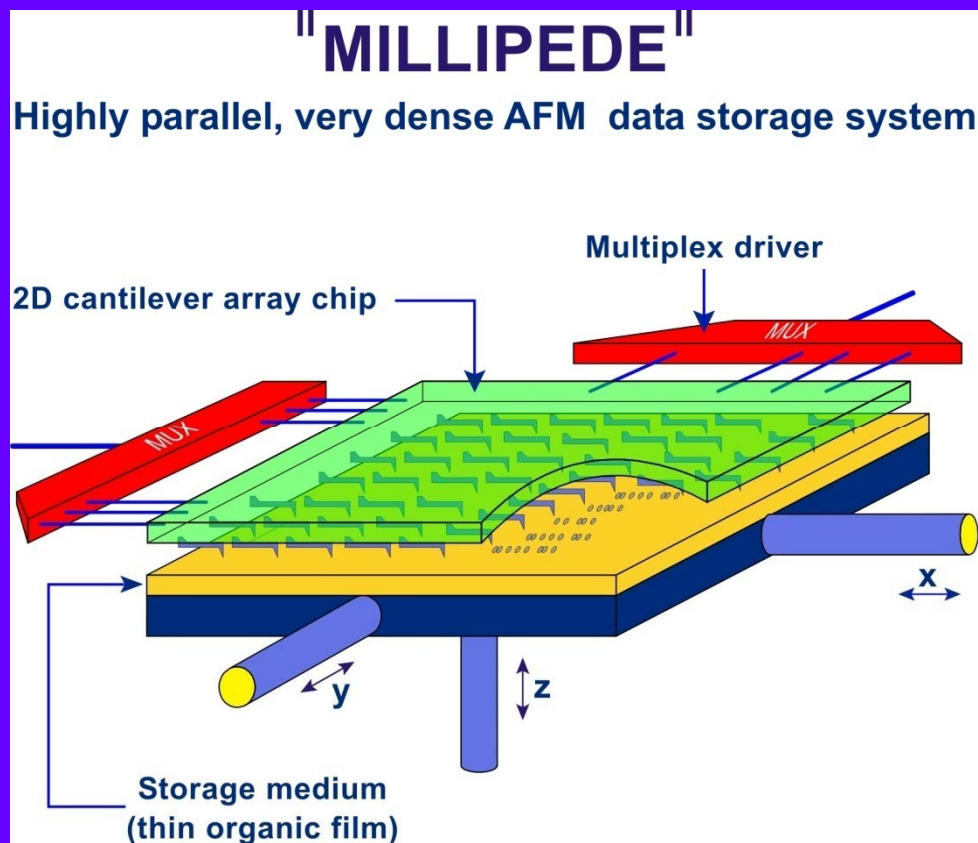
Image of polycarbonate film on silicon surface



# Nanodrive



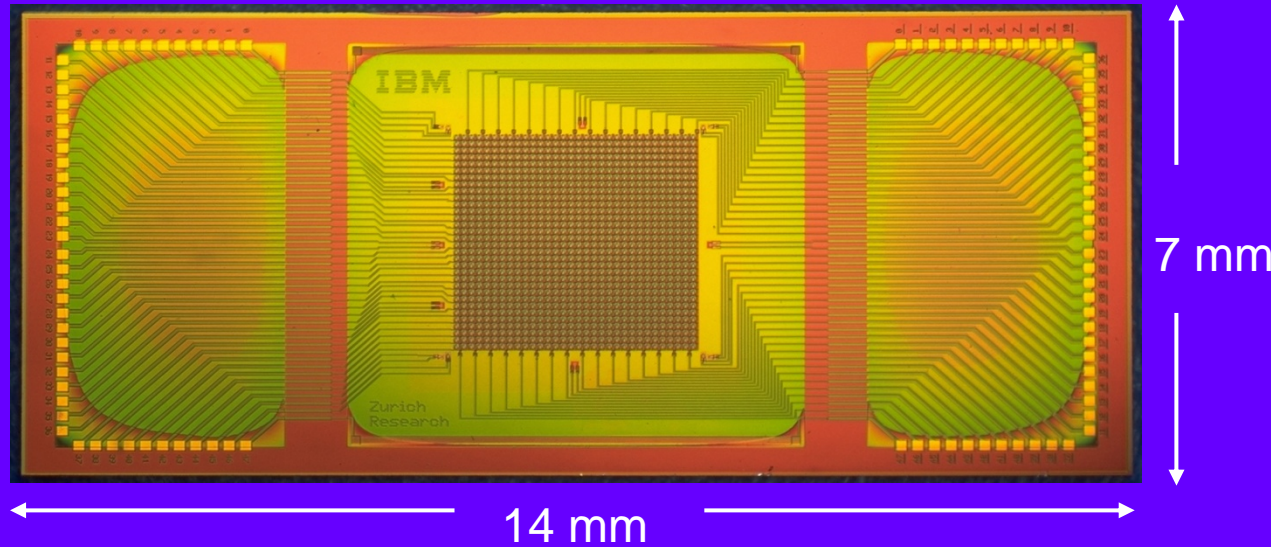
# Millipede



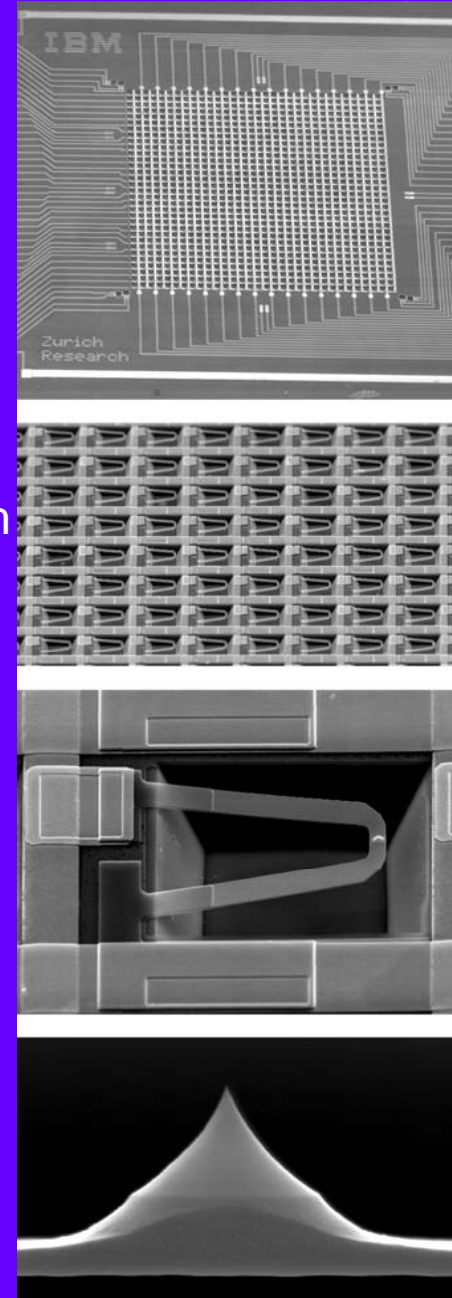
**The Millipede concept:** for operation of the device, the storage medium - a thin film of organic material (yellow) deposited on a silicon "table" - is brought into contact with the array of silicon tips (green) and moved in x- and y-direction for reading and writing. Multiplex drivers (red) allow addressing of each tip individually.

IBM-Zurich

# Cantilever array



**Millipede cantilevers and tips:** electron microscope views of the 3 mm by 3 mm cantilever array (top), of an array section of 64 cantilevers (upper center), an individual cantilever (lower center), and an individual tip (bottom) positioned at the free end of the cantilever which is 70 micrometers (thousands of a millimeter) long, 10 micrometers wide, and 0.5 micrometers thick. The tip is less than 2 micrometers high and the radius at its apex smaller than 20 nanometers (millionths of a millimeter).

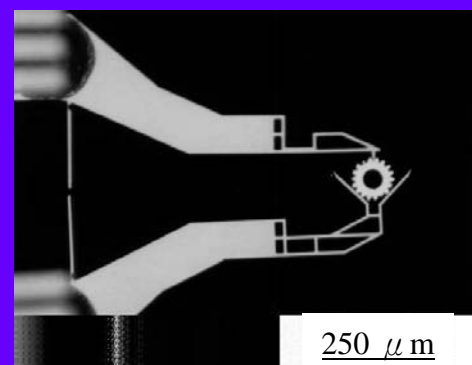
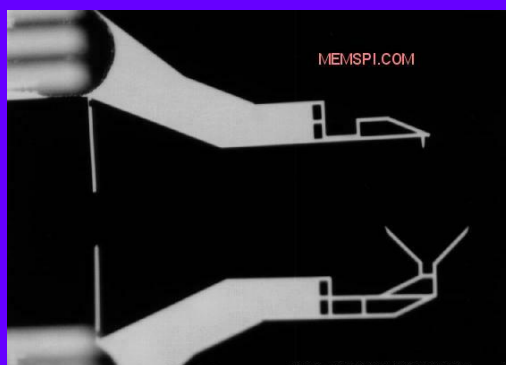
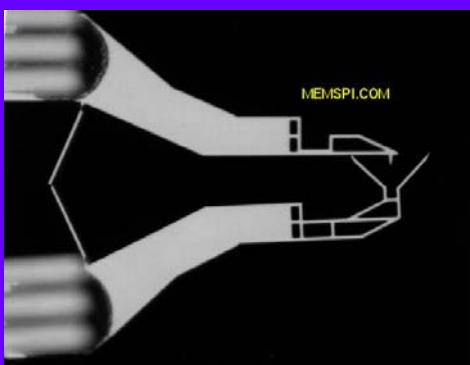




# Considerations for making nanoscale tools

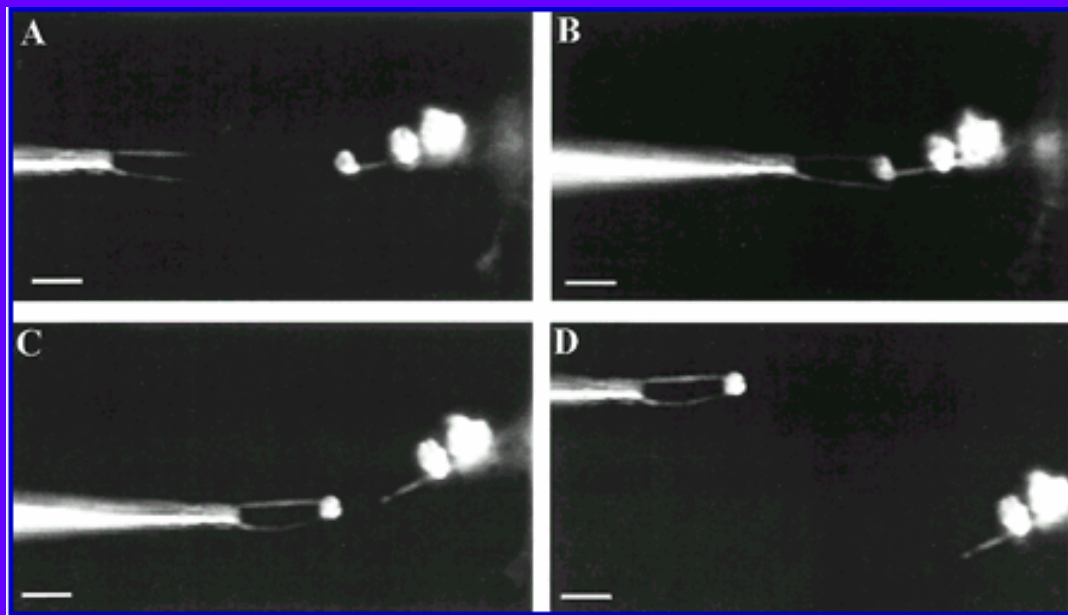
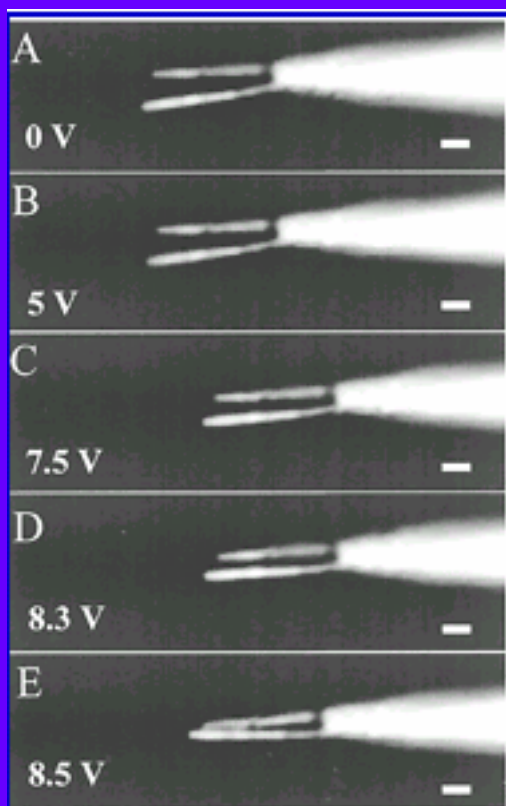
1. Size compatibility
2. Force compatibility
3. Mechanical Properties
4. Chemical Properties
5. Precision movement
6. Integratable coarse movement
7. Environment interferences

# Maneuvering tools





# Operation of nano tweezers

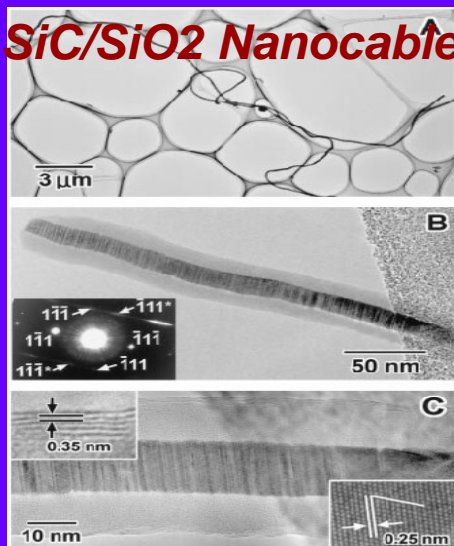


P. Kim and C.M. Lieber,  
*Science* 286, 2148 (1999).



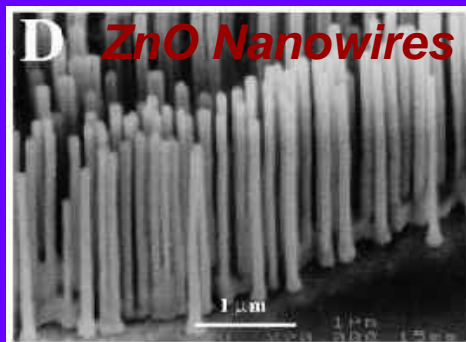
# Nano wires and belts

**SiC/SiO<sub>2</sub> Nanocable**

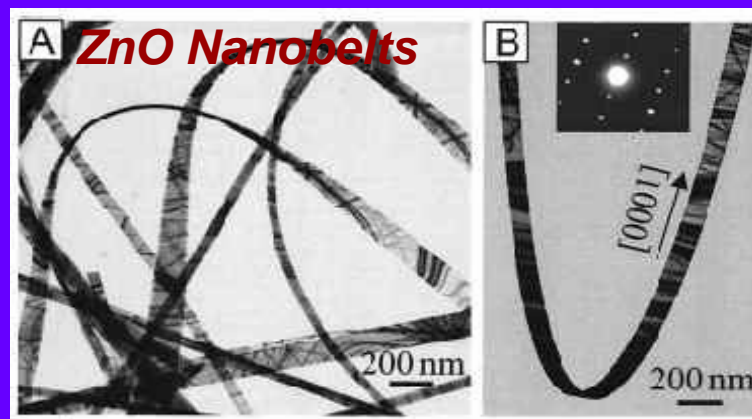


*Science* 281, 973 (1998)

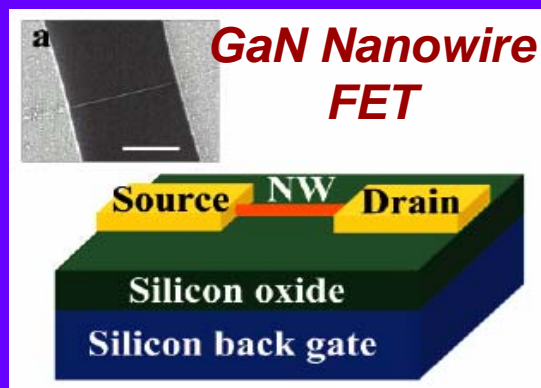
**D ZnO Nanowires**



*Science* 292, 1897 (2001)



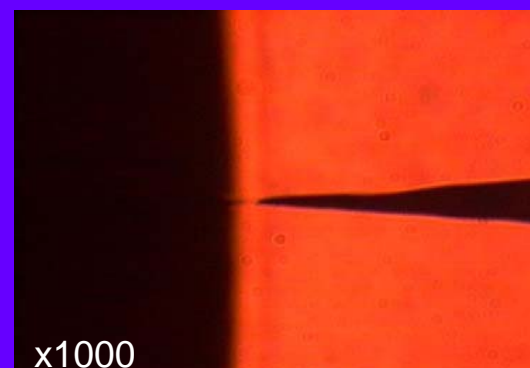
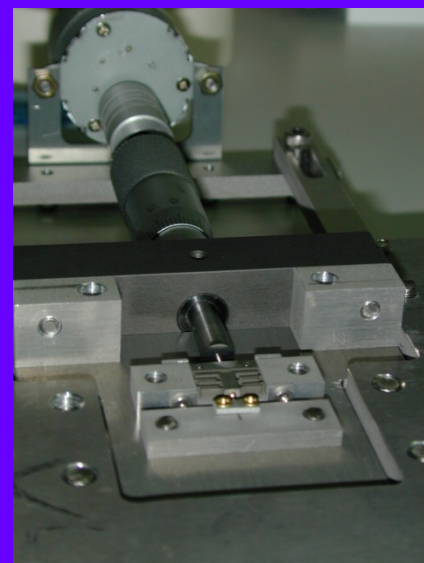
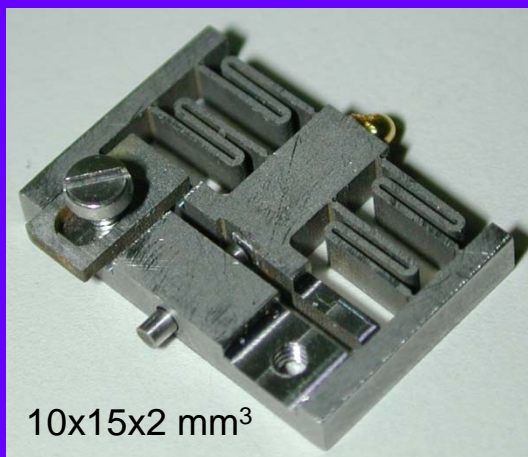
*Science* 291, 1947 (2001)



*Nano Lett.* 2, 101 (2002)



# Precision stepper





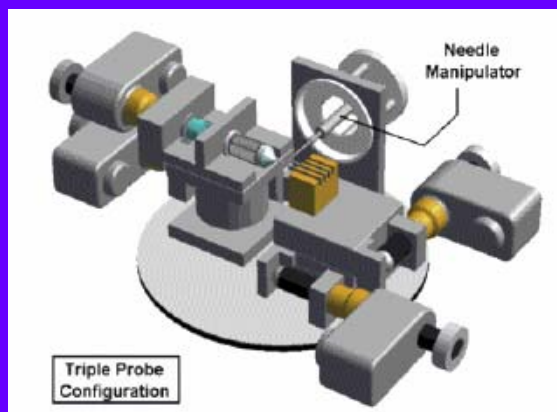
# Considerations for environmental factors

Temp., humidity & ventilation control

Dust filtering

EMI shield

Acoustic isolation



Vibration damping





## *Goals for next 5-10 years*

- Instruments for analysis of supramolecules, biomolecules, and polymers.
- 3-D structure determination.
- Nanostructure chemical identification.
- *In situ* functional measurements.
- Functional parallel probe arrays.
- Standardization and metrology.
- New nano-manipulators.



## References

1. *Scanning Tunneling Microscopy II*, eds. R. Wiesendanger and H.-J. Guntherodt (Springer-Verlag, Berlin, 1992).
2. M. Stark and R. Guckenberger, *Rev. Sci. Instru.* 70, 3614 (1999).

Related links :

<http://www.di.com/>

<http://www.topometric.com/>

# Core Facilities Program

## National Taiwan University

FE-SEM, FESEM attachment-EBIC, STEM, TEM, STEM+EDS, STEM+EELS, STEM+Monochromator, Ion Miller, Cryo stage, Heating, cooling and low-T holder, AFM

## National Chung-Cheng University

Cryo-bio-AFM/NSOM, HR-TEM(EDX), ICP reactive ion ether, High Pressure Stainless reactor, MOCVD, Nanoparticle Evaporator, SPM, Vibrating Sample Magnetometer, Low temperature I-V/C-V probe station, DIP-PEN, Fluorescence bio detection system, Physical Properties Measuring System

## National Cheng-Kung University

HRTEM, Micro PL+ Micro-Raman, FESEM, SPM, LPCVD, MBE, Nano Imprint, 快速雷射光刻系統

## National Sun-Yet-Sen University

Nano-laboratory for Kaohsiung and Ping-Tung Area

## Academia Sinica

Dual beam focused ion beam sys., E- beam writer, Inductively coupled plasma etcher, Advanced in-situ characterizing nanofabrication System

## ITRI

NTRC Common Laboratories

## Taiwan United University System (Tsing-Hua)

UST-Core Facility for Nano Lithography and Nano Biotechnology

## Taiwan United University System (Chiao-Tung)

Low Temperature / High Magnetic Field System, High-resolution transmission electron microscope, Veeco Gen II (MBE), ICP-RIE System of III-V Compound Device Production, X-Ray diffractometer, Sb based molecular beam epitaxy

