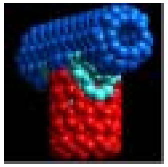
Ames Research Center

Nanotechnology: Impact on Metrology



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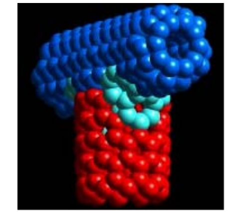
Moffett Field, CA 94035
m.meyyappan@nasa.gov

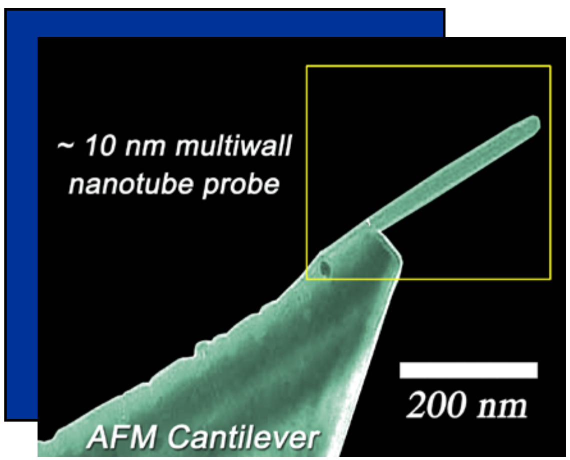
Acknowledgement: Jeff Sun, Bin Yu.

CNT in Microscopy



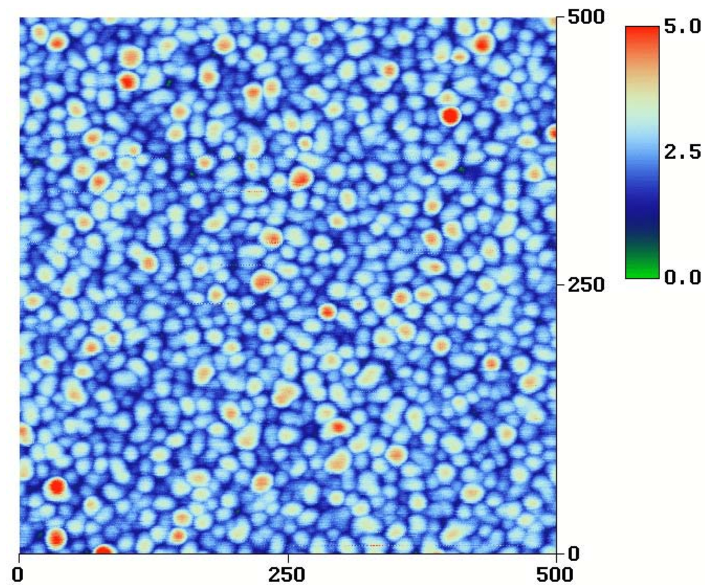
Atomic Force Microscopy is a powerful technique for imaging; also CD metrology, nanomanipulation, as platform for sensor work, nanolithography...

Conventional silicon and other tips wear out quickly.
CNT tip is robust, offers amazing resolution.



Simulated Mars dust

2 nm thick Au on Mica imaged with SWNT



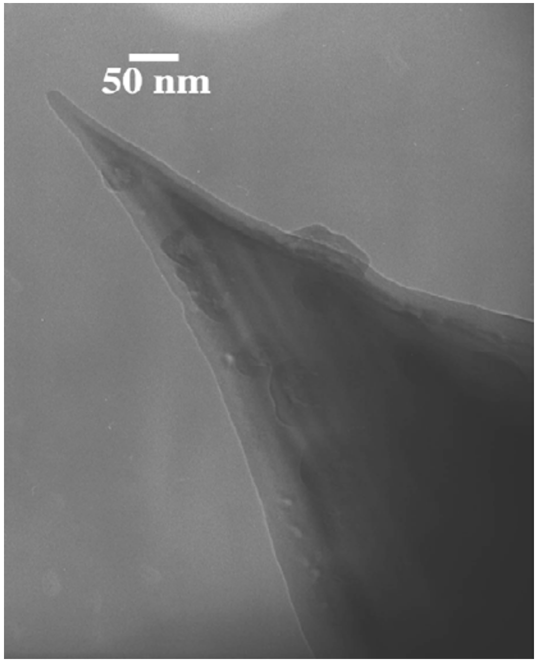
NASA Ames Research Center
Ramsey Stevens, Lance Delzeit, Cattien Nguyen

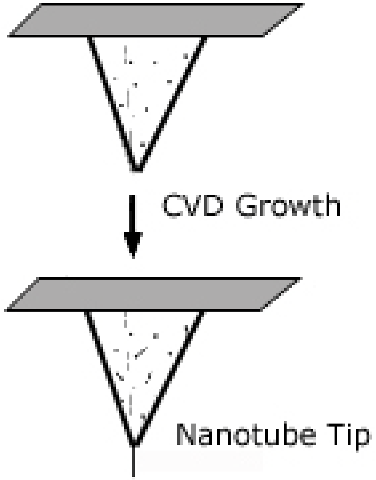
Written using multiwall tube

Nguyen et al., Nanotechnology, **12**, 363 (200

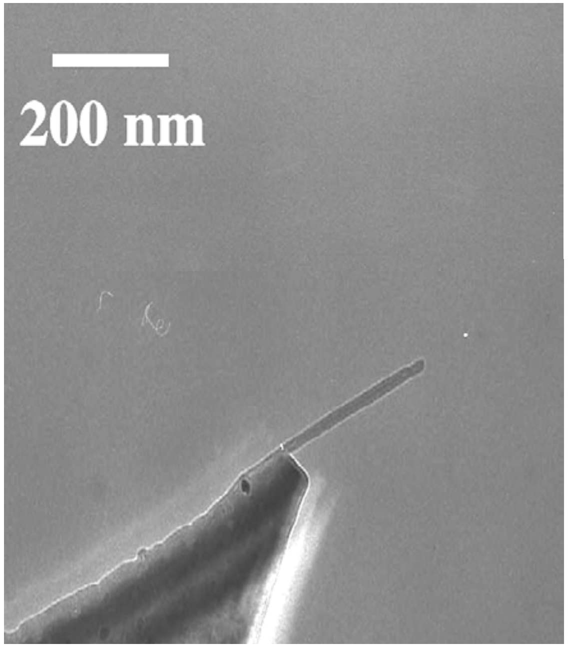
Fabrication of CNT Probes

Transition metal catalyst is deposited from liquid phase or sputtered on the tip of the cantilever





Carbon nanotube is grown in thermal CVD or plasma reactor



AFM Imaging with Single Wall Nanotube Tips

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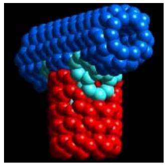
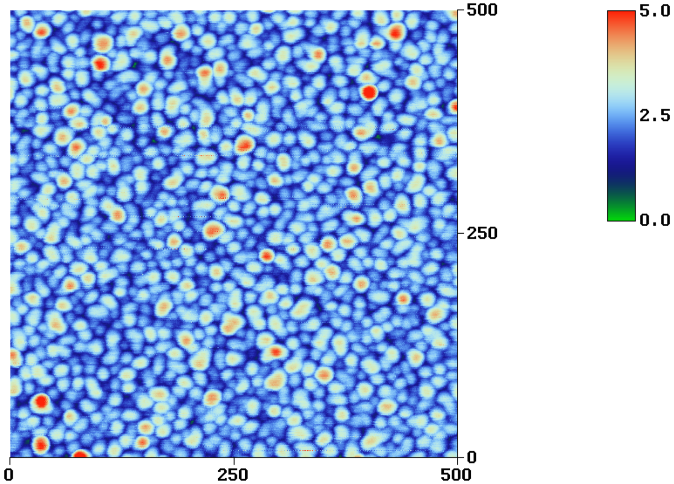
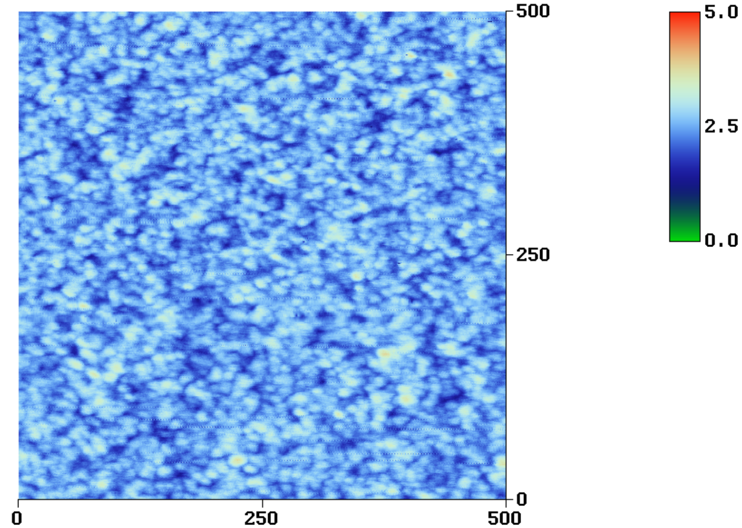
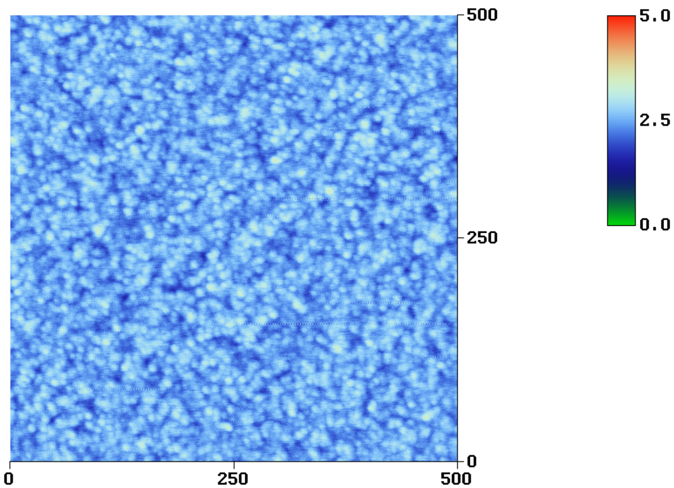




2 nm thick Au on Mica



Si_3N_4 on Silicon substrate



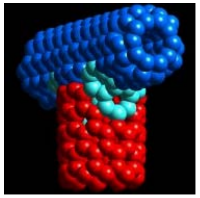
5 nm thick Ir on Mica

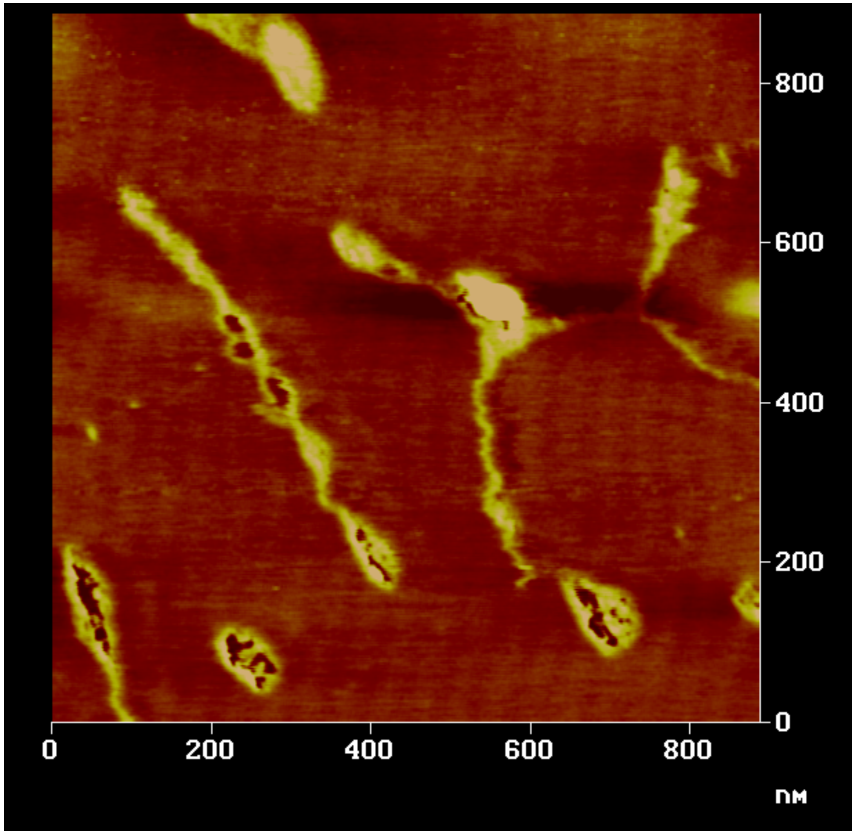
- Remarkable nanoscale resolution is evident

- More importantly, the same high resolution is maintained with continued use of the probe for a long time; probe doesn't wear out easily.

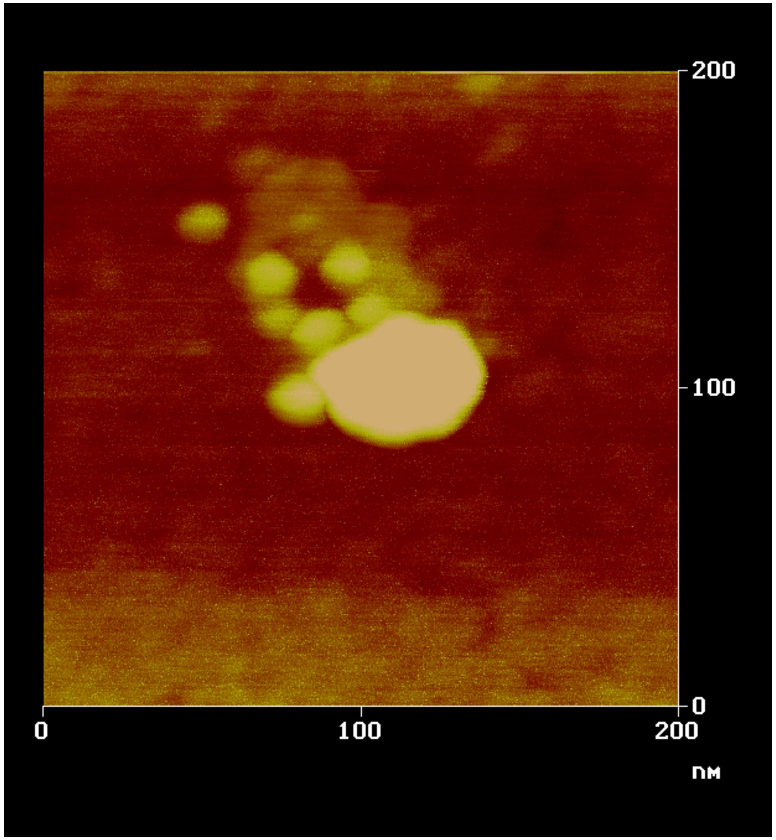
Nguyen et al., Nanotechnology, 2001, Vol. 12, p. 363.

High Resolution Imaging of Biological Materials





DNA



PROTEIN

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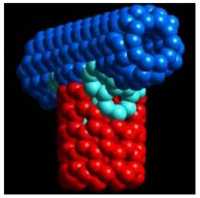
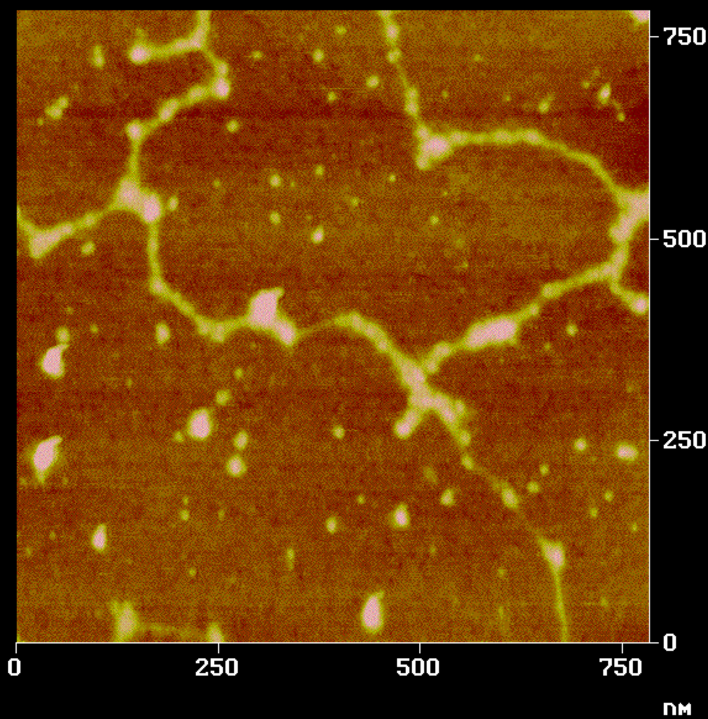
Imaging in Aqueous Environments





DNA on mica in 20 mM Tris HCl and 10 mM magnesium chloride solution (near physiological conditions)

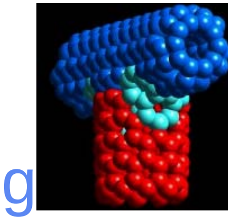
- The hydrophobic nature of the CNT graphitic sidewall is chemically incompatible with aqueous solutions. Probes are unstable when submerged in solution.

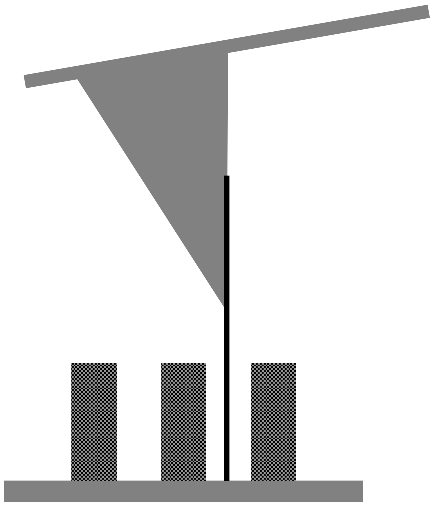
- The CNT probe is treated with a ethylene diamine coating, rendering it hydrophilic.

R.M. Stevens et al., IEEE Trans. Nanobioscience Vol. 3, pp. 56-60 (2004).

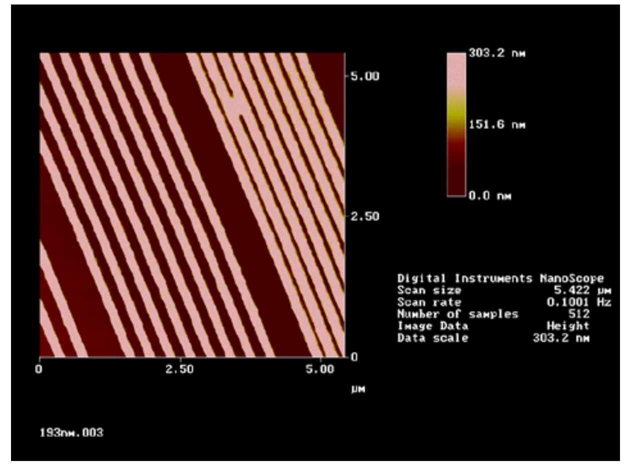
MWNT Scanning Probe:

Profilometry in Semiconductor Manufacturing

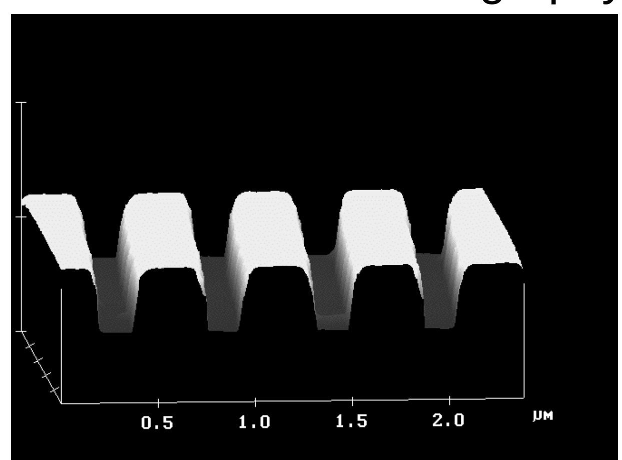




193 nm IBM Version 2 Resist

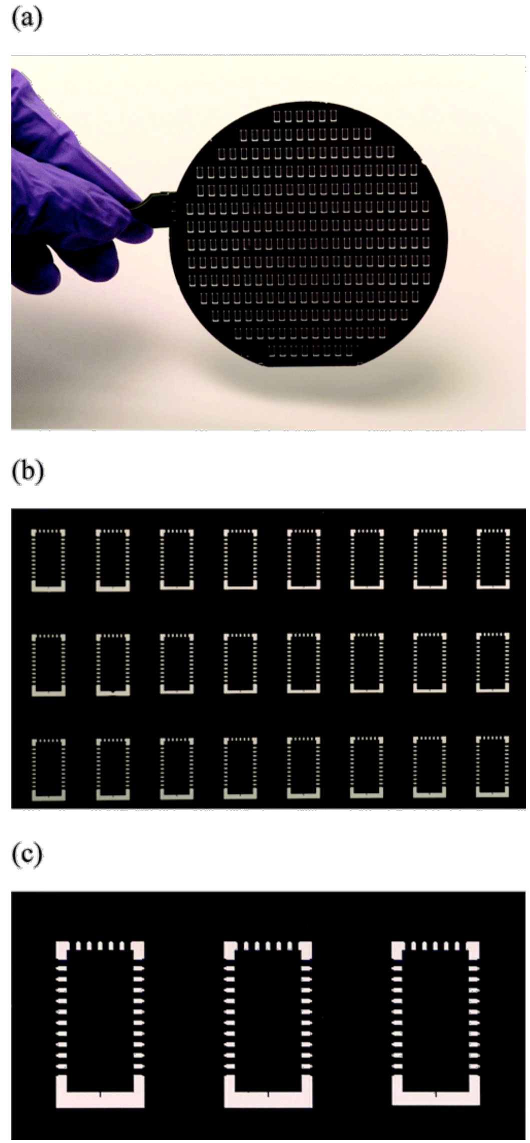


DUV Photoresist Patterns Generated by Interferometric Lithography



Nguyen et al.,
App. Phys. Lett.,
81 (5), 901 (2002).

Mass Production of CNT Probes



4” wafer with 244 probes

8 x 3 array

Each cantilever is 1.6 mm x 3.44 mm in size

Ye et al, NanoLett. 4, 1301 (2004).

Process Flow for CNT Probe Fabrication

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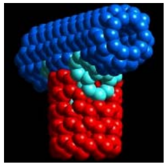
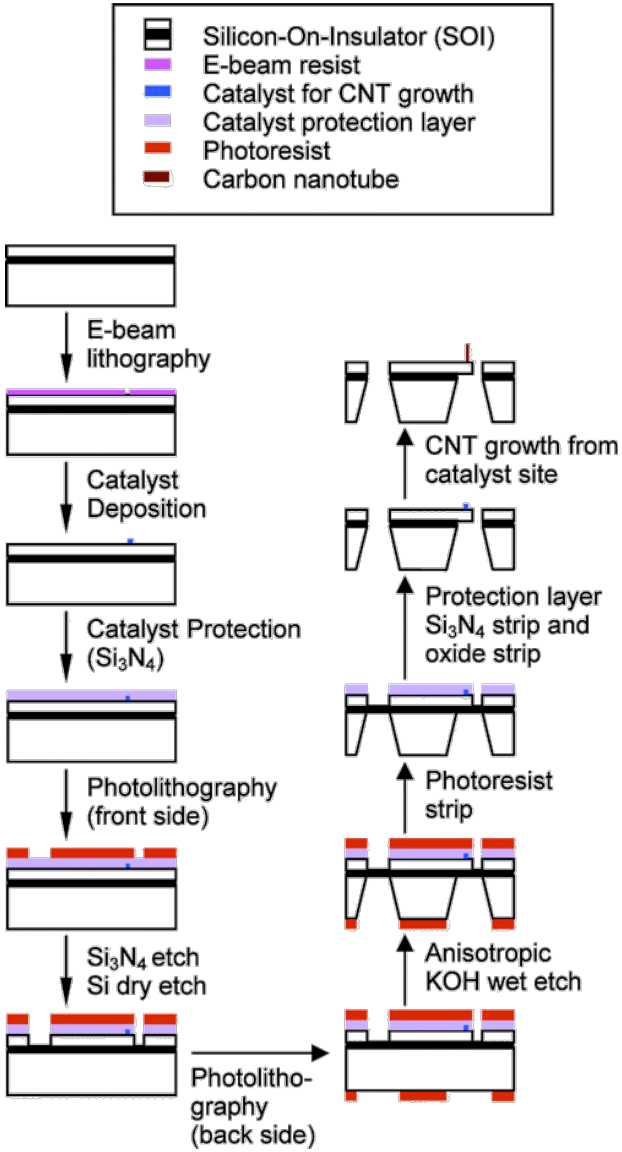
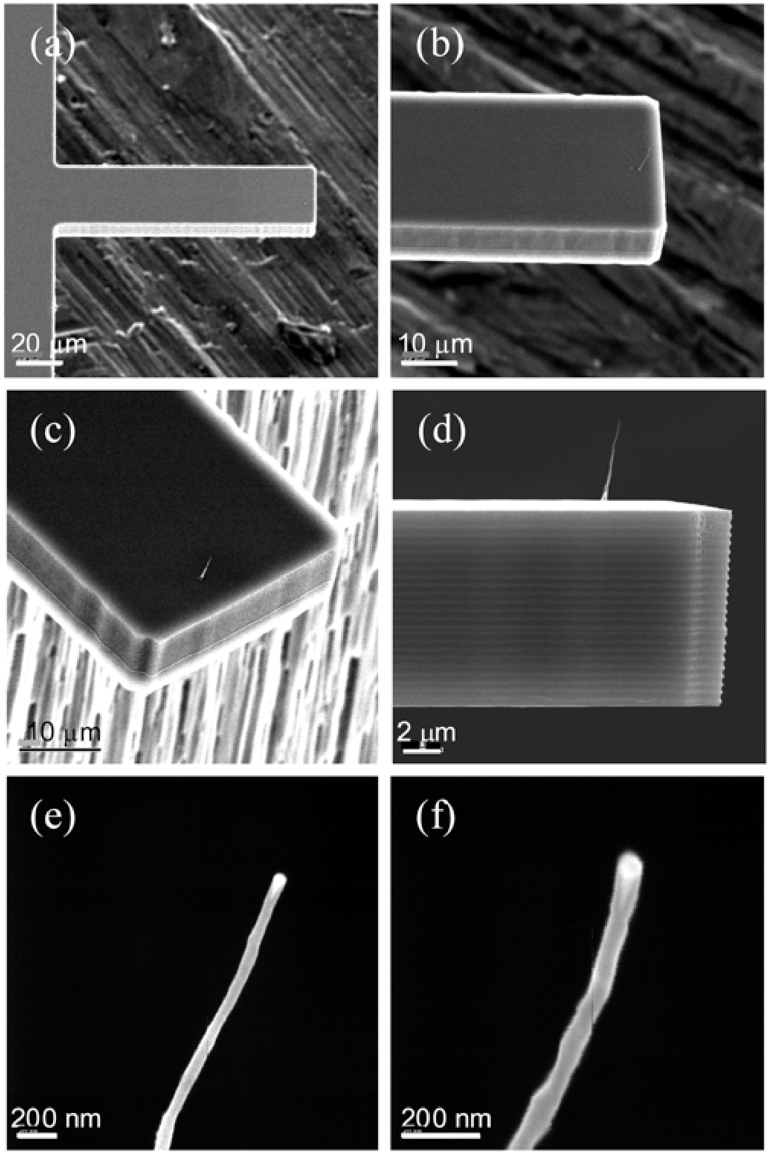
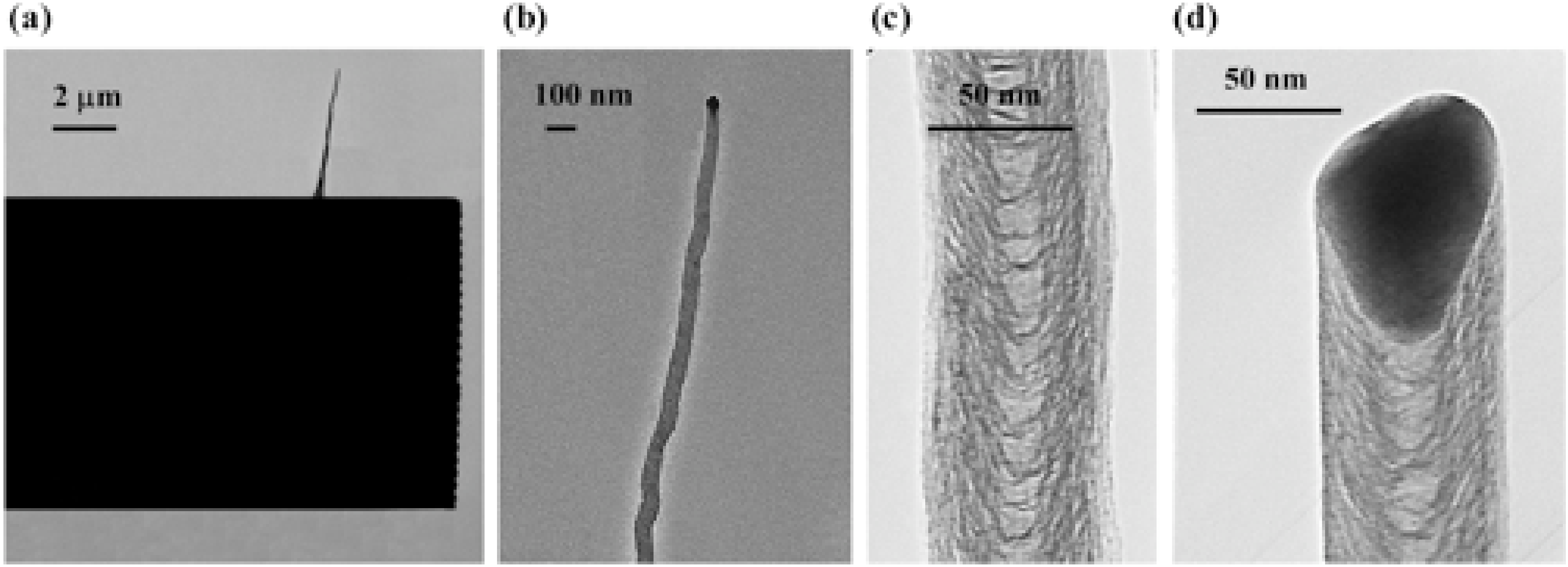




CNT AFM Probe



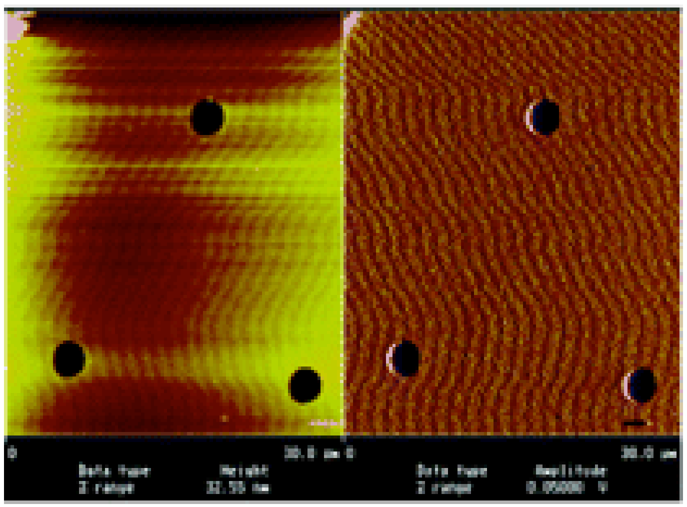
Cross-sectional TEM Image



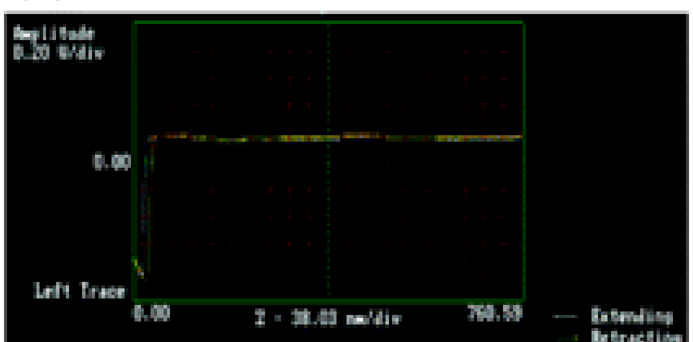
Imaging using the CNT Probe

2 µm dia hole, 400 nm deep in Si_3N_4

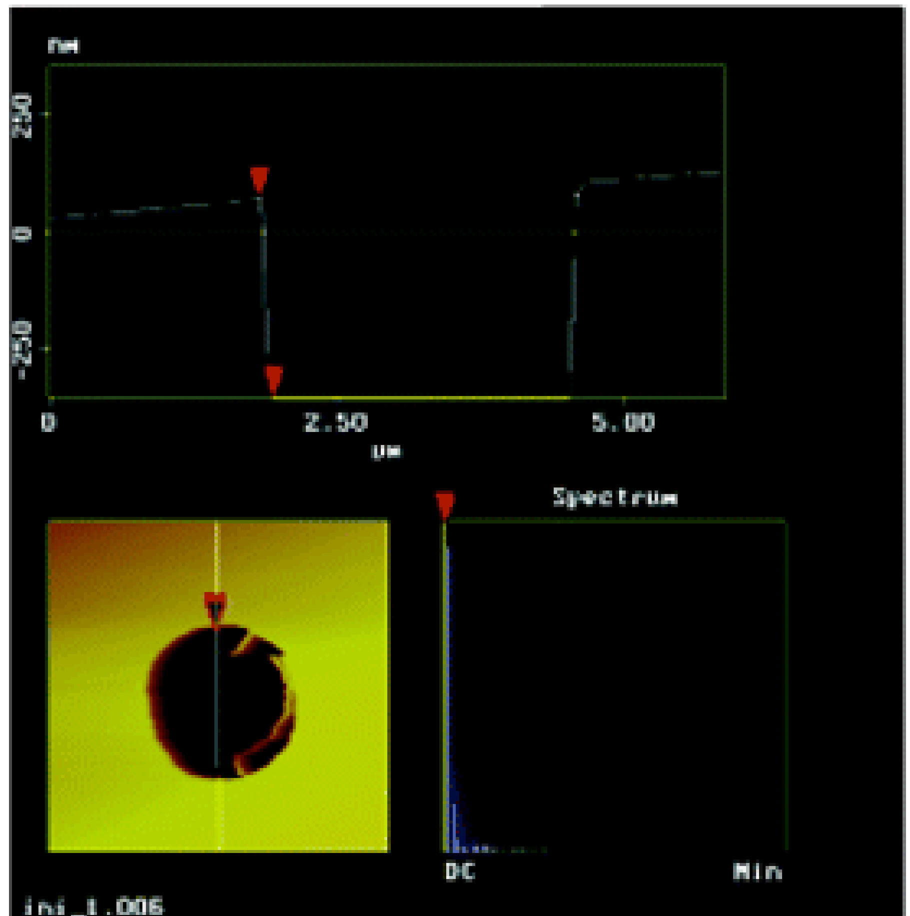
(a)



(b)



(c)



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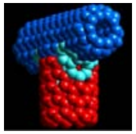
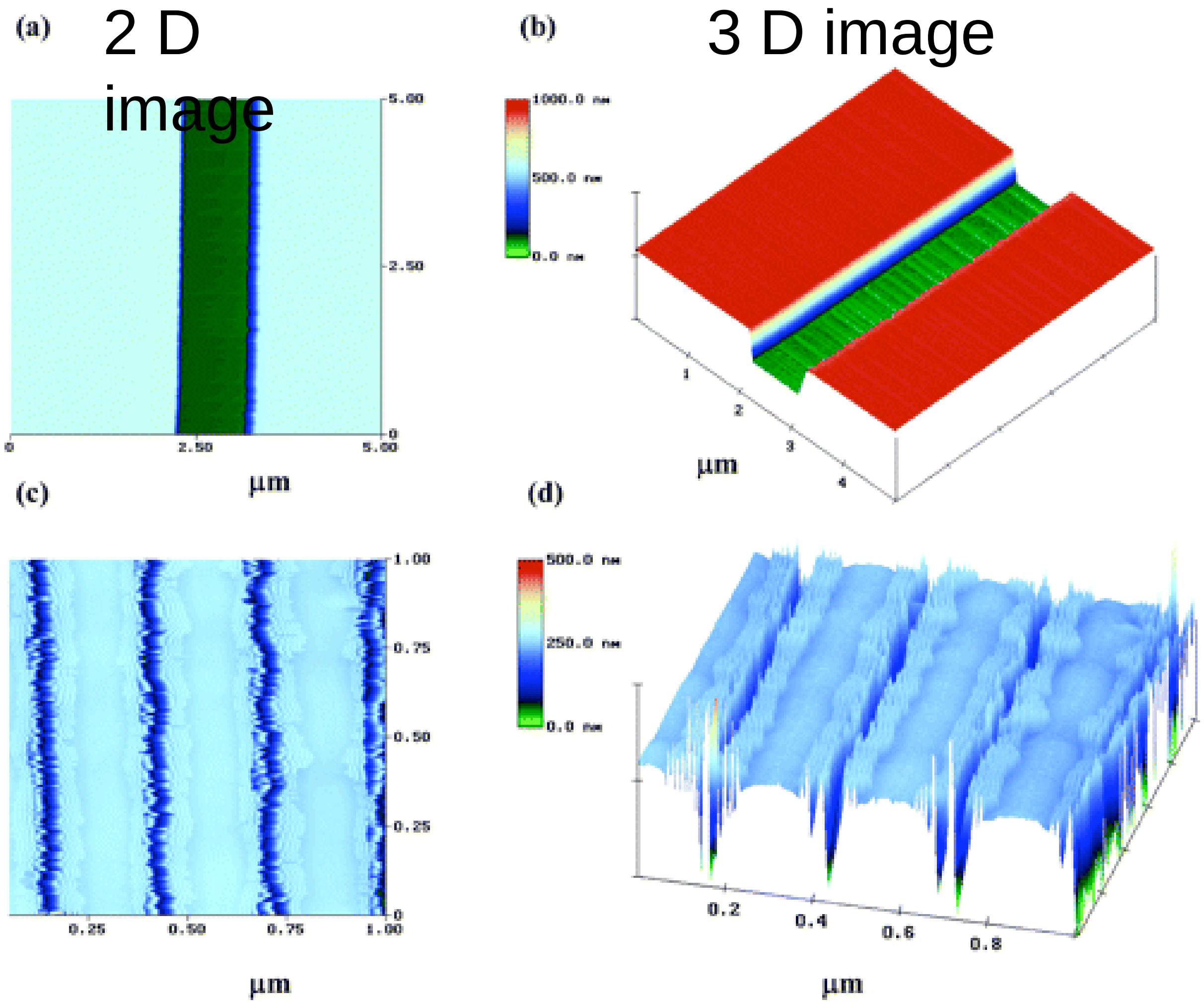
Imaging Trenches using CNT Probe





One micron trench (above) and 90 nm trench (below). Tip is 60 nm in diameter and 5 µm long.

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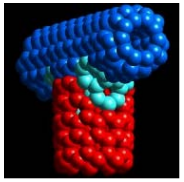
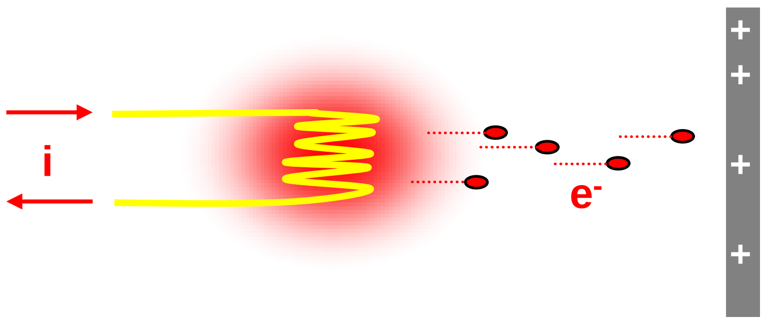
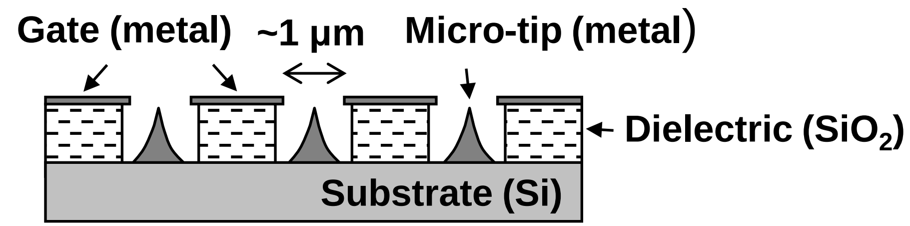
Electron Emitters



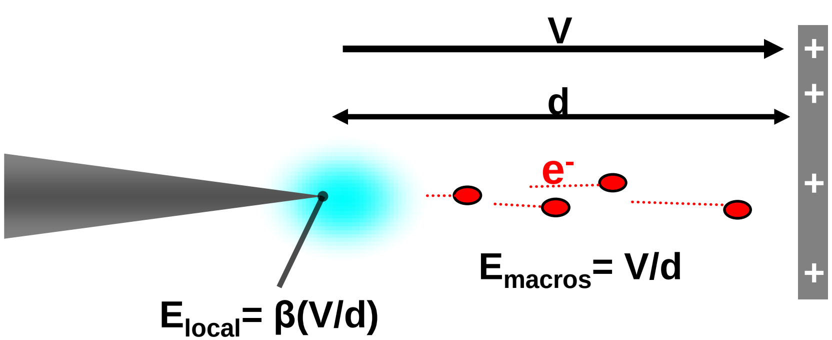


Thermionic Emitter - W filament operating at 1000°C



Spindt Cathode - sharp Si tips

Limited success: expensive to scale up



Cold Cathode Emitters

Threshold electric field at 10 mA/cm² (W. Zhu et al)

Material	Field (V/µm)
Mo and Si tips	50-100
P-type semi conducting diamond	130
Undoped defective CVD diamond	30-120
Amorphous diamond	20-40
CS-coated diamond	20-30
Graphite powder (<1mm)	17
Nanostructured diamond	3-5 (unstable >30mA/cm²)
Carbon nanotube (SWNT film)	1-3 (stable at 1A/cm²)

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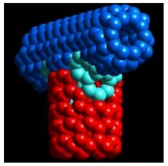
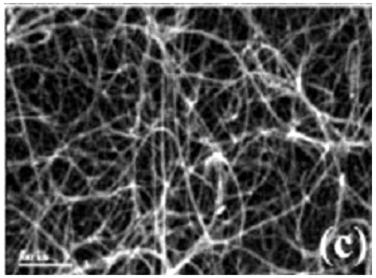
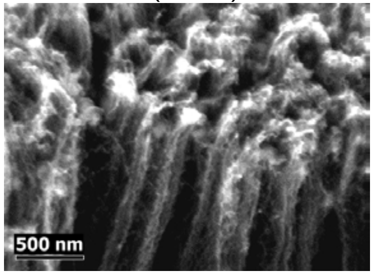
Carbon Nanotubes in Field Emission Applications

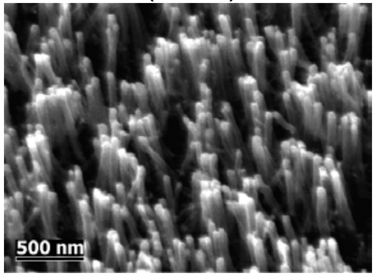




Single wall nanotube (SWNT)

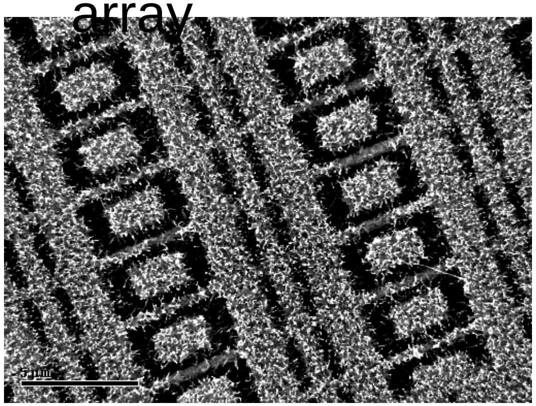


Multi wall nanotube (MWNT)



Nanofibers

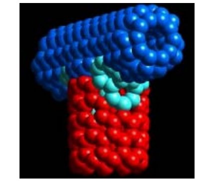
- Stable crystalline graphite structure
- Low turn-on fields
- Extremely sharp
- Very good conductor (up to 10^9 A/cm^2 local current density)
- Thermal tolerance (emission reported at 2000K)
- Mechanically robust
- Can be used as single emitter or large film / array



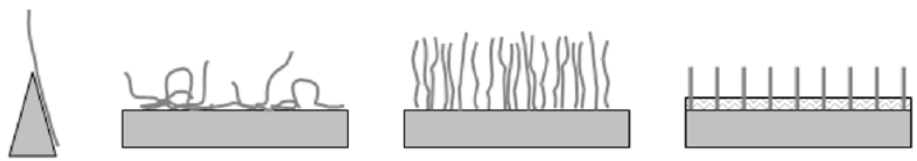
Applications

- Flat panel displays
- Lighting source
- X-ray tubes
- Instrumentation: SEM, mass spectrometer

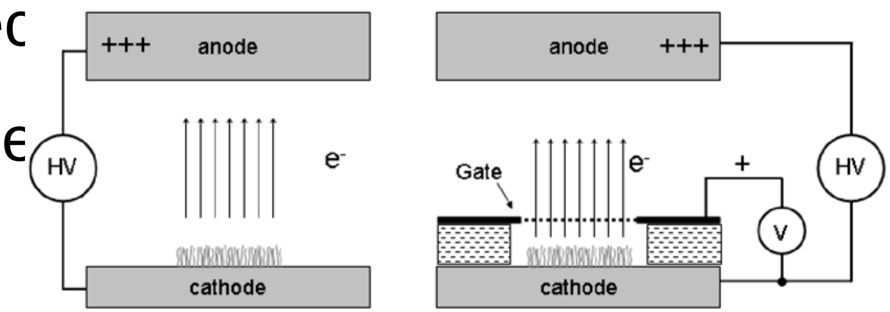
Structure of CNT Field Emitters



- Nature of nanotubes (SWCNTs, MWCNTs, CNFs…)
- Clean emitting sites vs. adsorbates (wafer vapor, oxygen…)
- Microstruct



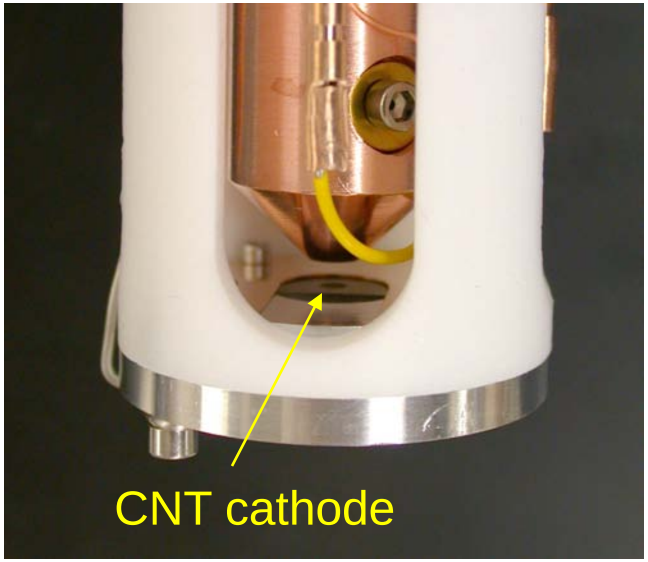
- Screening effec
- Diode vs. triode



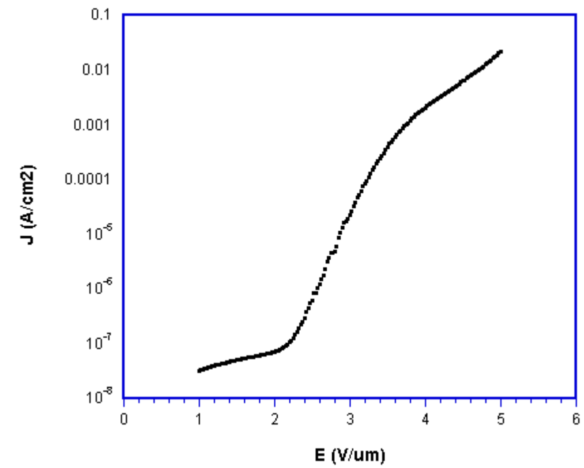
High voltage required and/or gap needs to be

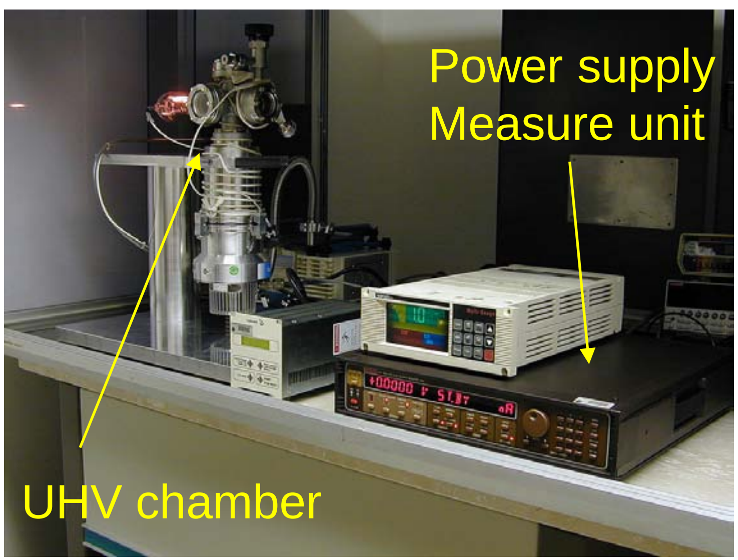
Current is controlled by gate voltage, independent of

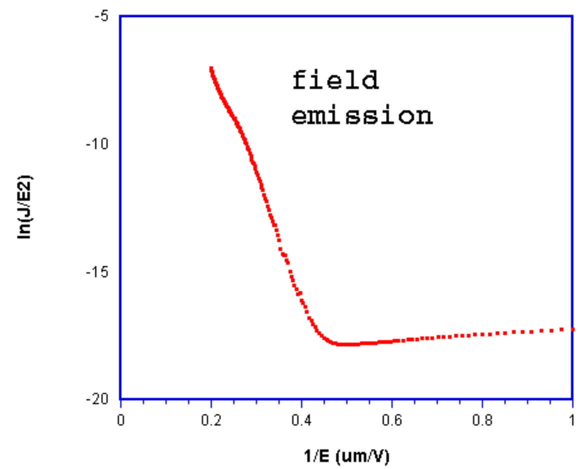
Characterization of FE Properties

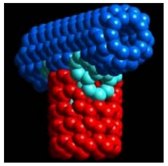


Gap adjustment





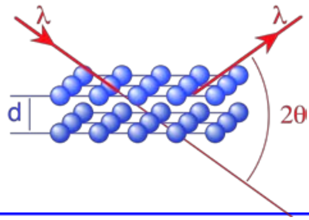


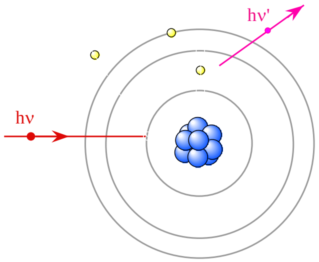


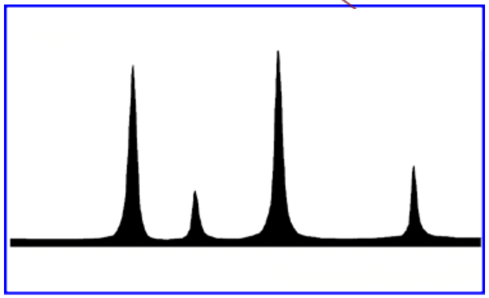
CheMin X-Ray Diffraction/ X-Ray Fluorescence

Chemistry & Mineralogy

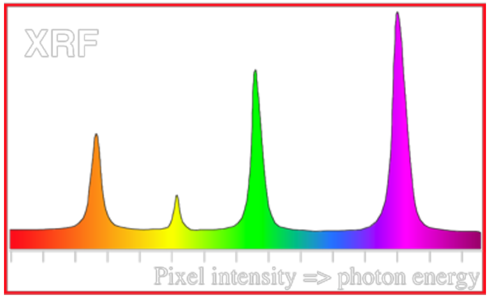
- 1 DETECTOR
- 2 SIMULTANEOUS ANALYSES
- NO MOVING PARTS



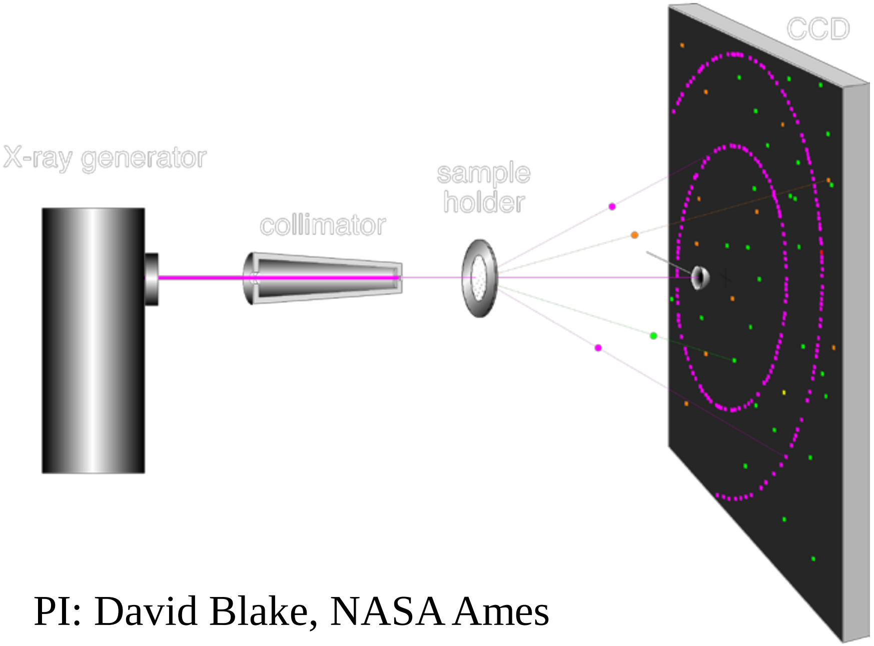


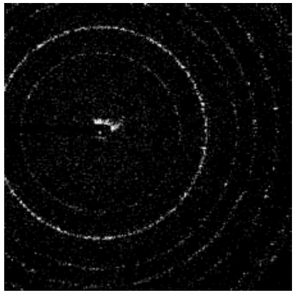


X-ray diffraction



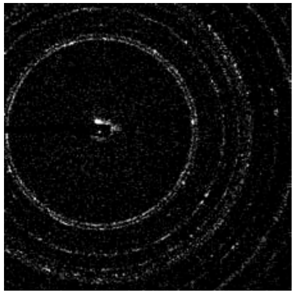
X-ray fluorescence





Calcite

$CaCO_3$



Aragonite

PI: David Blake, NASA Ames

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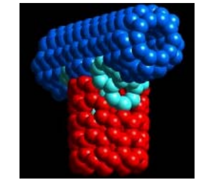
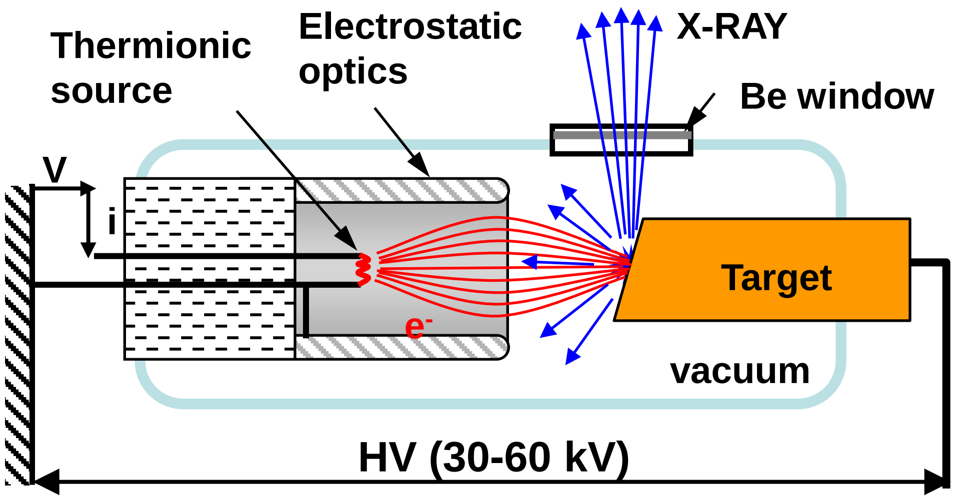
X-ray Tube





CNT X-ray Tubes

- Beam spread of electrons and X-rays narrower
- Lower power
- Reduced heat load
- Lower maintenance, longer life

X-Ray Wavelength (nm)

Target	Kβ 1	Kβ 2	Kα 1	Kα 2
Fe	0.17566	0.17442	0.193604	0.193998
Ni	0.15001	0.14886	0.165791	0.166175
Cu	0.139222	0.138109	0.154056	0.154439
Zr	0.070173	0.068993	0.078593	0.079015
Mo	0.063229	0.062099	0.070930	0.071359

Miniature CNT X-ray Tube in Flight Instrument

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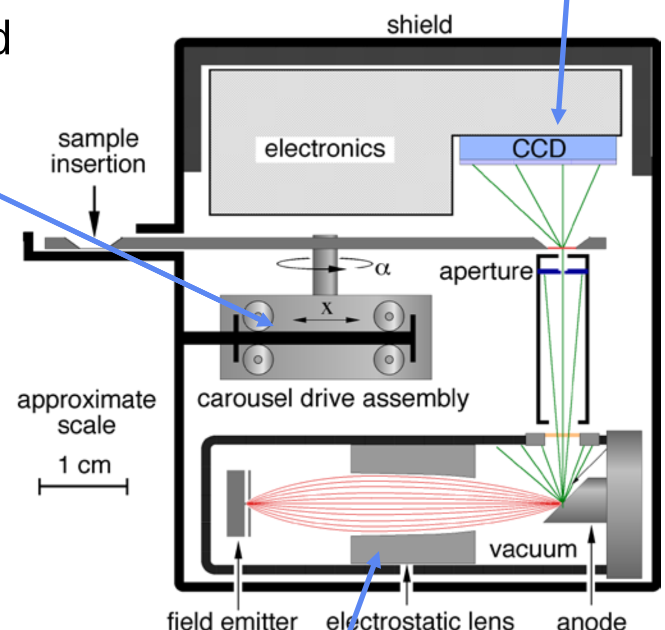
Integration in miniature X-ray tube (Oxford XTG Inc.)

Flight instrument layout:

CCD detector optimized for X-ray detection

Various sample delivery configurations are studied



miniature microfocused X-ray tube

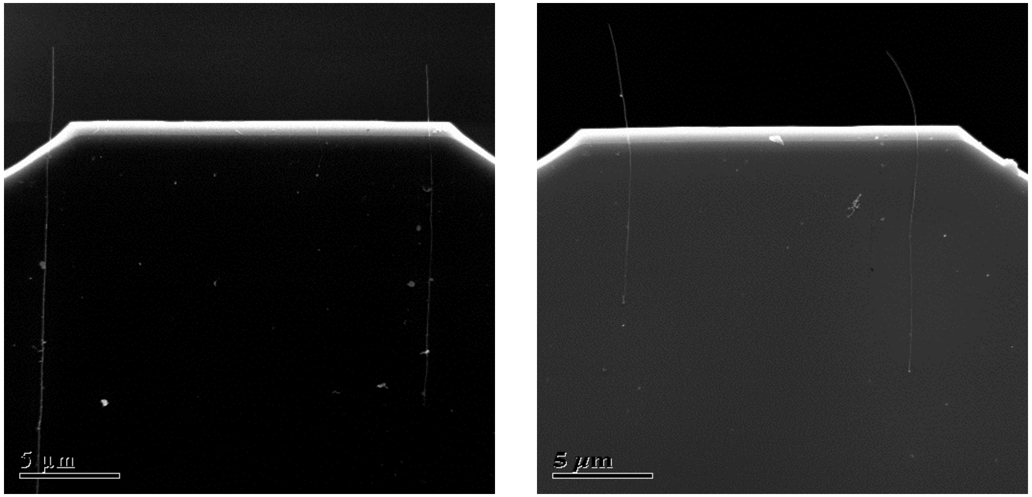
- volume <1 liter
- mass < 1 kilogram
- power < 5 watts

PI: David Blake

Future Issues in X-ray Tube Design

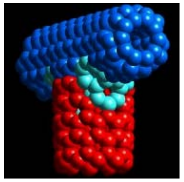
- Investigate field screening effect in a definitive manner
  - as a function of emitter separation
  - as a function of CNT length

  (Theory: field screening is minimized at separation > 2L)

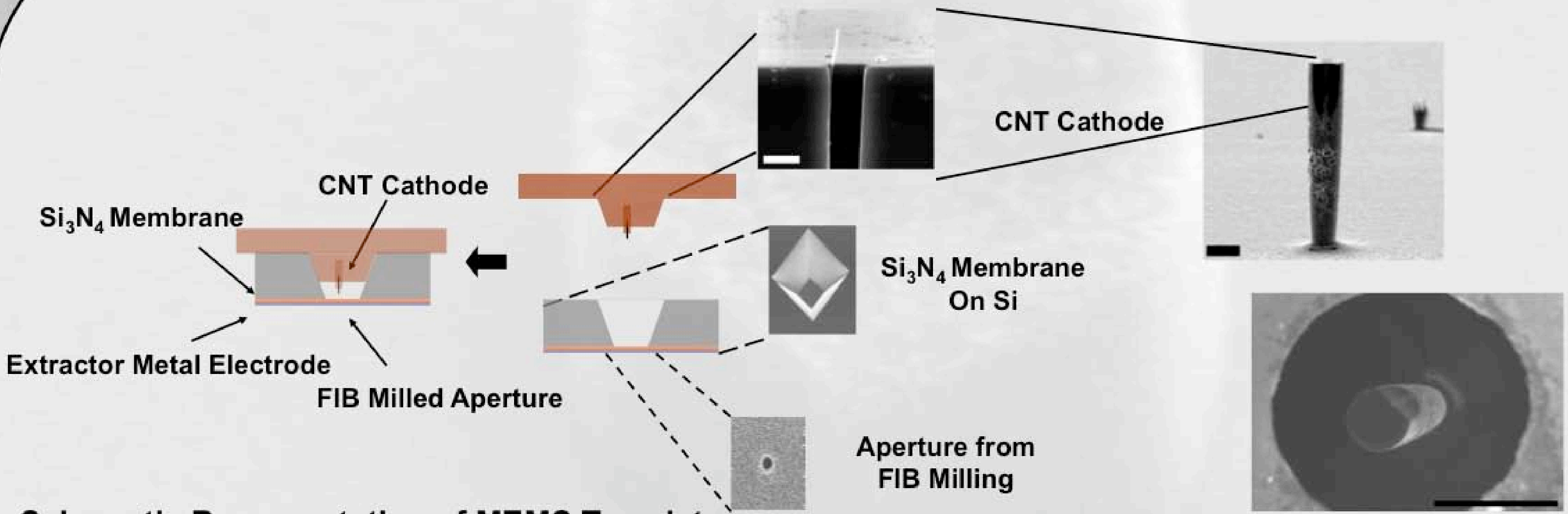


- Robustness and stability of the CNT emitter
- Electron optics optimization, anode target cooling (for baggage screening)
- Power supply design (takes up 90% of the volume), Packaging

Single Electron Gun for a Microcolumn SEM



Fabrication by MEMS Structural Templating Technique*

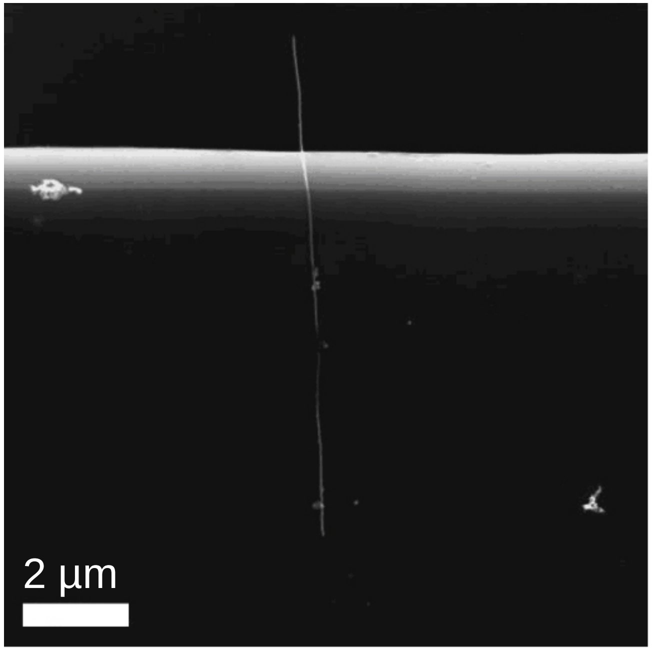


Schematic Representation of MEMS Template Fabrication of CNT Guns

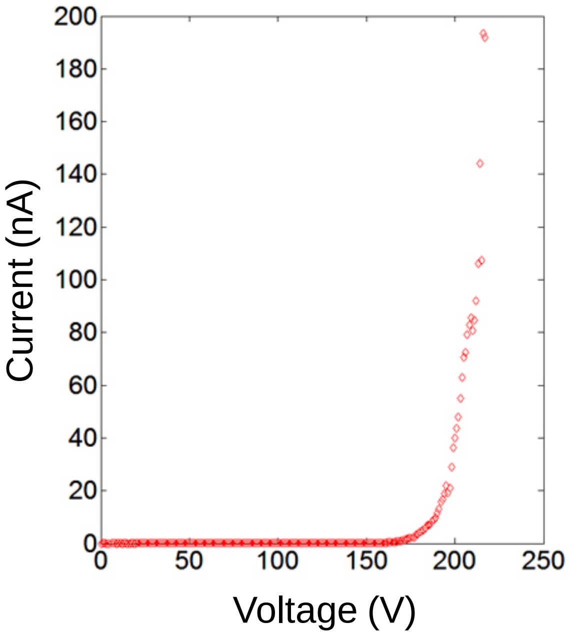
Top-down SEM image of MEMS Cathode with CNT emitter well-aligned with the Aperture

Silicon Based Carbon Nanotube Cathode

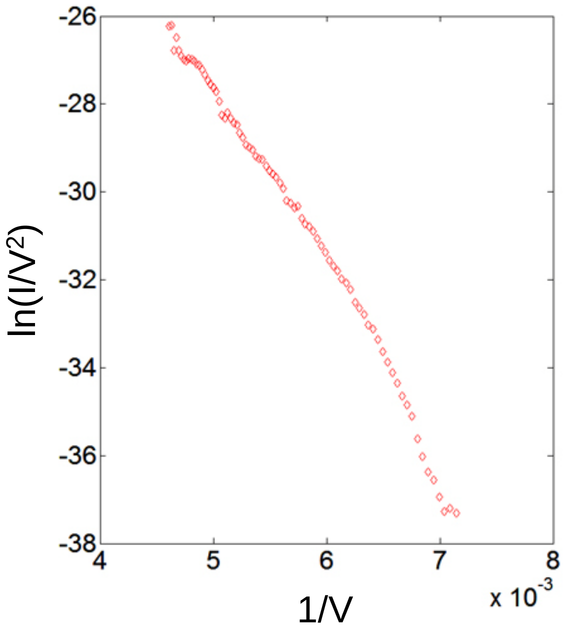
- MEMS based carbon nanotube cathode
  - Si structure coated with 25 nm Ni
  - Point electron source



MEMS based nanotube cathode.

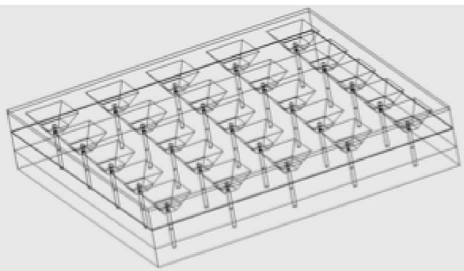


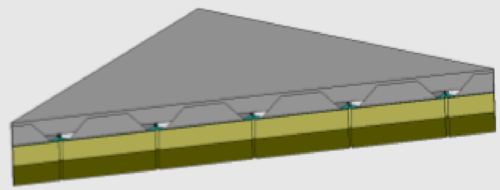
Field emission current-voltage data.



Fowler-Nordheim plot.

High Throughput Maskless Electron Beam Lithography

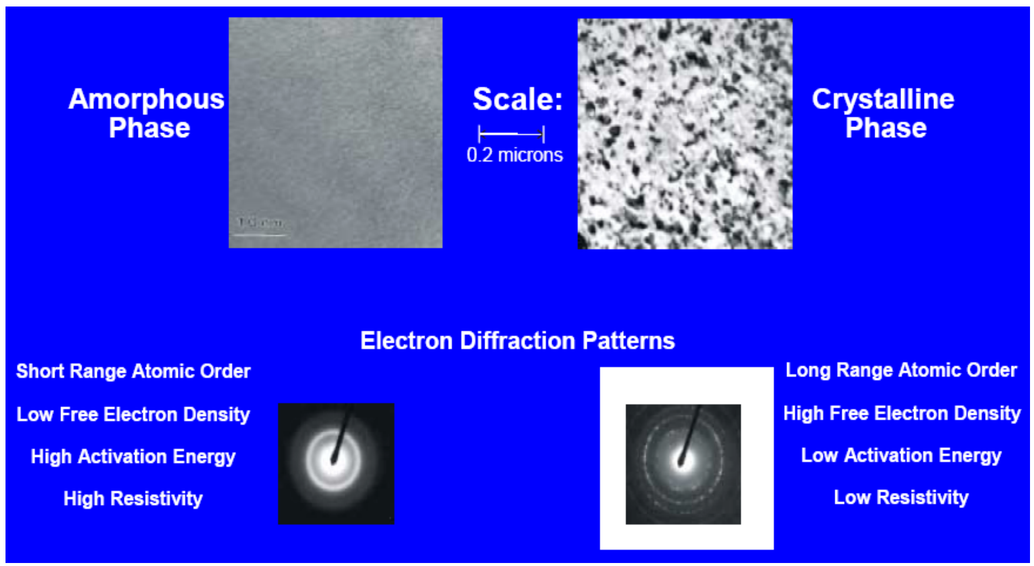




5X5 Array of MEMS-based CNT electron guns fabricated by templating technique demonstrated above. Each gun in the array will be individually electrically addressable in order to precisely control dose exposure using voltage-controlled variable resistor circuit model that we developed above

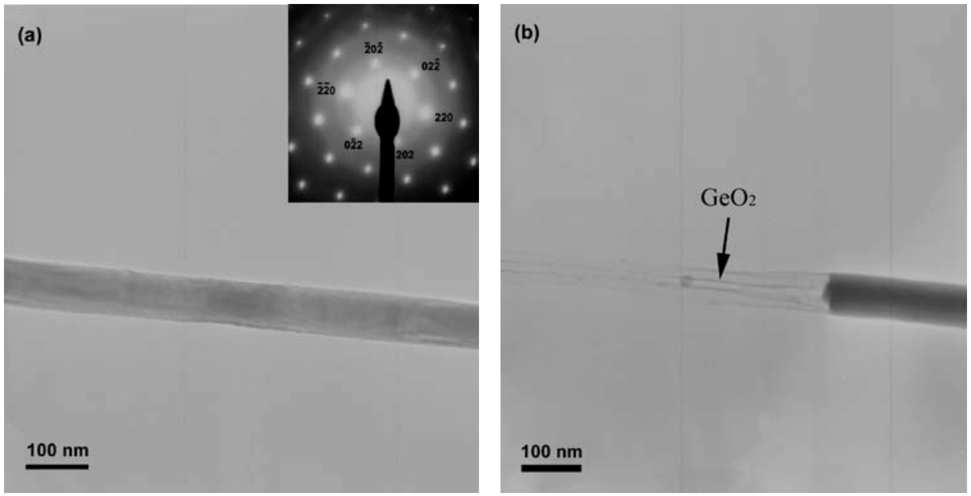
Phase Change Materials

- Phase change materials date back to 1960s
  - Mainstream optical storage media (CD-RW, DVD-RW)
- Common phase-change material candidates
  - GeTe, GeSbTe, In_2Se_3, InSb, SbTe, GaSb, InSbTe, GaSeTe, …



\- Thermally induced phase change (orderly single crystalline or polycrystalline C-phase vs.
less orderly amorphous α-phase)

PCM Nanowires: Melting Point



	GeTe (d=70nm)	In_2Se_3 (d=40nm)
Bulk T_m	725°C	890°C
Nanowire T_m	390°C	680°C
Reduction	46%	24%

- The melting temperature of the phase-change nanowire is identified as the point at which (1) the electron diffraction pattern disappears and (2) the nanowire starts to evaporate.
- This property is diameter-dependent: reduction even more significant for smaller diameters

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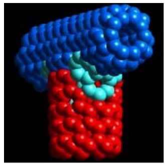

Summary

- Nanomaterials, because of a change in properties relative to their bulk counterparts, can have an impact on construction of probes, diagnostic equipment and sensors.

- For example, we have used carbon nanotubes successfully in AFM-based metrology, and miniaturization of analytical equipment.