Chapter 18
Nanostructures

Reducing dimension

- 2D—surfaces, interfaces and quantum wells
- 1D—carbon nanotubes, quantum wires and conducting polymers
- 0D—nanocrystals, nanoparticles, lithographically patterned quantum dots

- Crystalline structures

Creation of nanostructures

- Top-down—lithographic patterning
- Bottom-up—growth and self-assembly

- Boundary at 50nm?

Top-down nanofabrication

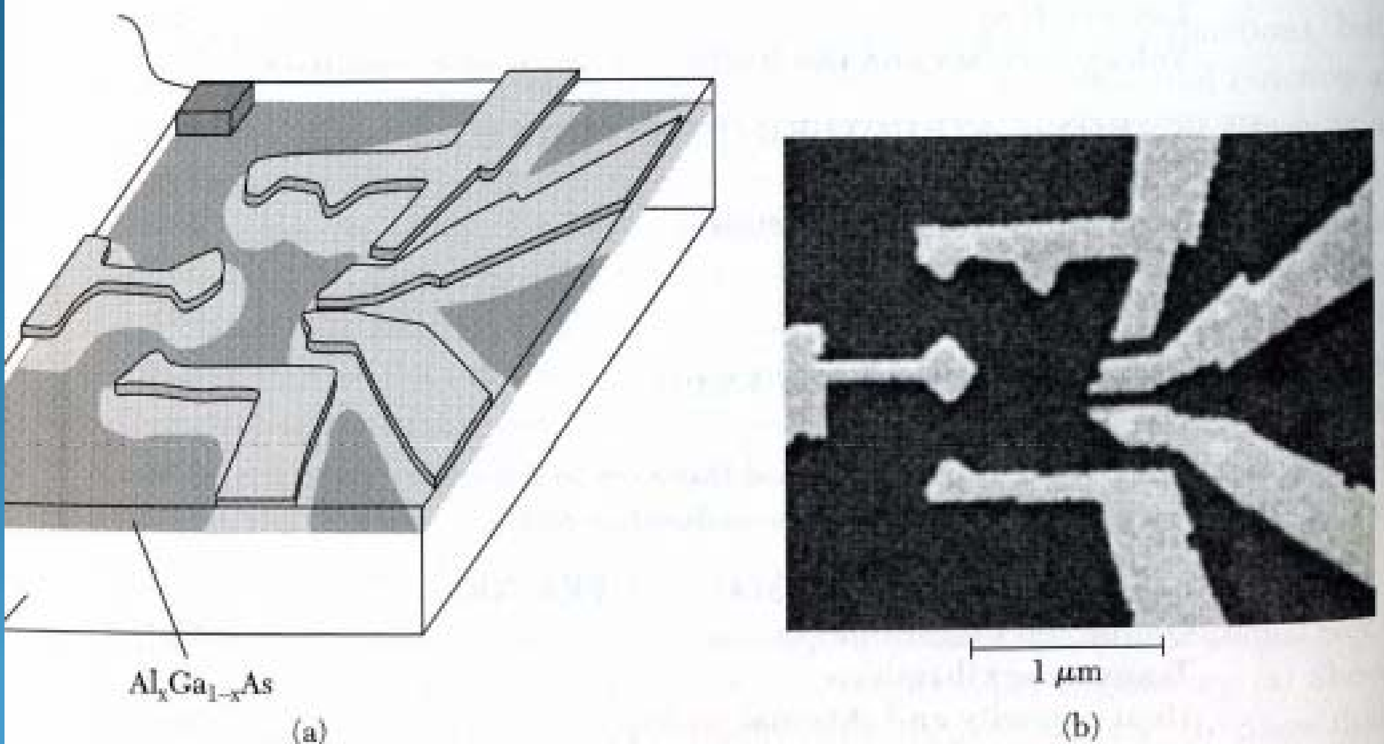


Figure 1 Schematic and scanning electron microscope (SEM) image of a gate electrode pattern on a GaAs/AlGaAs heterostructure used to create a quantum dot of complex shape in the underlying two-dimensional (2D) electron gas. (Courtesy of C. Marcus.)

Bottom-up Nanofabrication

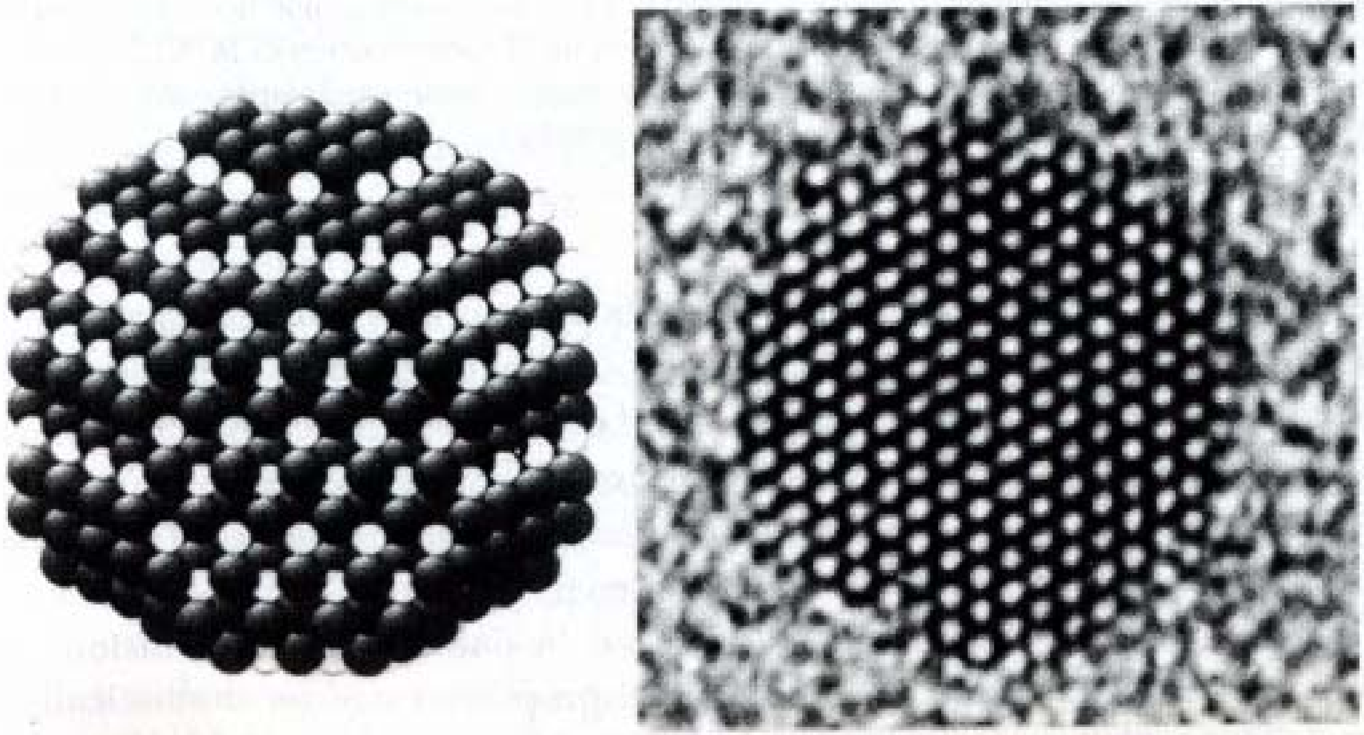


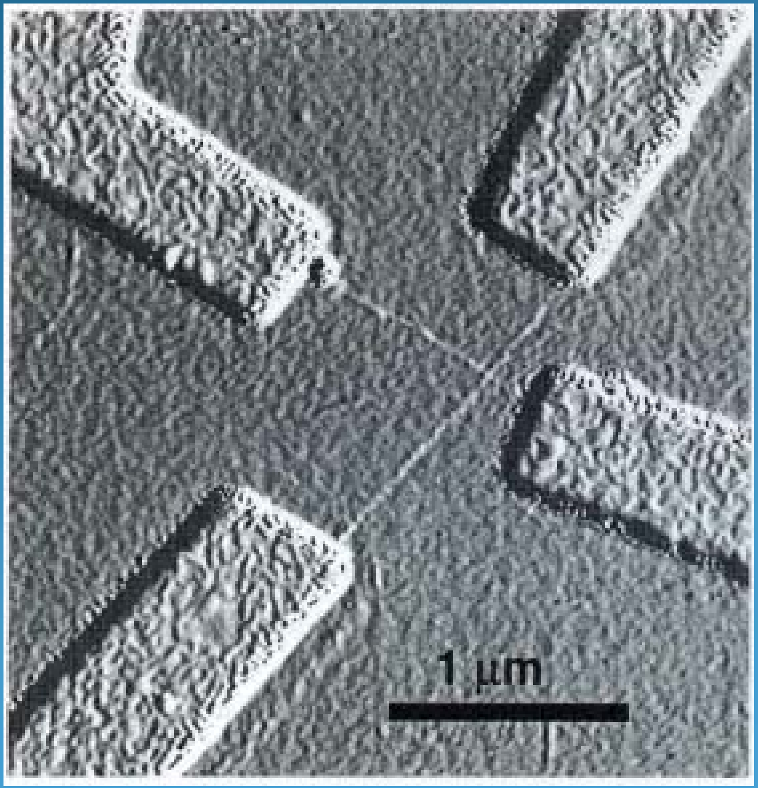
Figure 2 Model and transmission electron microscope (TEM) image of a CdSe nanocrystal. Individual rows of atoms are clearly resolved in the TEM image. (Courtesy of A. P. Alivisatos.)

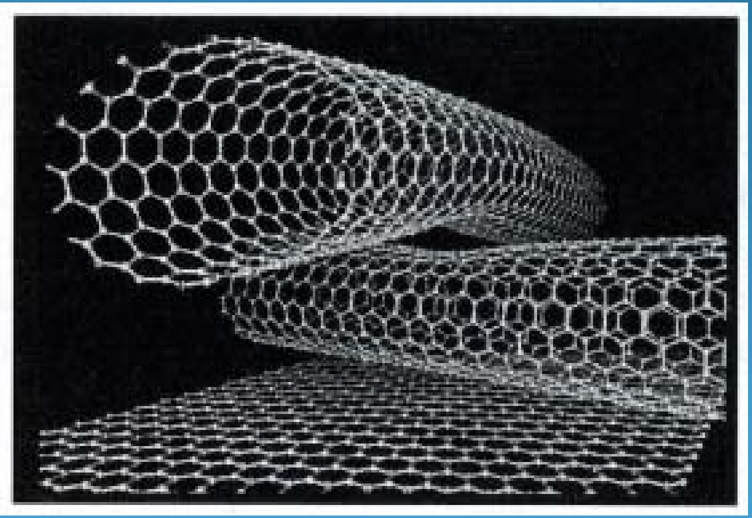
Surface to bulk ratio

$$N_{surf} / N \cong 3a / R$$

- For R = 6a ~1nm, more than ½ of the atoms are on the surface.
- Gas storage
- Catalysis
- Stability—melting
- Quantized electronic and vibrational excitation—important in the 1-100nm ragan

Carbon Nanotubes and electrodes





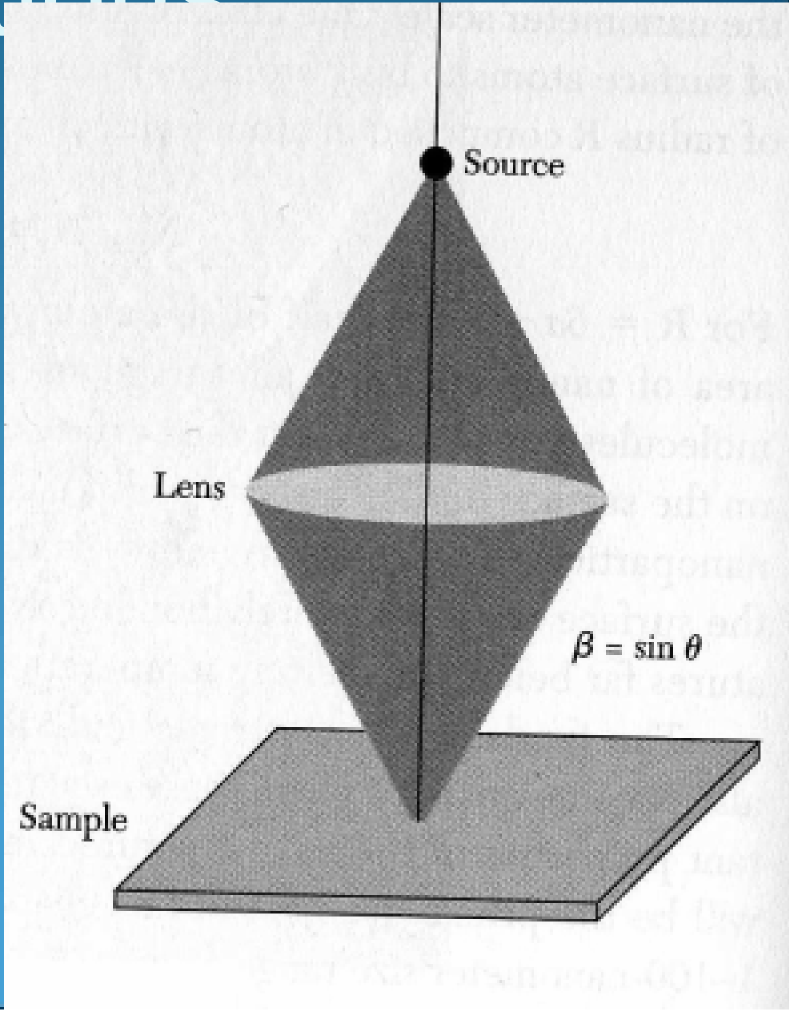
IMAGING TECHNIQUES FOR NANOSTRUCTURES

- Need new methods to image and probe nanostructures
- Limitation in X-ray diffraction for nanostructures
- Real-space probes
- Focal and Scanning probes

Focal Microscopy—resolution limitations

- Wavelike nature of the particles
- Heisenberg Uncertainty Principles
- Resolution:

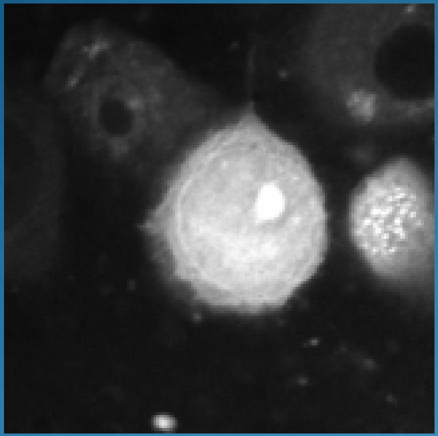
$$d \approx \lambda / 2\beta$$

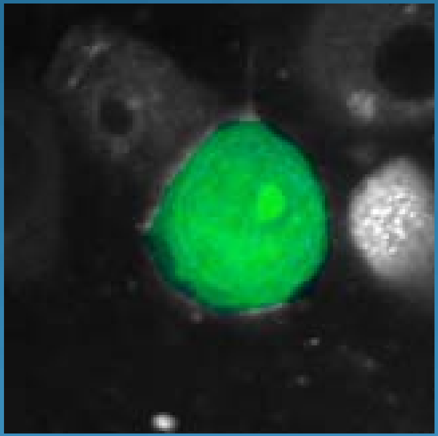


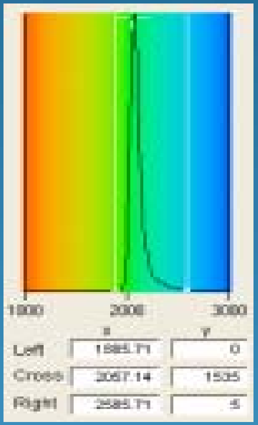
Optical Microscopy

- Resolution limited
- Live specimen
- Low damage
- Rich spectroscopy content
- Confocal microscopy
- Fluorescence microscopy

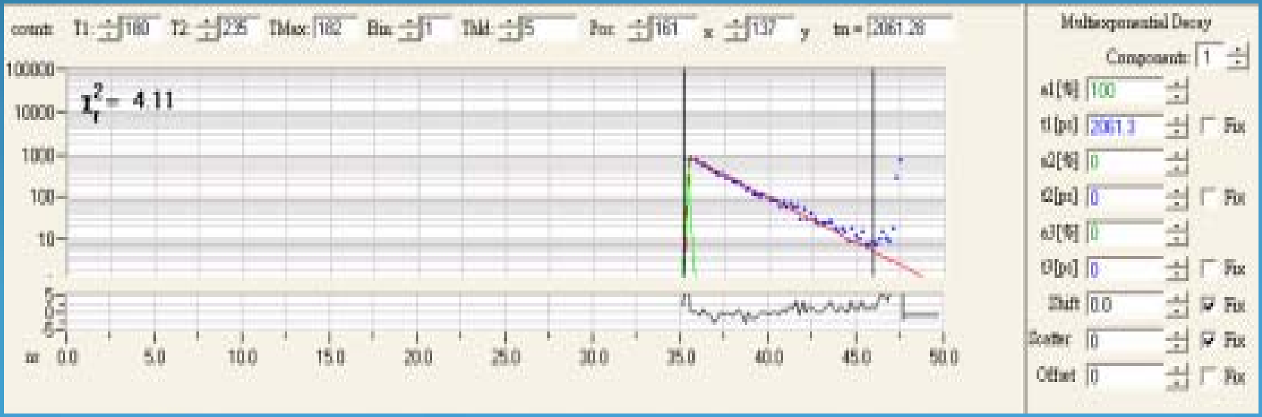
eGFP in HeLa cells — single exponential fit



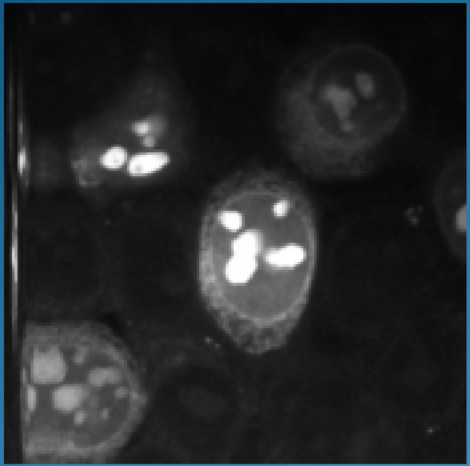


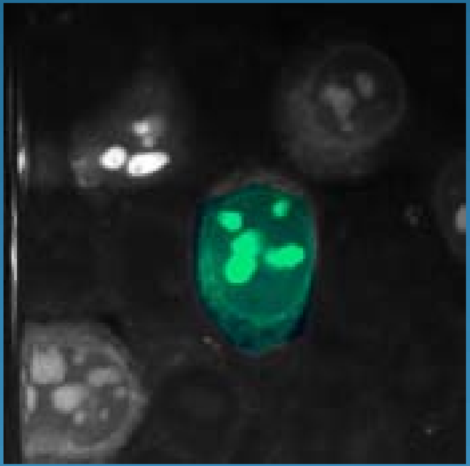


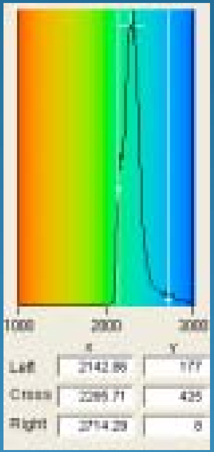
Lifetime (ps)	
1	2208
2	2213
3	2162
4	2157
5	2208
6	2042
Average	2165



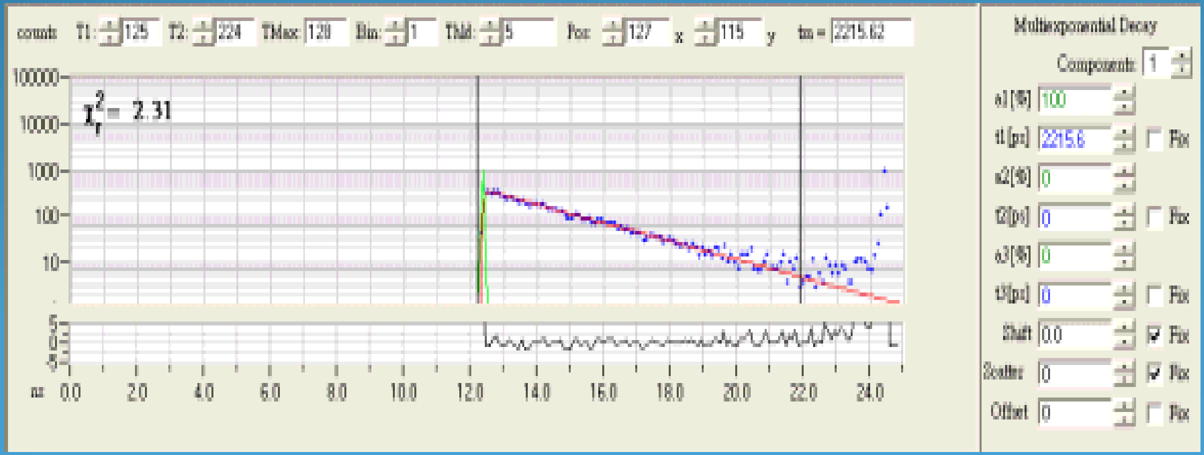
Core protein (of Dengue Virus) – eGFP single exponential fit





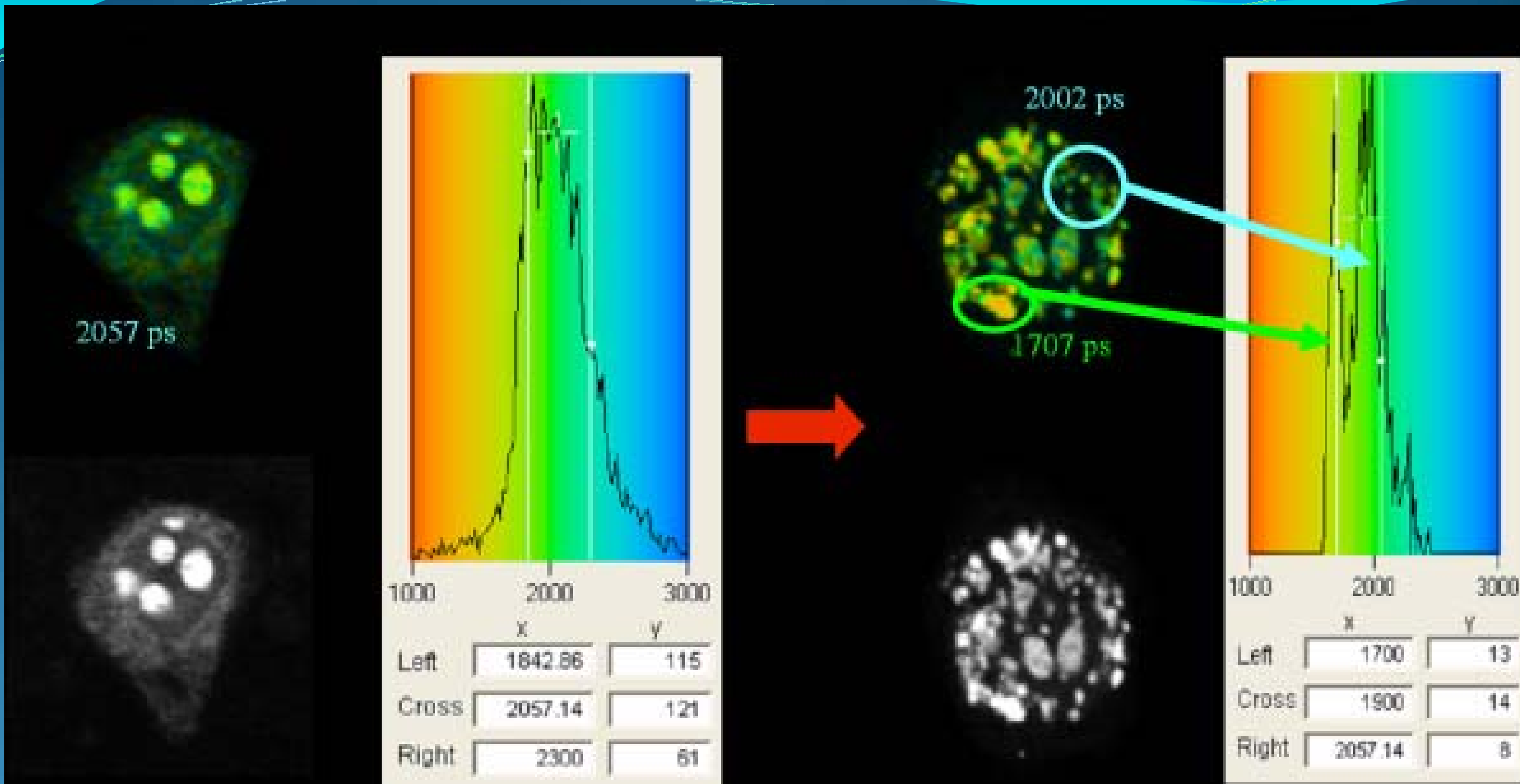


lifetime (ps)	
1	2270
2	2228
3	2057
4	1971
average	2131 ps



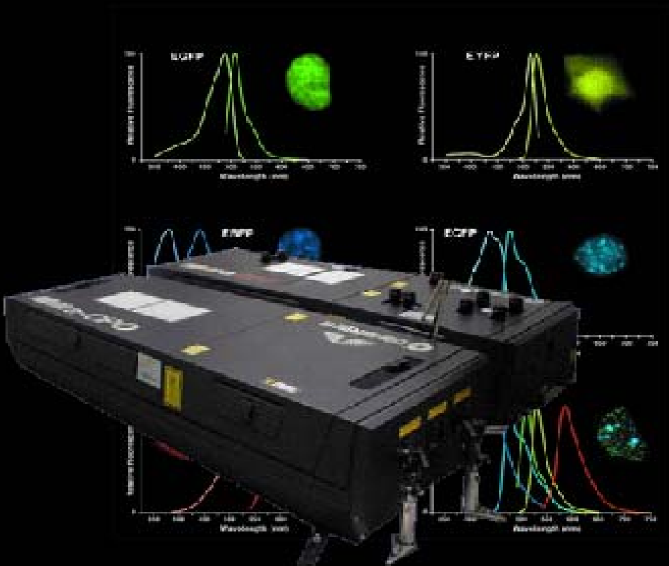
eGFP

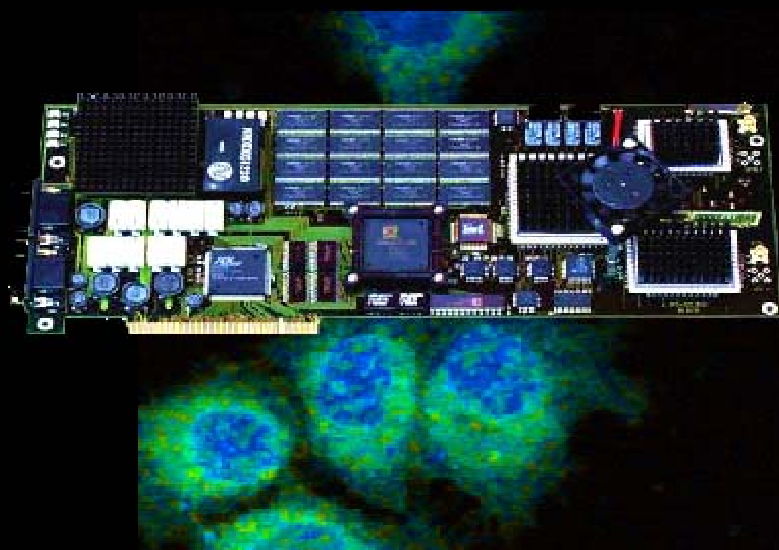
eGFP & dsRed2

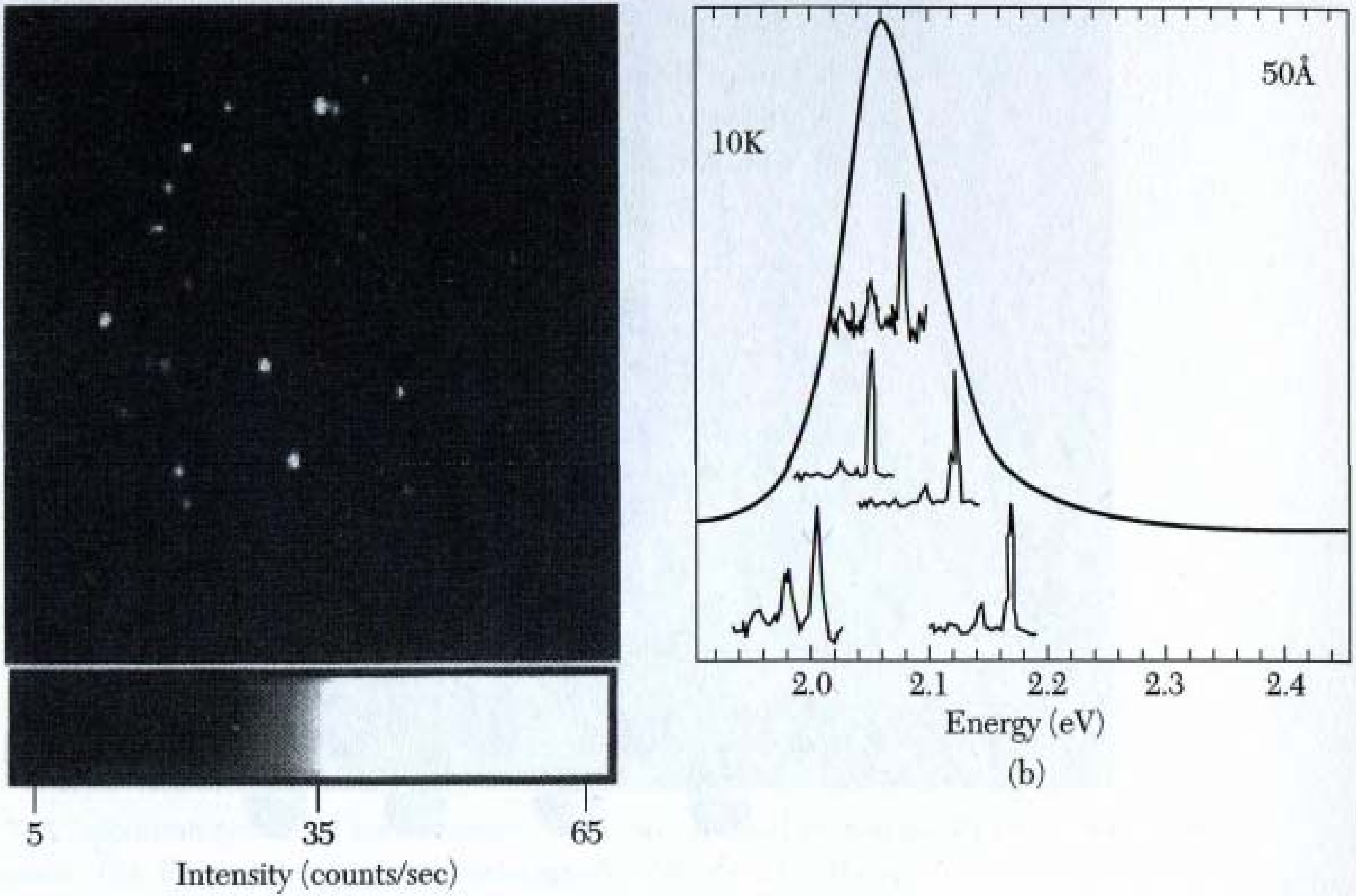


Laser wavelength	800nm
Laser power	19mW
Objective	40x/1
Scanning rate	10kHz
Collection time	30 sec

sample	HeLa Cell	
Fluorophores	eGFP	eGFP & dsRed (FRET)
lifetime	2057 ps	1700 ps



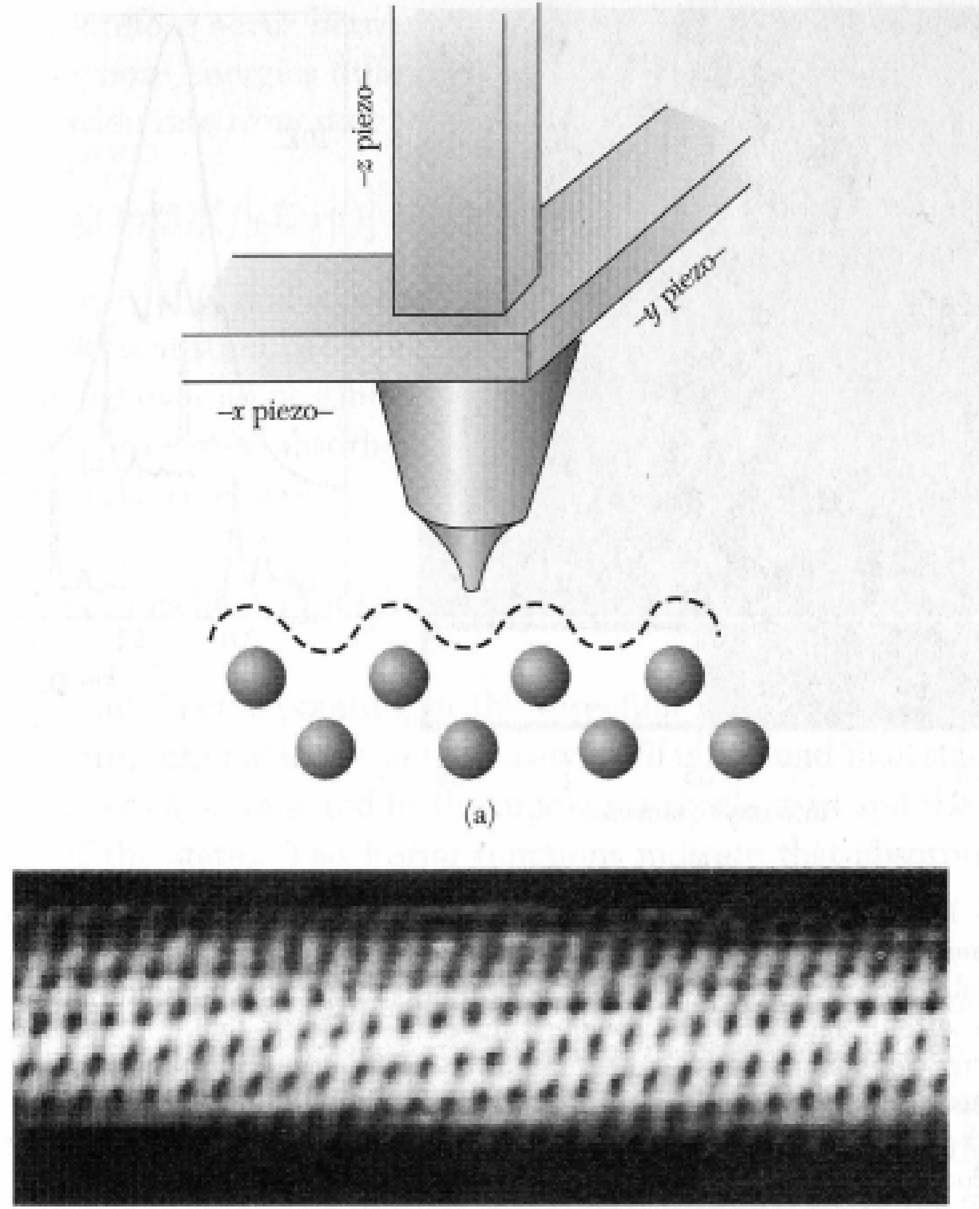




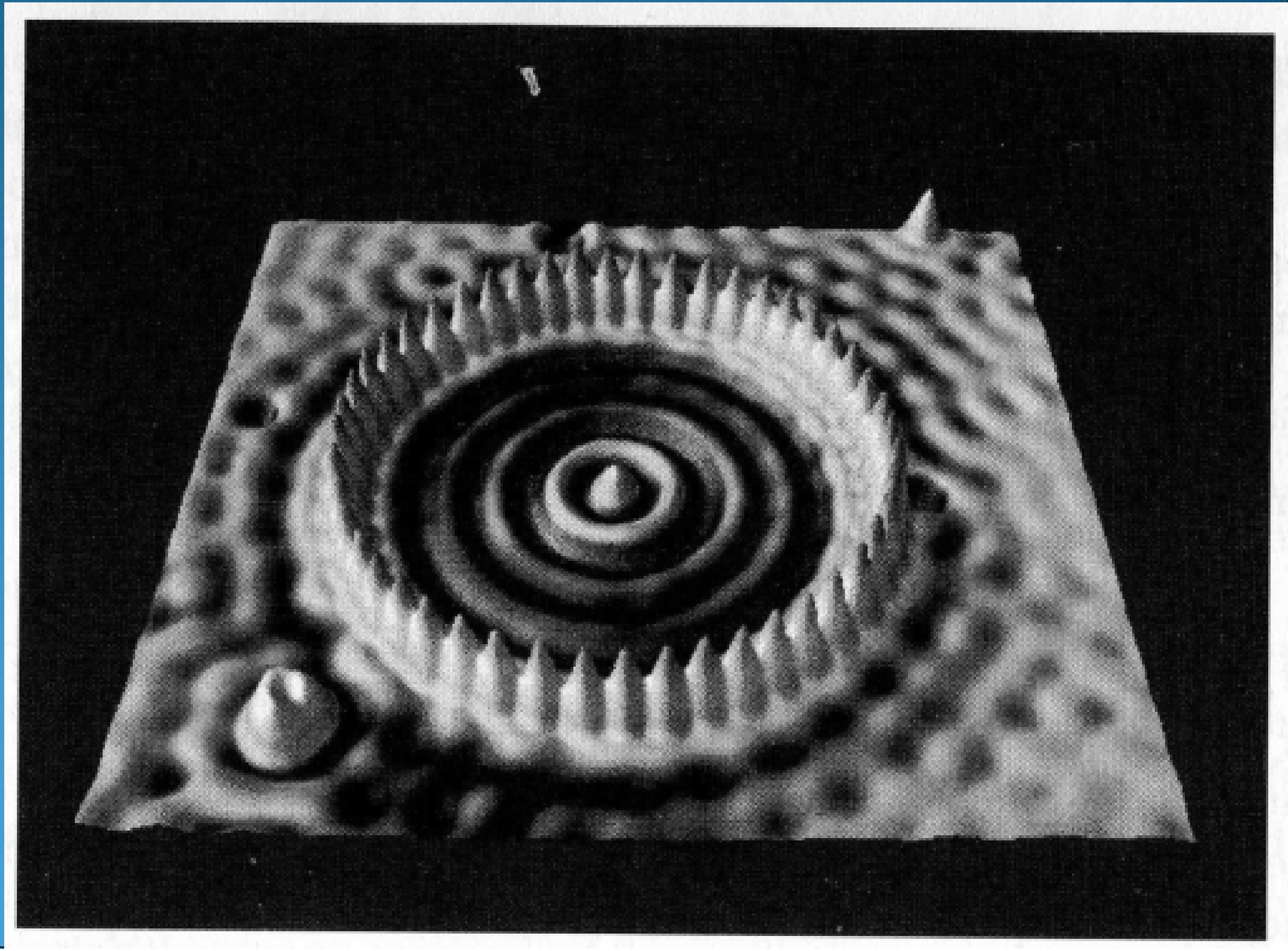
(a) (b)

Scanning Probe Microscopy

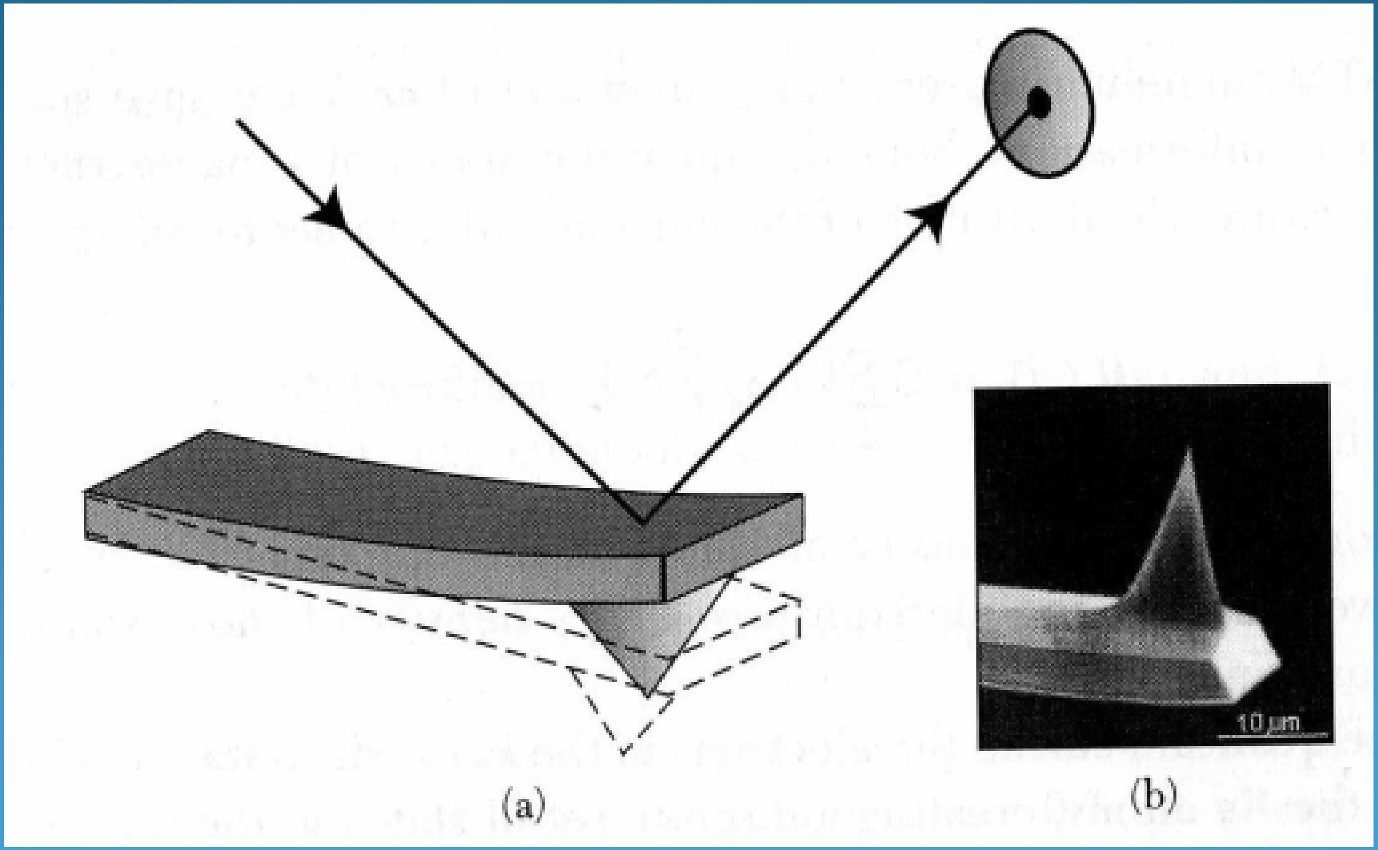
- AFM, atomic force microscopy
  - contact AFM
  - non-contact AFM
  - dynamic contact AFM
- EFM, electrostatic force microscope
- ESTM electrochemical scanning tunneling microscope
- FMM, force modulation microscopy
- KPFM, kelvin probe force microscopy
- MFM, magnetic force microscopy
- MRFM, magnetic resonance force microscopy
- NSOM, near-field scanning optical microscopy (or SNOM, scanning near-field optical microscopy)
- PSTM, photon scanning tunneling microscopy
- SECM, scanning electrochemical microscopy
- SCM, scanning capacitance microscopy
- SGM, scanning gate microscopy
- SICM, scanning ion-conductance microscopy
- SPSM spin polarized scanning tunneling microscopy
- SThM, scanning thermal microscopy
- STM, scanning tunneling microscopy
- SVM, scanning voltage microscopy



(a)
(b)



Atomic Force Microscopy



Advantages of scanning probe microscopy

- The resolution of the microscopes is not limited by diffraction, but only by the size of the probe-sample interaction volume (i.e., point spread function), which can be as small as a few picometres.
- The interaction can be used to modify the sample to create small structures (nanolithography).

Disadvantages of scanning probe microscopy

- The scanning techniques are generally slower in acquiring images, due to the scanning process. As a result, efforts are being made to greatly improve the scanning rate.
- The maximum image size is generally smaller.

Density of States imaging

- As long as measured in STM current is determined by the tunneling processes through tip-sample surface gap its value depends not only on the barrier height but on the electron density of states also. Accordingly obtained in STM images are not simply images of sample surface relief (topography), these images can be hardly affected by the density of electronic states distribution over the sample surface. Good example of Local Density of States (LDOS) influence on the STM image is well-known image of highly oriented pyrolitic graphite (HOPG) atomic lattice. Only half atoms are visible in STM. Similar case is image of GaAs atomic lattice.
- LDOS determining can also help to distinguish chemical nature of the surface atoms. LDOS acquisition is provided simultaneously with the STM images obtaining. During scanning the Bias Voltage is modulated on the value dU, the modulation period is chosen to be much shorter than the time constant of the feedback loop in the STM.
- Suitable modulation of tunnel current dI is measured, divided by dU and presented as LDOS image. On Example the topography and LDOS image of HOPG sample are presented.

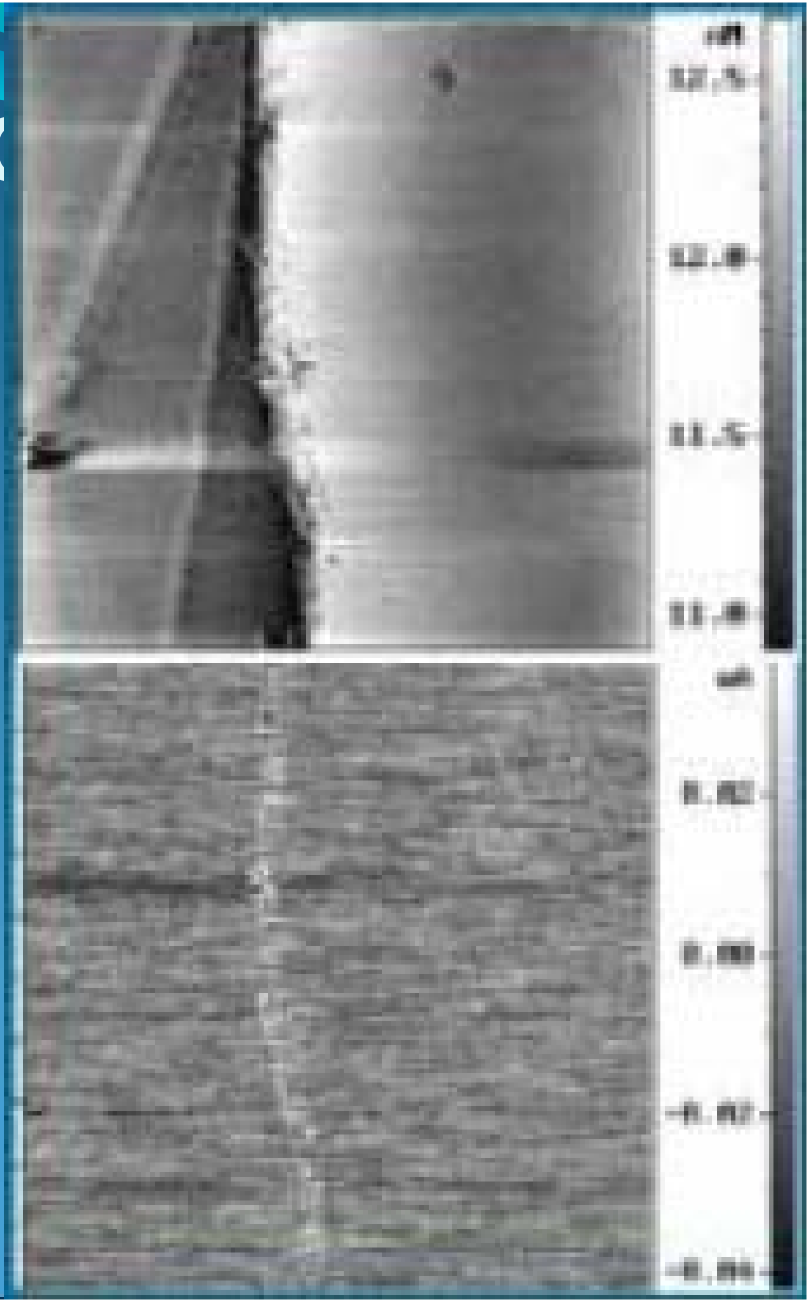
Local Density o imaging

- HOPG sample. Upper picture - topography image, lower picture - simultaneously obtained LDOS image.

  Scan size: 1 x 1 um

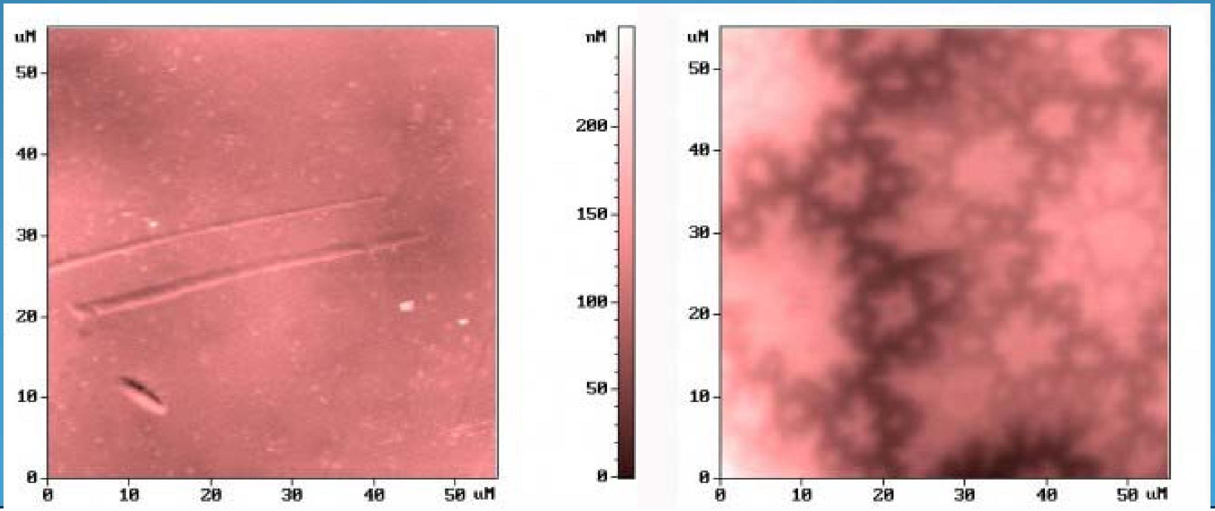
- http://www.ntmdt.ru/SPM-Techniques/Principles/

-



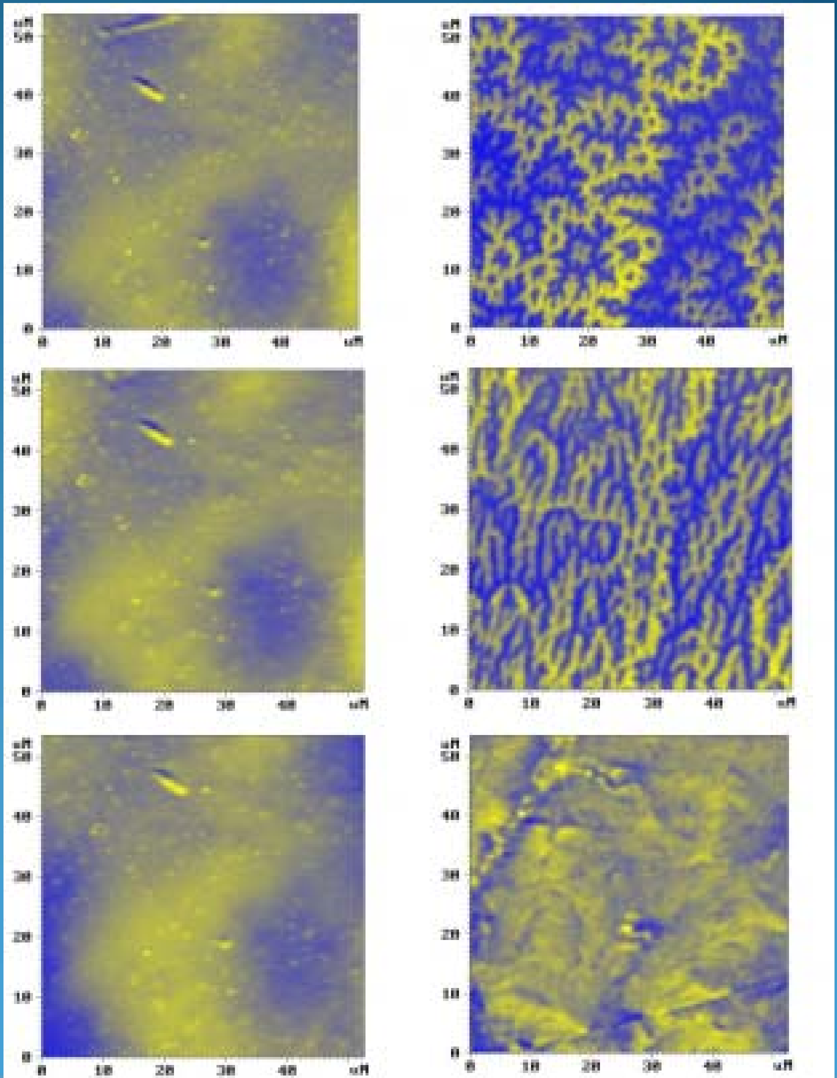
Magnetic domains of cobalt monocrystal

- Topography (left) and magnetic force distribution for same area (right) of cobalt monocrystal with strong perpendicular magnetocrystalline anisotropy. Right image corresponds to deflection of the nonvibrating cantilever. Domains of closure in the form of flowers are seen on magnetic image.



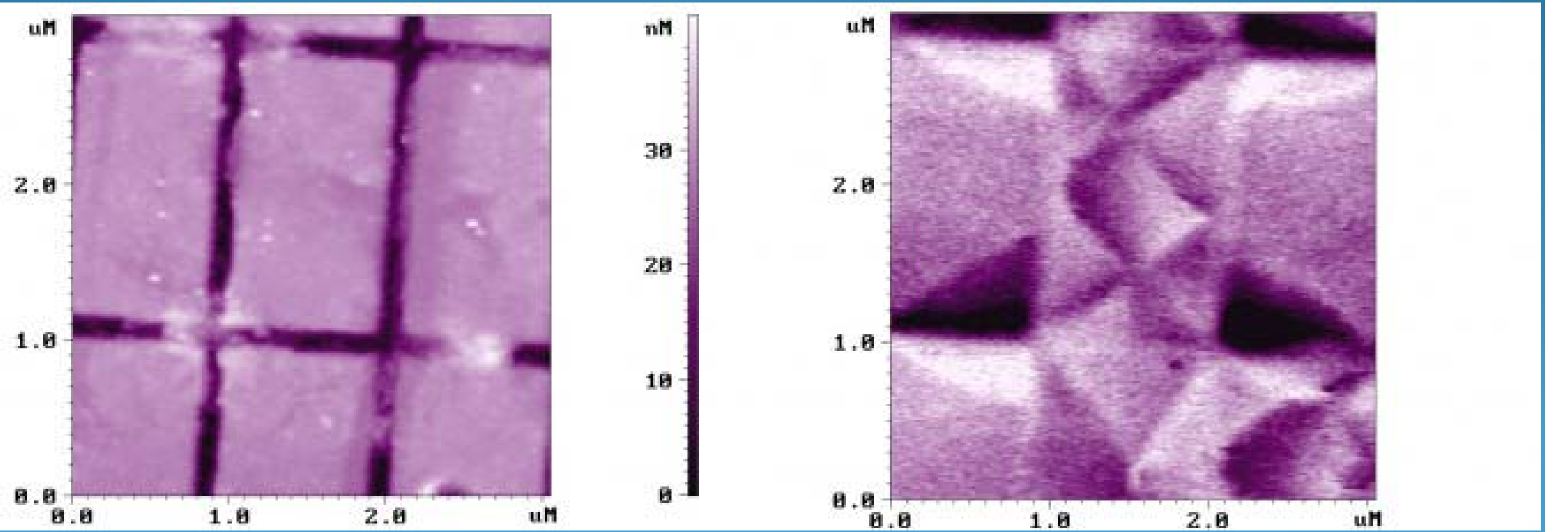
Magnetic phase transition in cobalt

- Topography (left column) and corresponding MFM images (right) of the cobalt monocrystal. Magnetic images show changes of the domain structure at heating (from top row to lower: 220C, 250C, 290C).



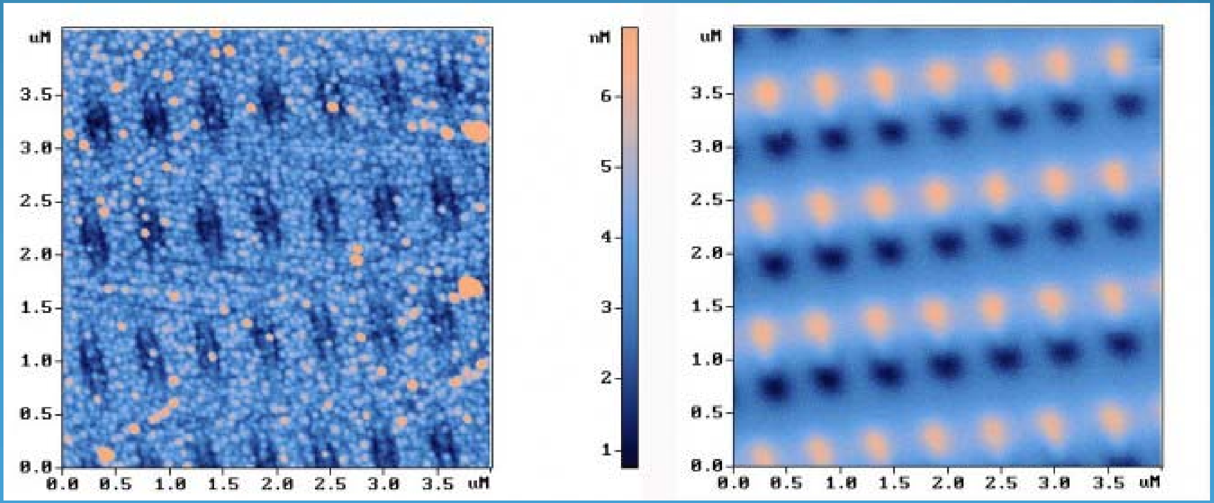
Magnetic structures of permalloy

- Left image is topography of the thin film permalloy rectangles, and right image shows corresponding domain structure.



Lattice of magnetic dipoles

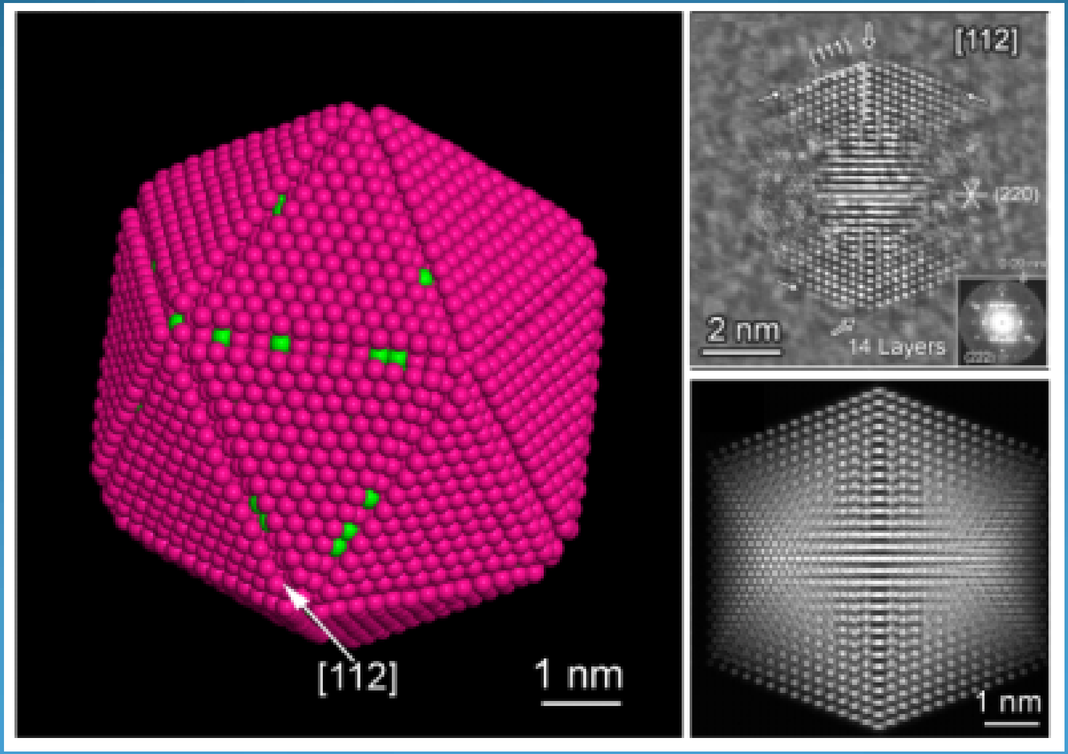
- Topography (left) and magnetic force microscopy image (right) of ferromagnetic islands in paramagnetic film. Magnetic image looks like a sub-micron dipole array. Dark and light areas on right image correspond to different poles. This is perspective data storage medium. Both images were obtained simultaneously by two-pass method.

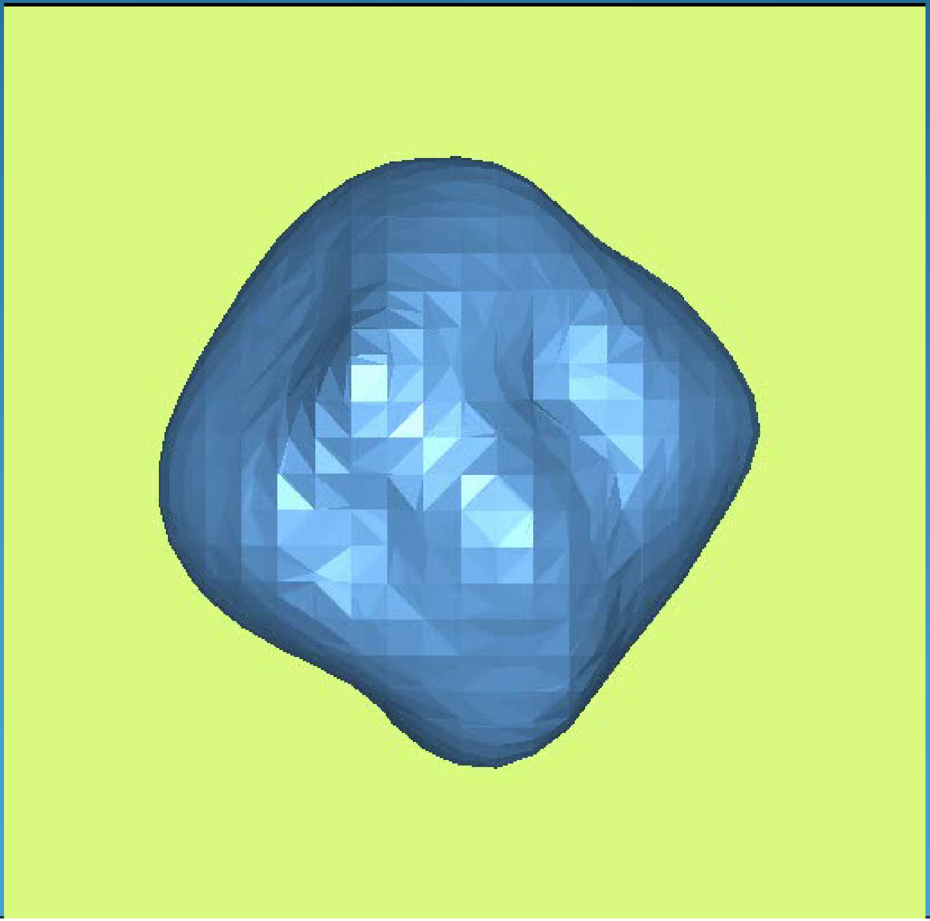


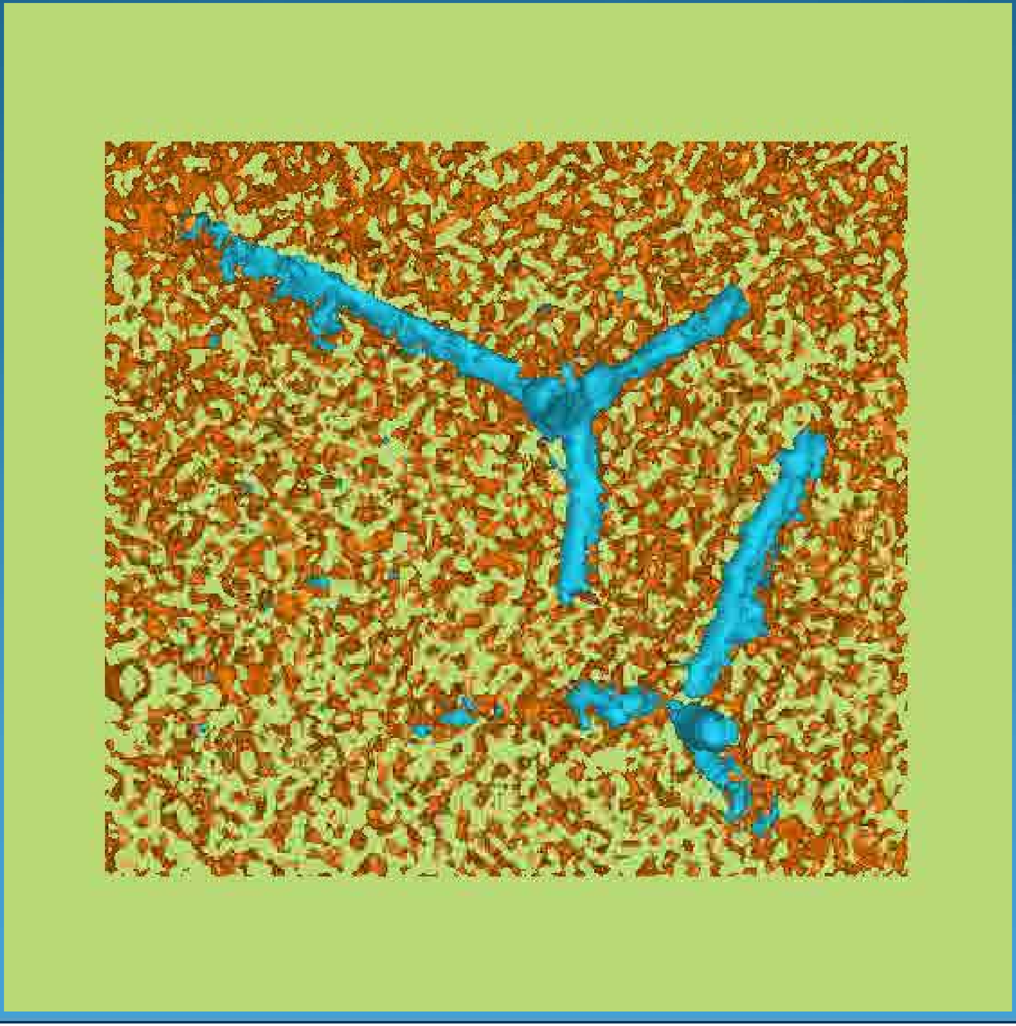
Force Modulation mode

Transmission Electron Microscopy

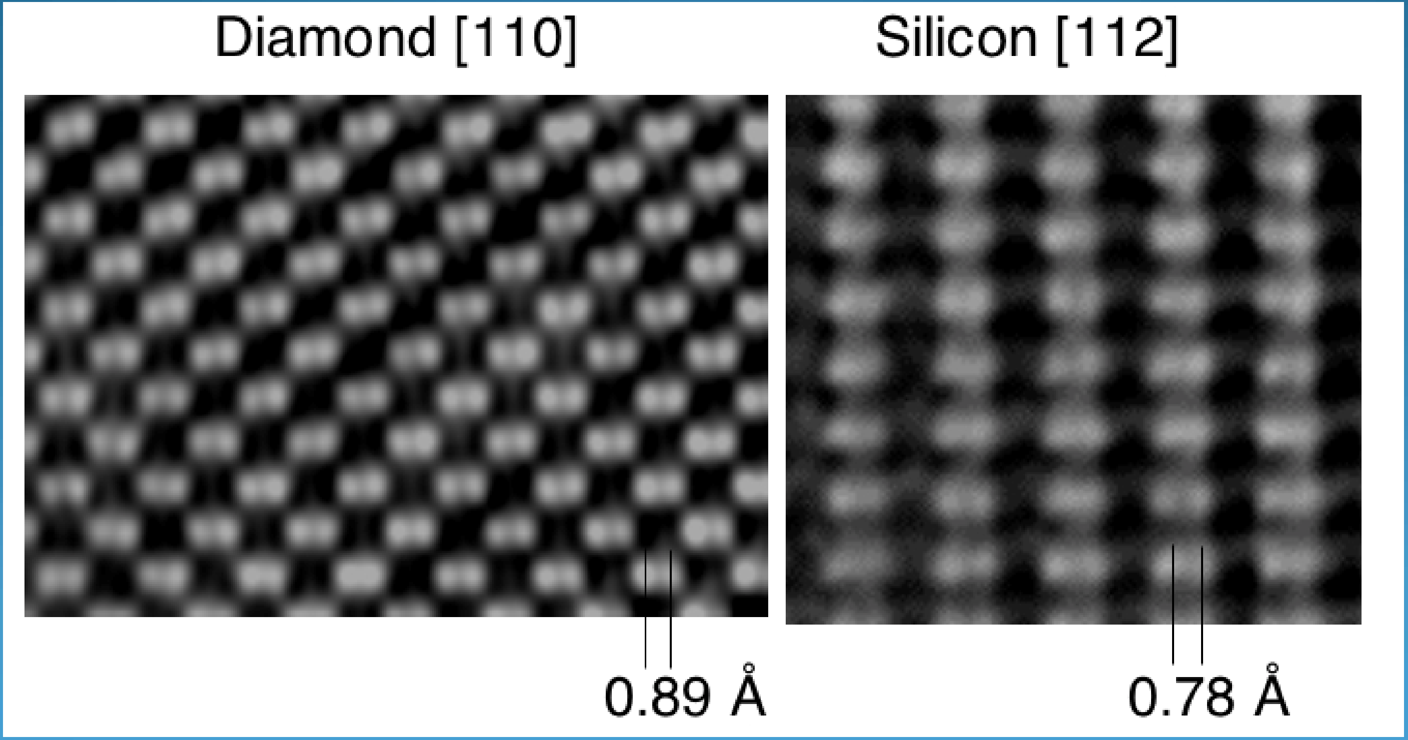
- http://www.matter.org.uk/tem/default.htm


[112]
1 nm
[112]
(220)
2 nm
14 Layers
1 nm

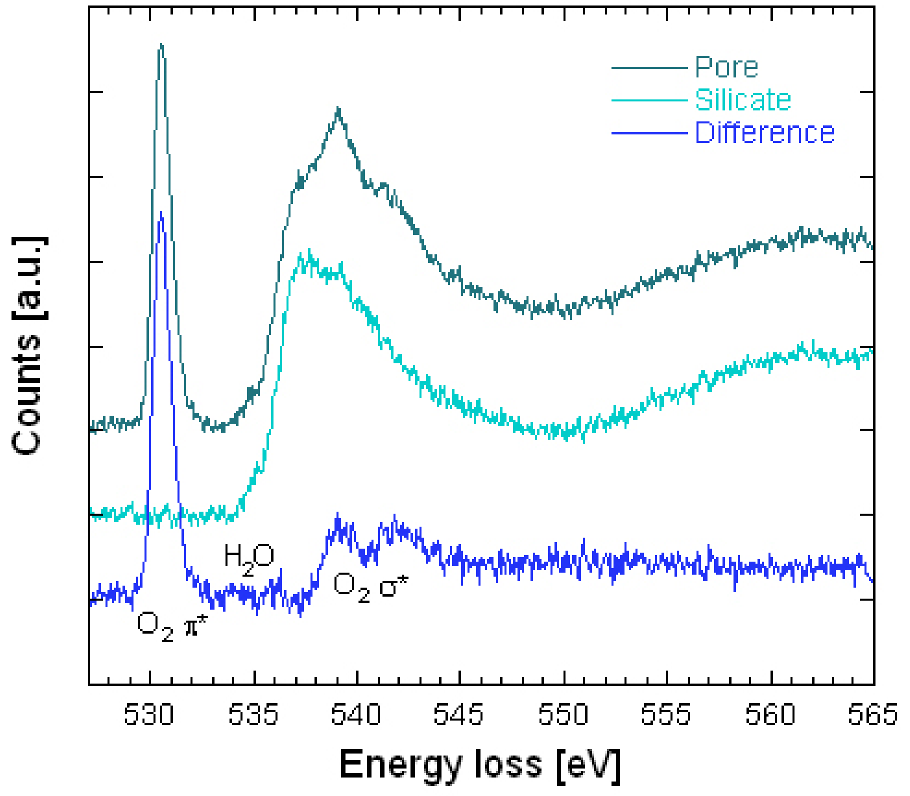


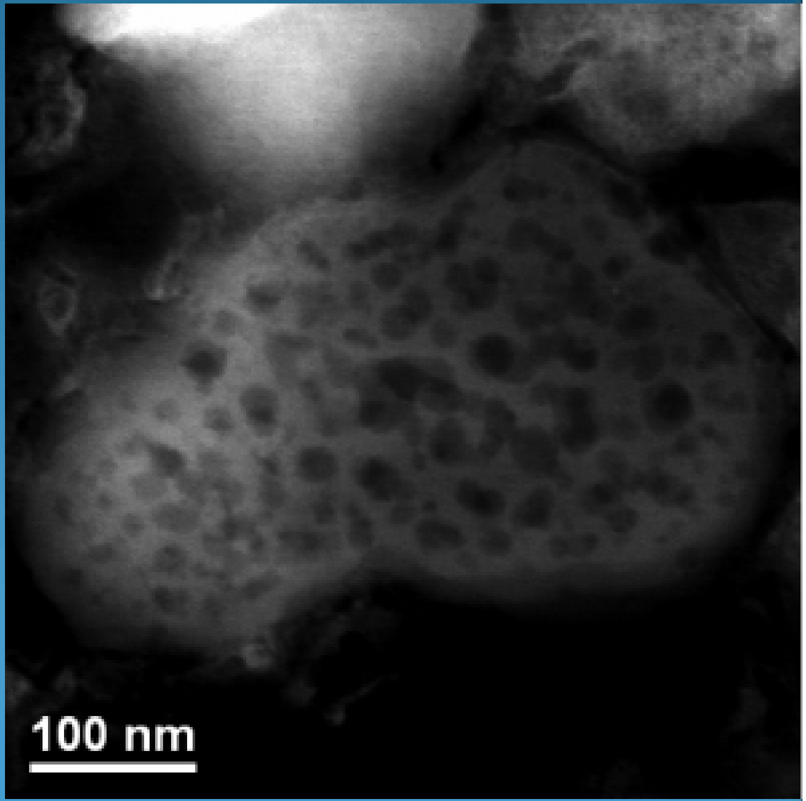


Atomic Resolution

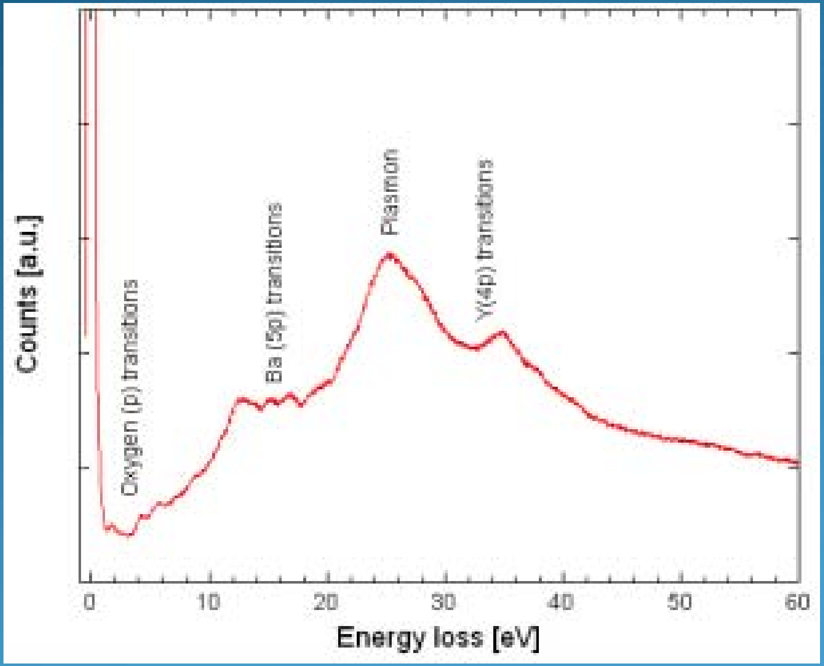


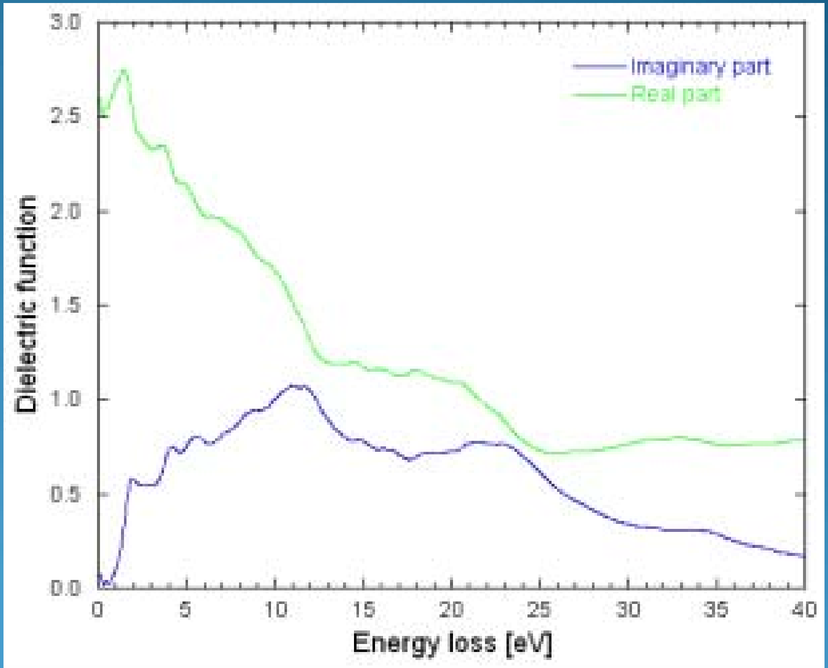
Core-Loss Spectroscopy





Low-Loss Spectroscopy





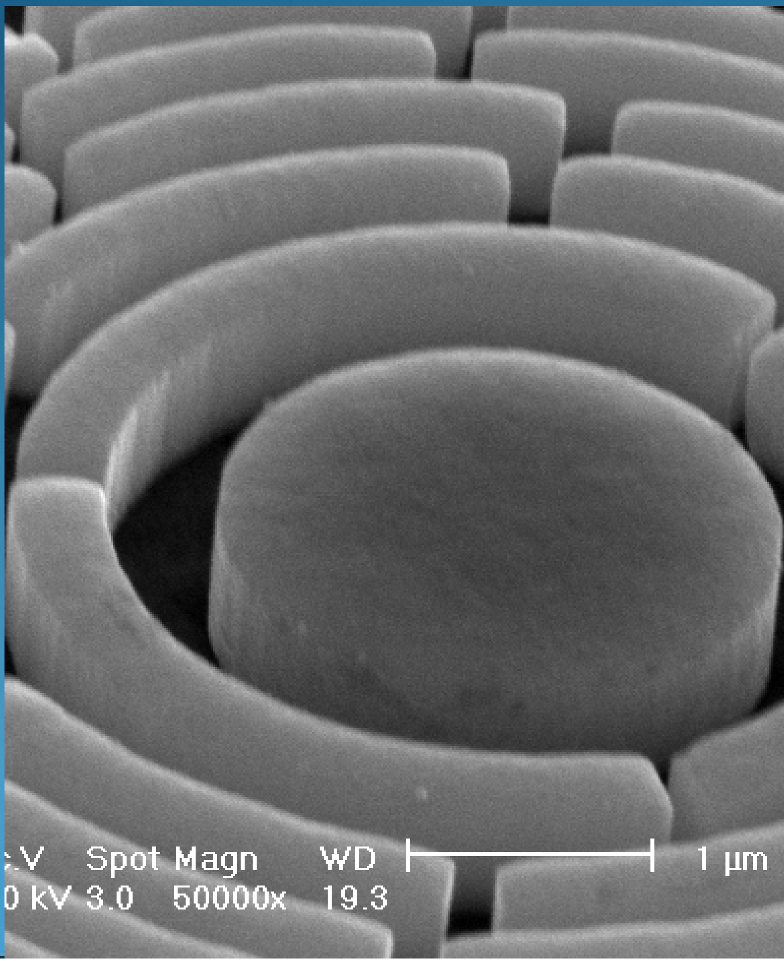
Phase Retrieval Devices

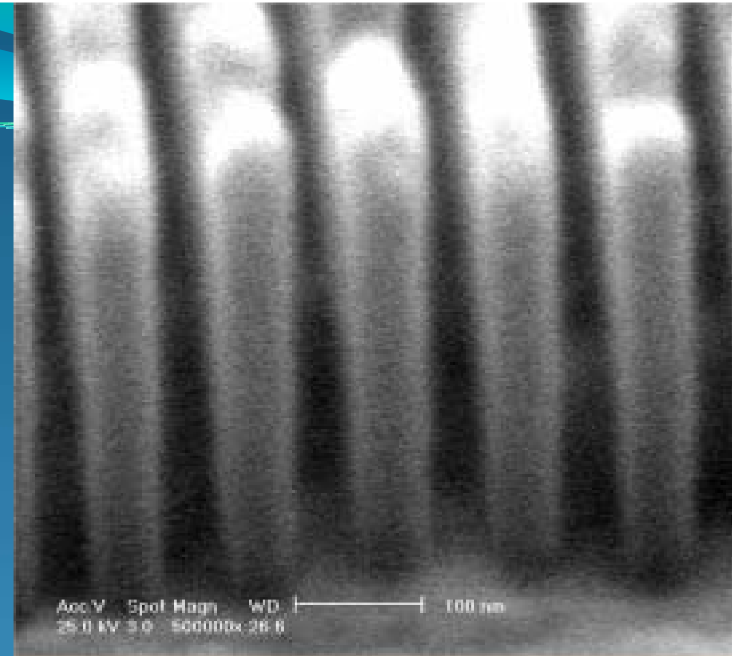
- X-rays
  - Zernike Phase plate
  - Interferometer phase grating
  - holography
- Electrons
  - Zernike Phase plate
  - holography

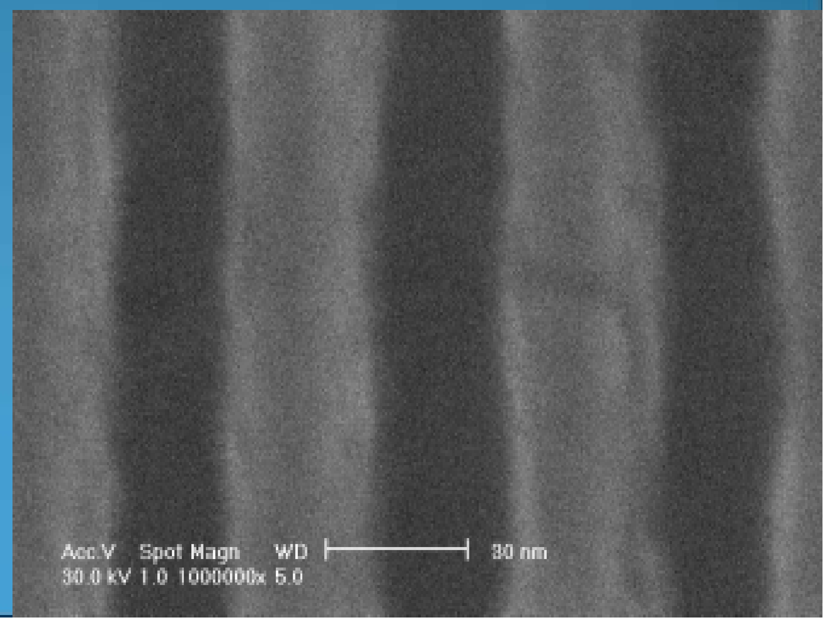
Nanofabrication:

45nm/300nm

Outmost zones

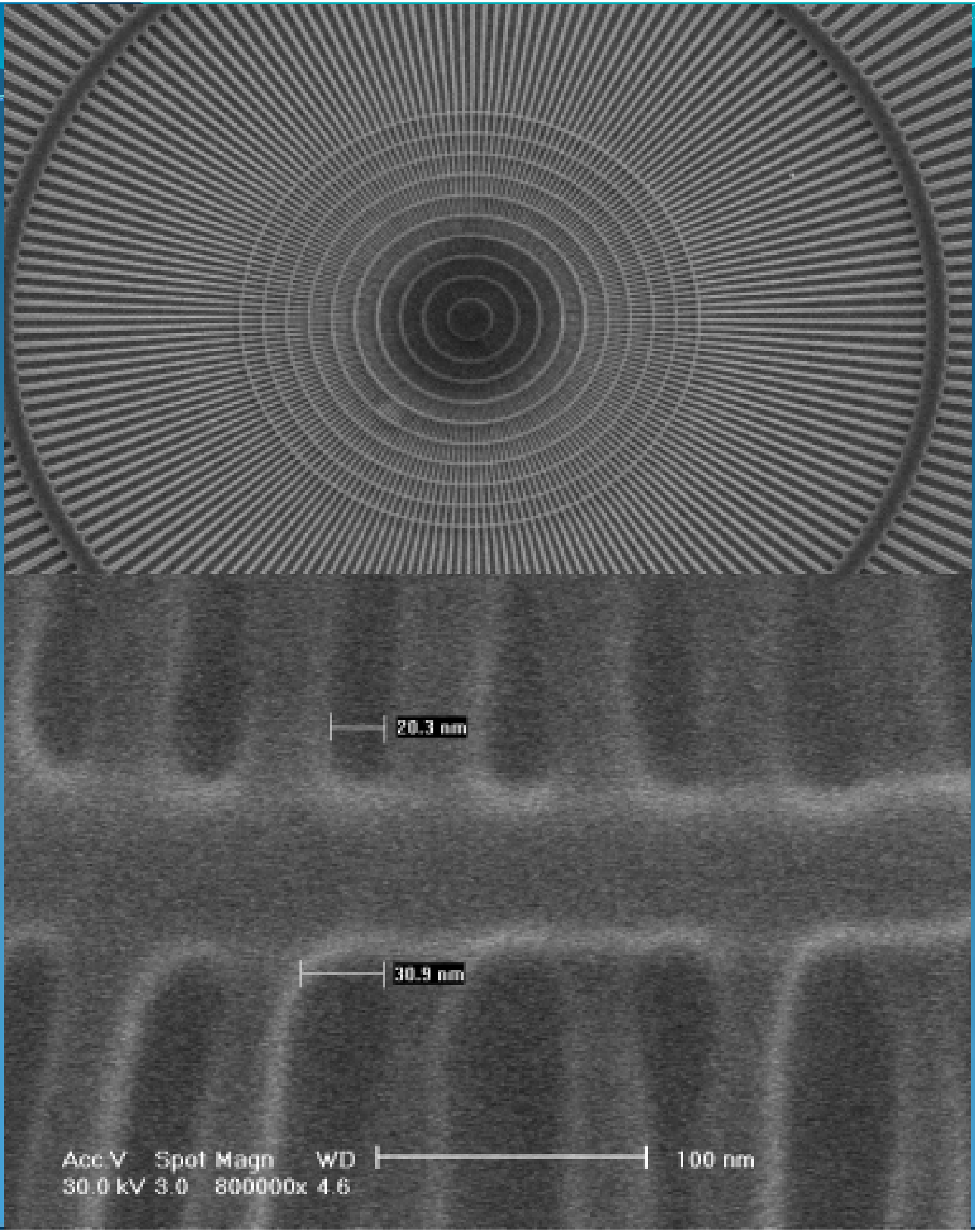






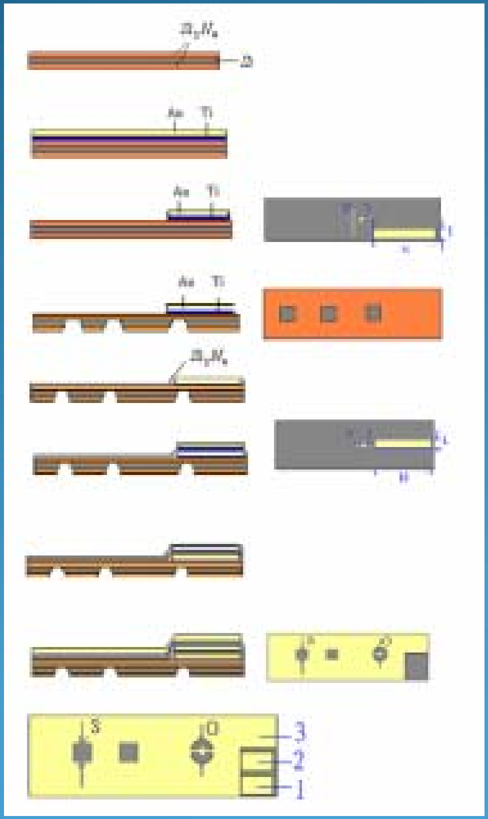
Test pattern

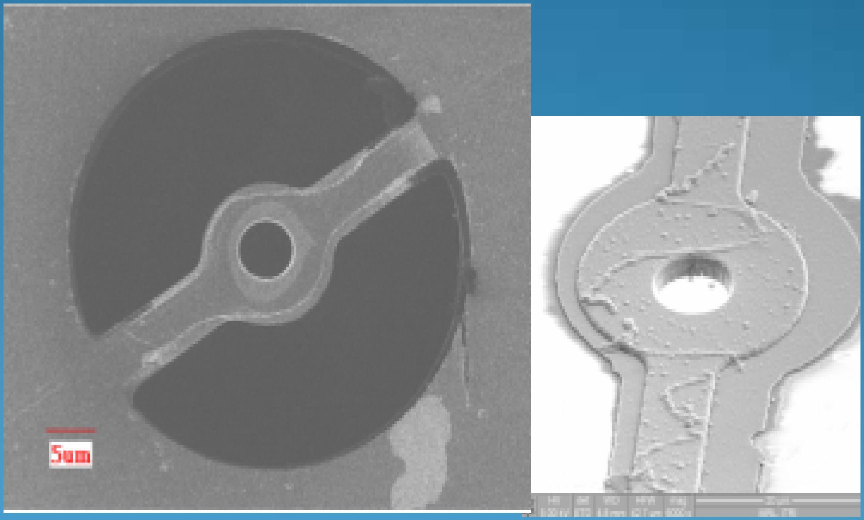
20 nm line width
400 nm thick
photoresist



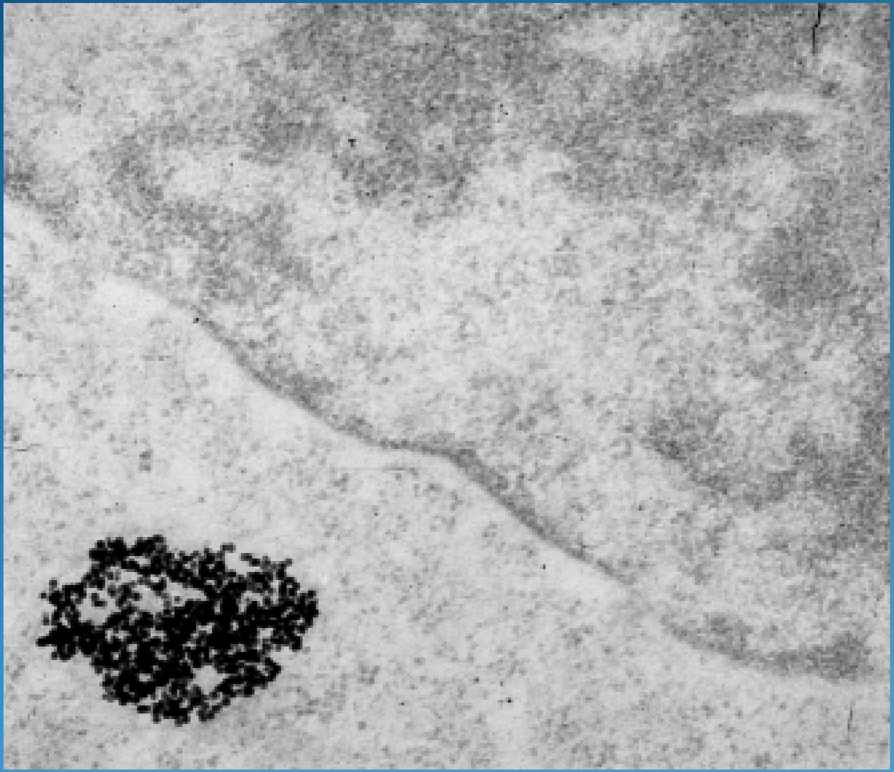
Zernike Type Electrostatic Phase plate manufactured by MEMS process

LPCVD low stress Si nitride

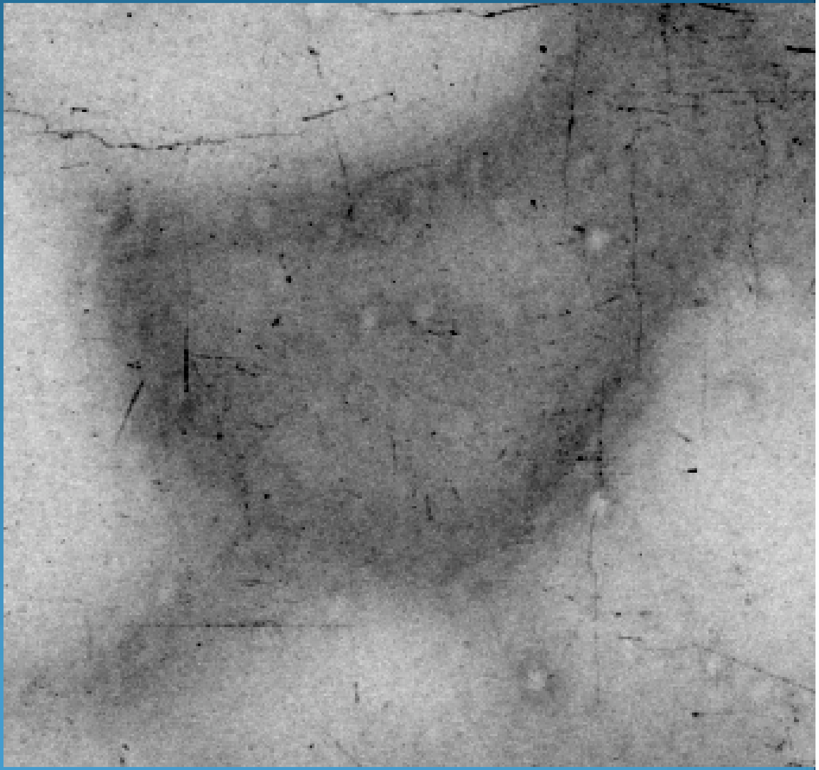




Phase enhanced TEM images of biology specimens

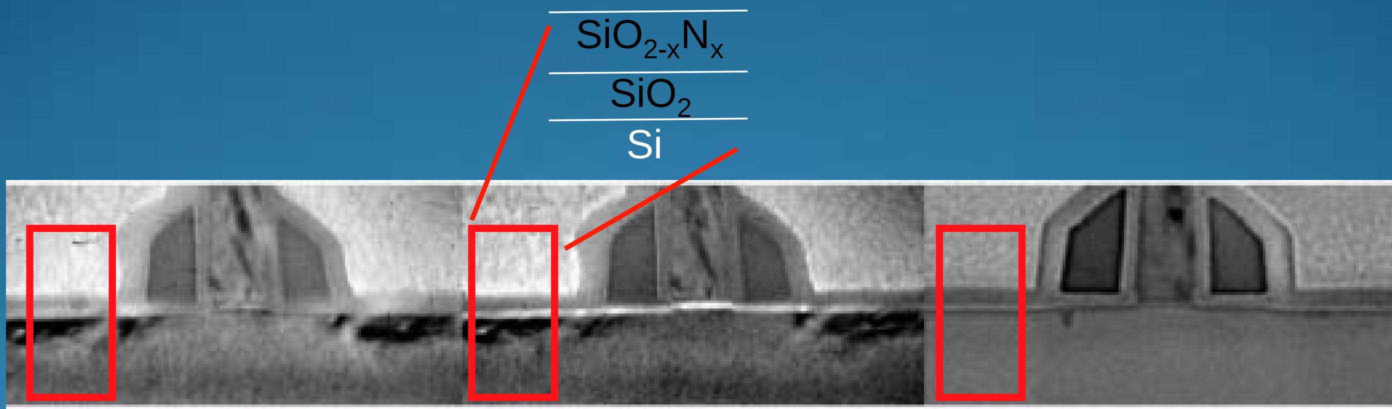


CT26 Cell



Drosophila brain

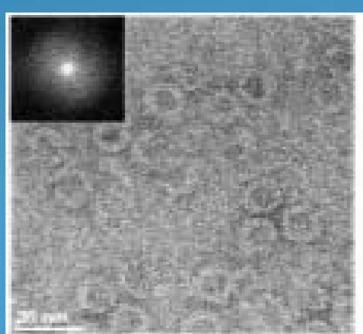
Phase Contrast Imaging



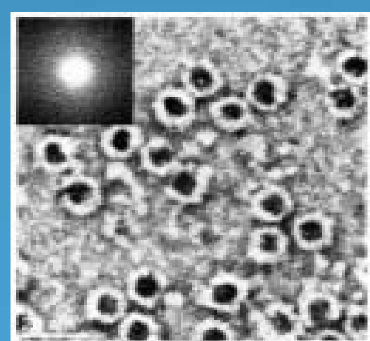
$\Delta f = 0nm$

Electro-static phase Plate

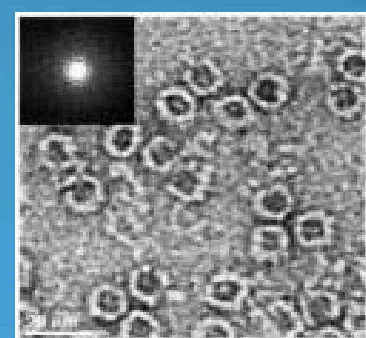
$\Delta f = -15360nm$
de-localization of image



$\Delta f = -120nm$



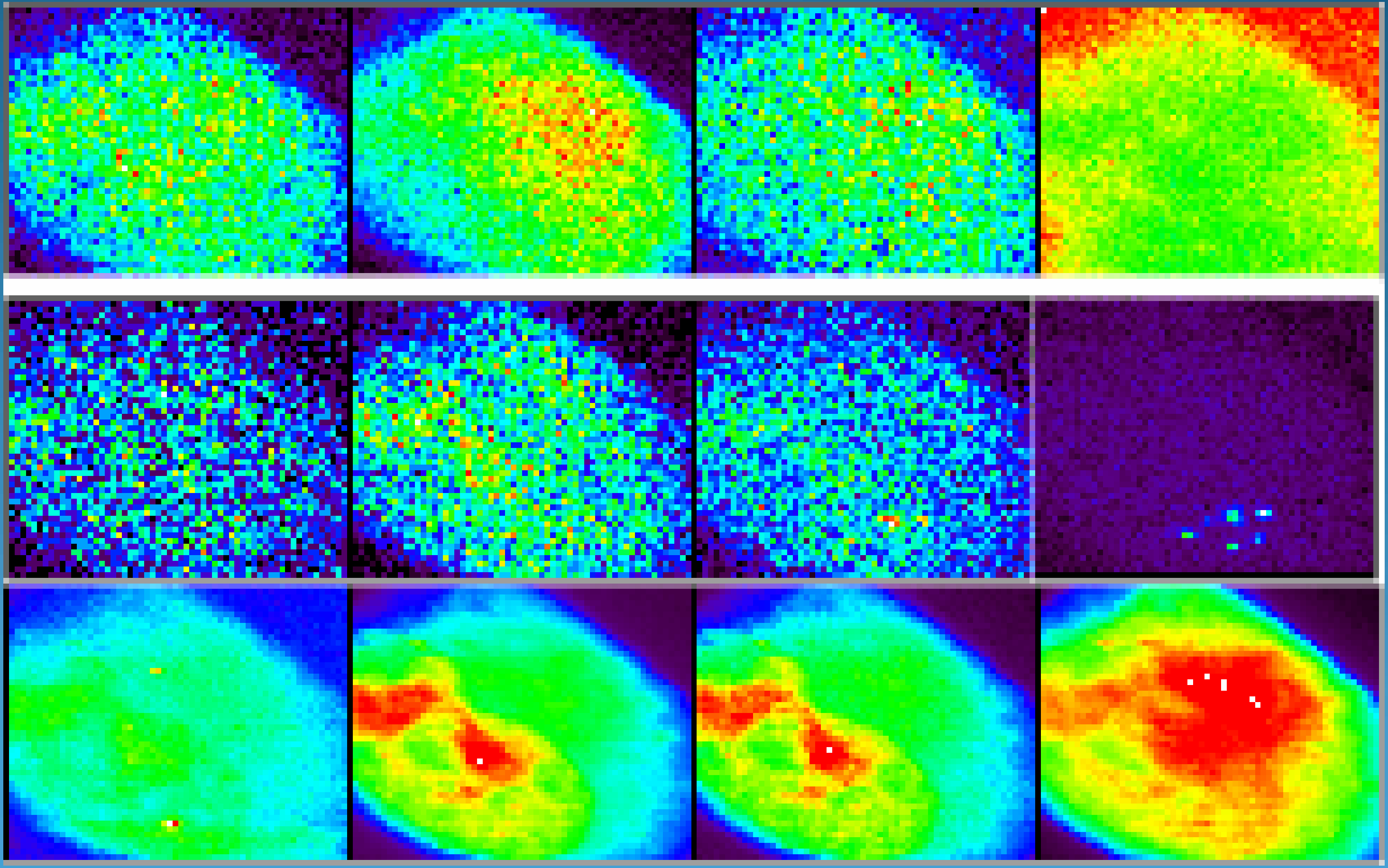
Carbon Film Phase Plate



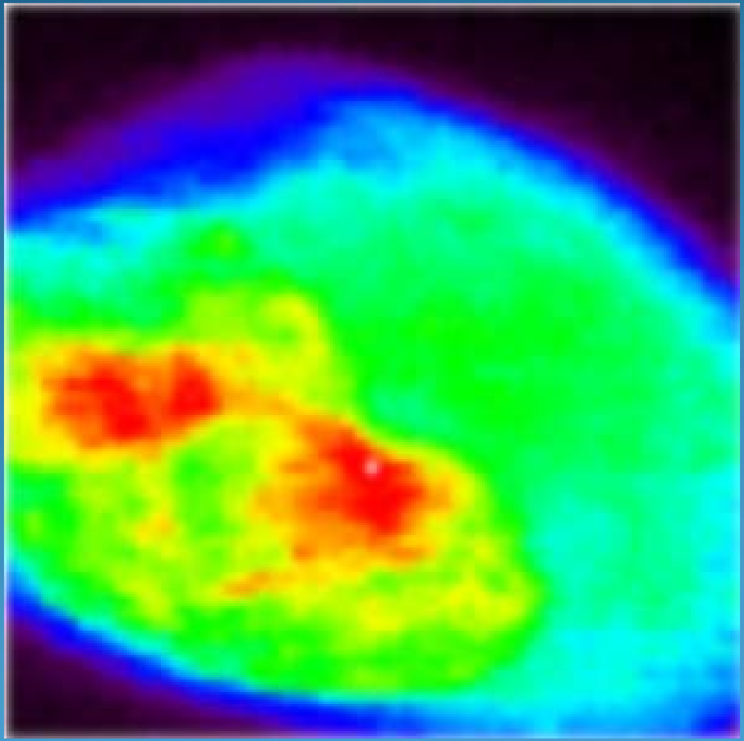
$\Delta f = -4050nm$

X-ray Fluorescence image of a single cell with 150 nm x-ray microbeam

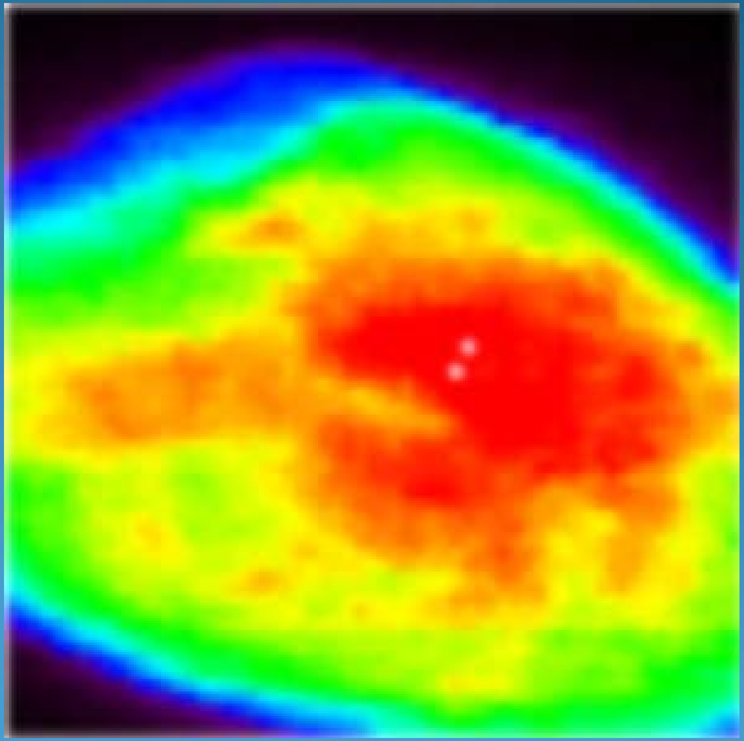
APS, Argonne National Laborator



Ca and Os distribution in a single cell

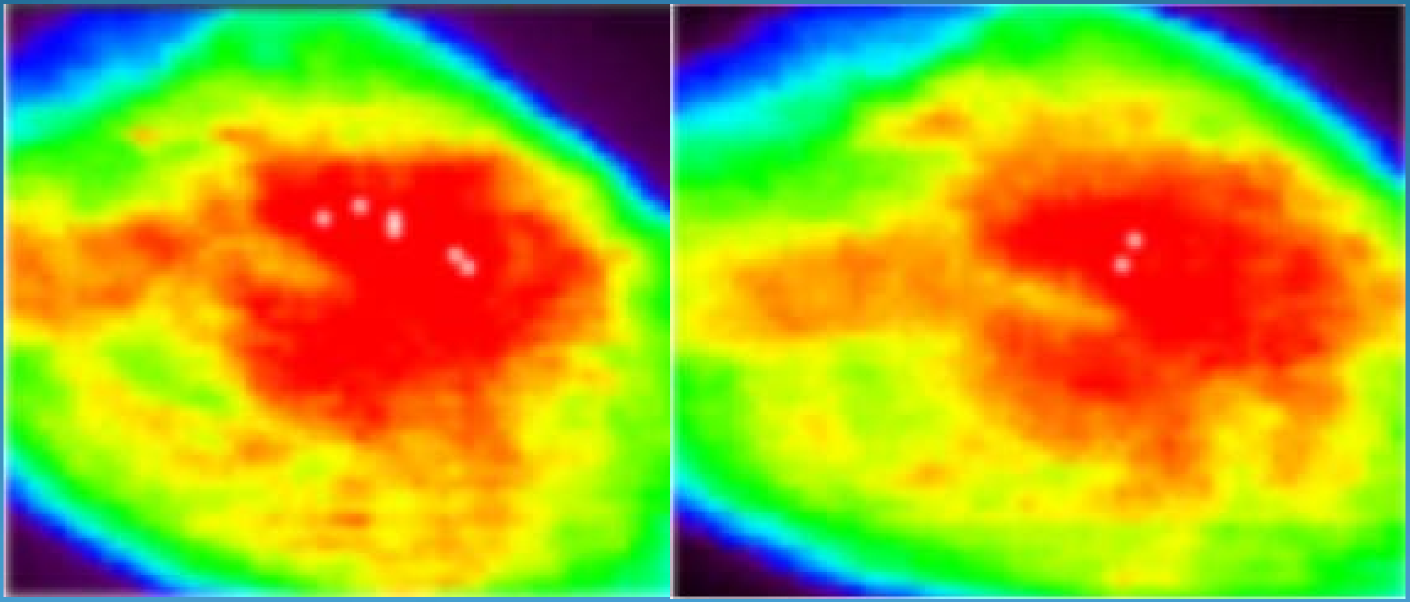


Ca



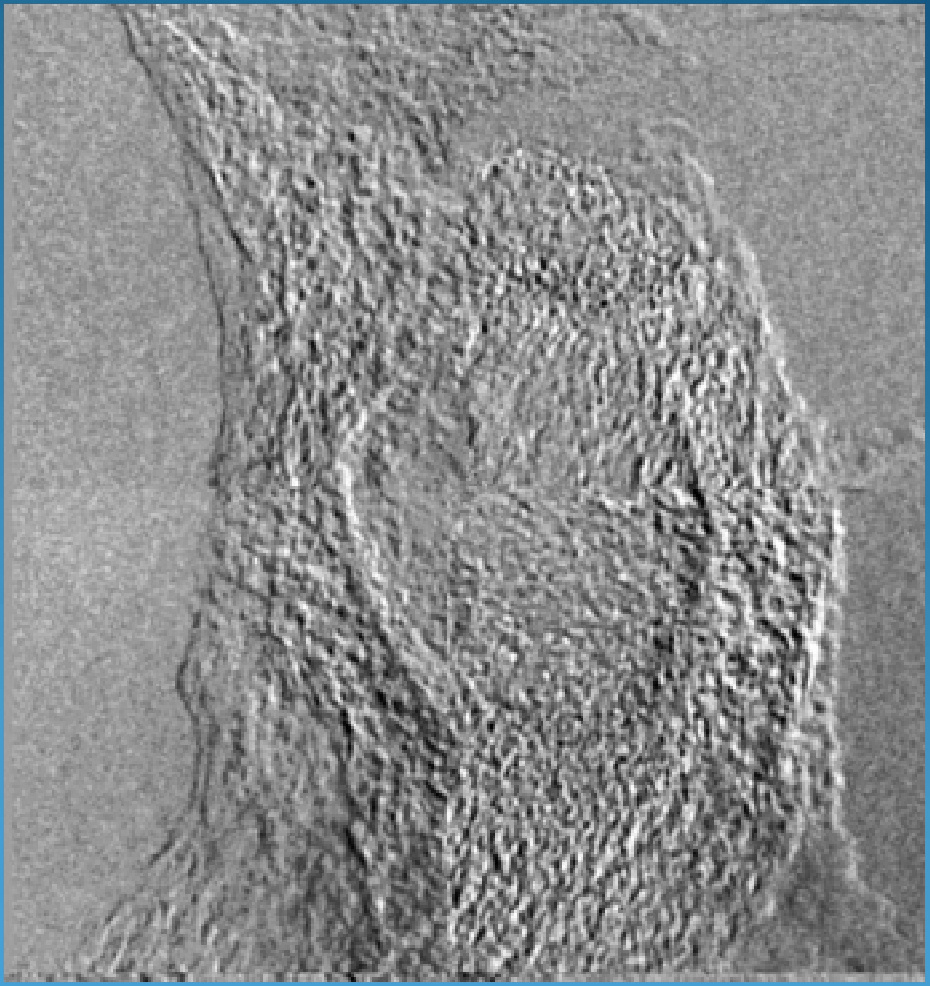
Os

Os distribution viewed from different angle—3D structure information can be obtained



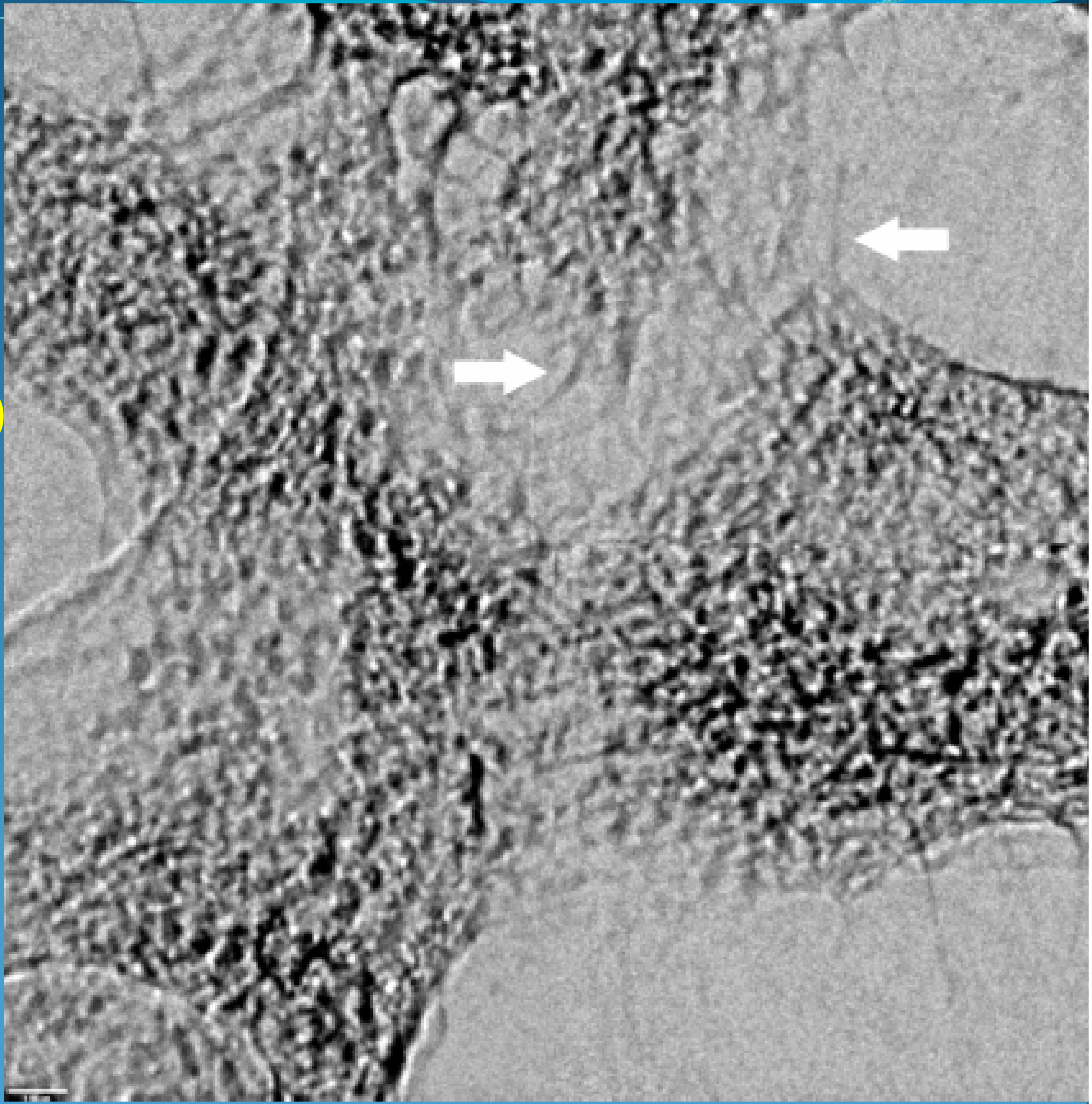
In collaboration with B. Lai, APS, Argonne

Human HaLa Cell
radiograph of 60nm resolution



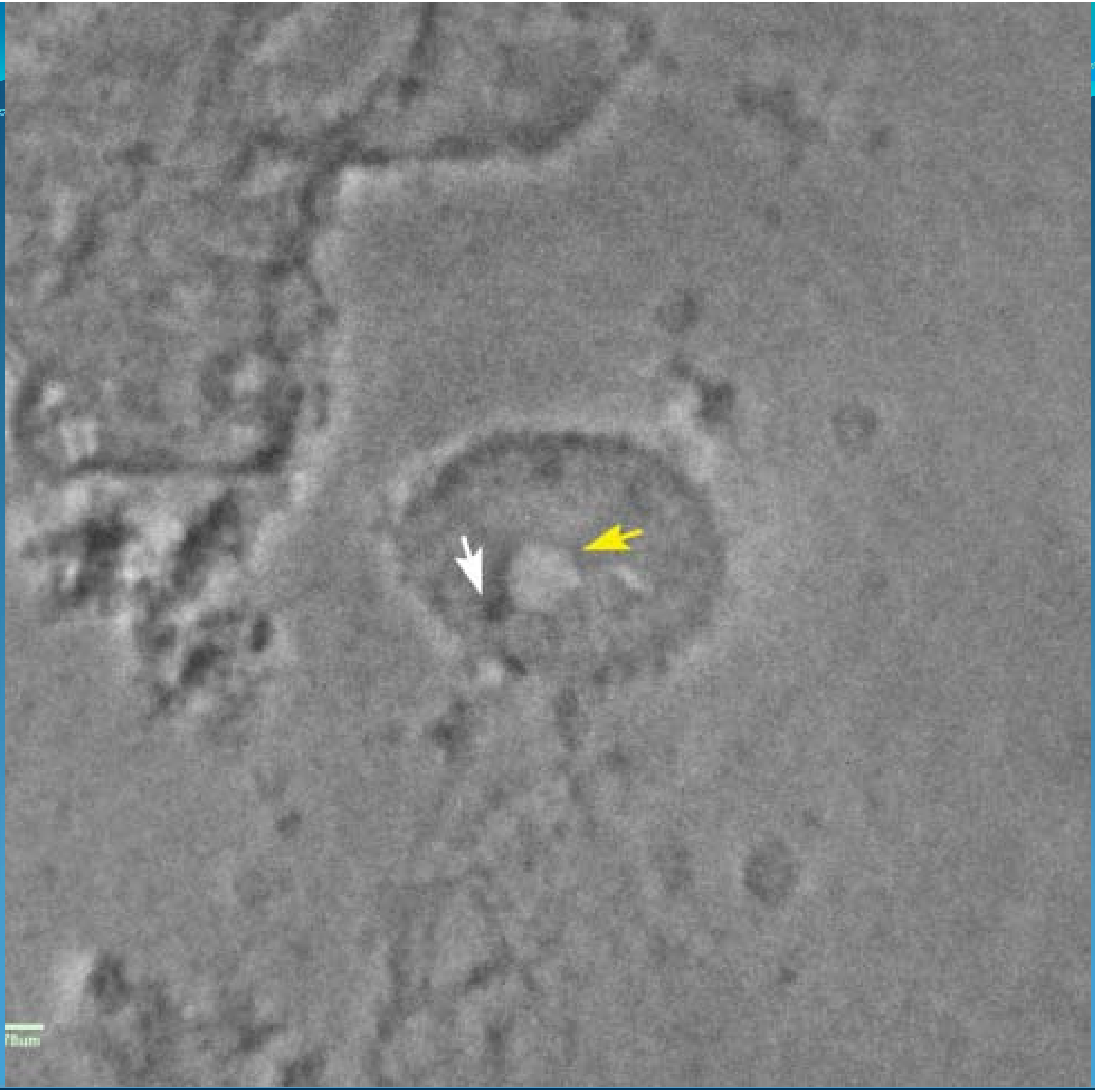
FOV: 30x30μm

Vimentin of the Cervix Tumor (HeLa) Cells Immuno-labelled with DAB Chromogen with Ni Enhancement

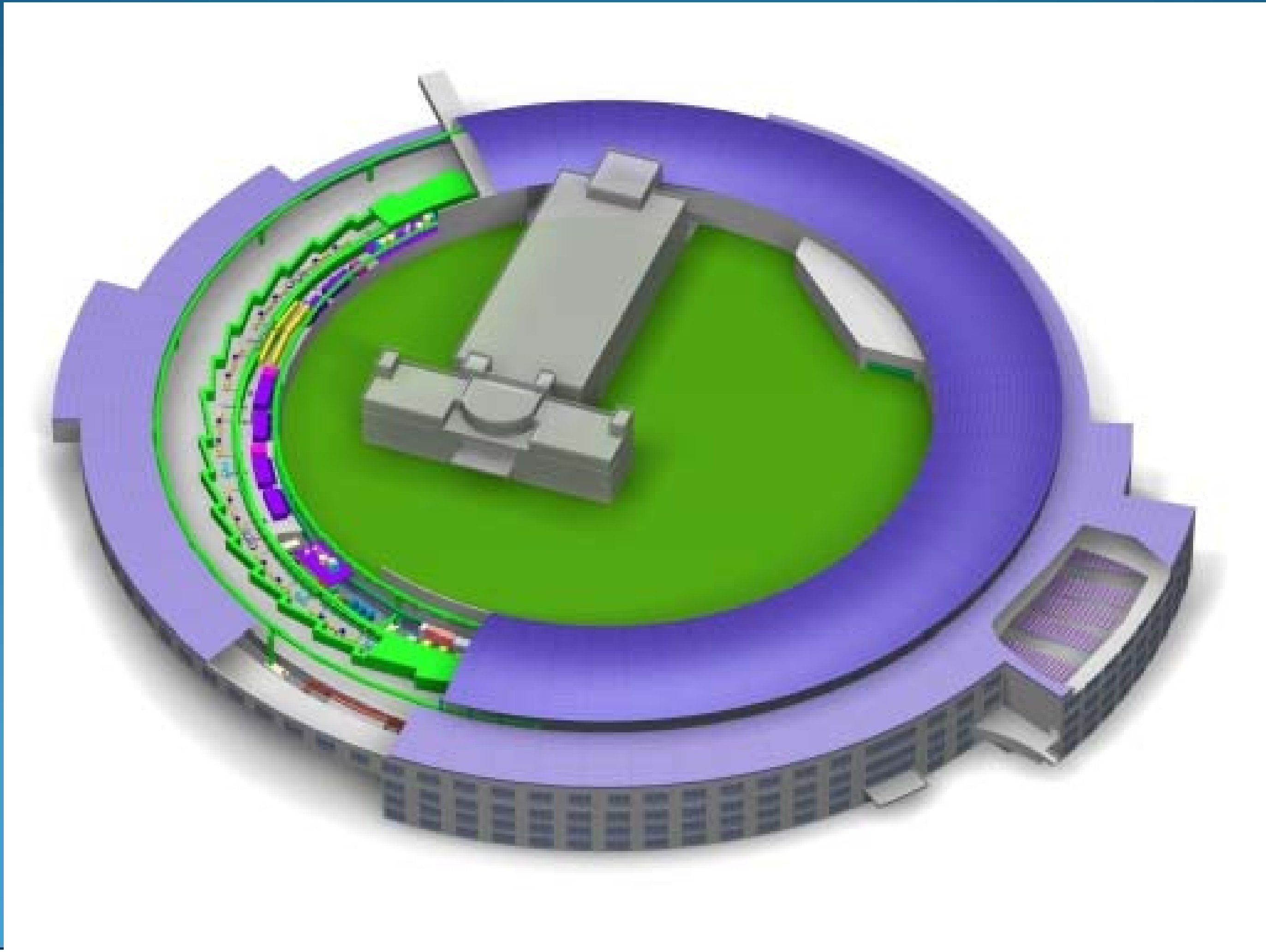


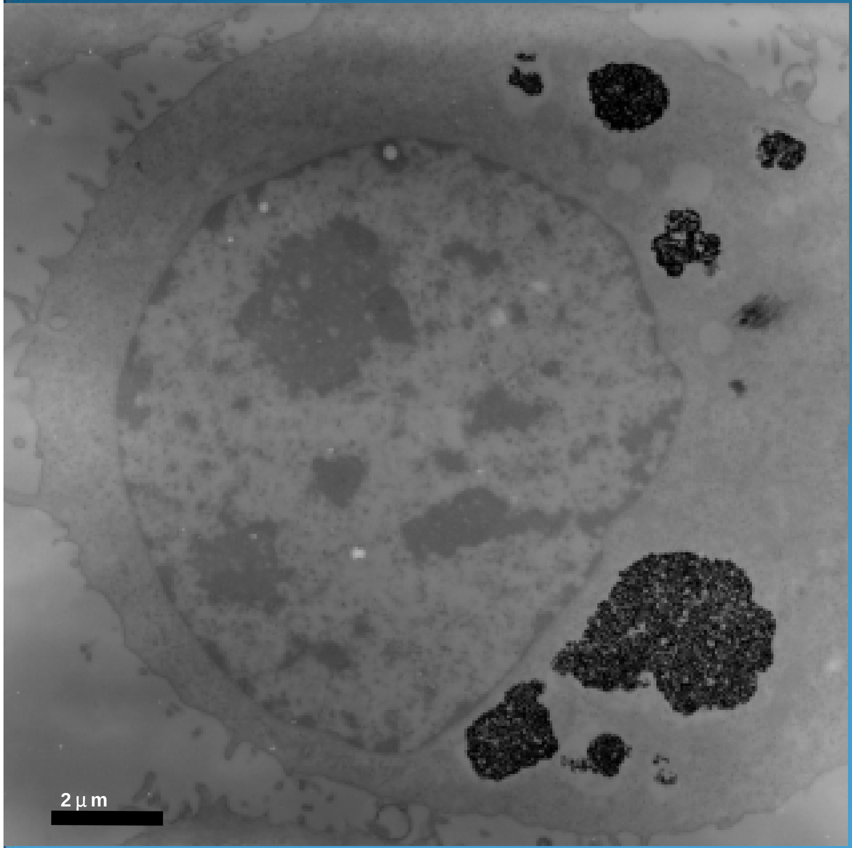
GFP-tagged Yeast

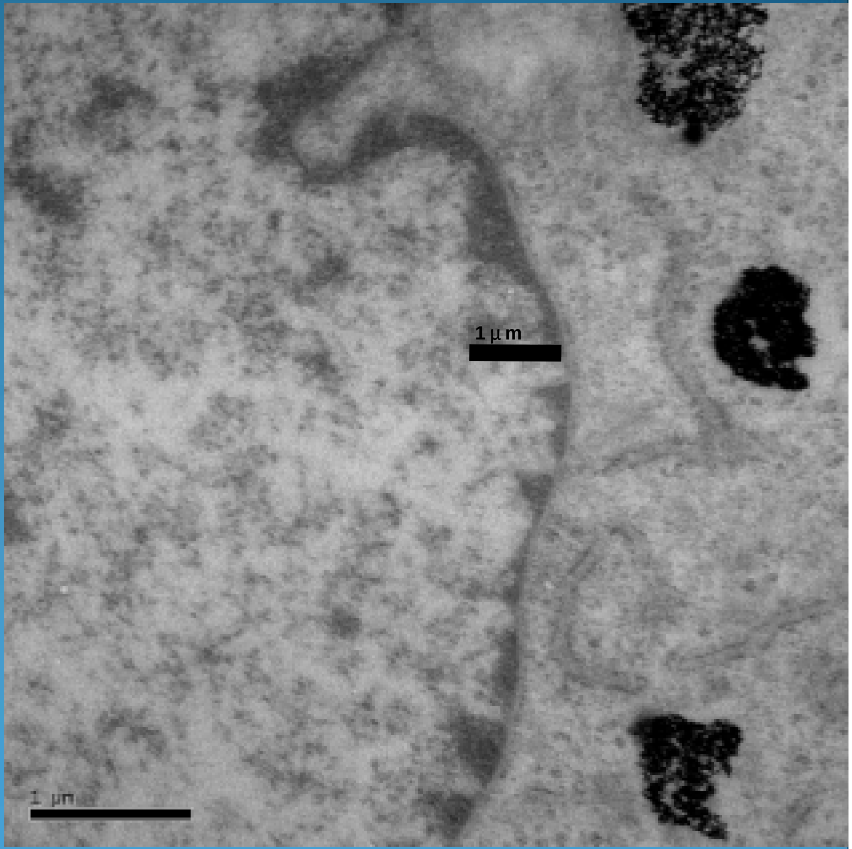
- Sup35-GFP-yeast
- DAB-nickel

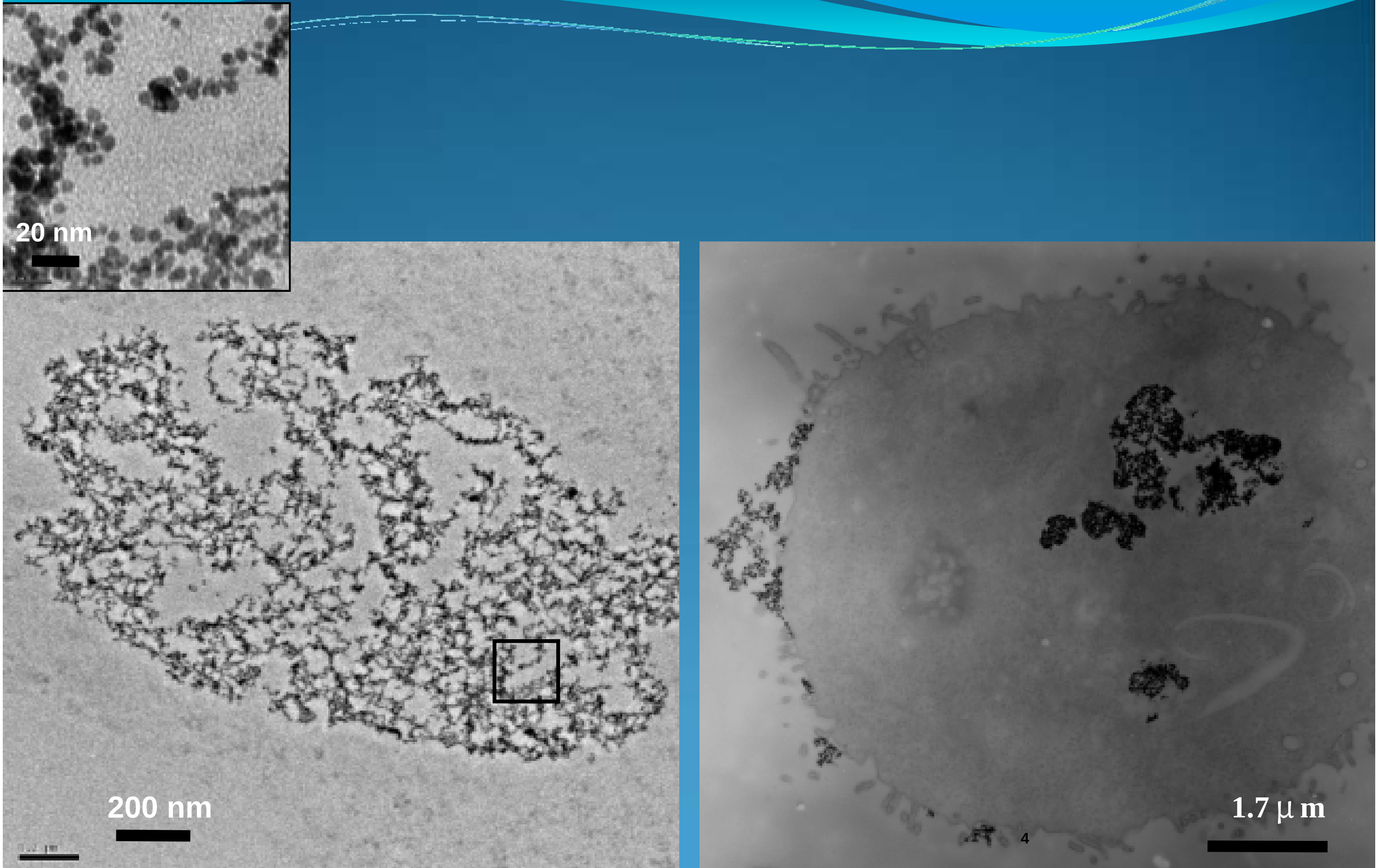


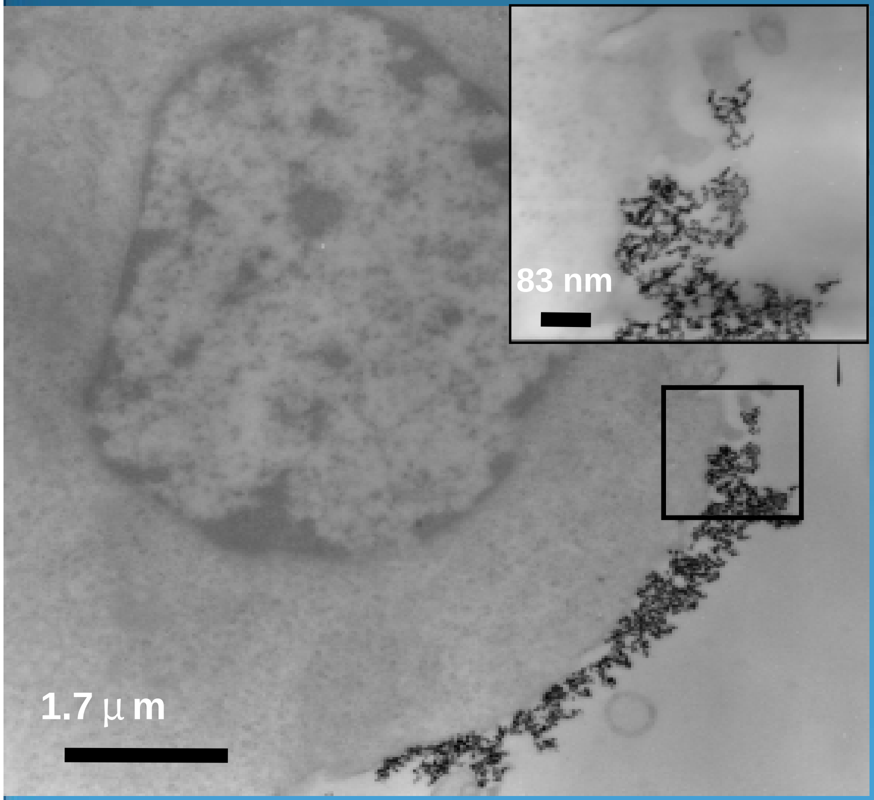
Taiwan Photon Source Project: 2006-2012

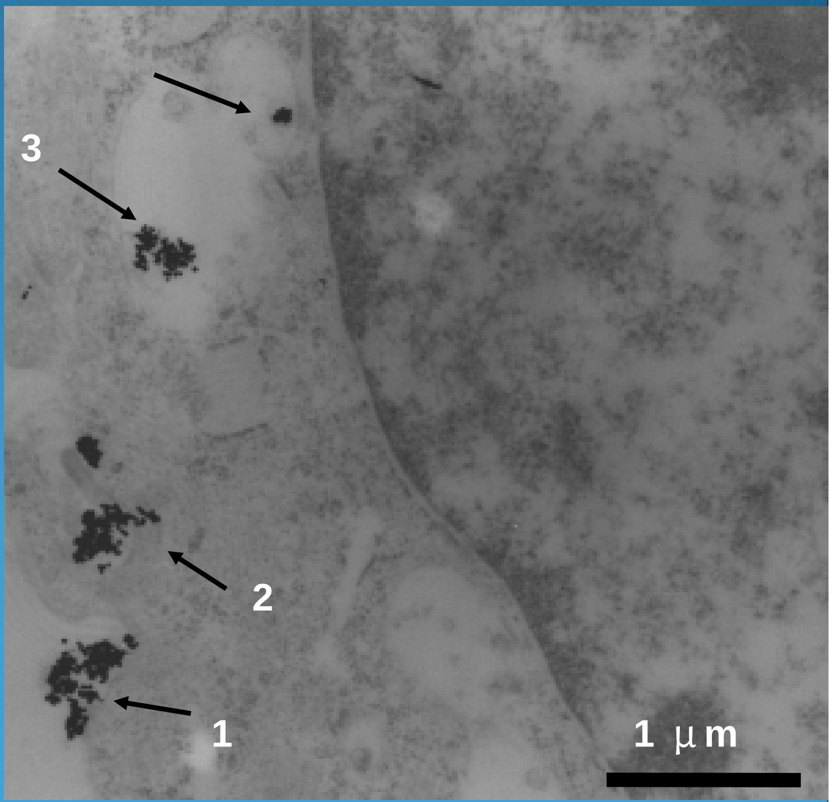


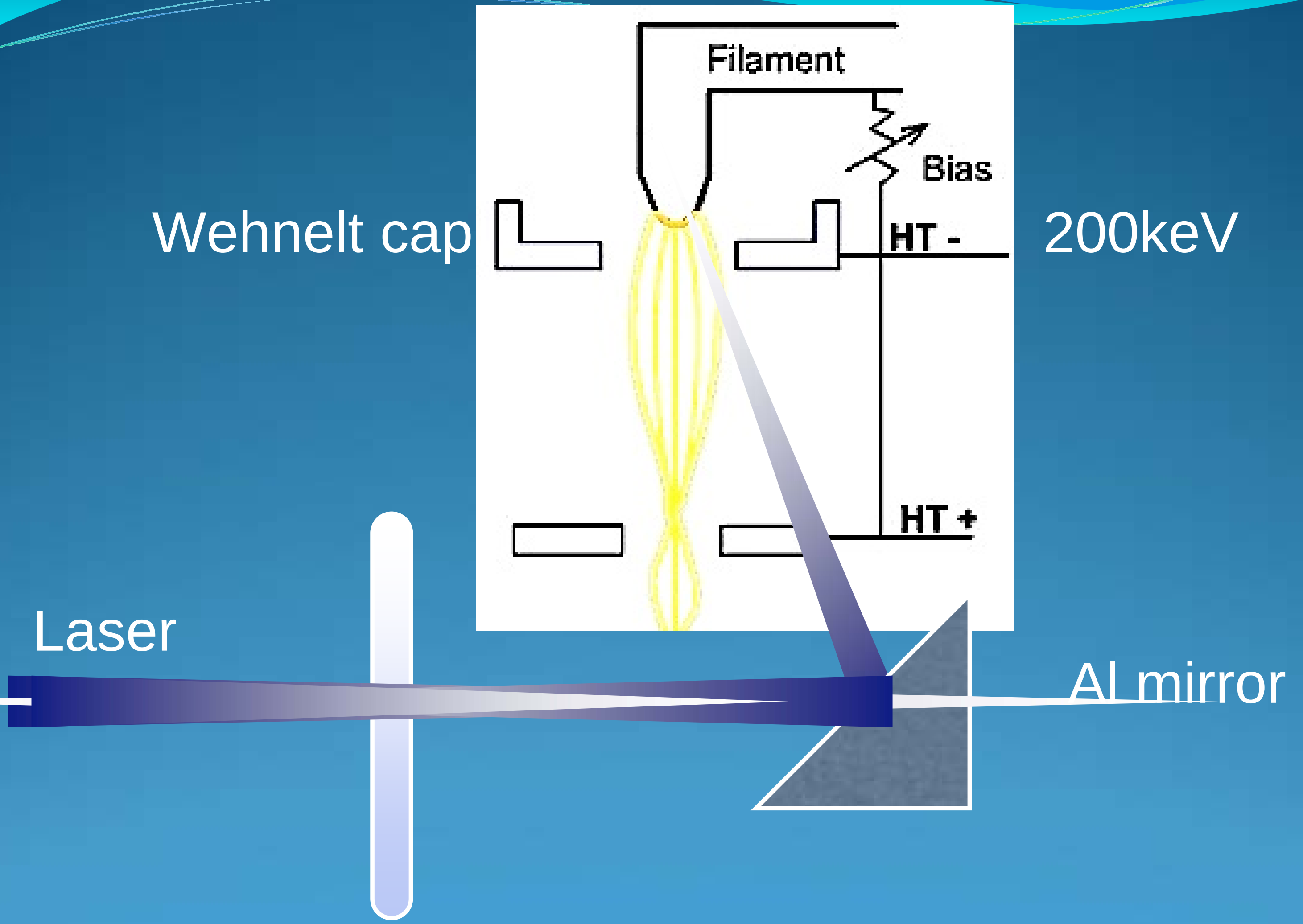

2 µm

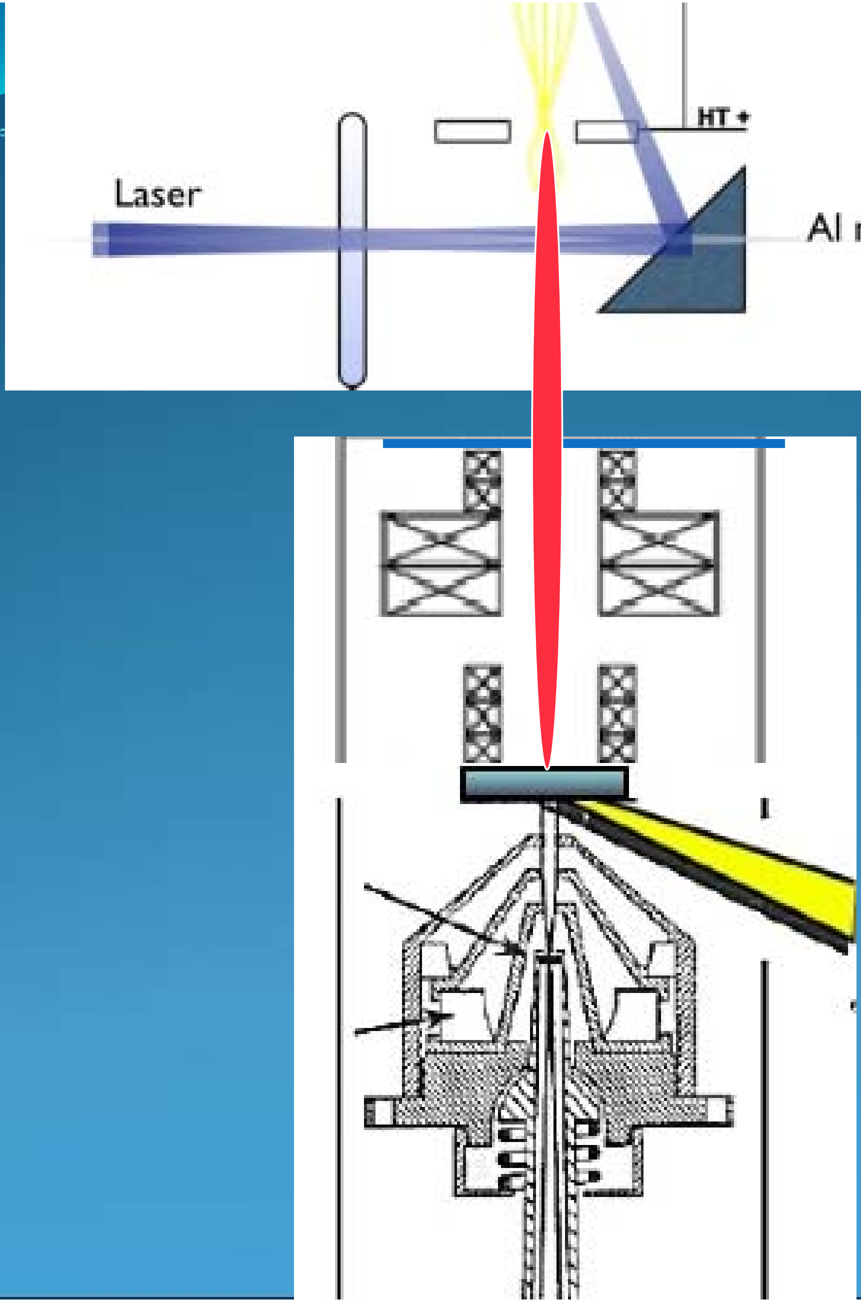

1 µm
1 µm


20 nm
200 nm
1.7 μ m
4

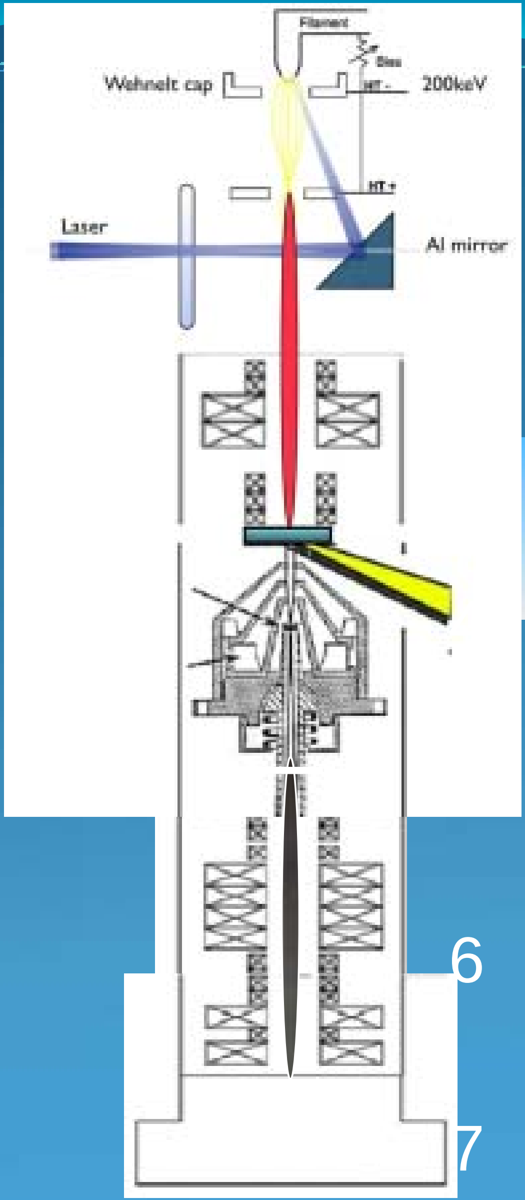

83 nm
1.7 µm

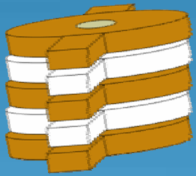

3
2
1
1 µm


Filament
Bias
Wehnelt cap
HT -
200keV
HT +
Laser
Al mirror



Synchrontron or Laser source

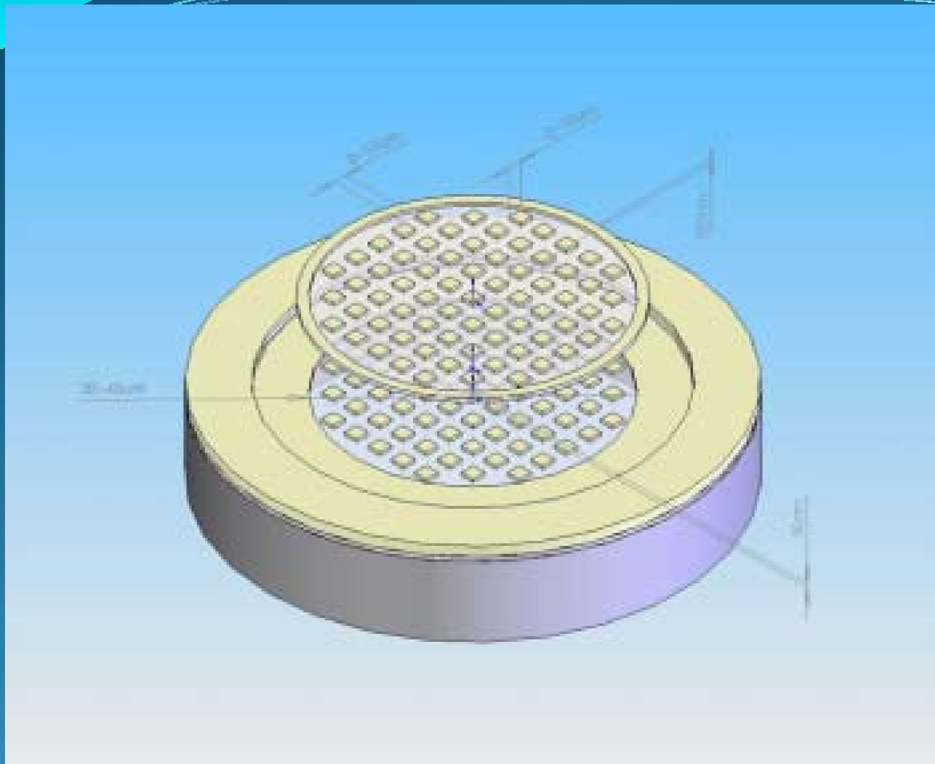


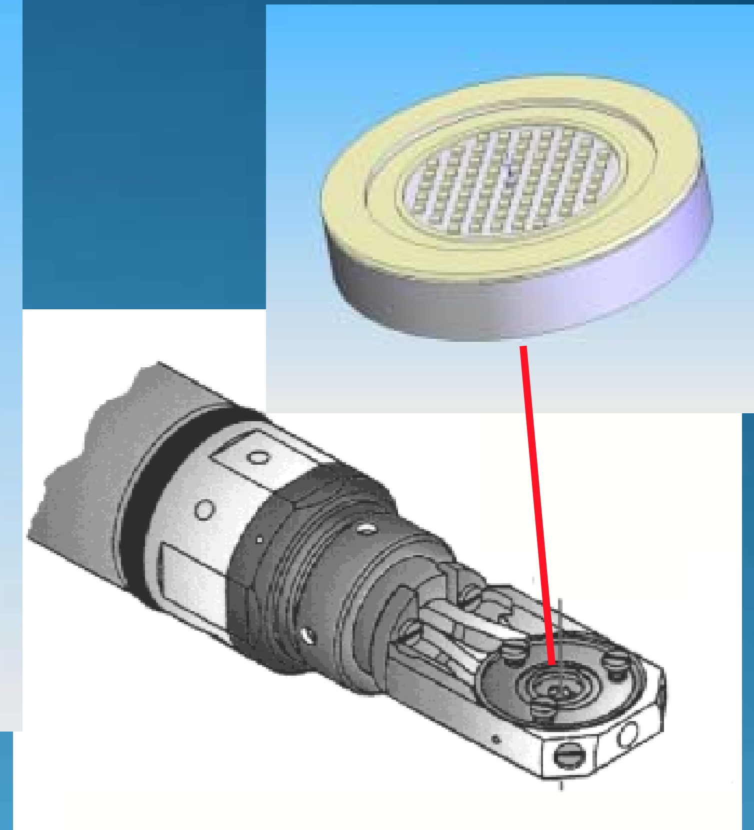


Project supported by IOP, AS

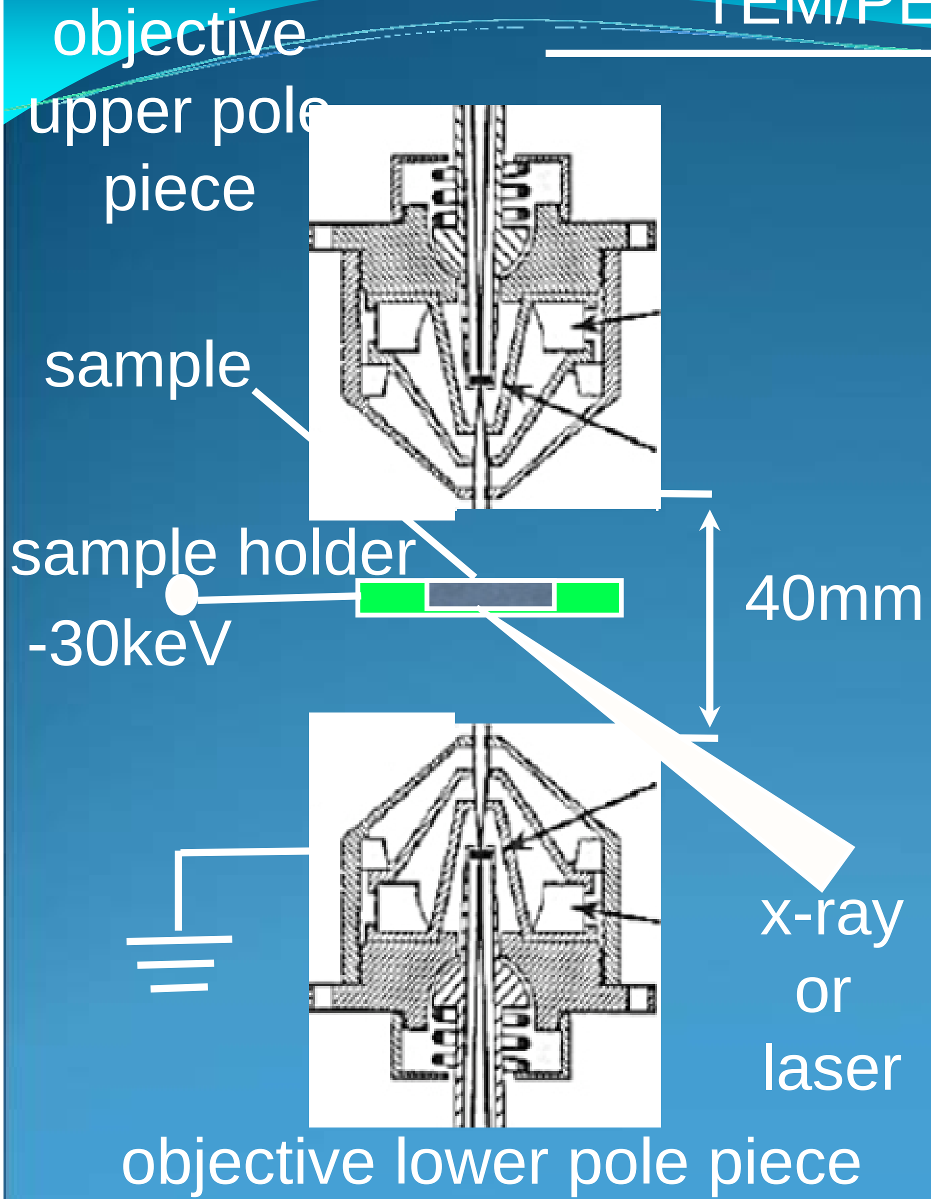
6

7





TEM/PEEM



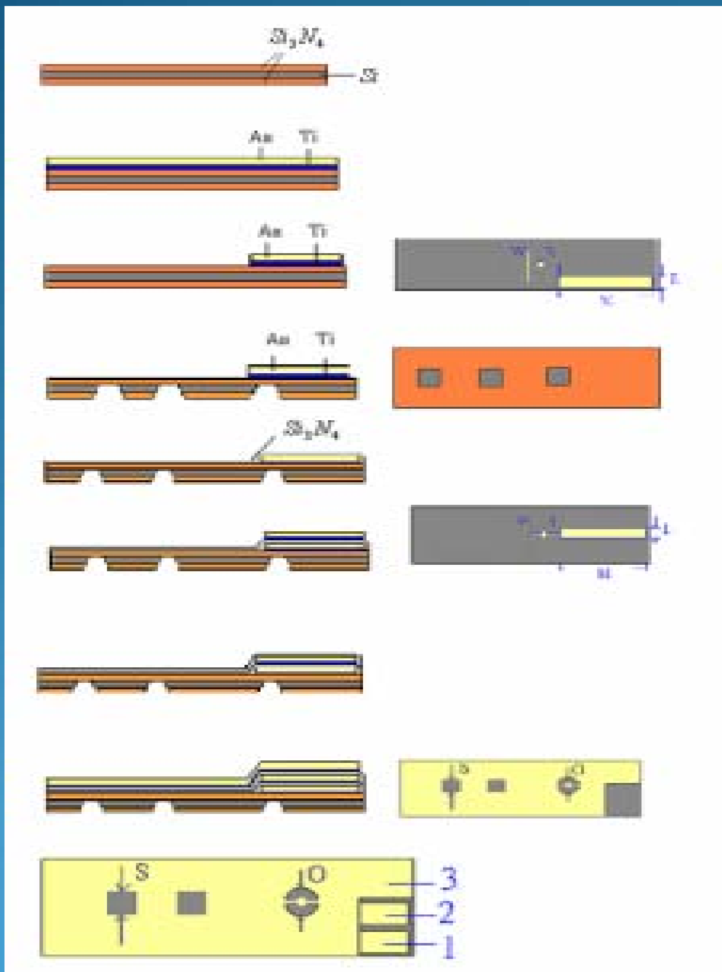
1) Larger Cs =40mm.
It can be corrected by Cs corrector, but it takes long time to re-design the objective lens and Cs corrector

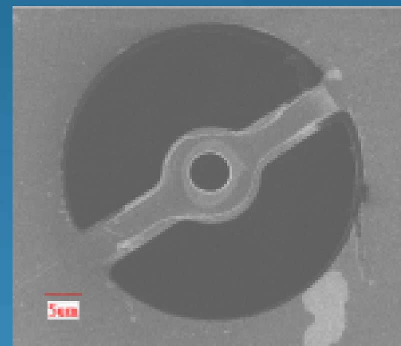
2) Larger Cc =30mm~40mm
it can not be corrected, until Cc corrector is available

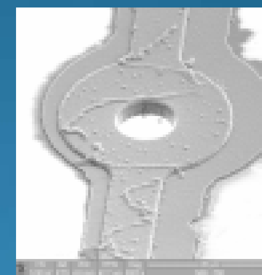
2000FX Cs=5mm
2010FEG Cs=1 mm

Process Flow by MEMS

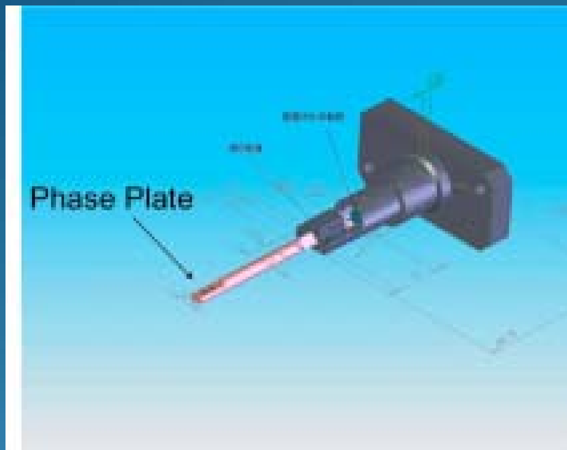
LPCVD low stress Si nitride





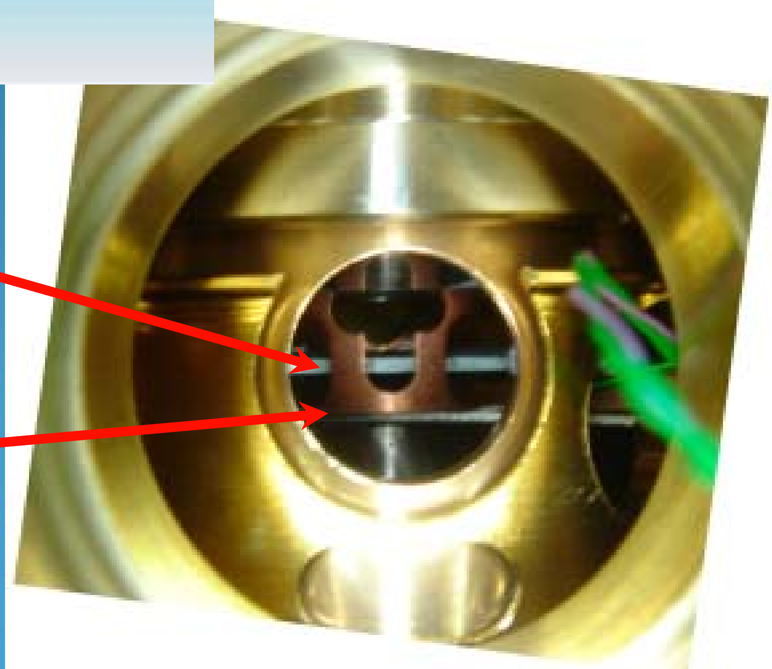


Installation of Phase Plate

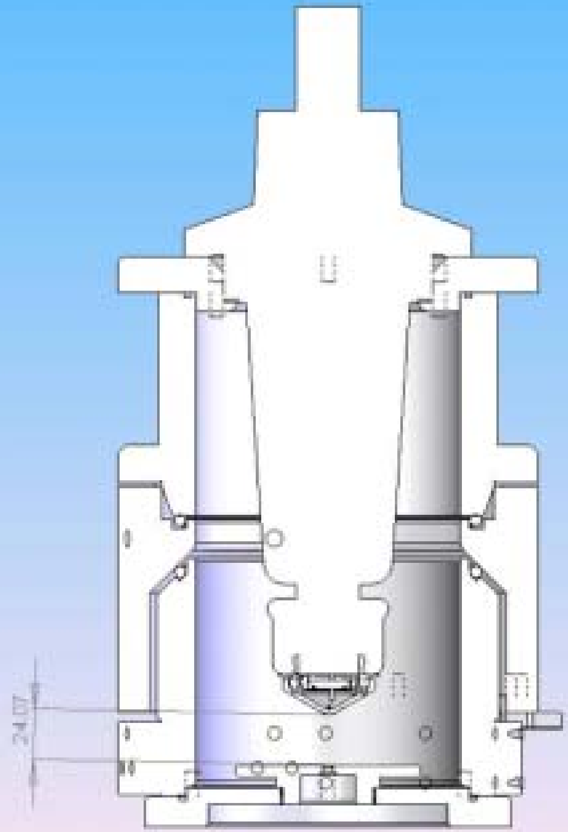


sample holder

phase plate holder



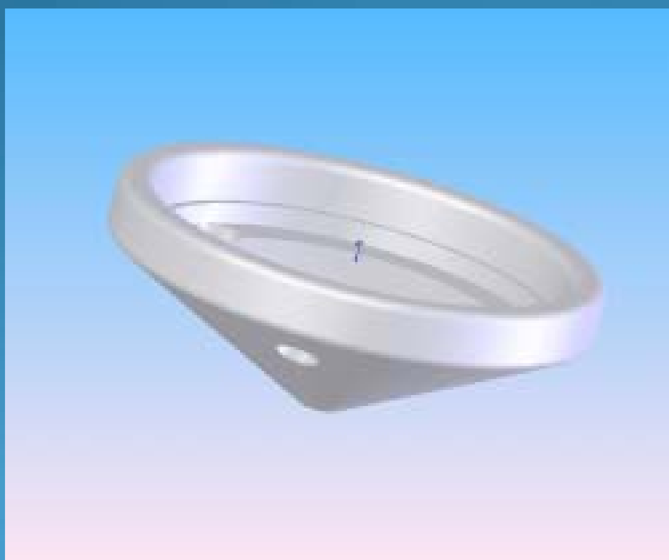
Electron Gun for Ultrafast TEM

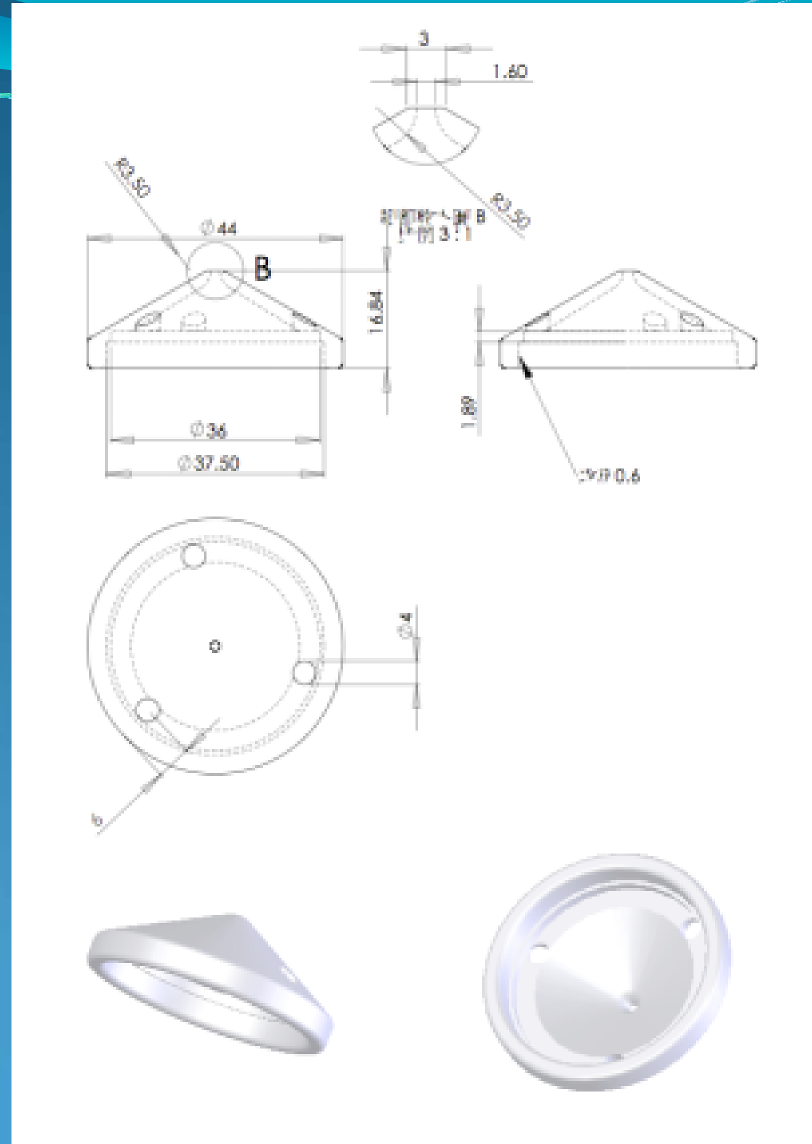


Cathode

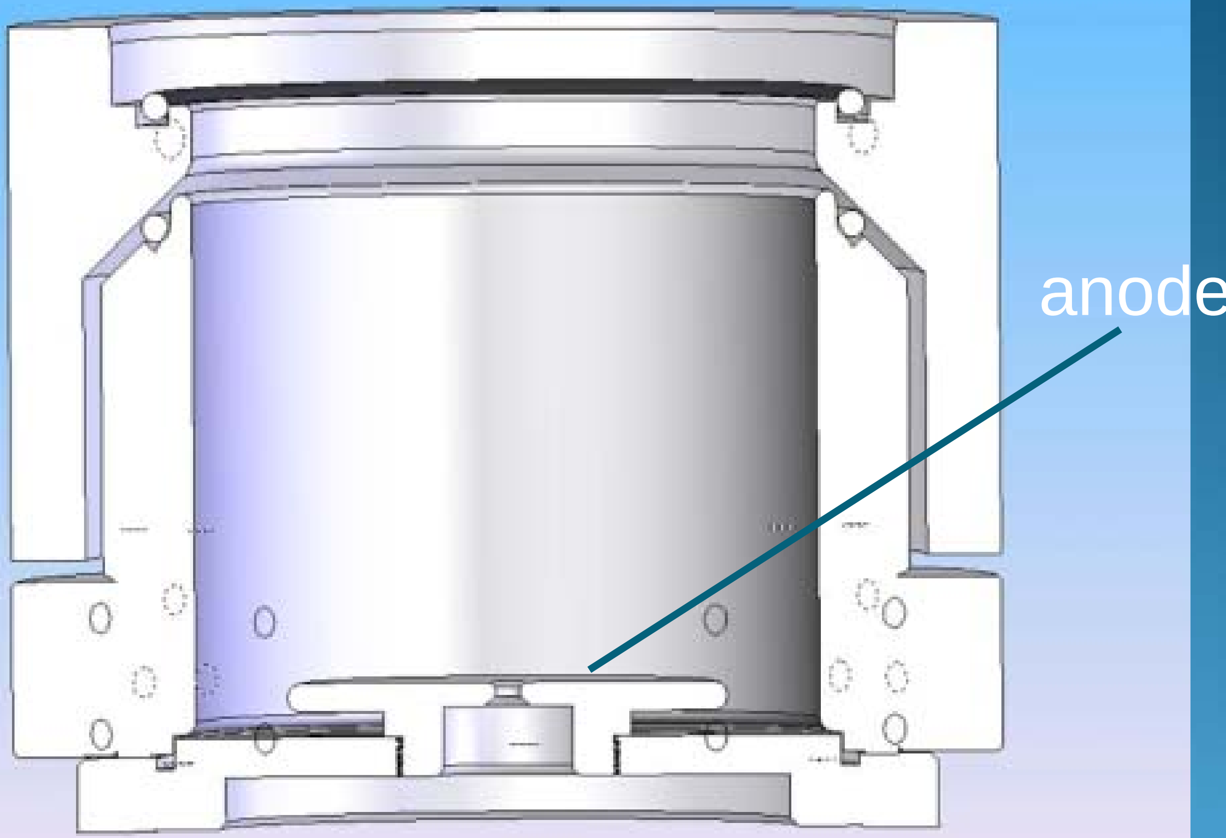
Anode

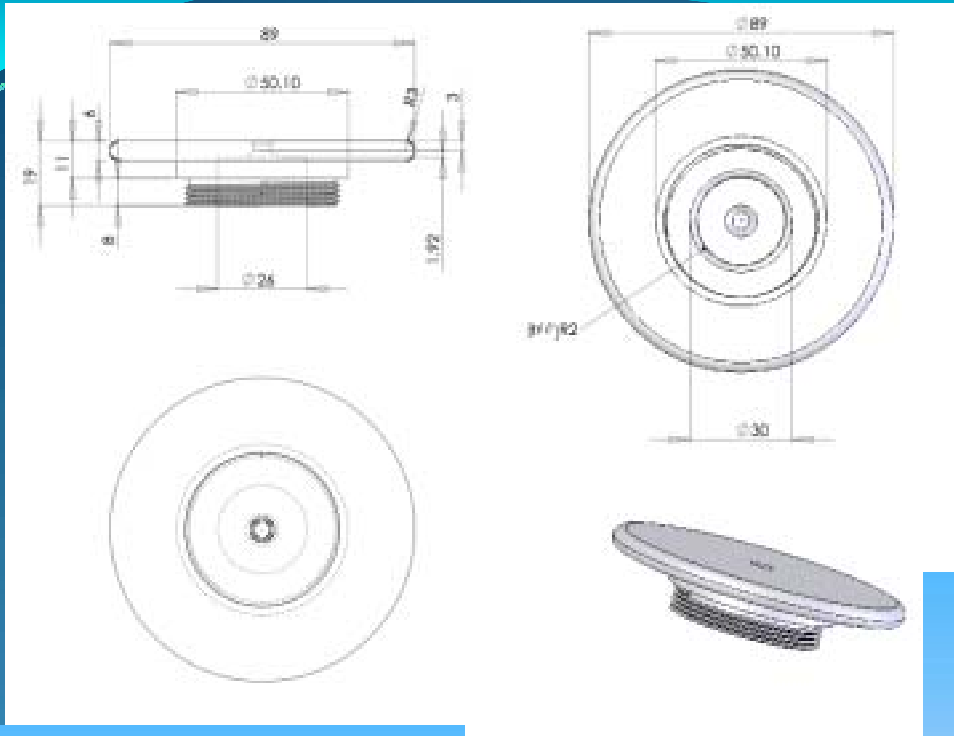
Wehnelt Cap





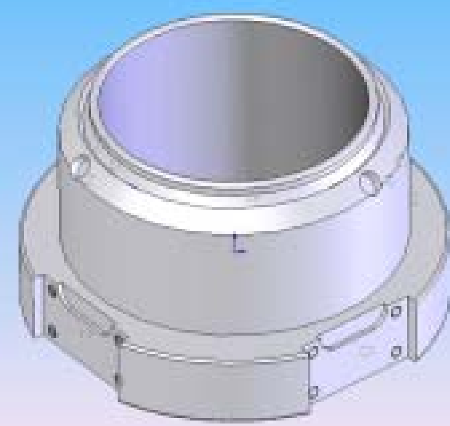
gun chamber





Anode

gun chamber



anode base