

Transmission Electron Microscopy

Fu-Rong Chen

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David B. Williams

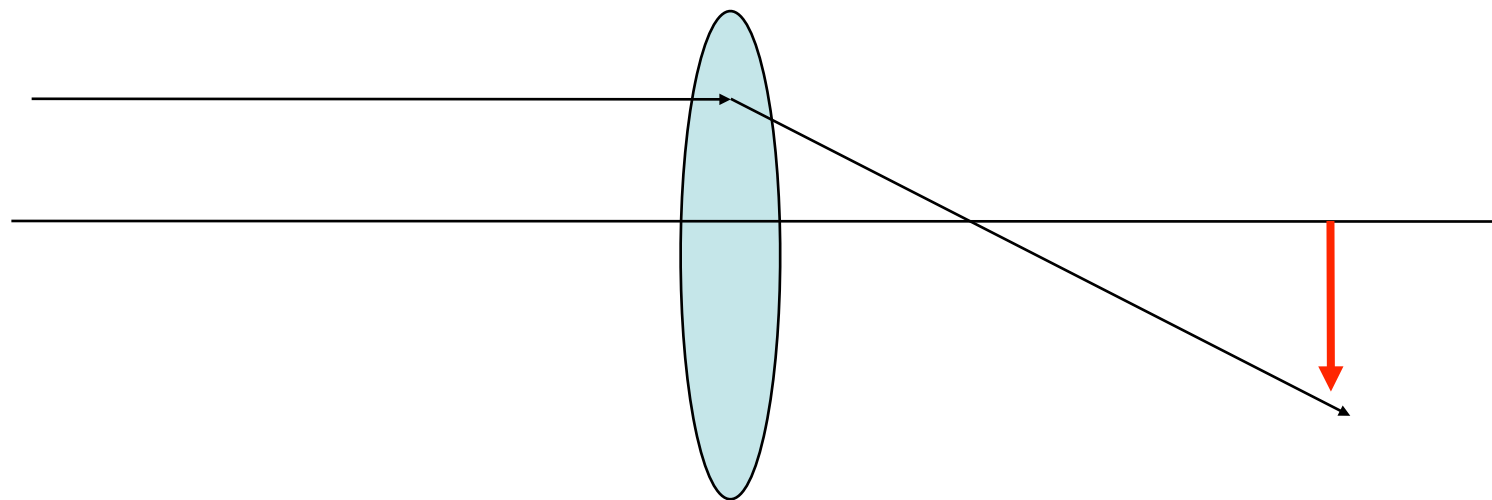
C. Barry Carter

Background : Solid State Physics
Materials Science

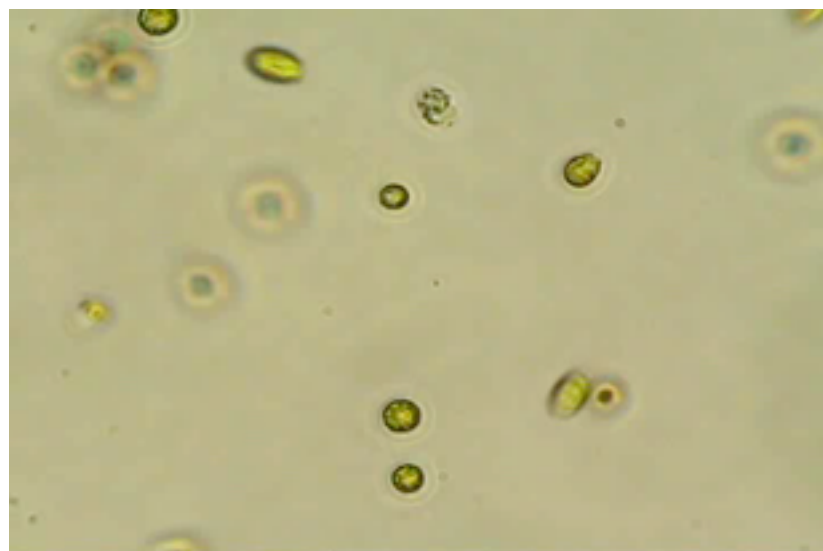
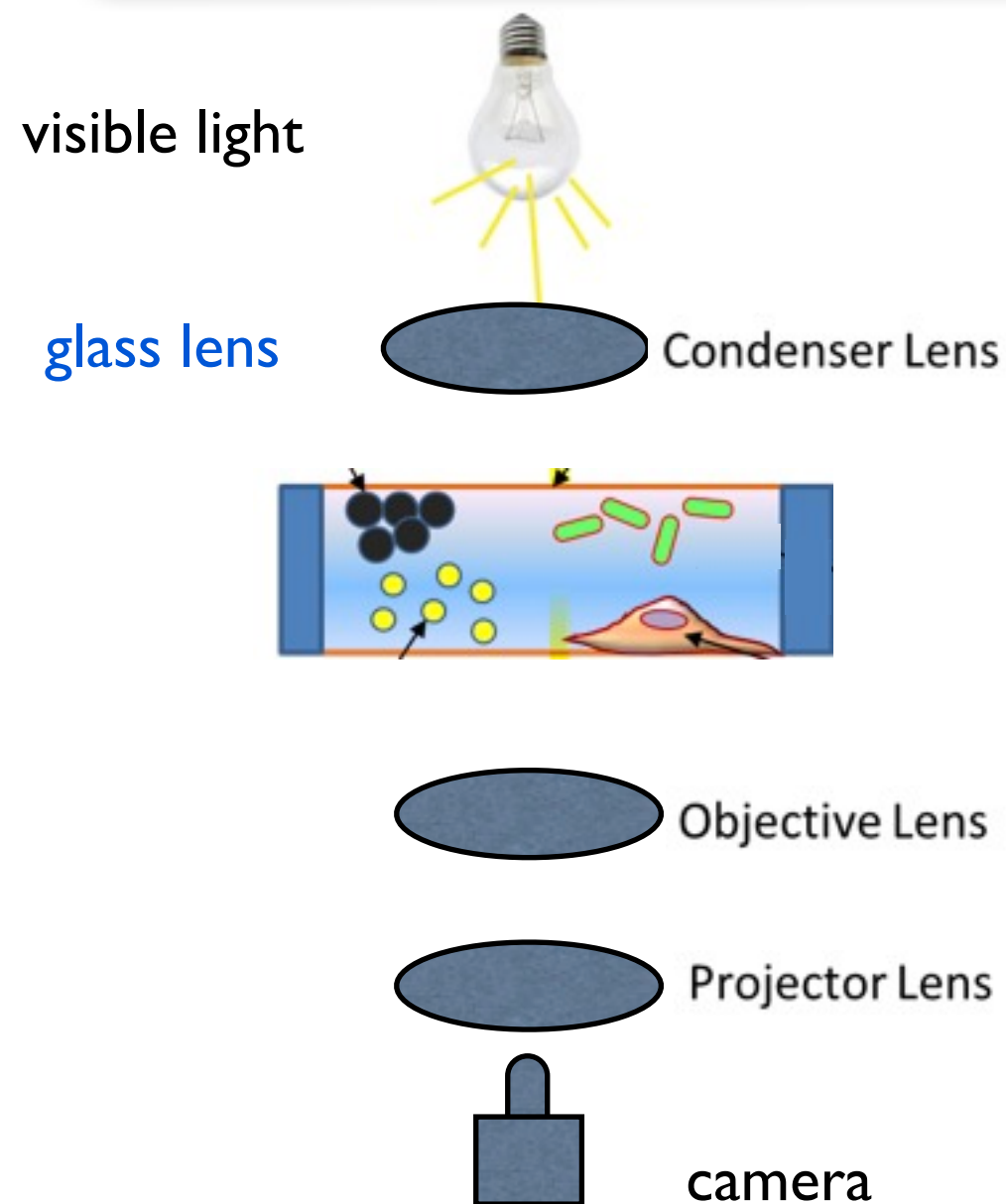
1.1 Why Electron Microscope?

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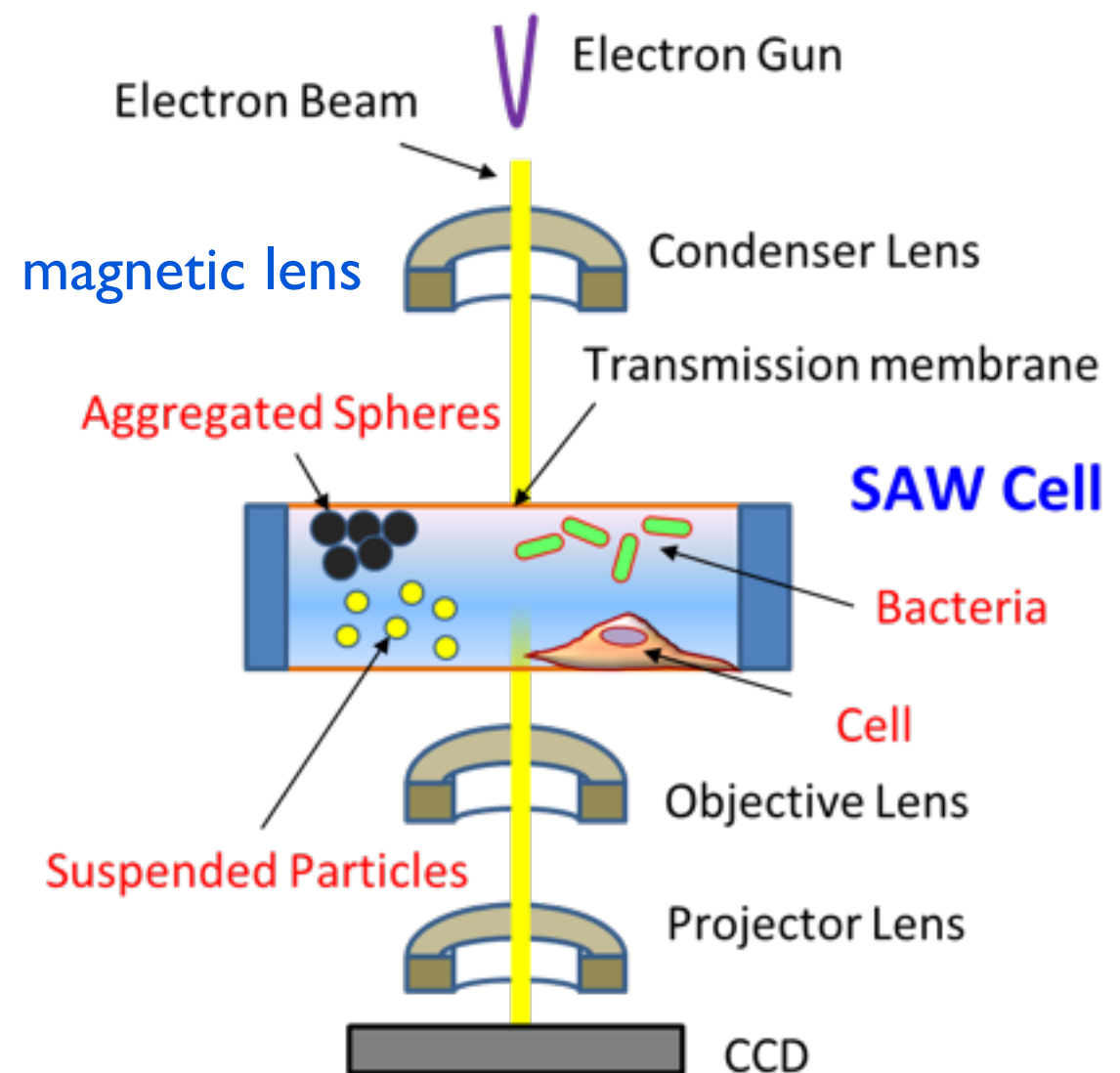
	<u>Optical Microscope</u>	<u>Electron Microscope</u>
source	visible light	electron
lens	glass	electro-magnetic lens



Optical Microscope



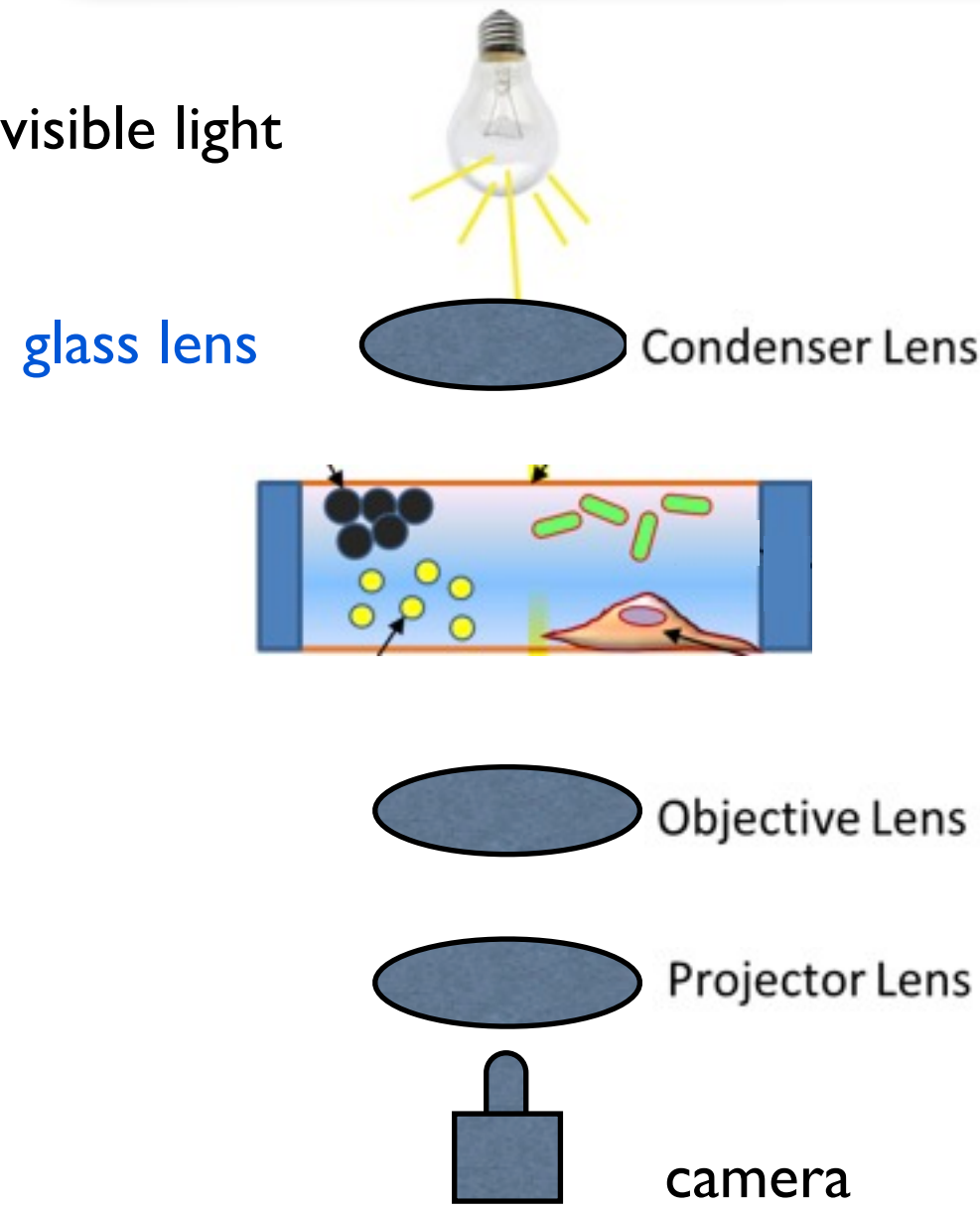
Electron Microscope



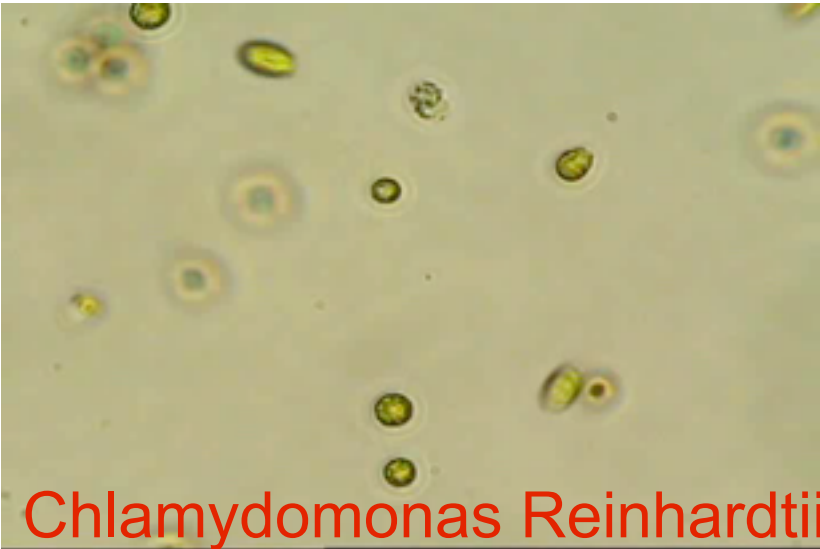
Liquid TEM
image

Optical Microscope

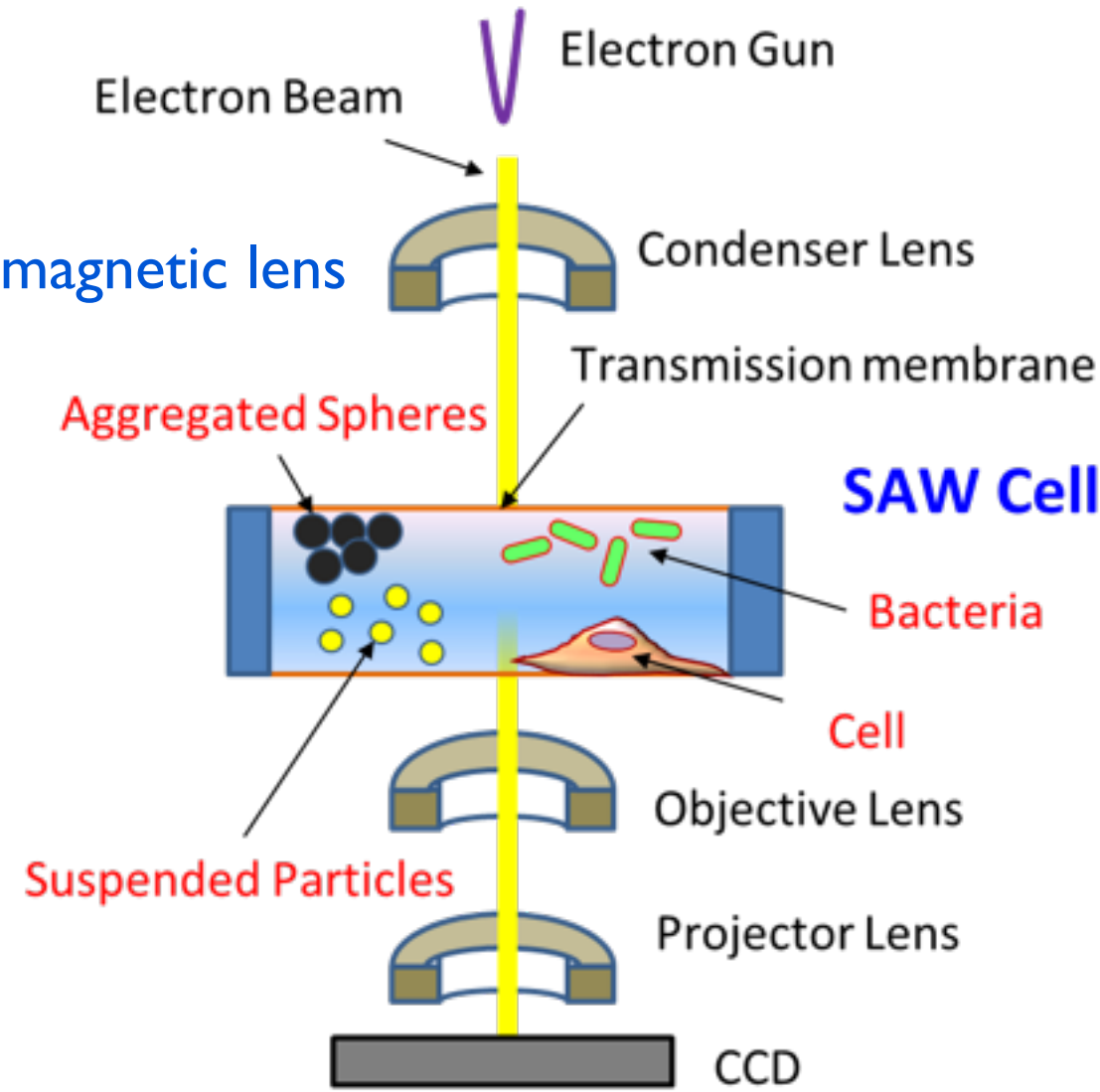
Electron Microscope



衣藻



Chlamydomonas Reinhardtii



Liquid TEM image

Ernst Ruska



Nobel Prize in Physics 1986



Ernst Ruska



Nobel Prize in Physics 1986



Better Electron Microscope

..... If we can see where the **atoms** are , then the analysis of chemical substance become very easy

The most difficulty is the power of electron microscope must be improved 100 times..



- Richard F. Feynman
- 12.29.1959 American Physics Society, CIT



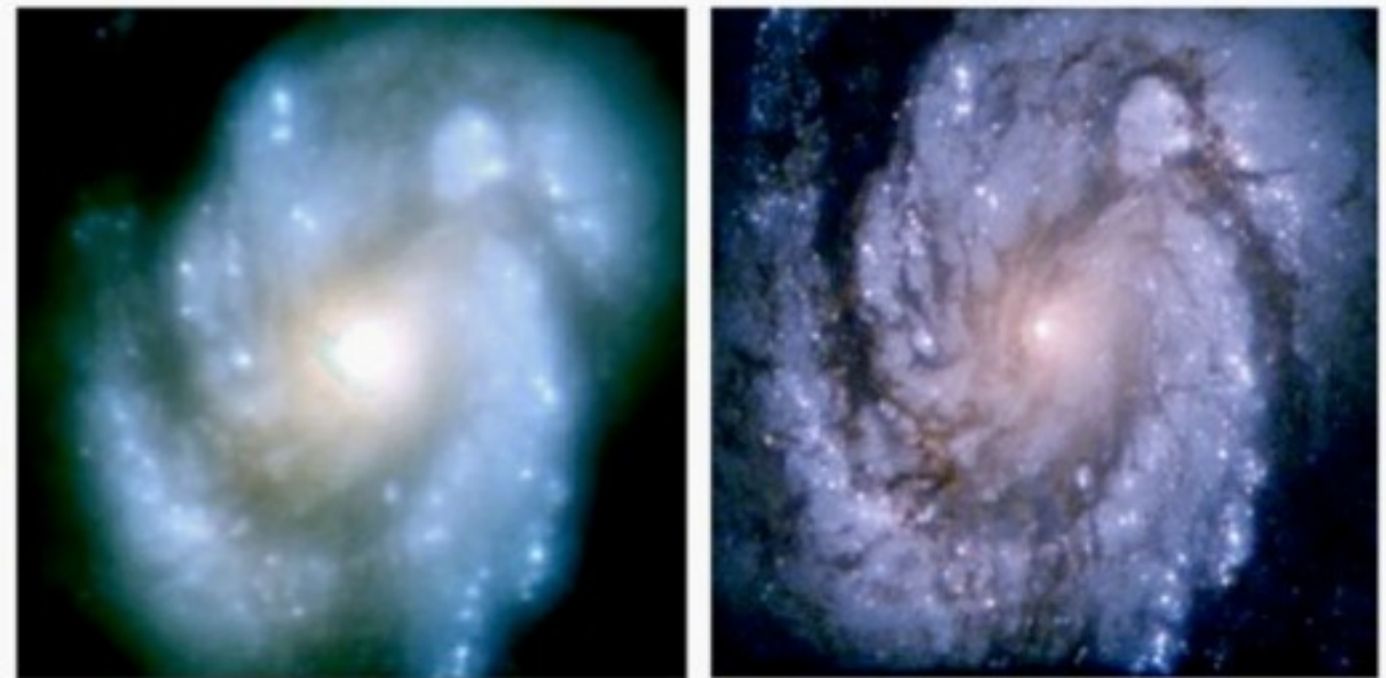
A. Resolution

NTHU



A. Resolution

NTHU





A. Resolution

NTHU

- visible light : $\lambda \sim 6000\text{\AA}$

Rayleigh resolution

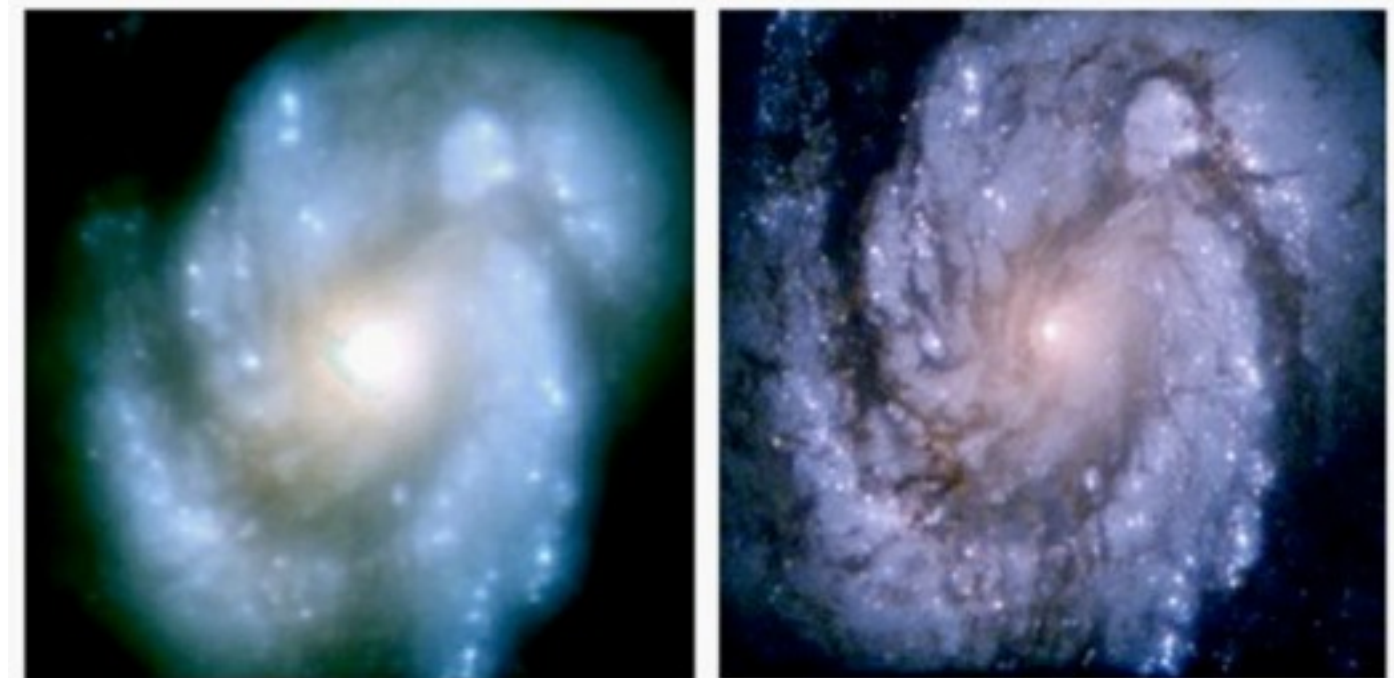
$$\delta = \frac{0.61\lambda}{\mu \sin \beta} \quad (1.1)$$

δ : Rayleigh resolution

λ : wavelength

μ : refractive index

β : semi-angle of lens



$\mu \sin \beta$: Numerical Aperature ~ 1



A. Resolution

NTHU

- visible light : $\lambda \sim 6000\text{\AA}$

Rayleigh resolution

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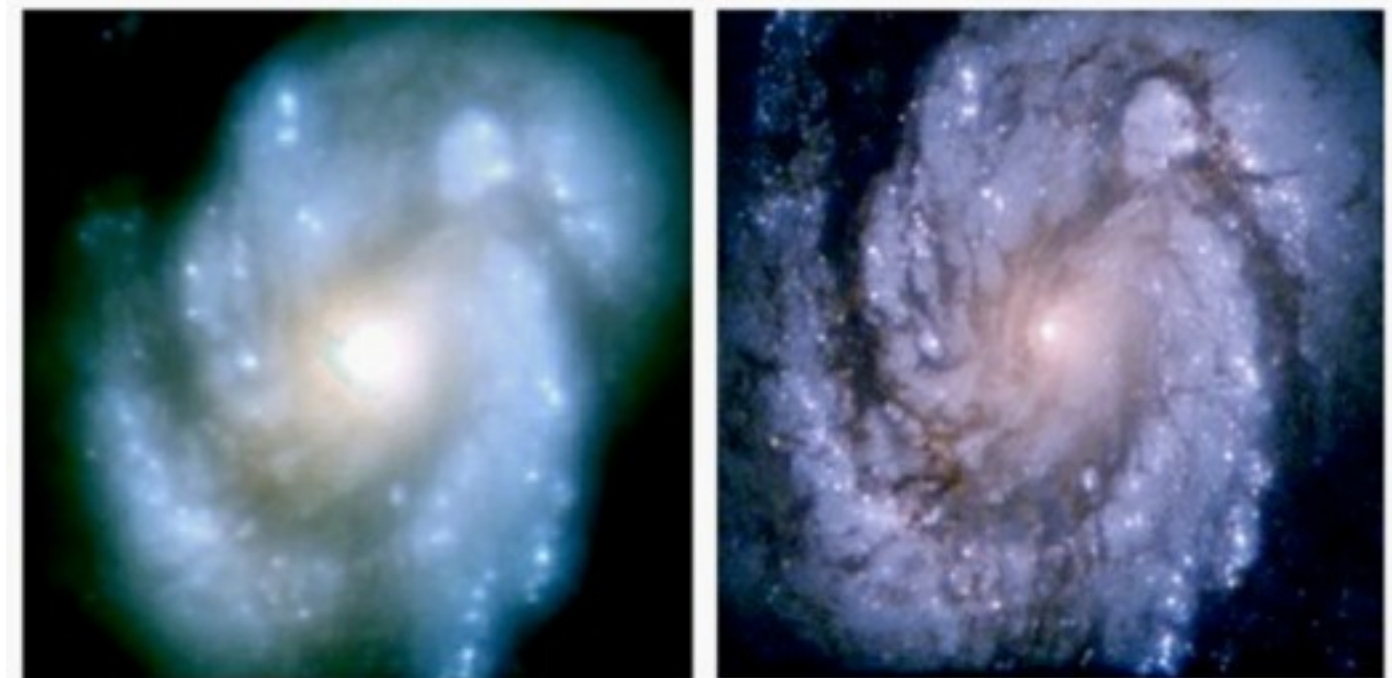
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- resolution of optical microscope is about half of wavelength $\sim 3000\text{\AA}$ (1000 atoms)



A. Resolution

NTHU

A the resolution of an optical microscope is limited by the wavelength °

- visible light : $\lambda \sim 6000\text{\AA}$

Rayleigh resolution

$$\delta = \frac{0.61\lambda}{\mu \sin \beta} \quad (1.1)$$

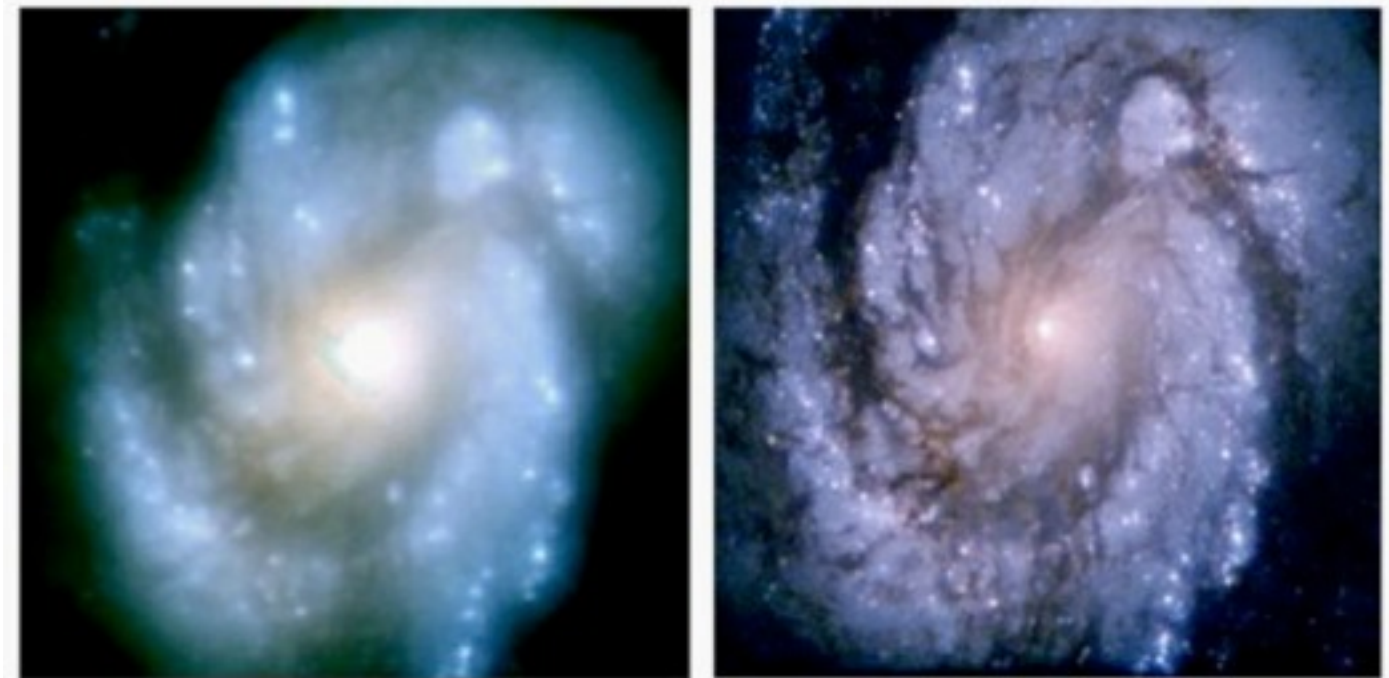
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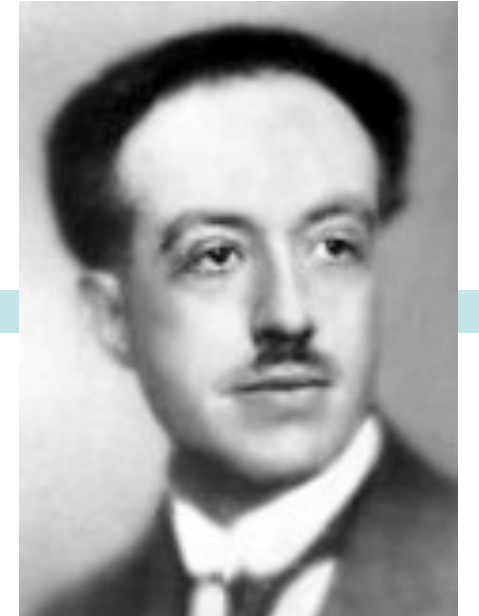
$\mu \sin \beta$: Numerical Aperture ~ 1



- resolution of optical microscope is about half of wavelength $\sim 3000\text{\AA}$ (1000 atoms)



Particle/ Wave Duality



Louis de Broglie
Nobel laureate in 1929

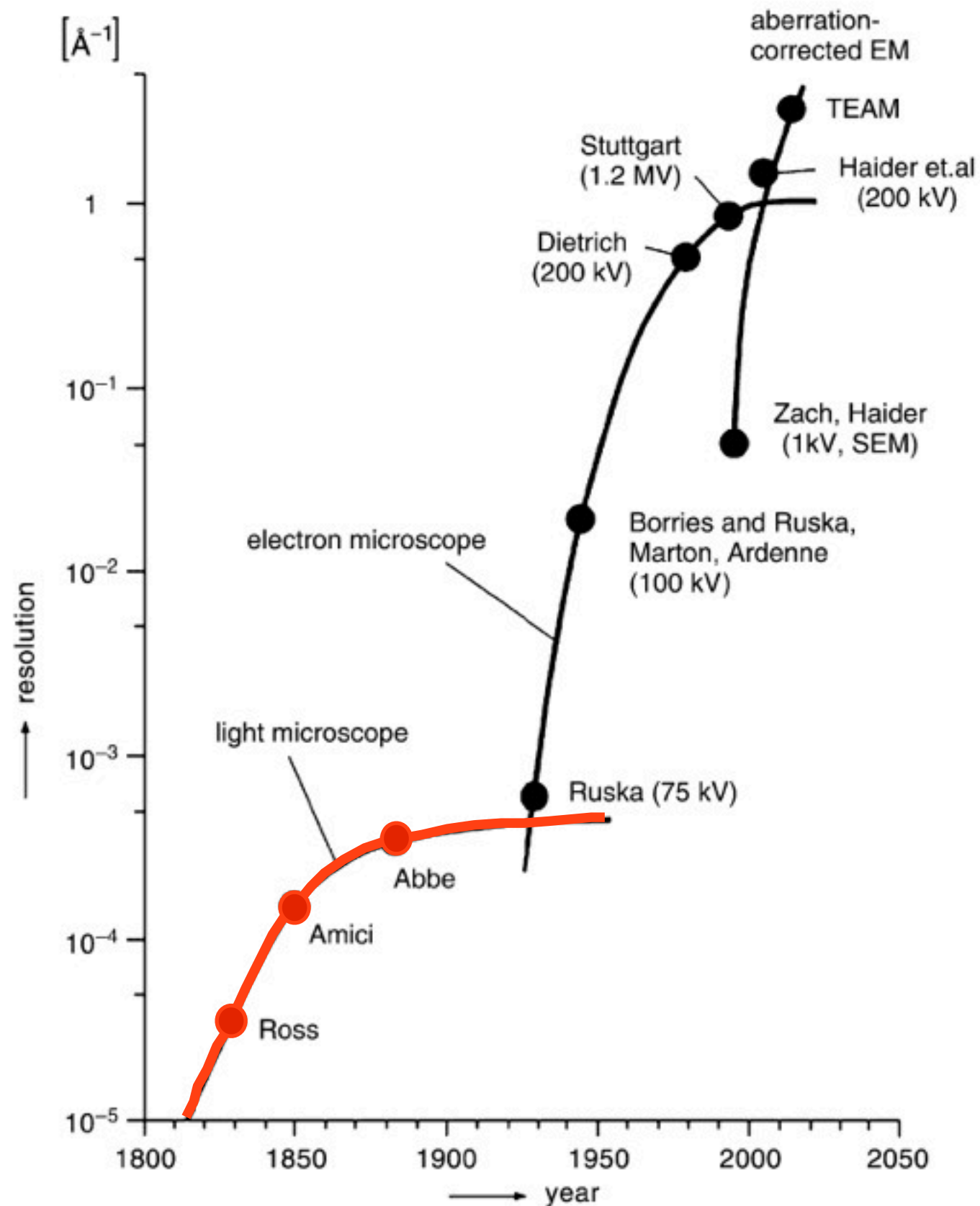
- de Broglie's matter/ wave theory

$$\lambda \sim \frac{1.22}{E^{1/2}}$$

$$E = \frac{1}{2} m_0 v^2$$

<u>E (kev)</u>	<u>$\lambda(\text{\AA})$</u>
100	0.037
200	0.025
300	0.0196
400	0.0169

Evolution of resolution in EM

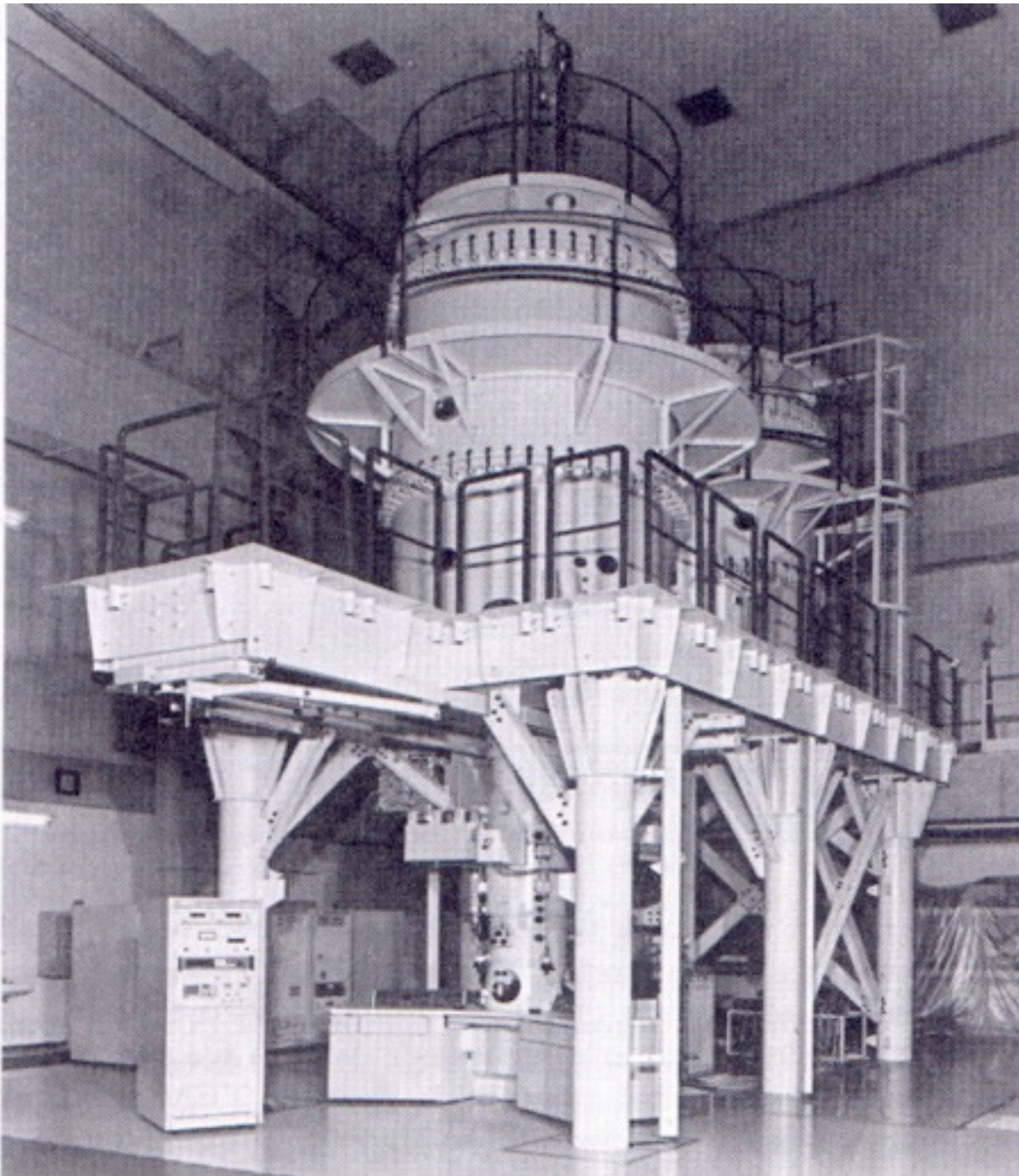


High and Median Voltage TEM



200keV

High and Median Voltage TEM

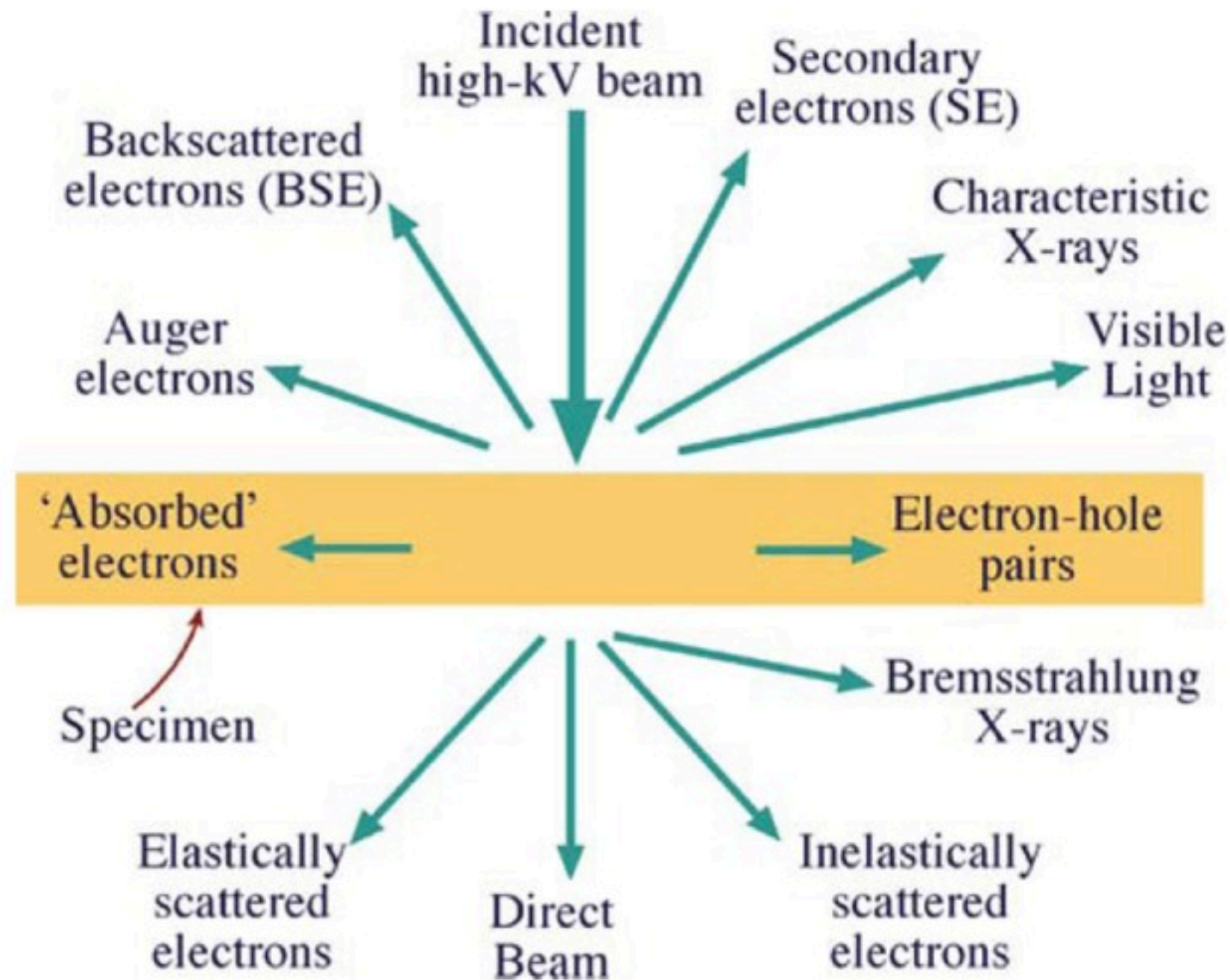


1MeV

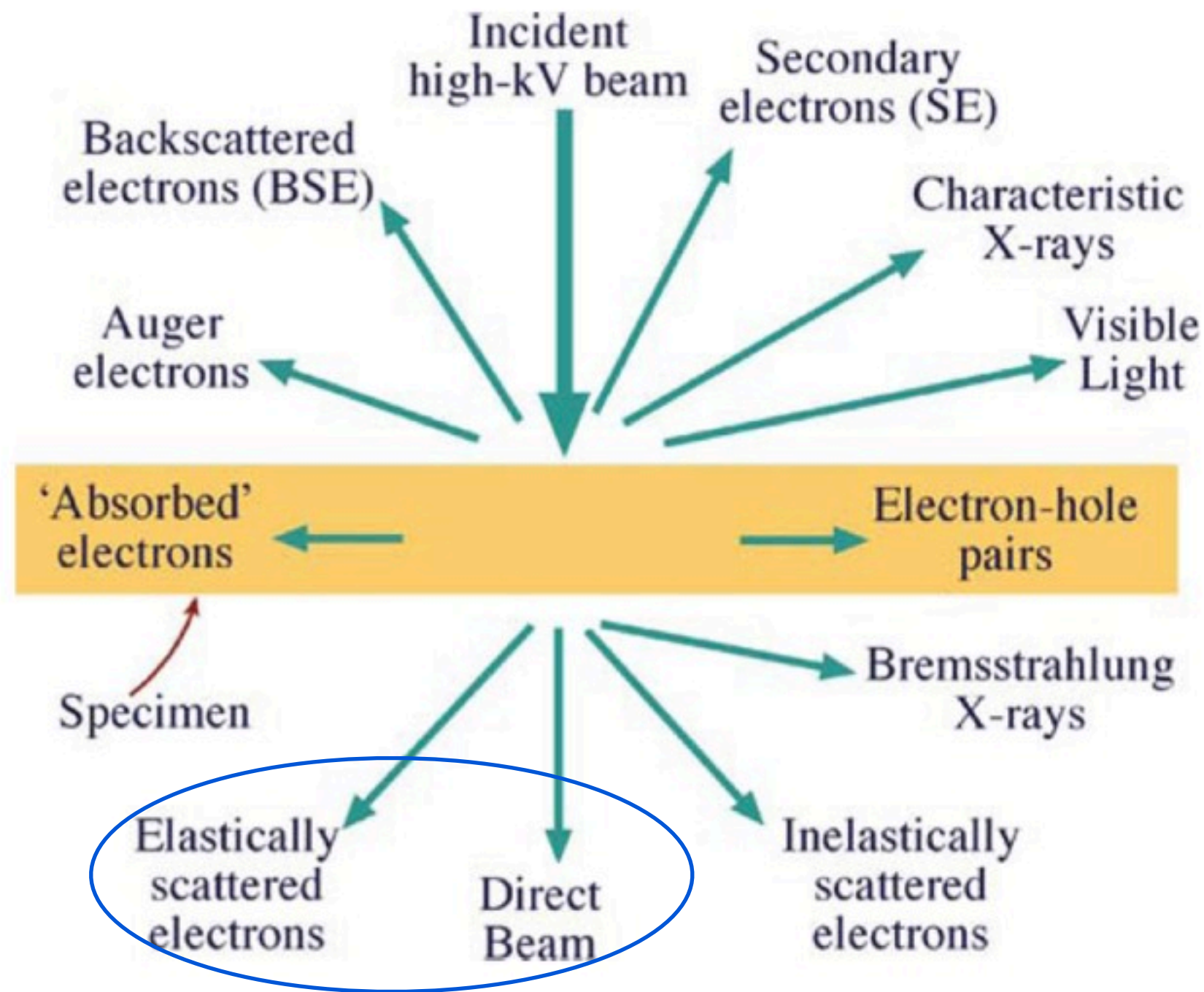


200keV

B. Microanalysis , (X-Ray) , (EELS)

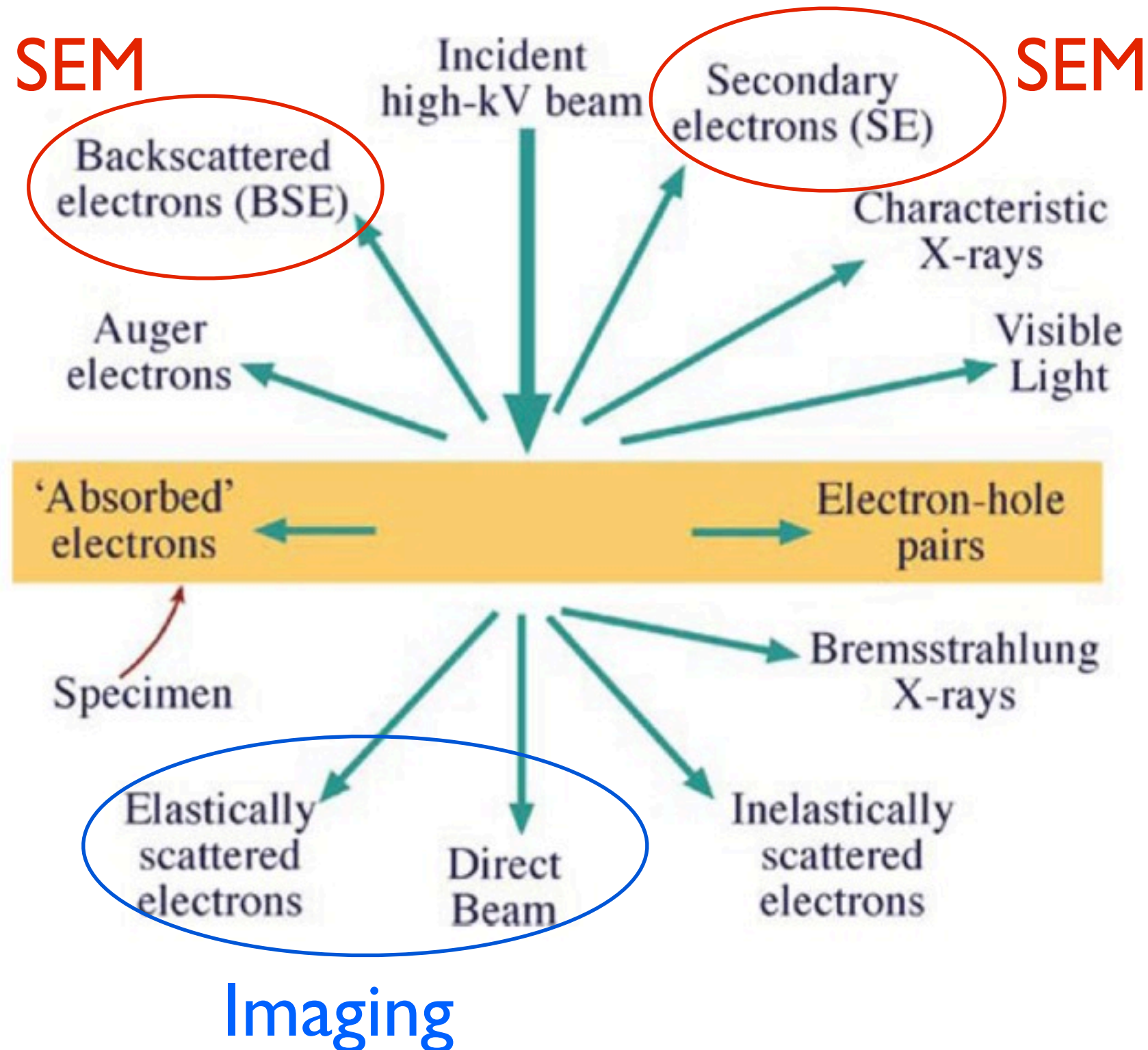


B. Microanalysis , (X-Ray) , (EELS)

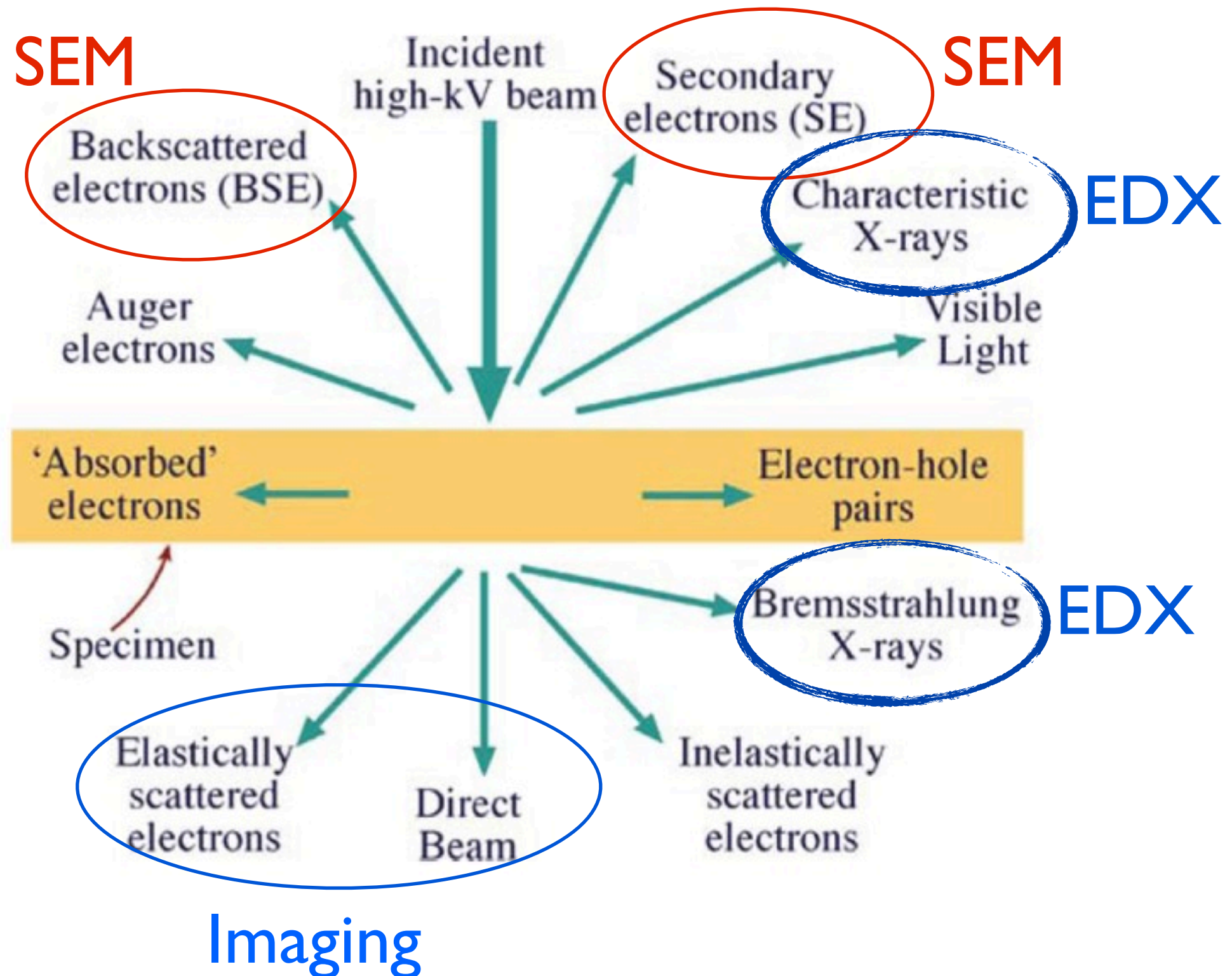


Imaging

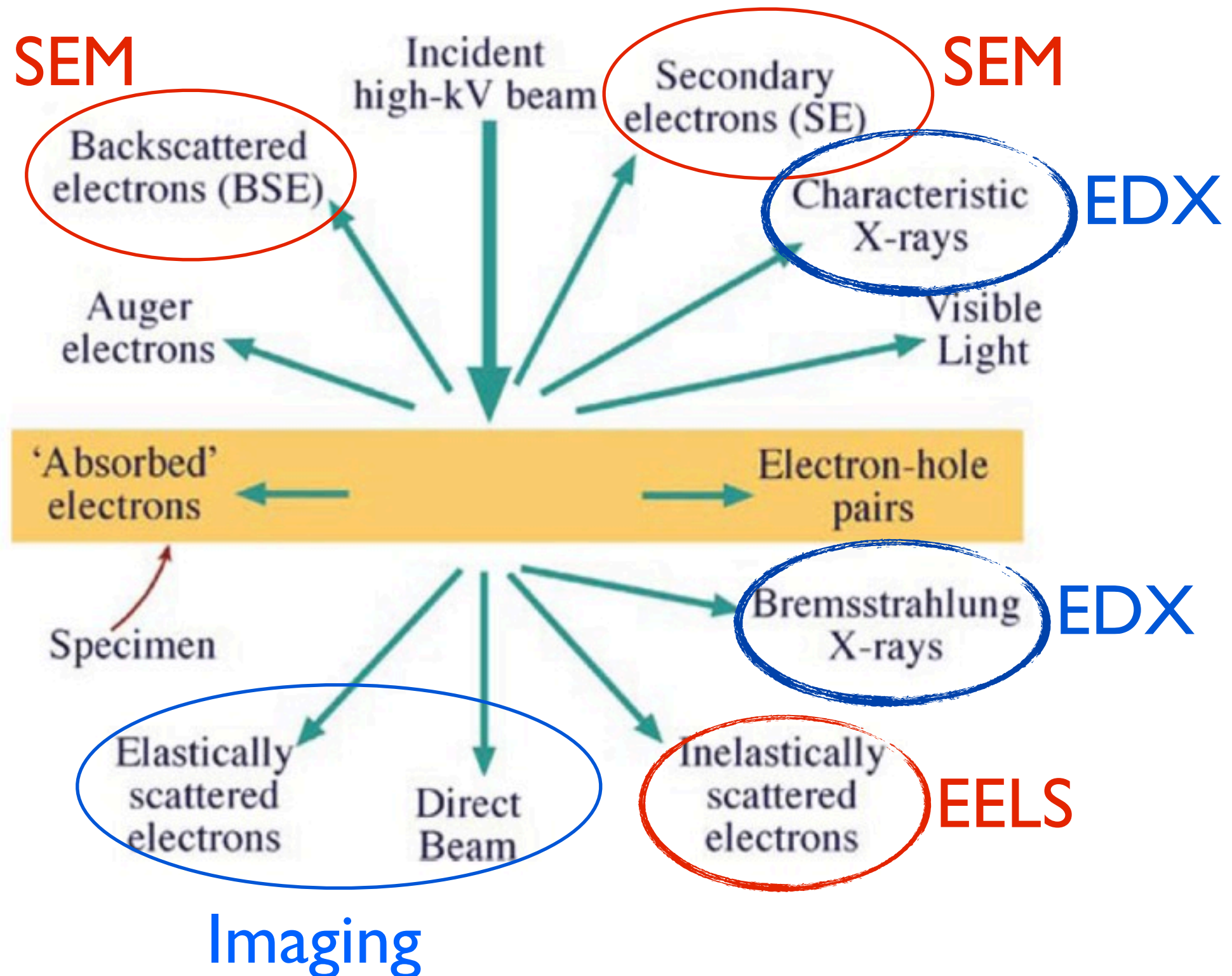
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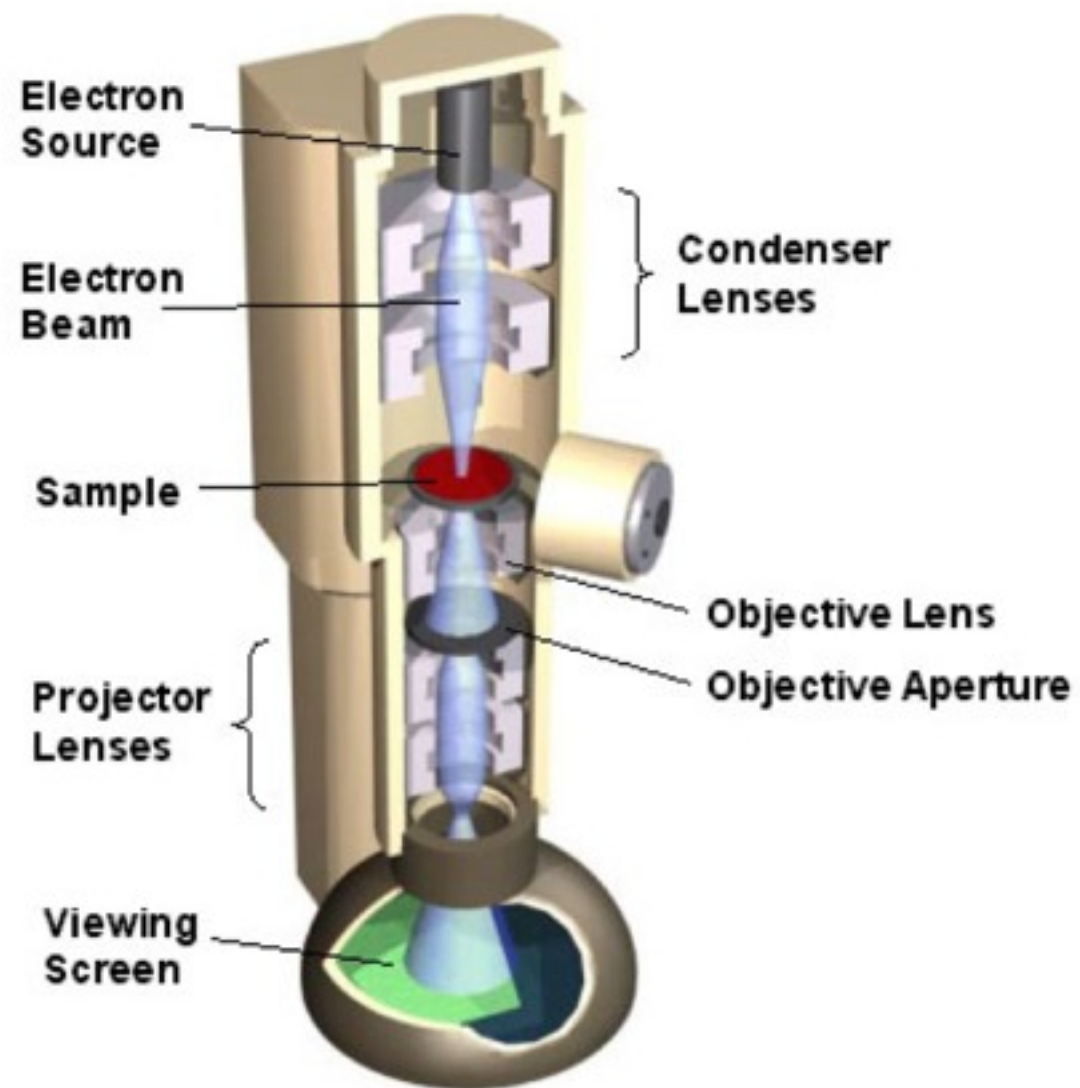


Typical TEM+EDX+EELS

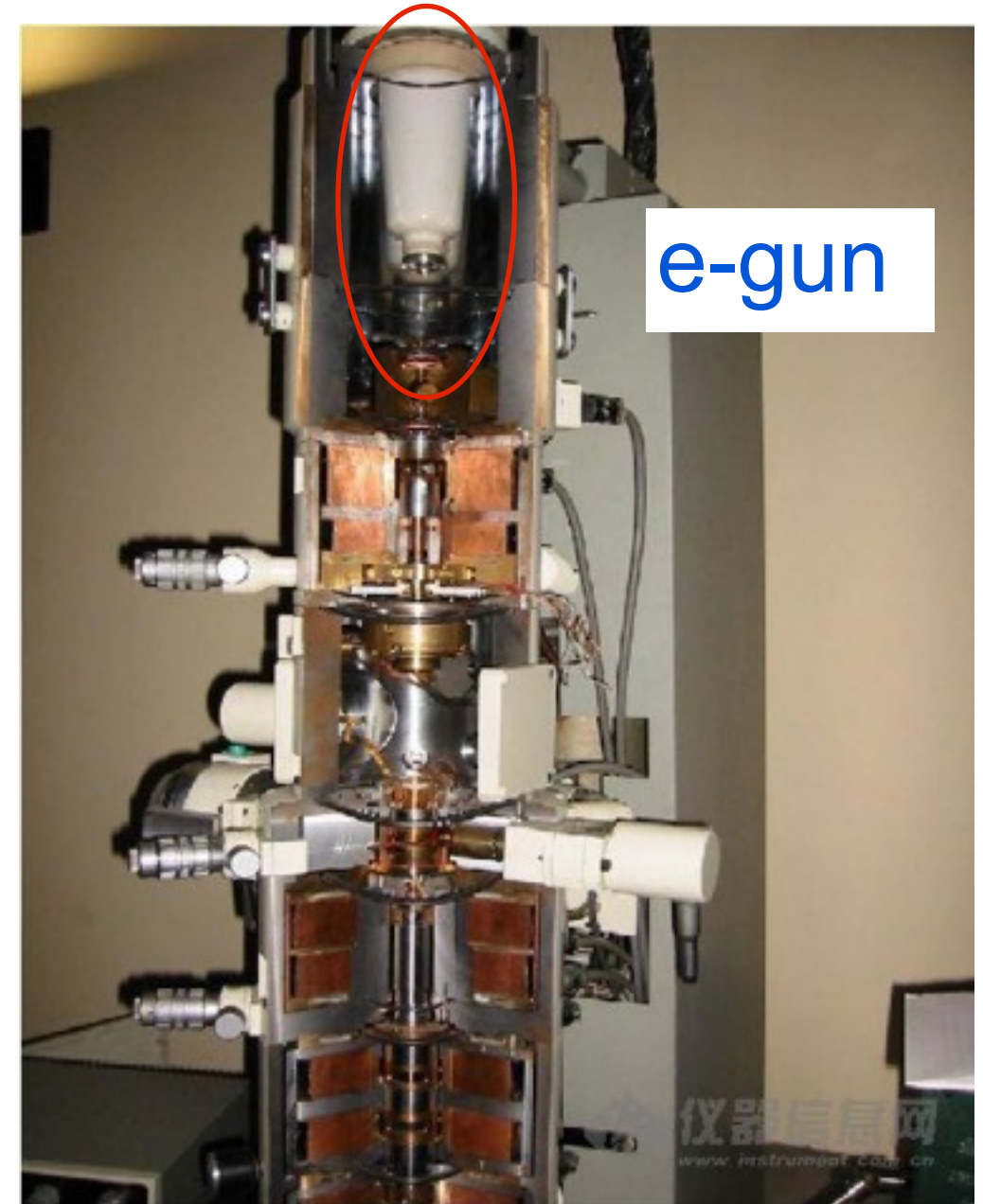
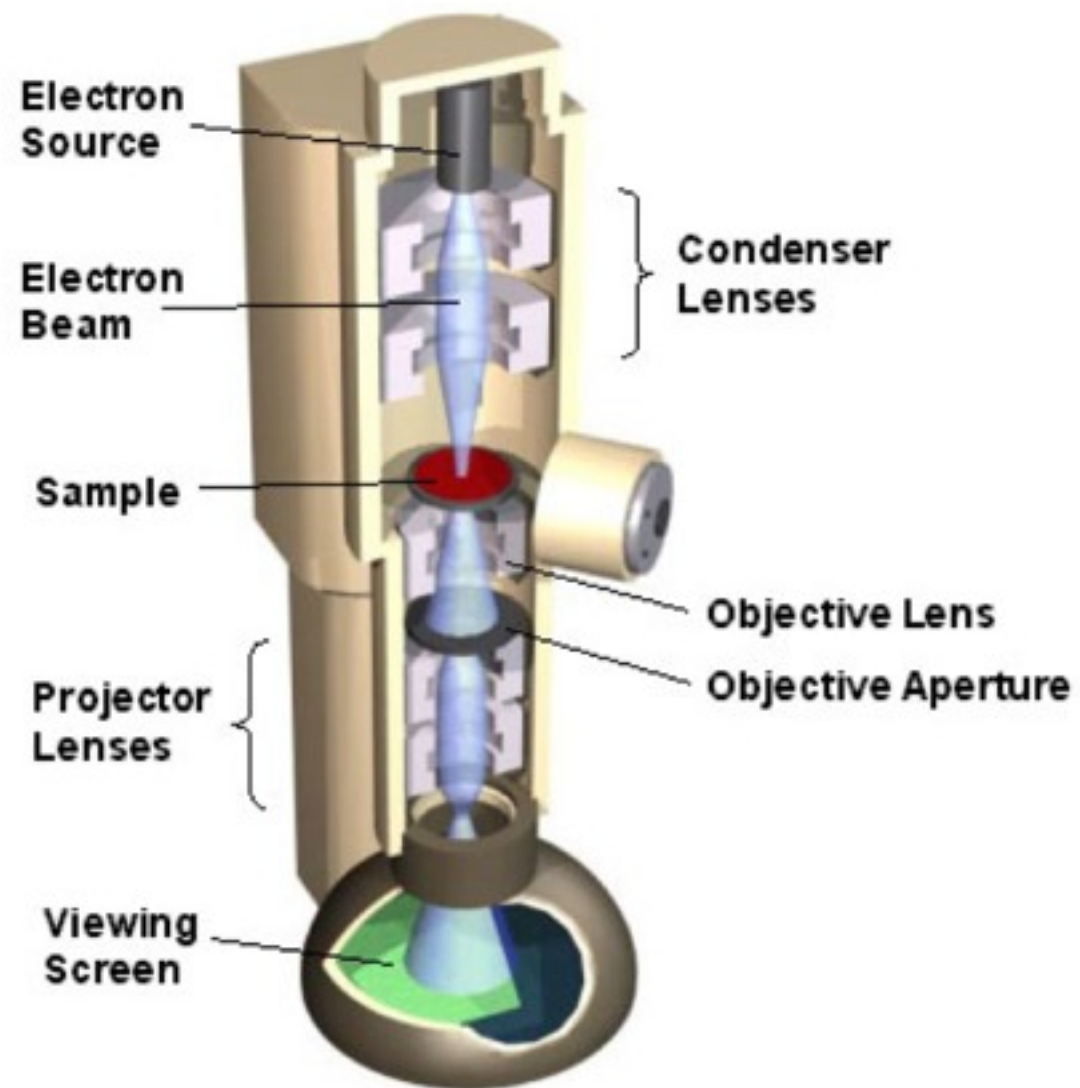


Typical TEM+EDX+EELS

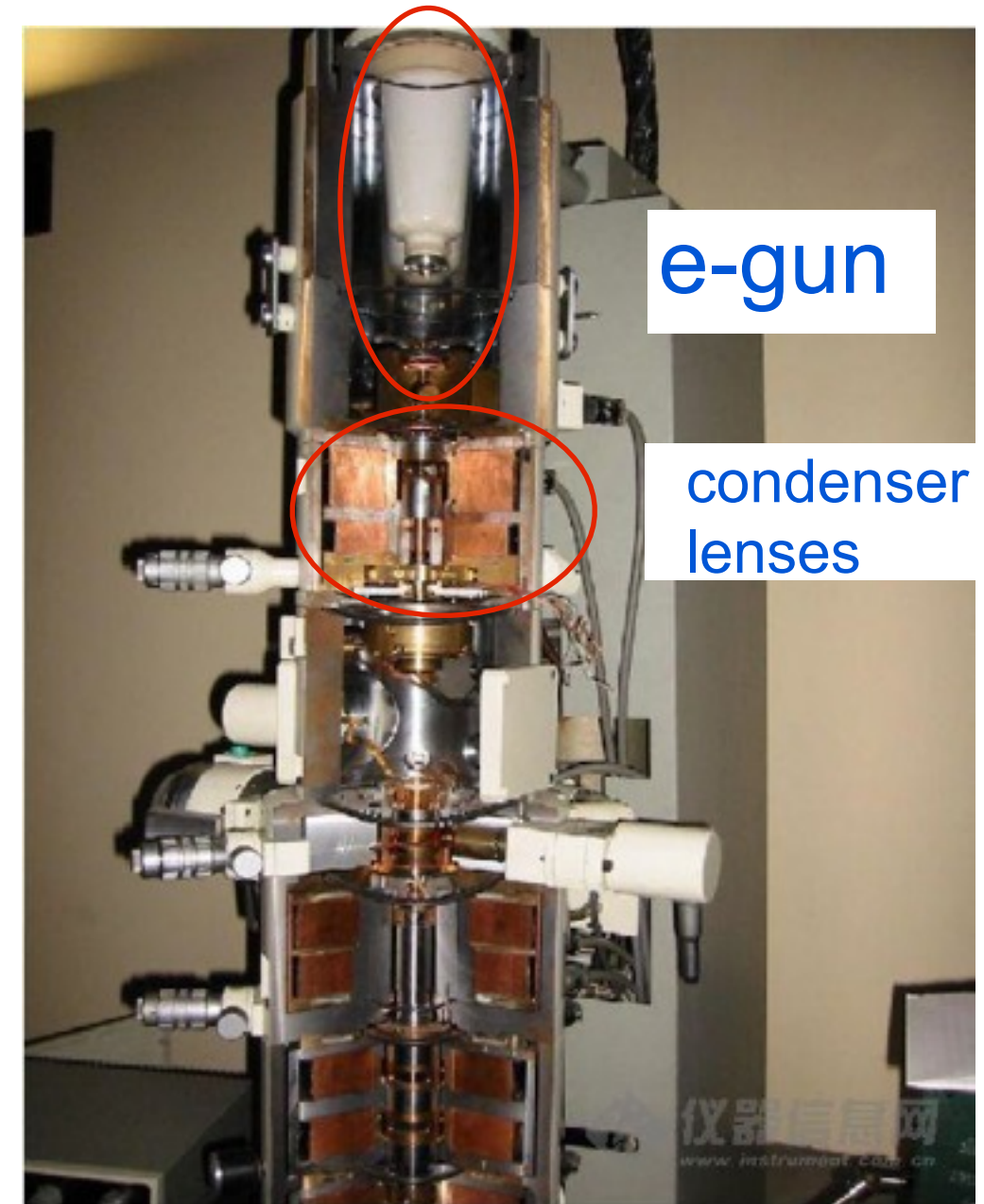
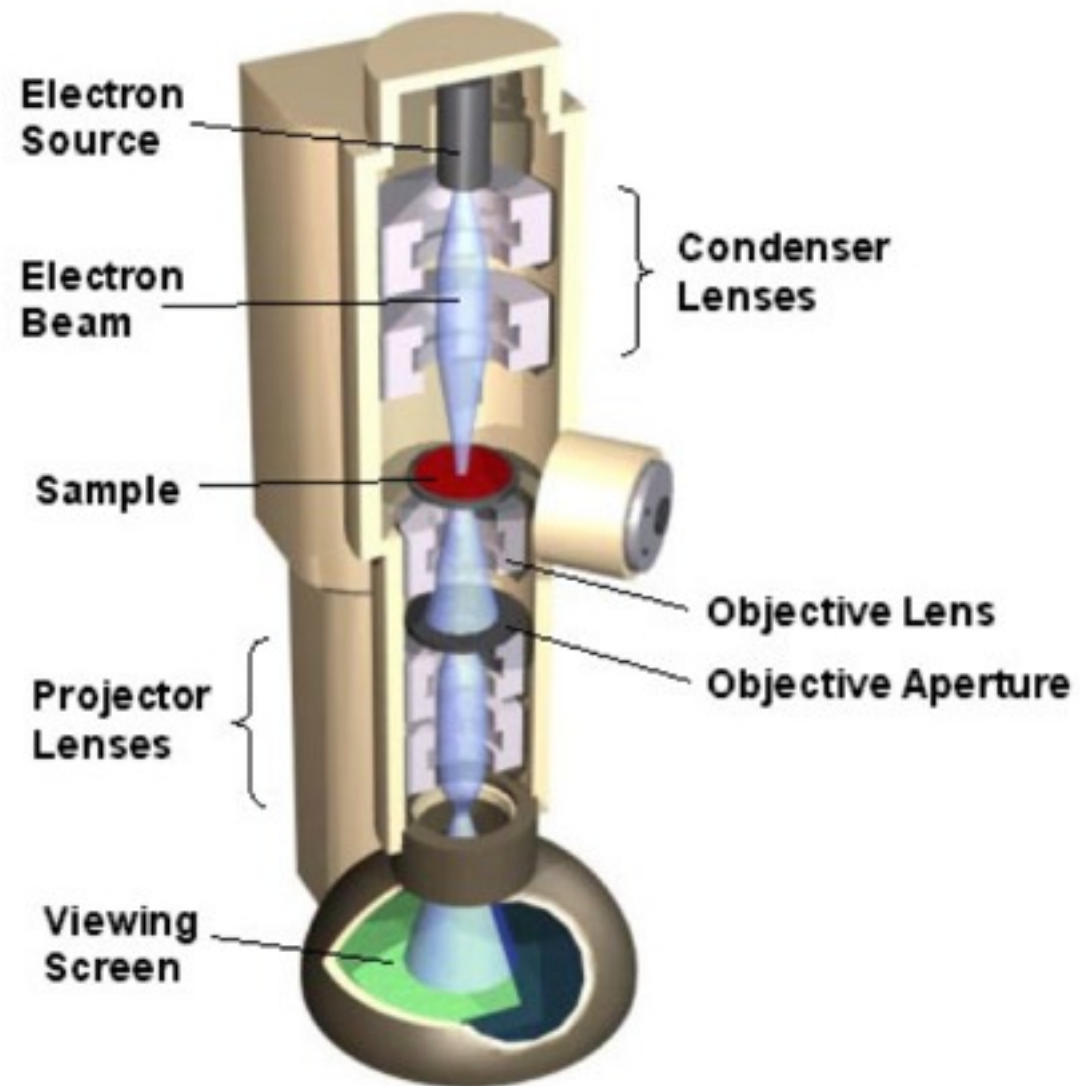
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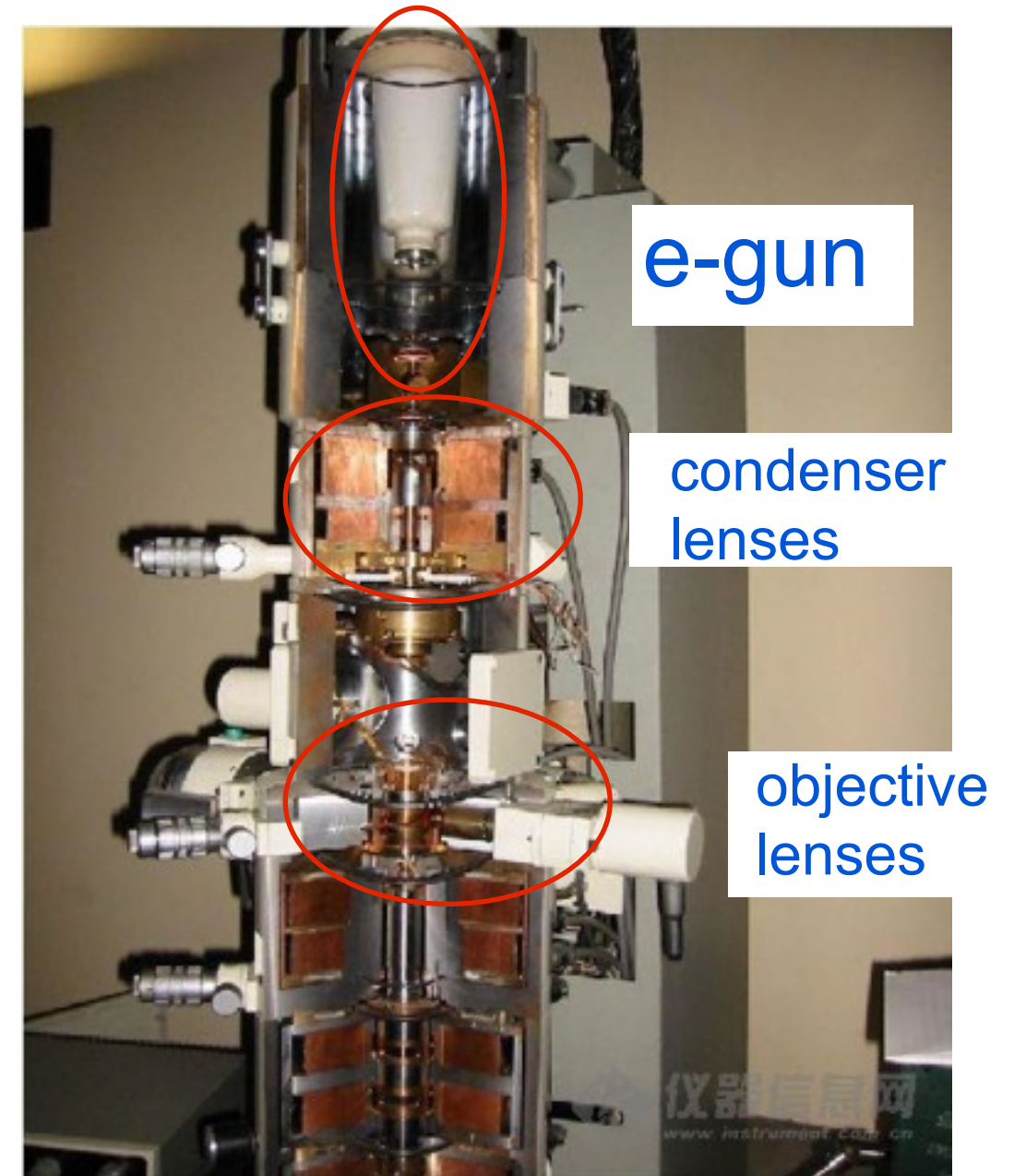
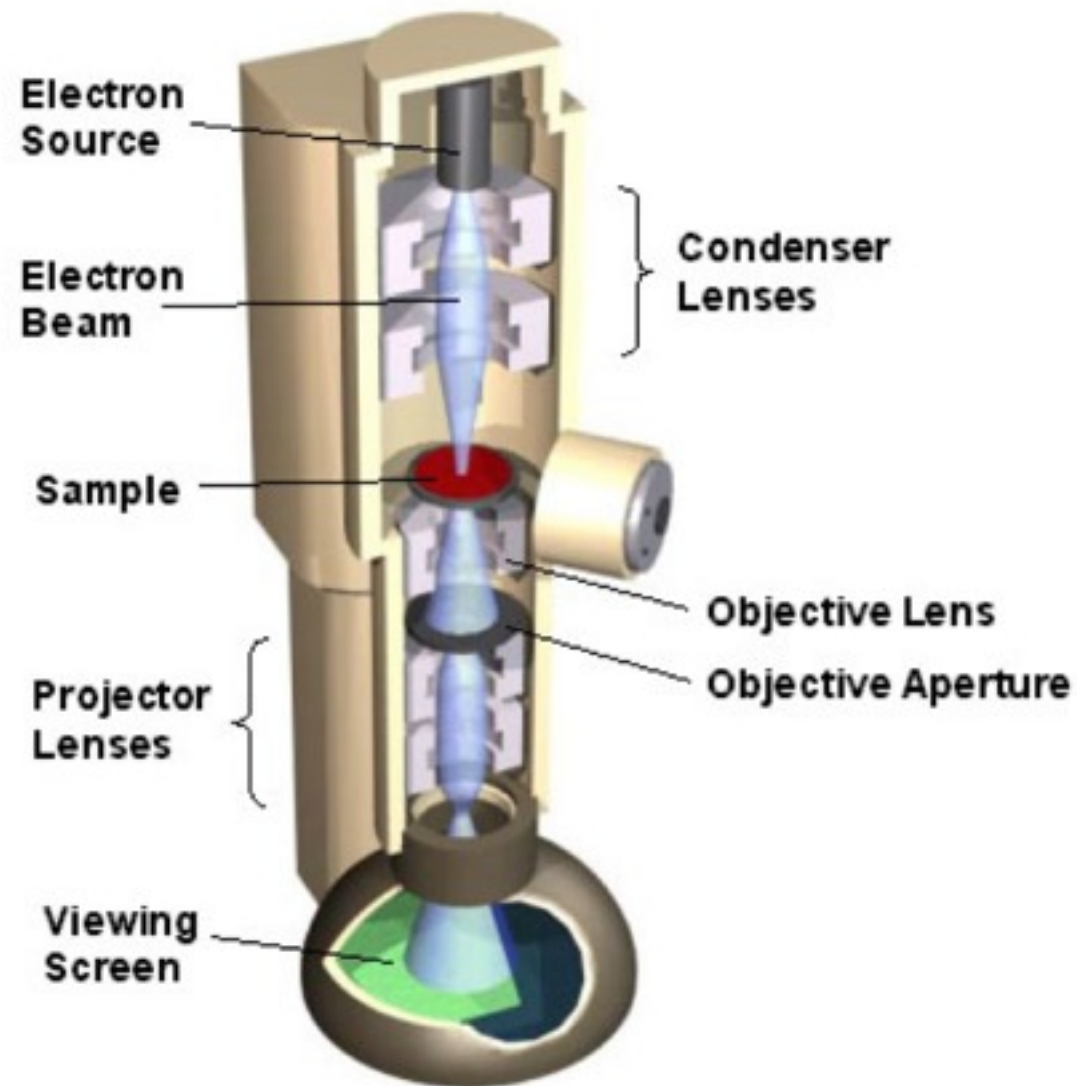
Typical TEM+EDX+EELS



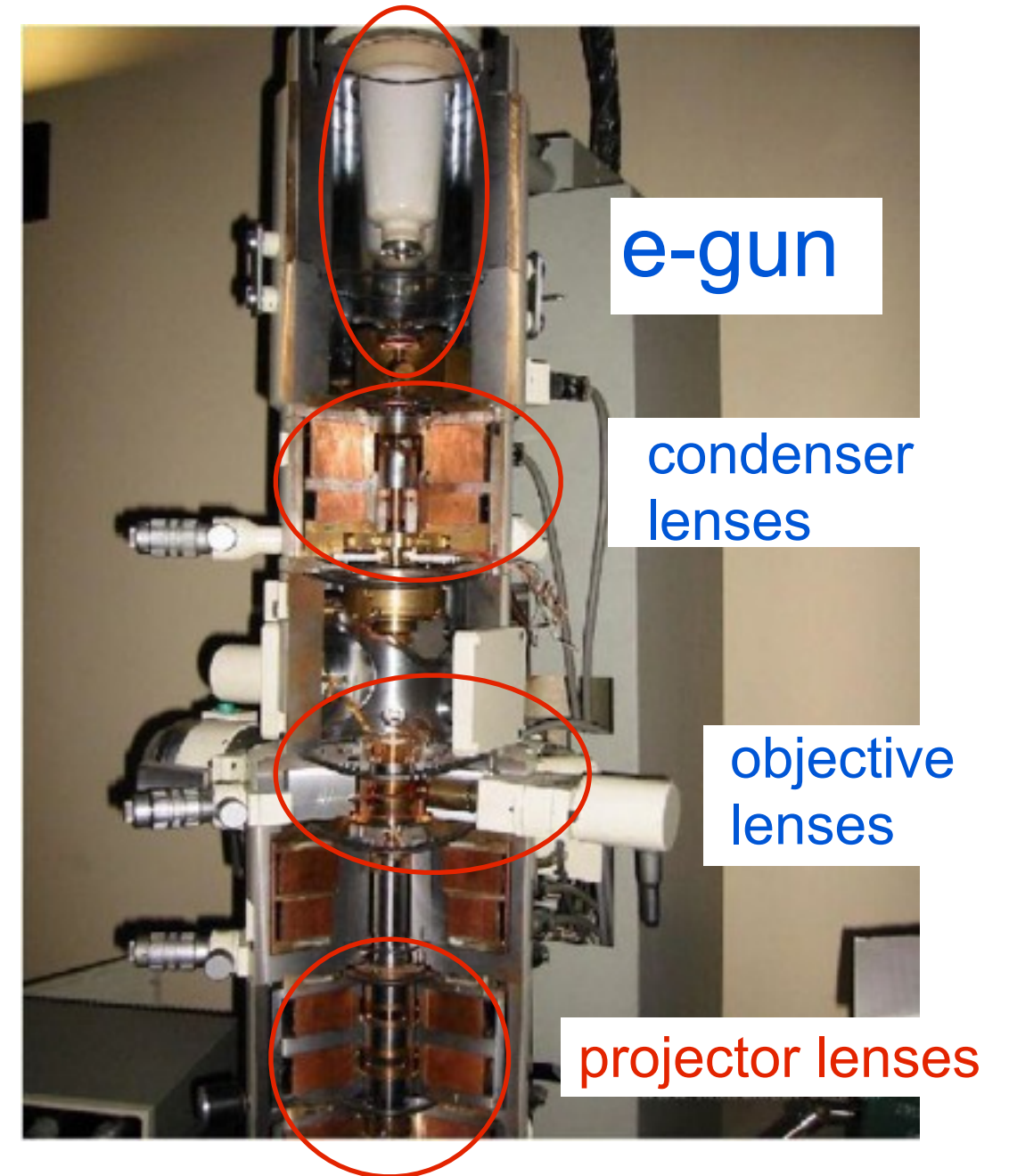
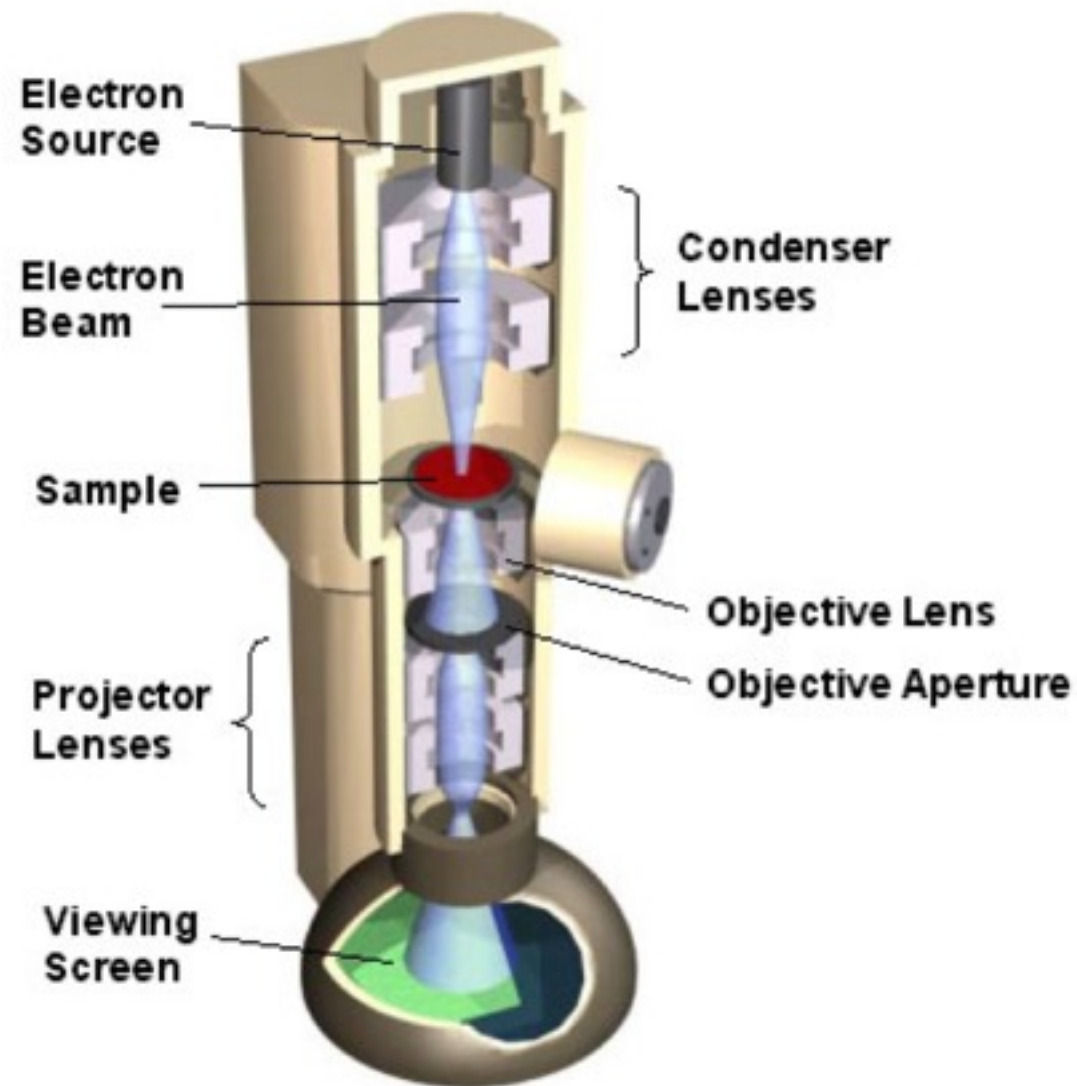
Typical TEM+EDX+EELS



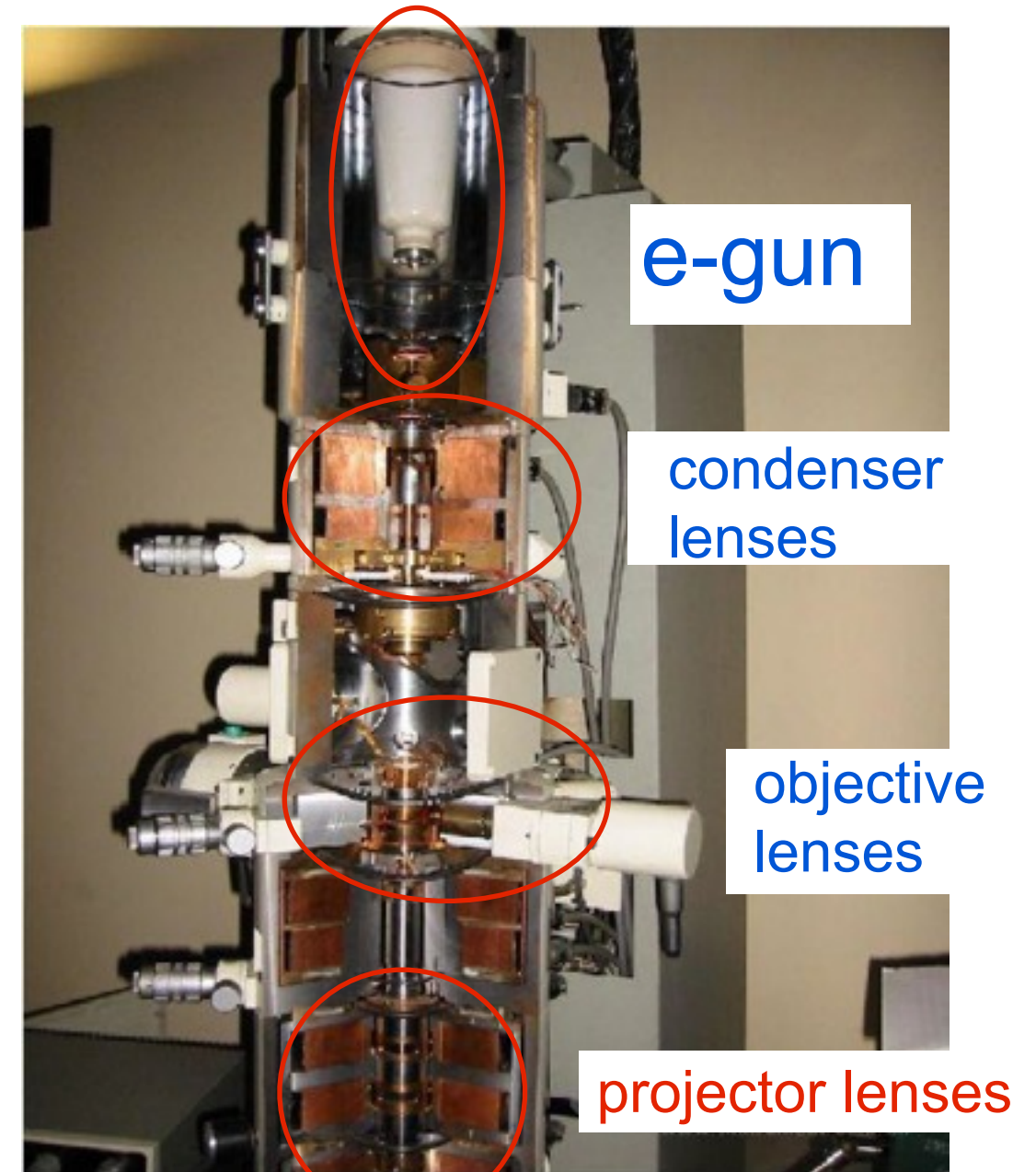
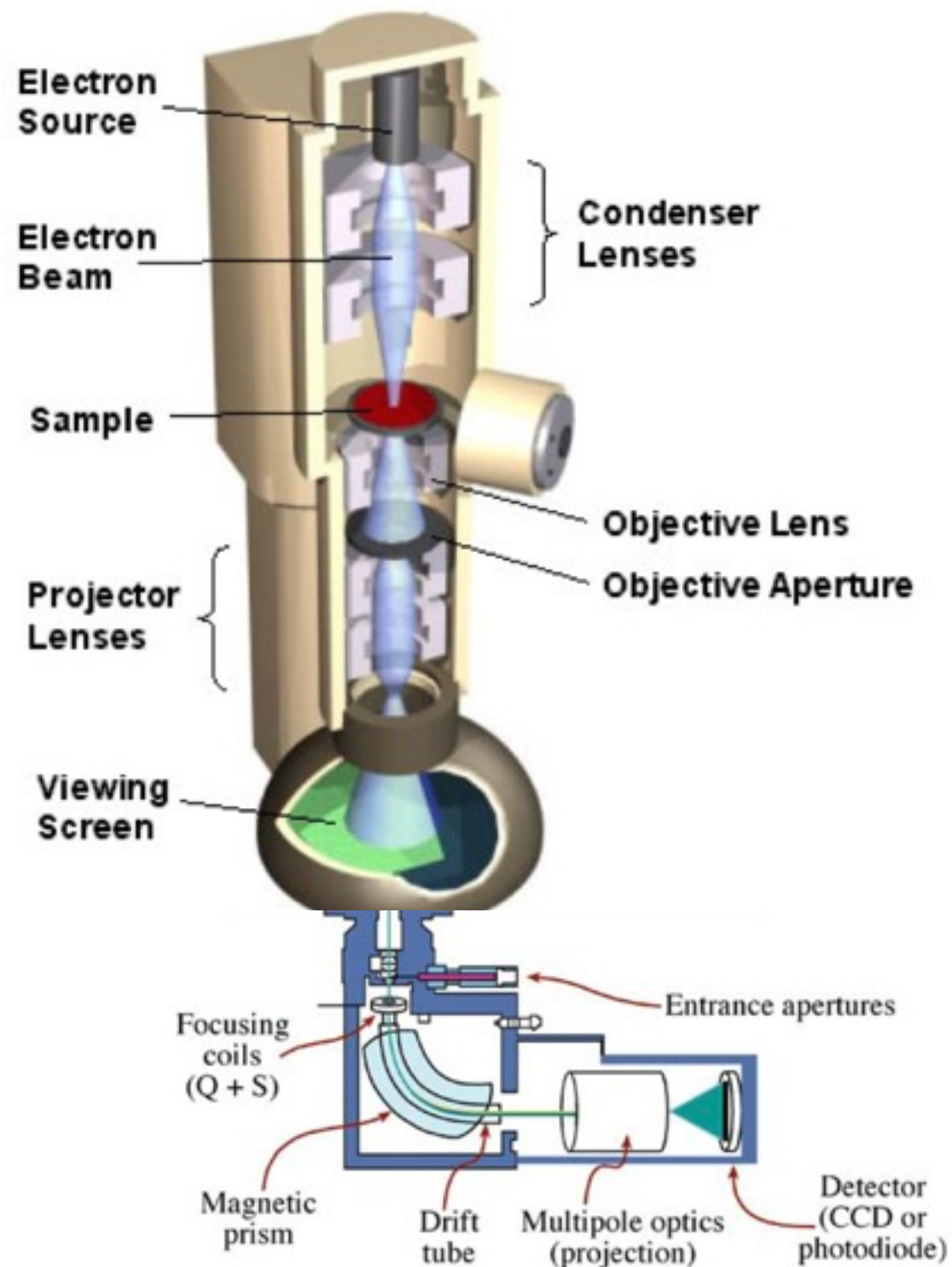
Typical TEM+EDX+EELS



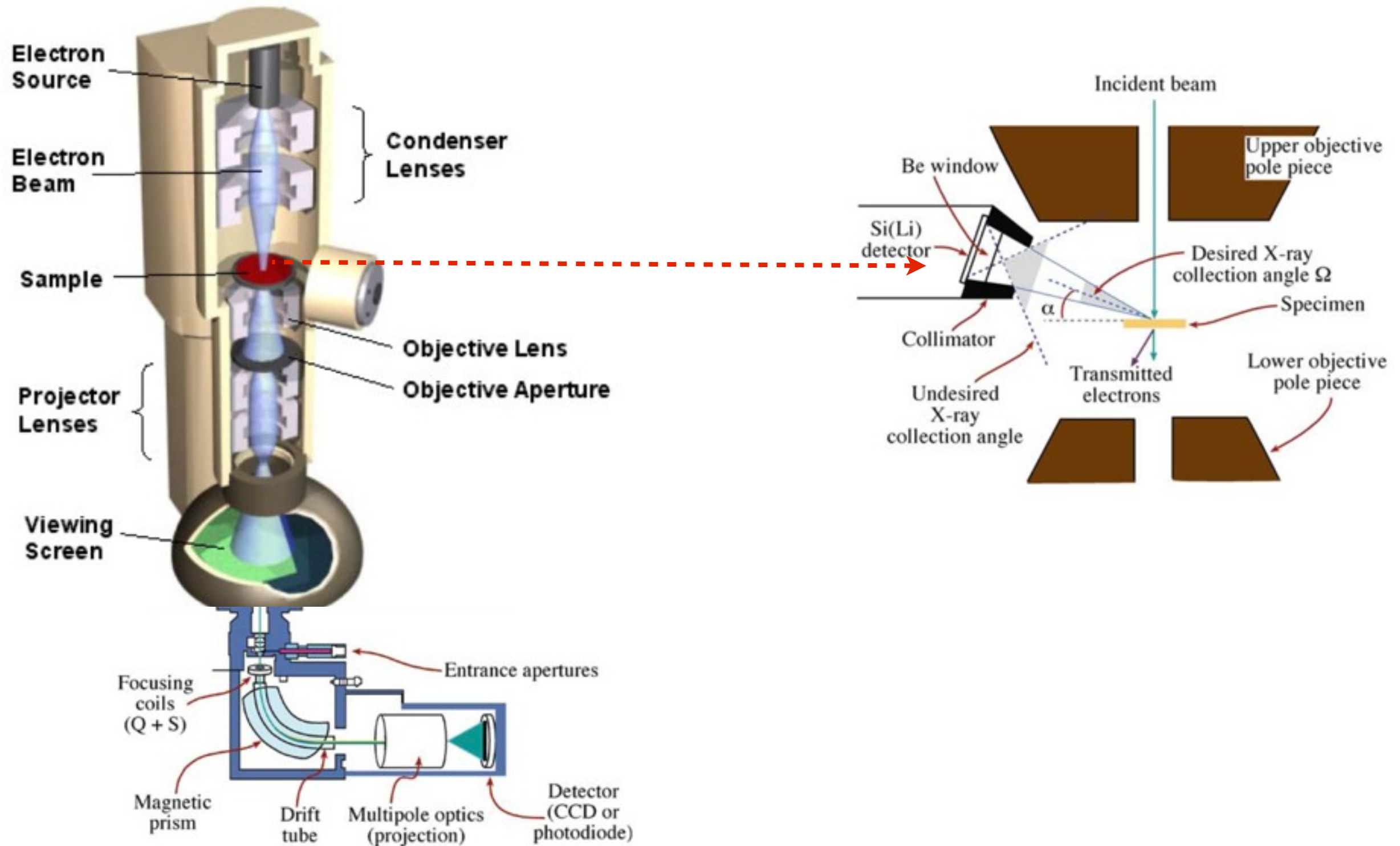
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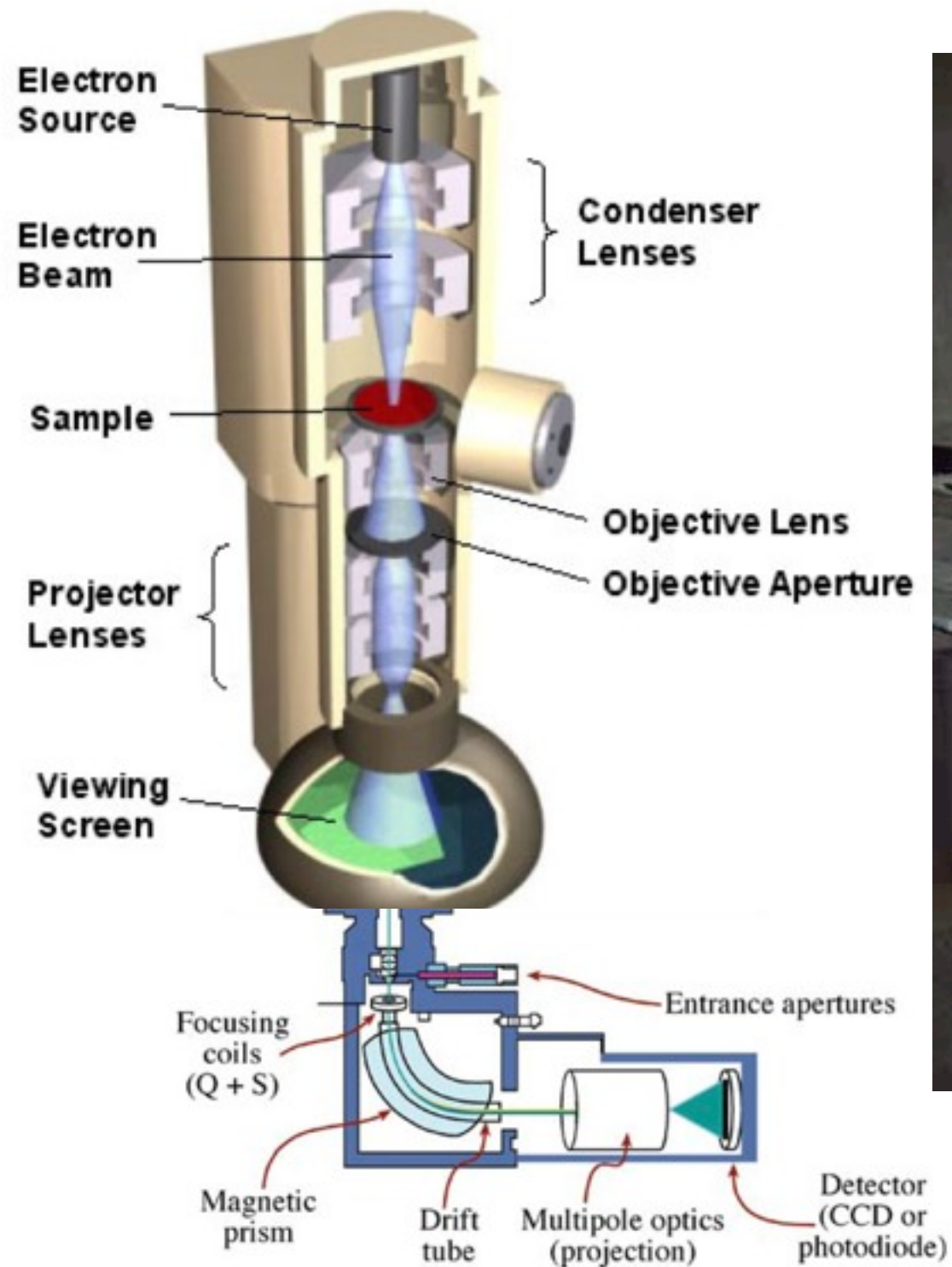
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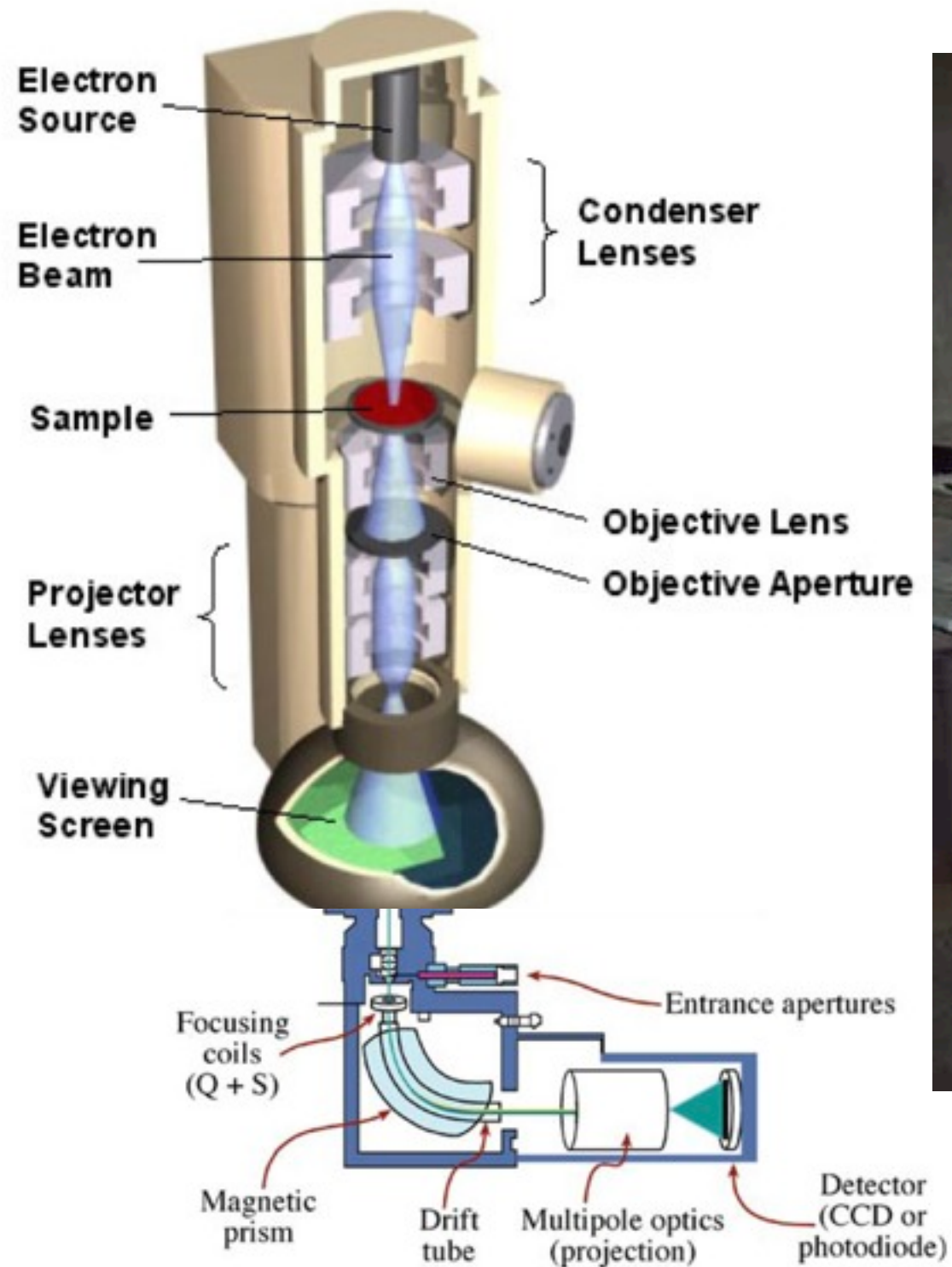
Typical TEM+EDX+EELS



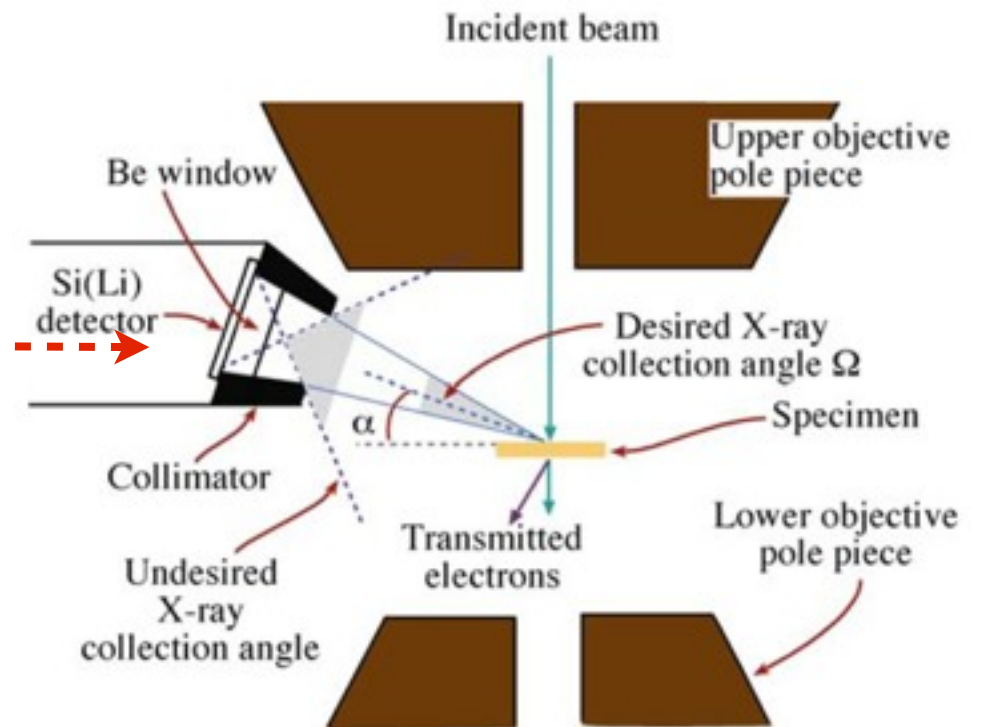
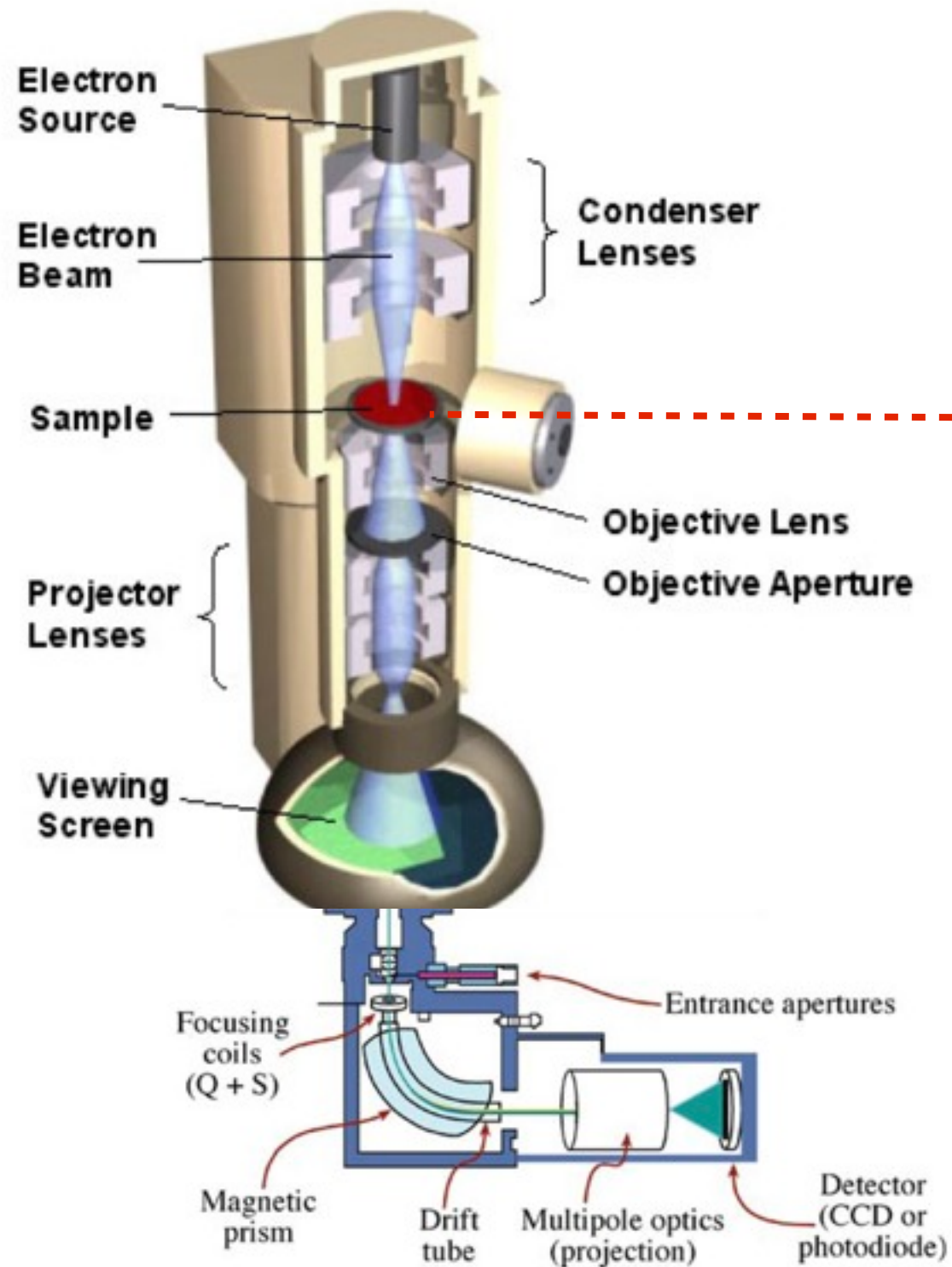
Typical TEM+EDX+EELS



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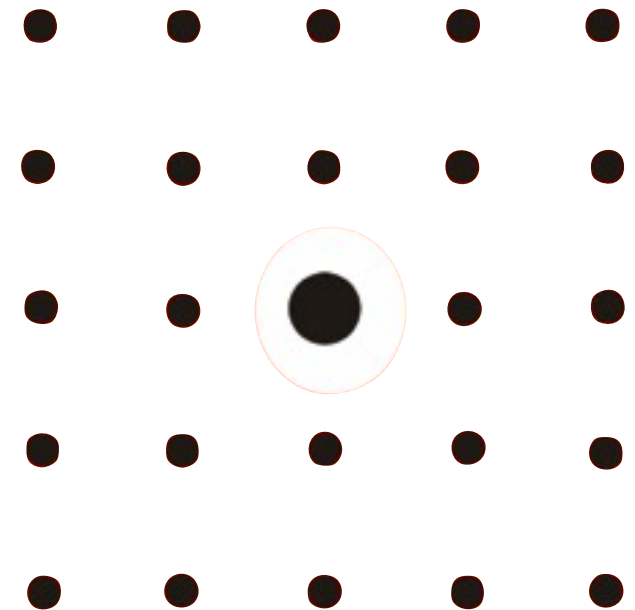
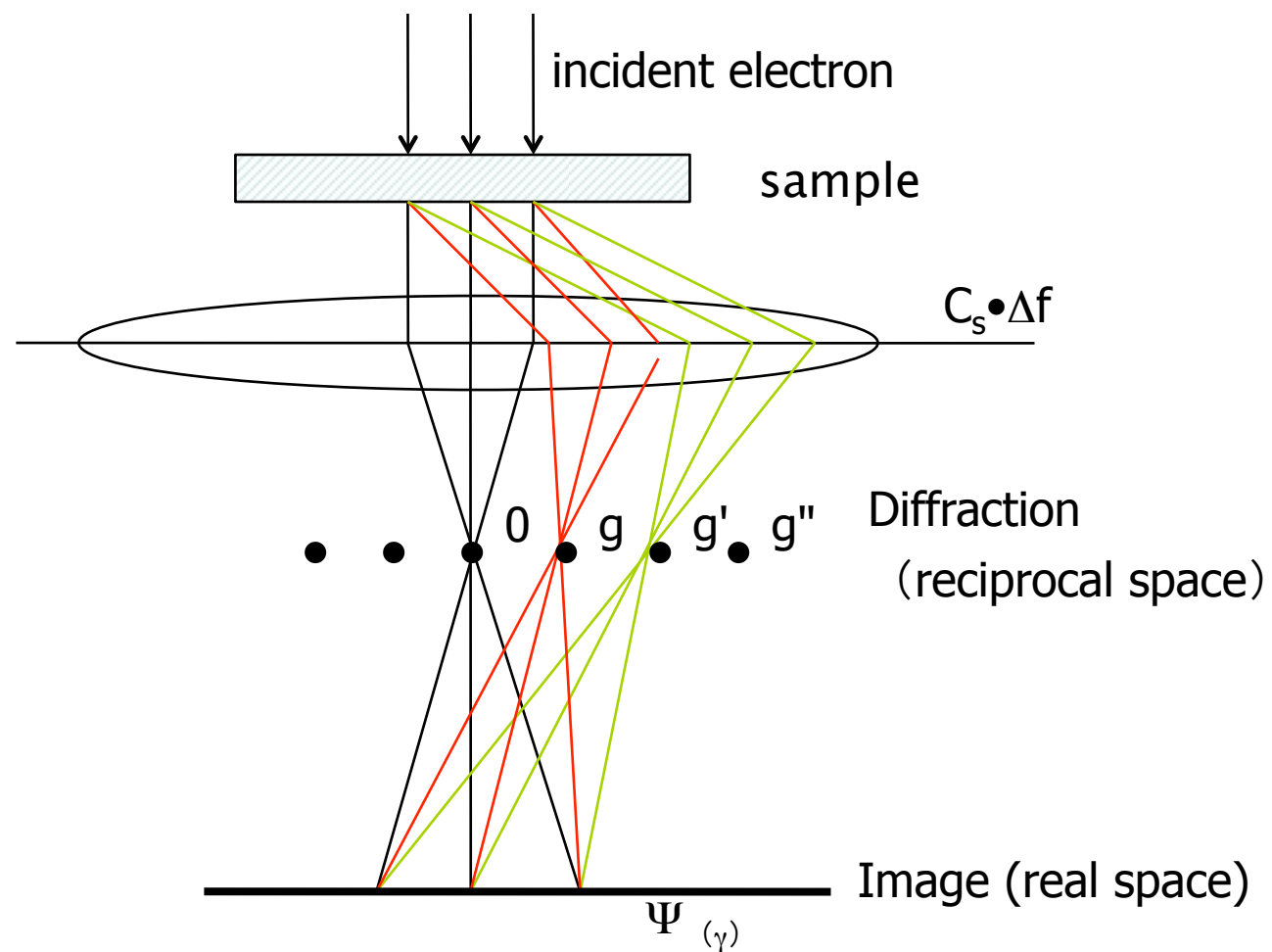
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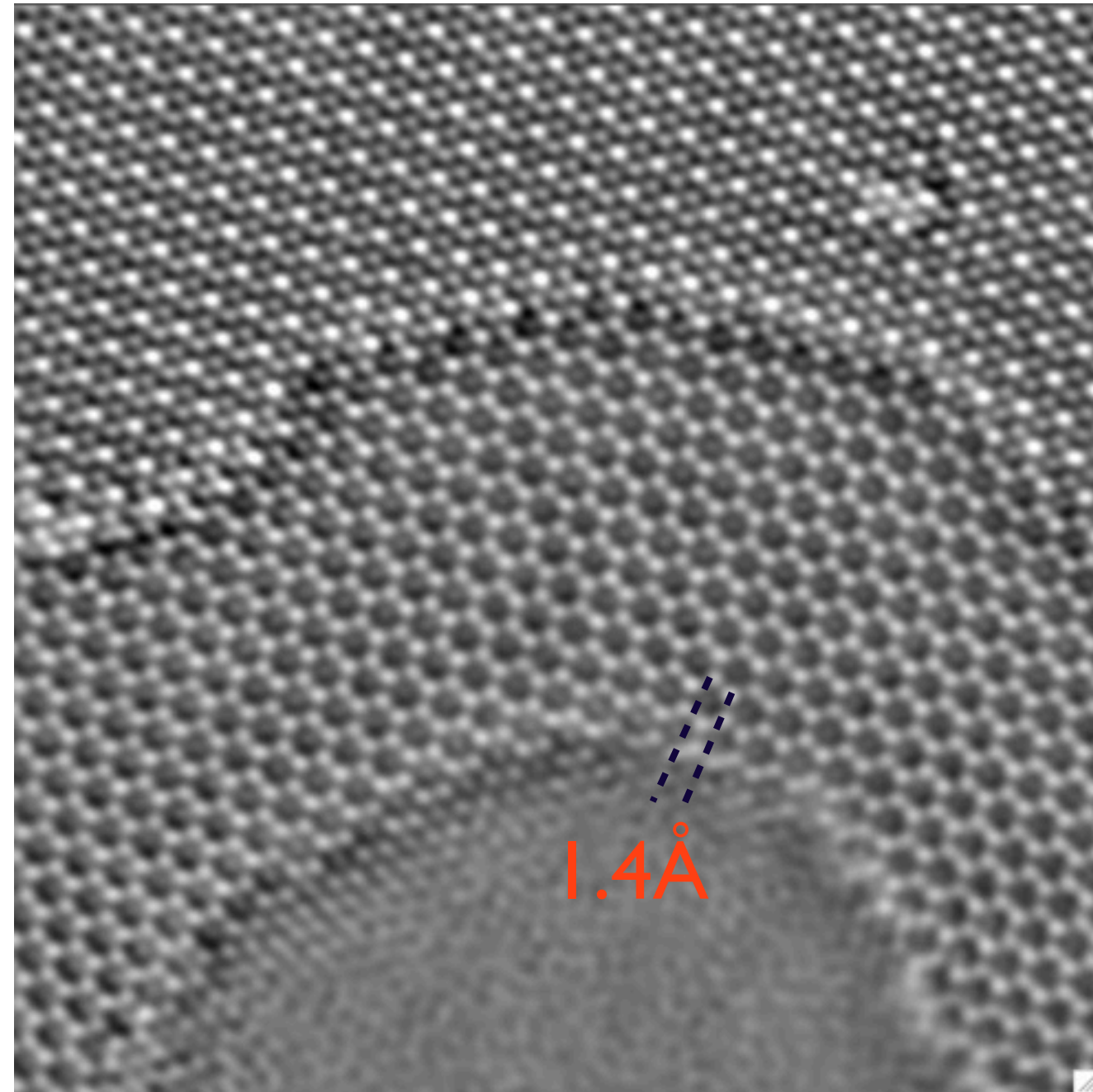
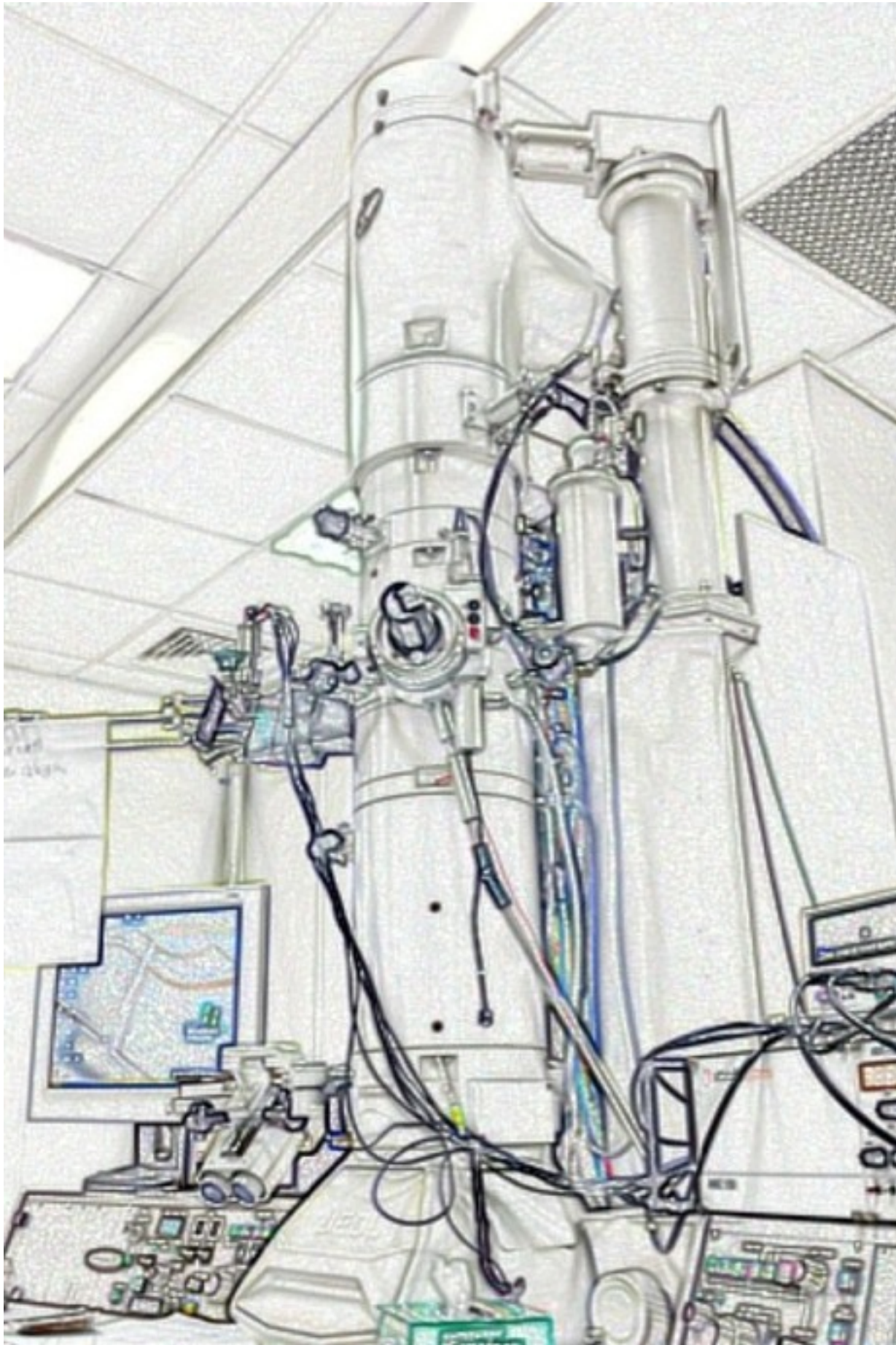
C. Diffraction

No lens for X-ray: Only diffraction can be seen

Electron microscope utilizes magnetic lens to see both image and diffraction



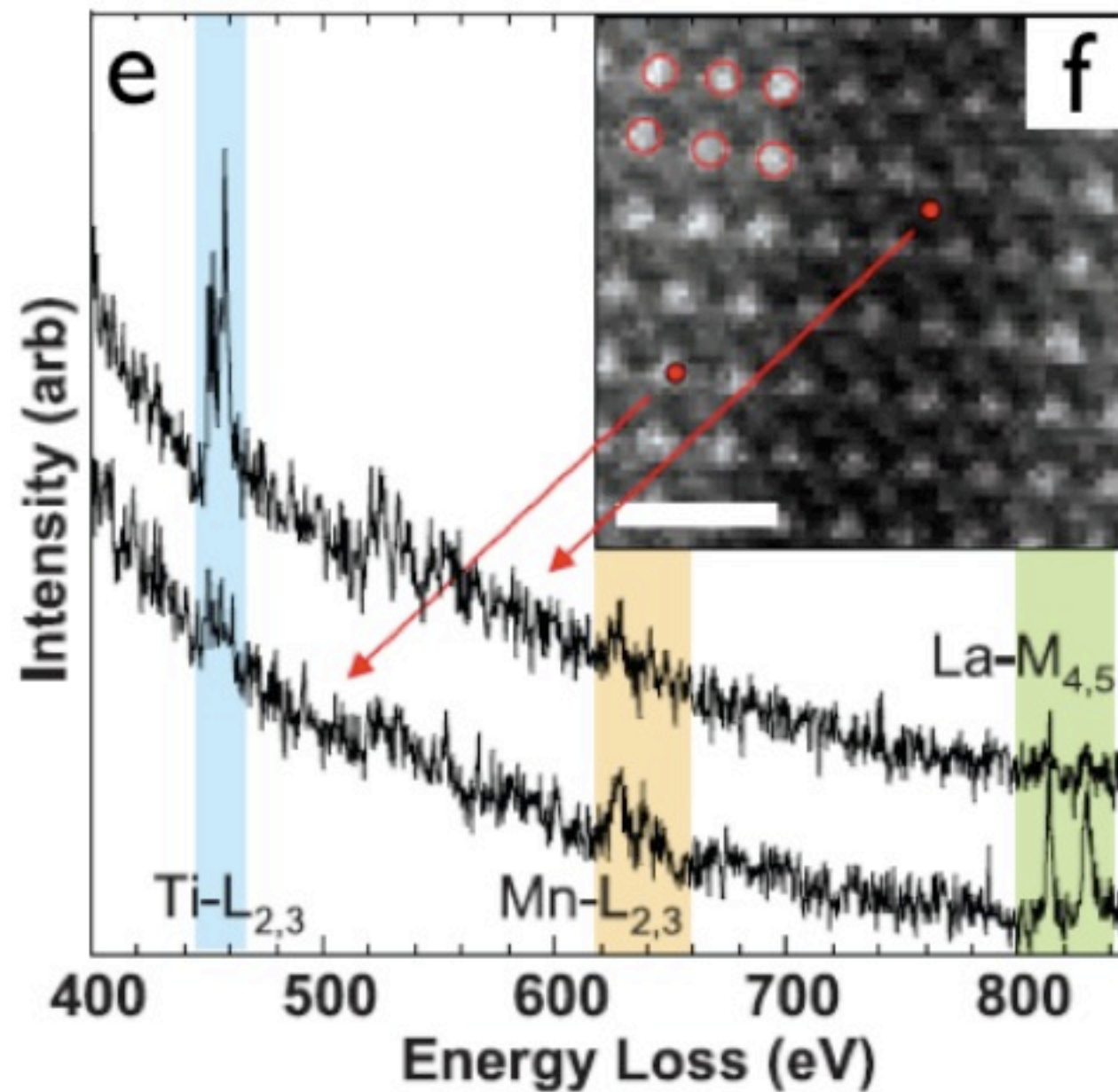
Imaging, Spectroscopy and Diffraction



Atomic Resolution

Imaging, Spectroscopy and Diffraction

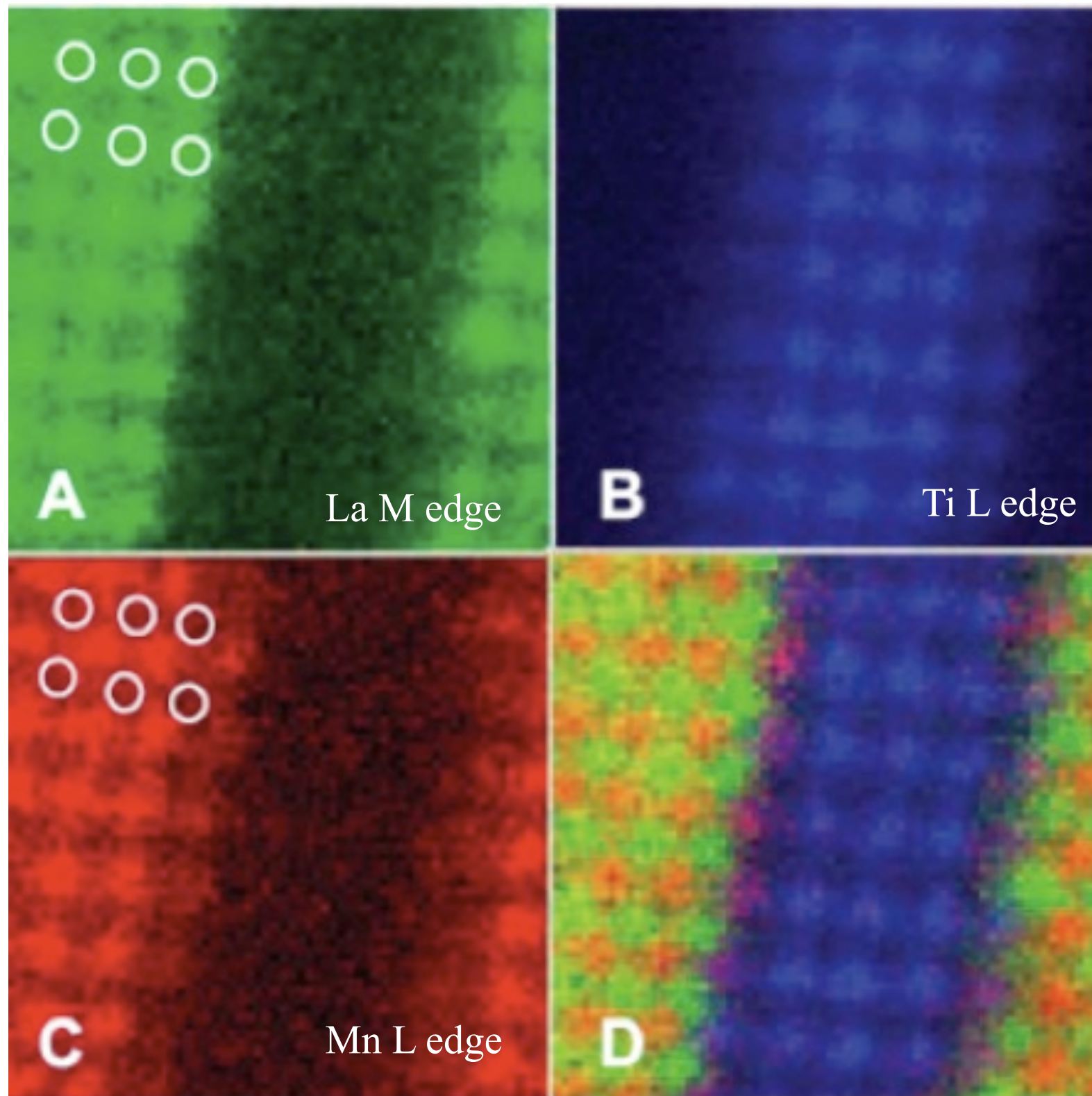
Imaging, Spectroscopy and Diffraction



Atomic resolution of EELS of $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{SrTiO}_3$ multilayer

- D. A. Muller, et al, SCIENCE **319** 1073-1075 (2008)

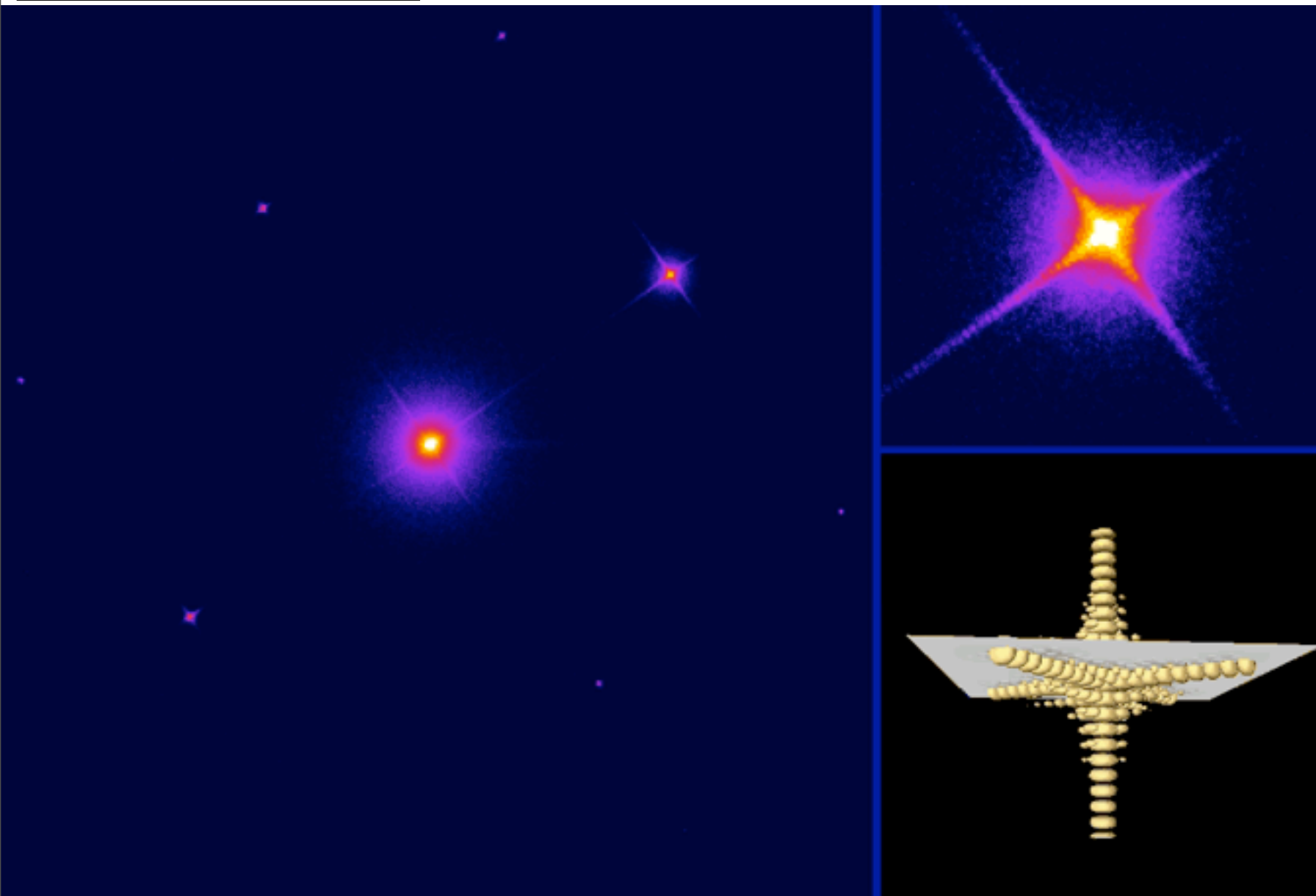
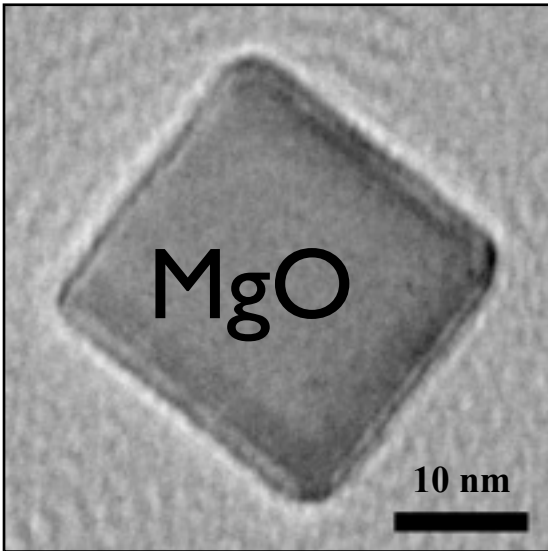
Imaging, Spectroscopy and Diffraction



Atomic resolution compositional and bonding maps

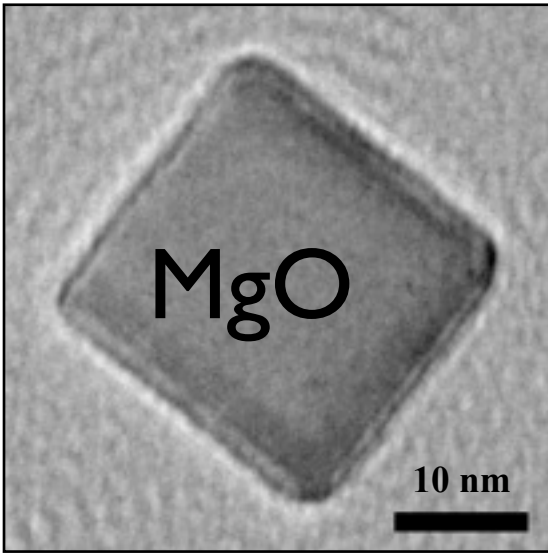
Imaging, Spectroscopy and Diffraction

Atomic-resolution phase recovery with dynamic support

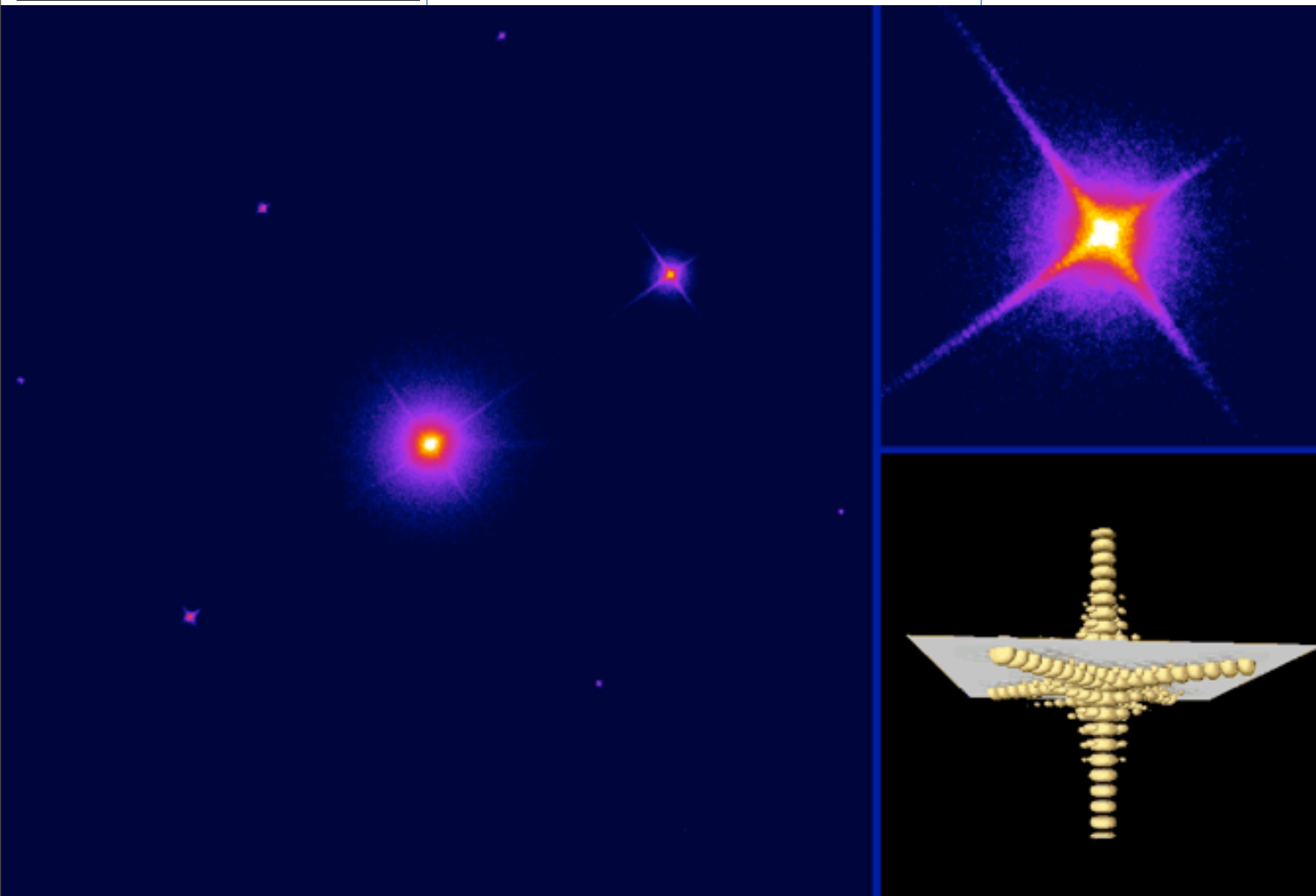
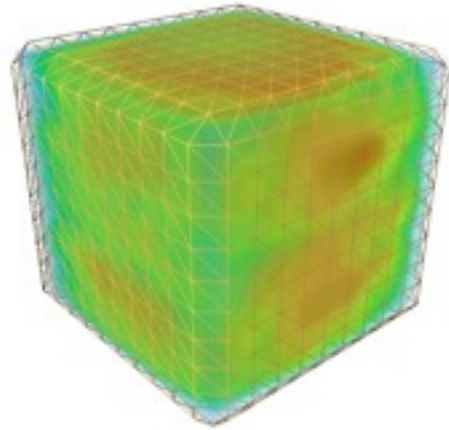


Imaging, Spectroscopy and Diffraction

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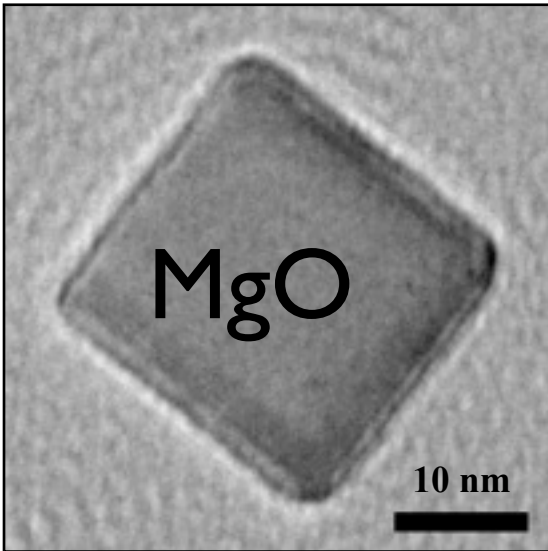


Electron Diffractive Tomography

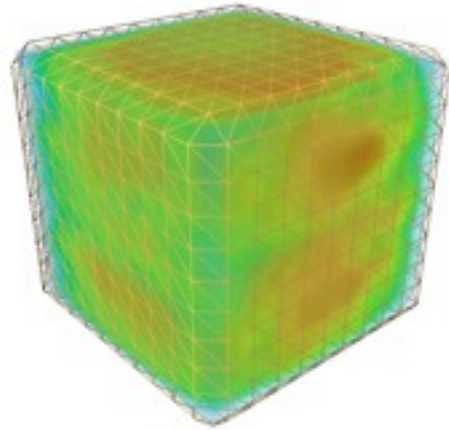


Imaging, Spectroscopy and Diffraction

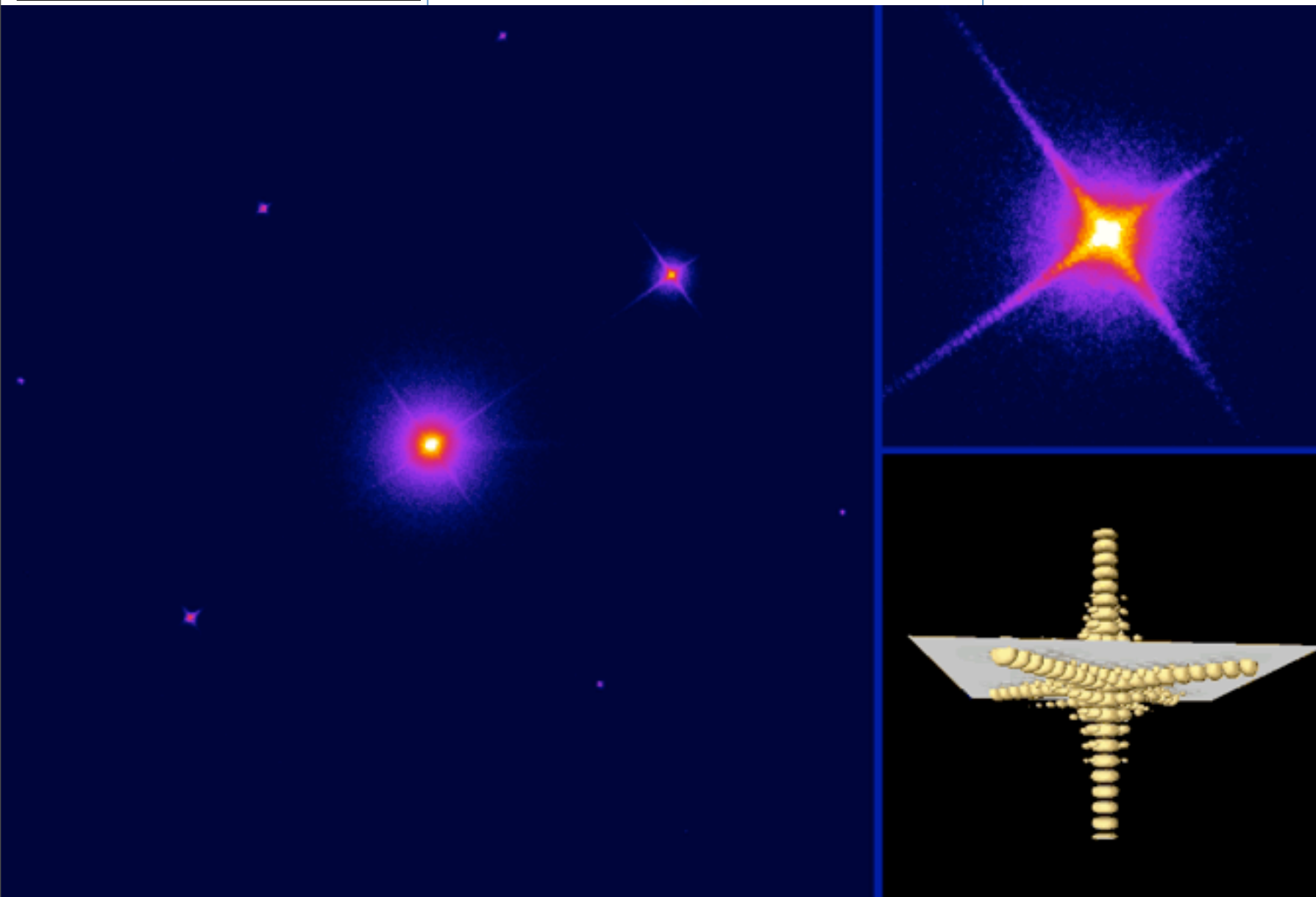
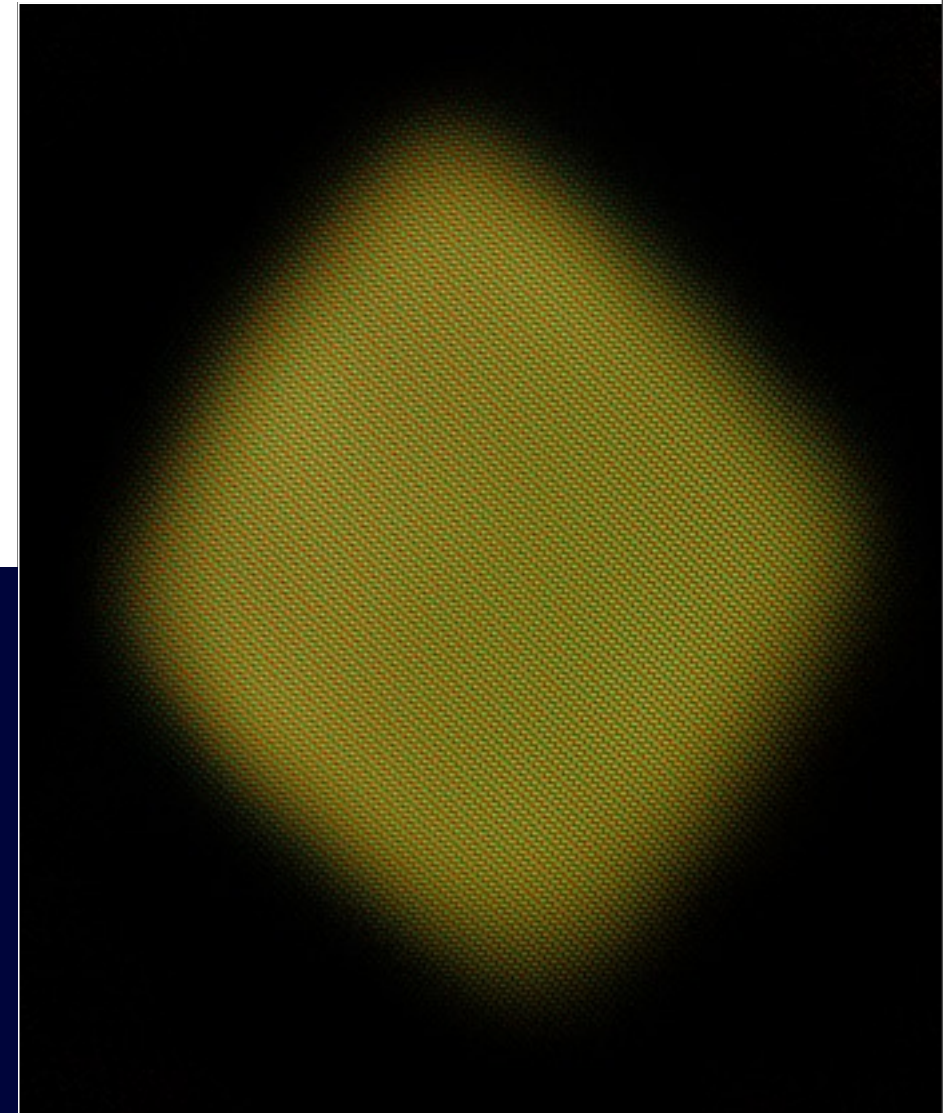
Atomic-resolution phase recovery with dynamic support



Electron Diffractive Tomography

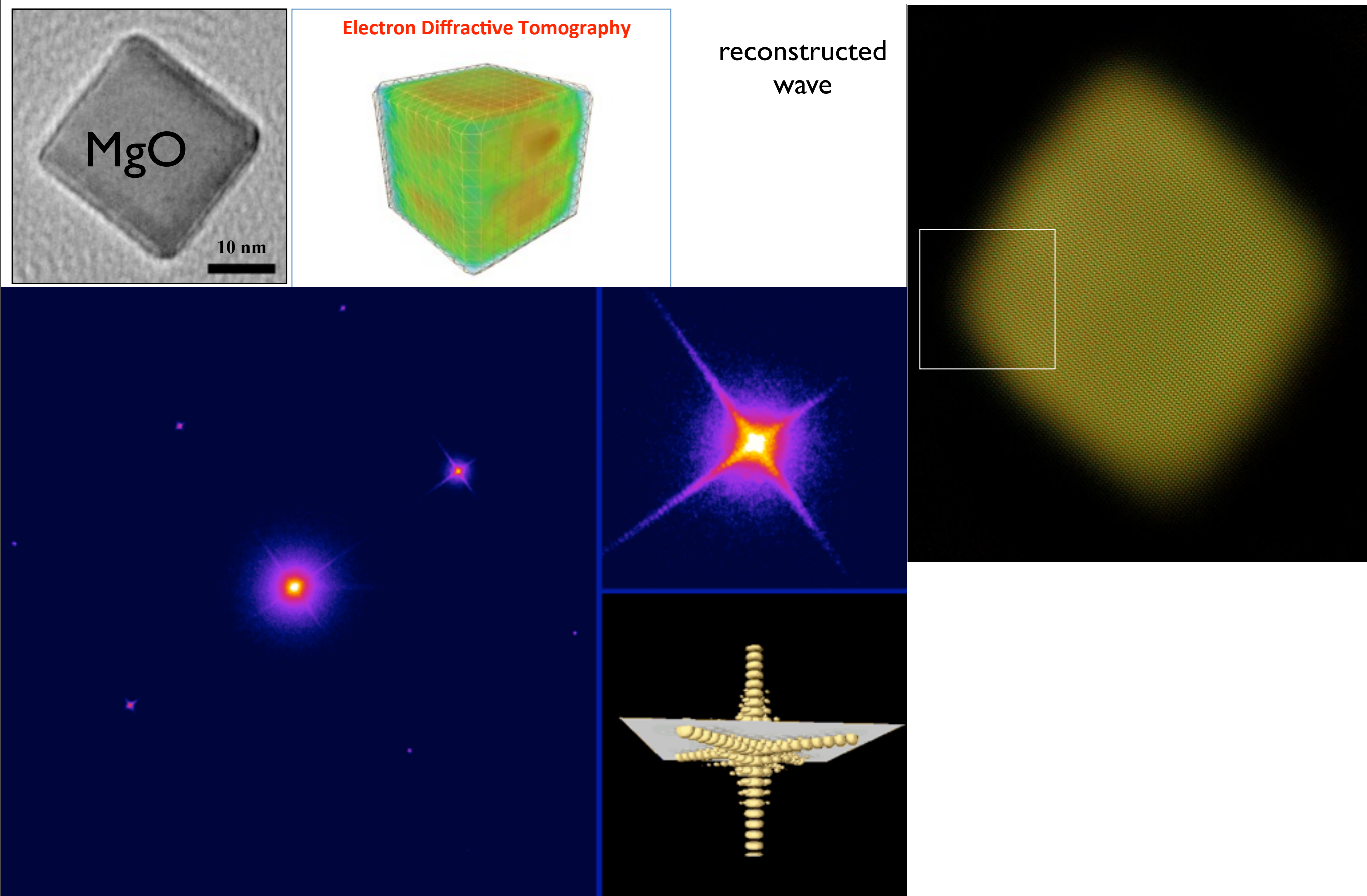


reconstructed
wave



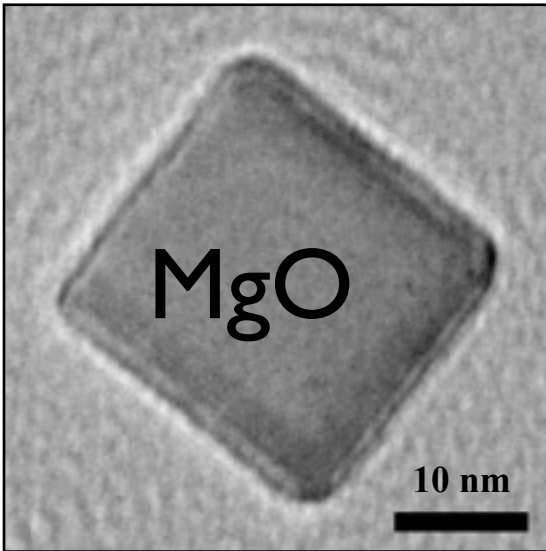
Imaging, Spectroscopy and Diffraction

Atomic-resolution phase recovery with dynamic support

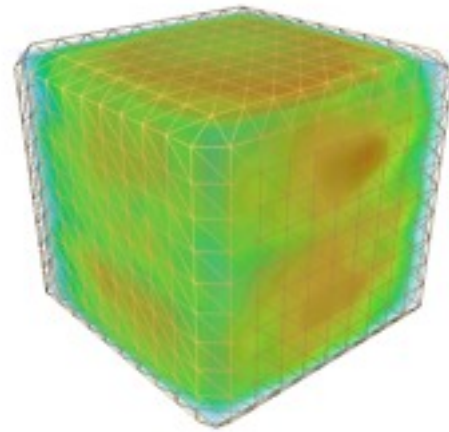


Imaging, Spectroscopy and Diffraction

Atomic-resolution phase recovery with dynamic support

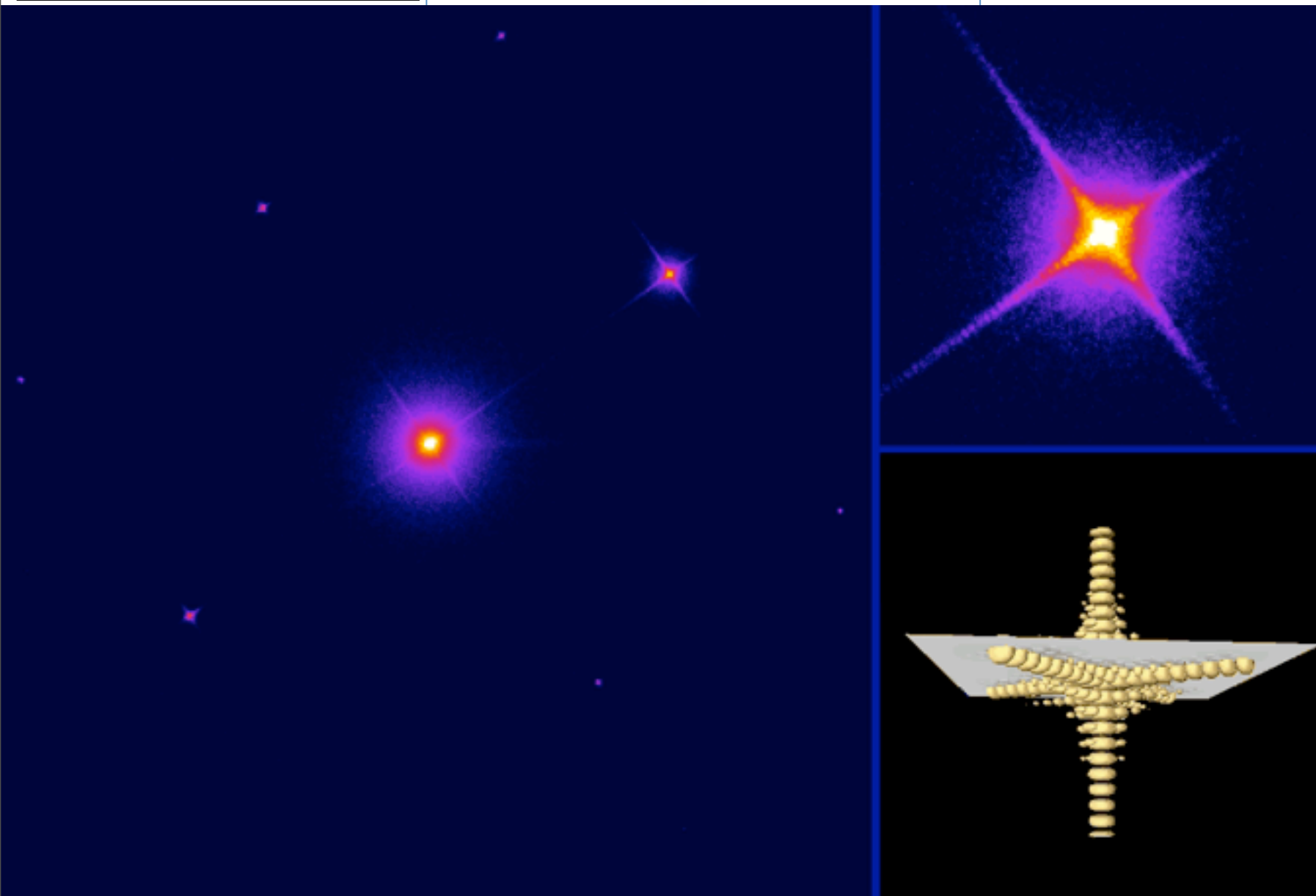
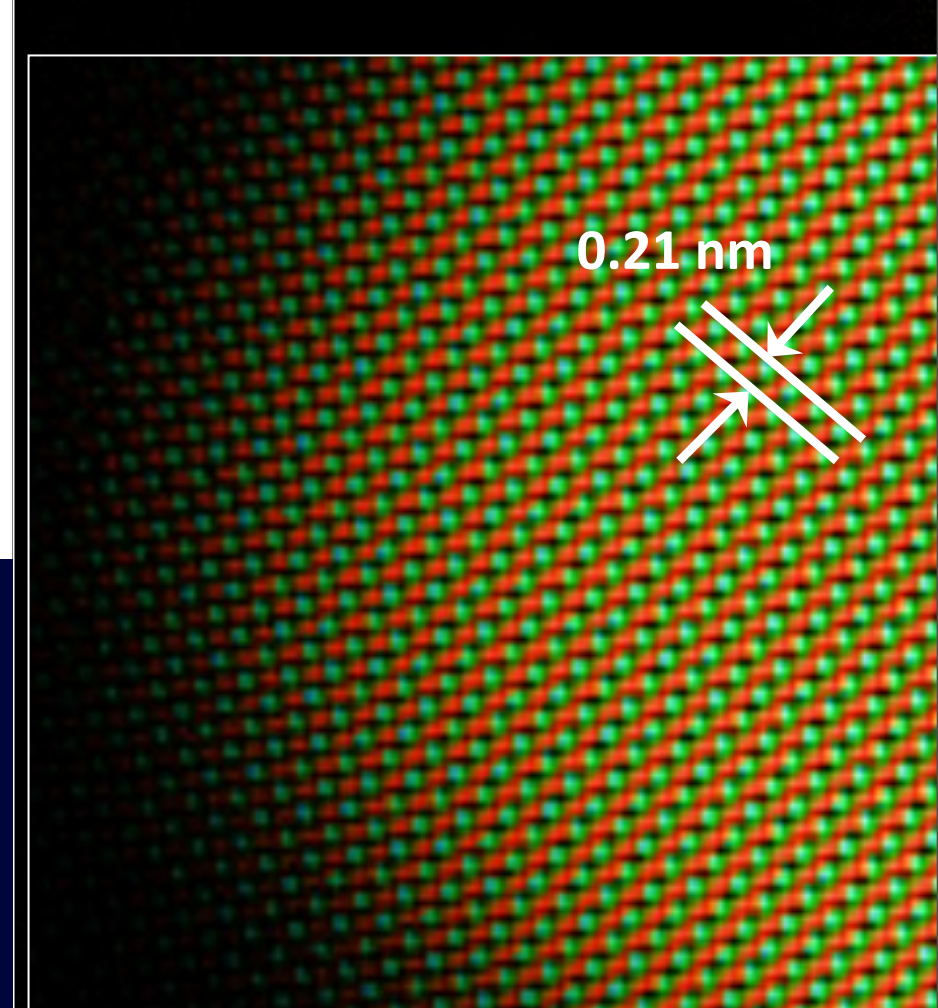


Electron Diffractive Tomography



reconstructed
wave

reconstructed
phase



1.2 limitation of TEM

- A 2D projection: Averaged from the thickn sample
-----> tomography
- B Radiation Damage — — polymer, bio-sample and ceramics °
- C “thin” sample is very difficult to be made
 $t < 50\text{nm} \sim 100\text{nm}$
- D only dry sample can be observed
- E . NO time resolved capability

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The Future of TEM: 3D atomic resolution
in wet environment with time resolved

Why 3D ?



Why 3D ?



Why 3D ?



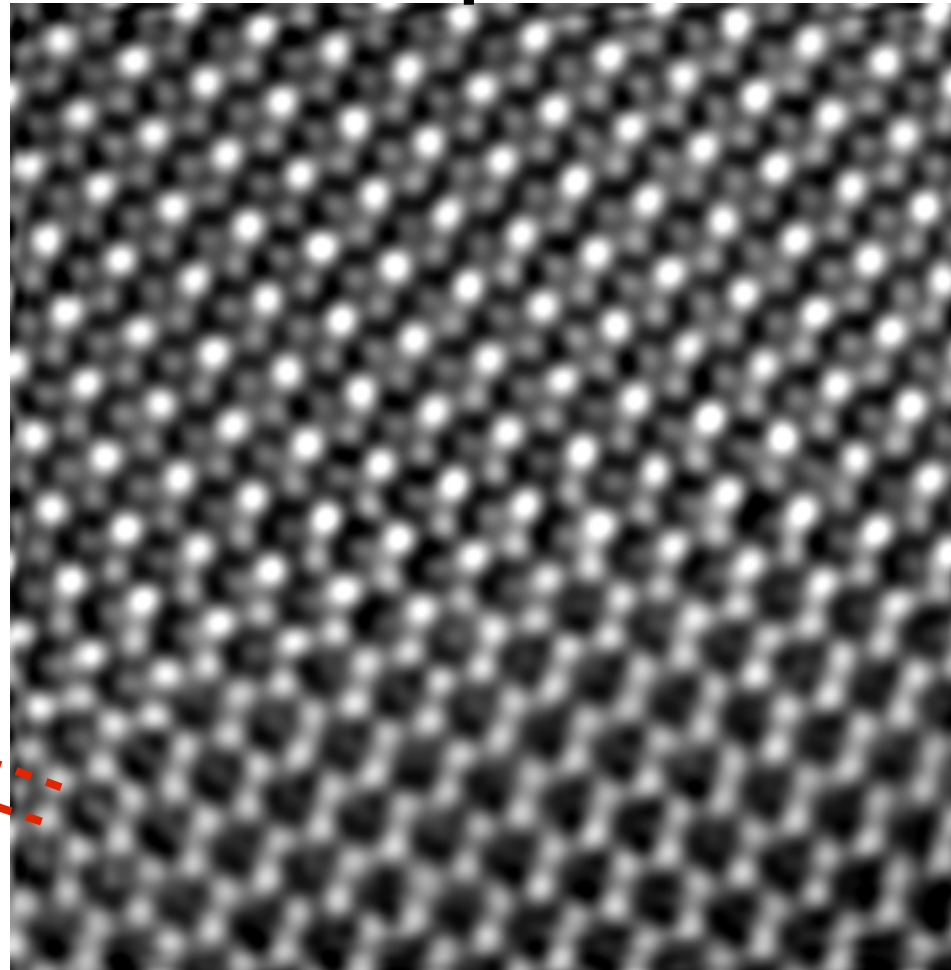
Why 3D ?



Atomic Resolution Tomography

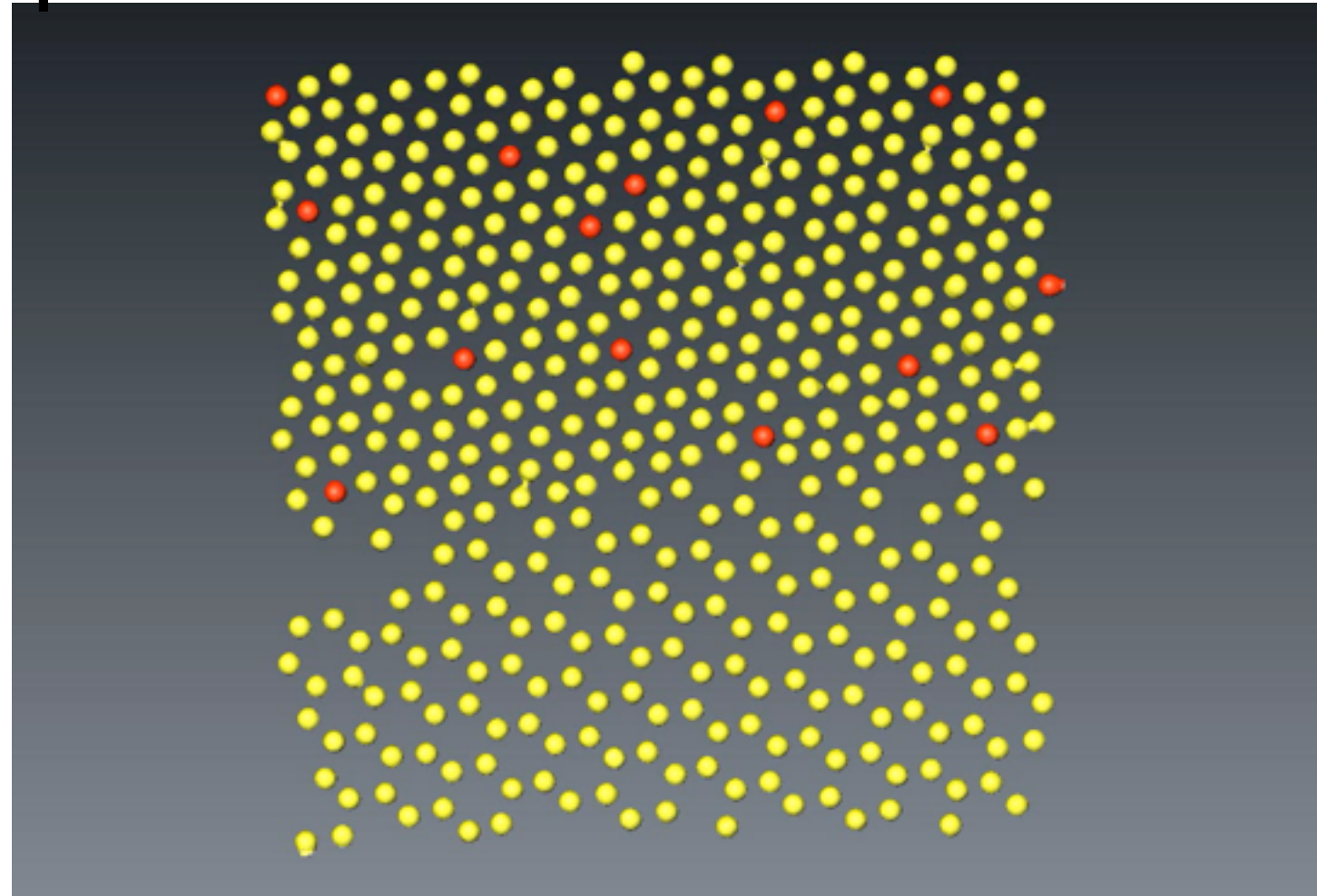
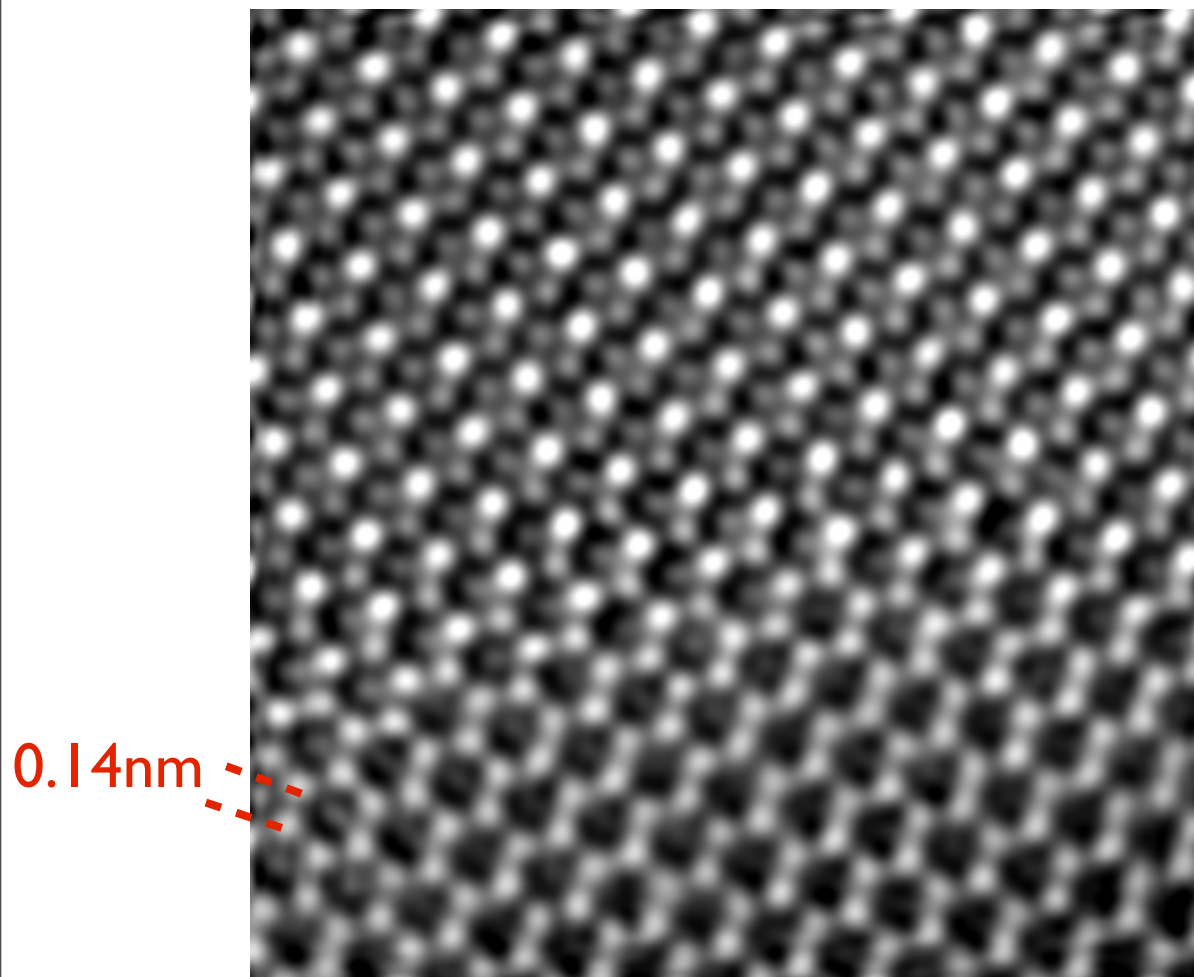
Graphene

0.14nm



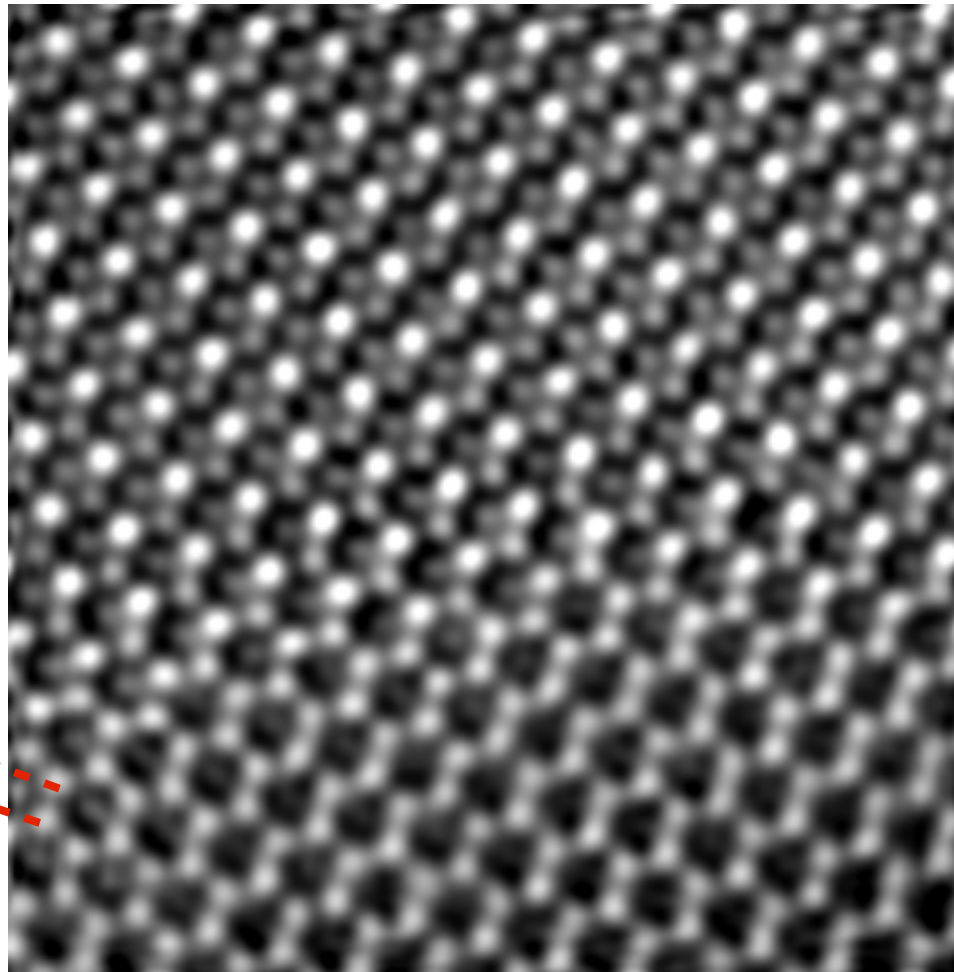
Atomic Resolution Tomography

Graphene

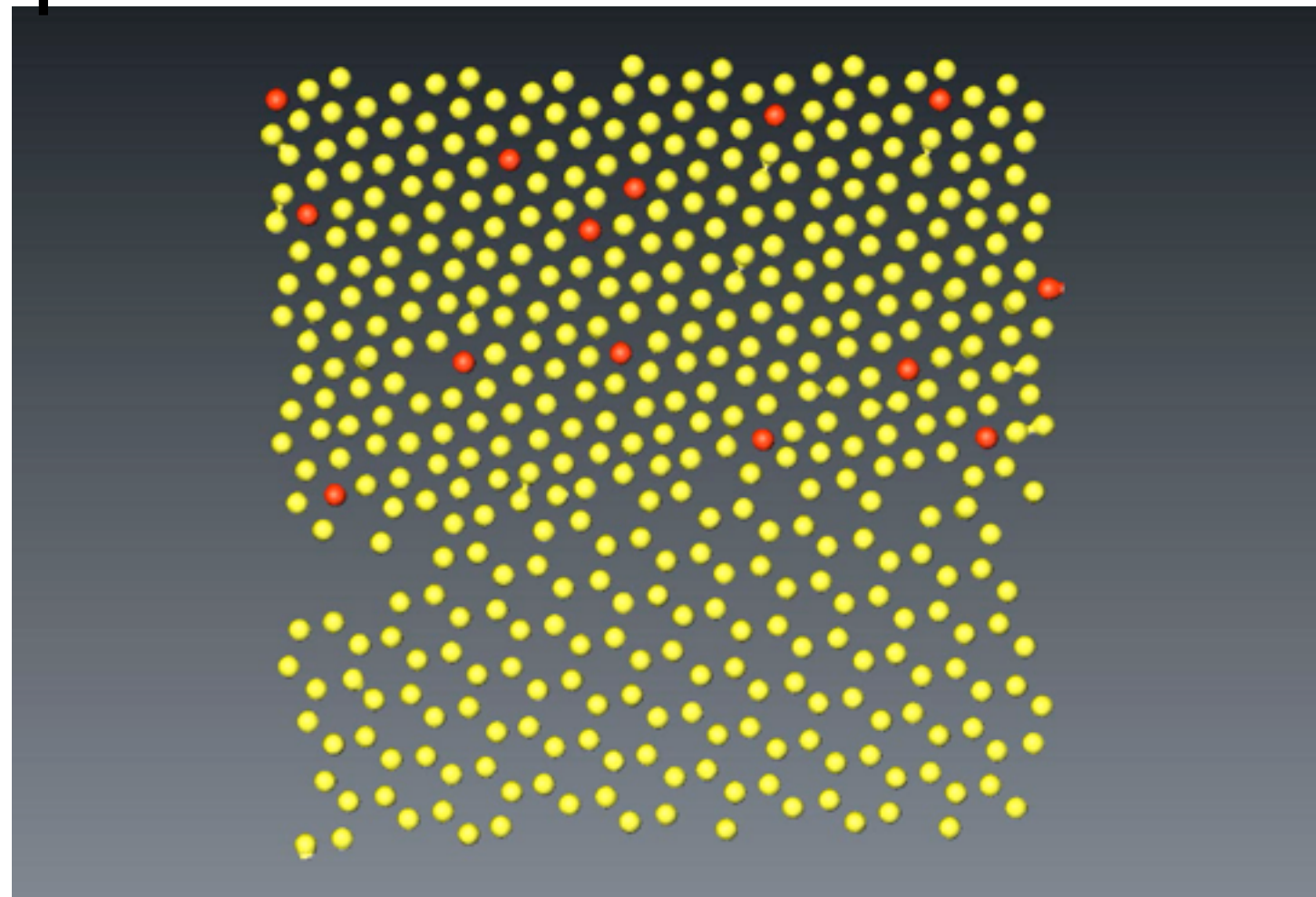


Atomic Resolution Tomography

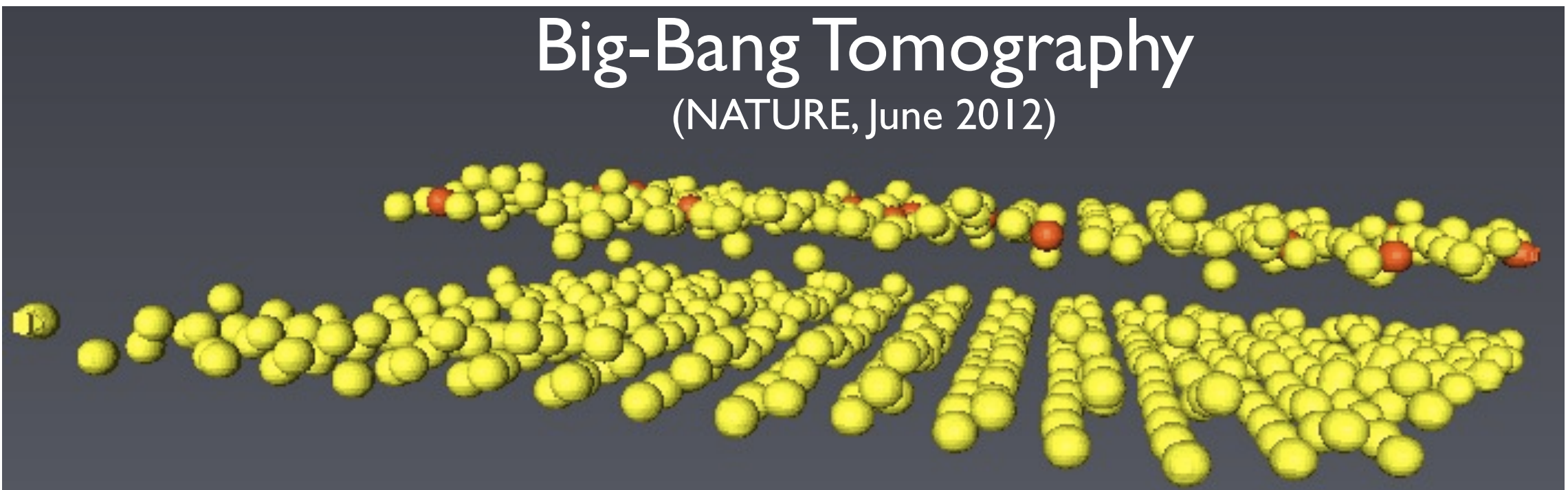
Graphene



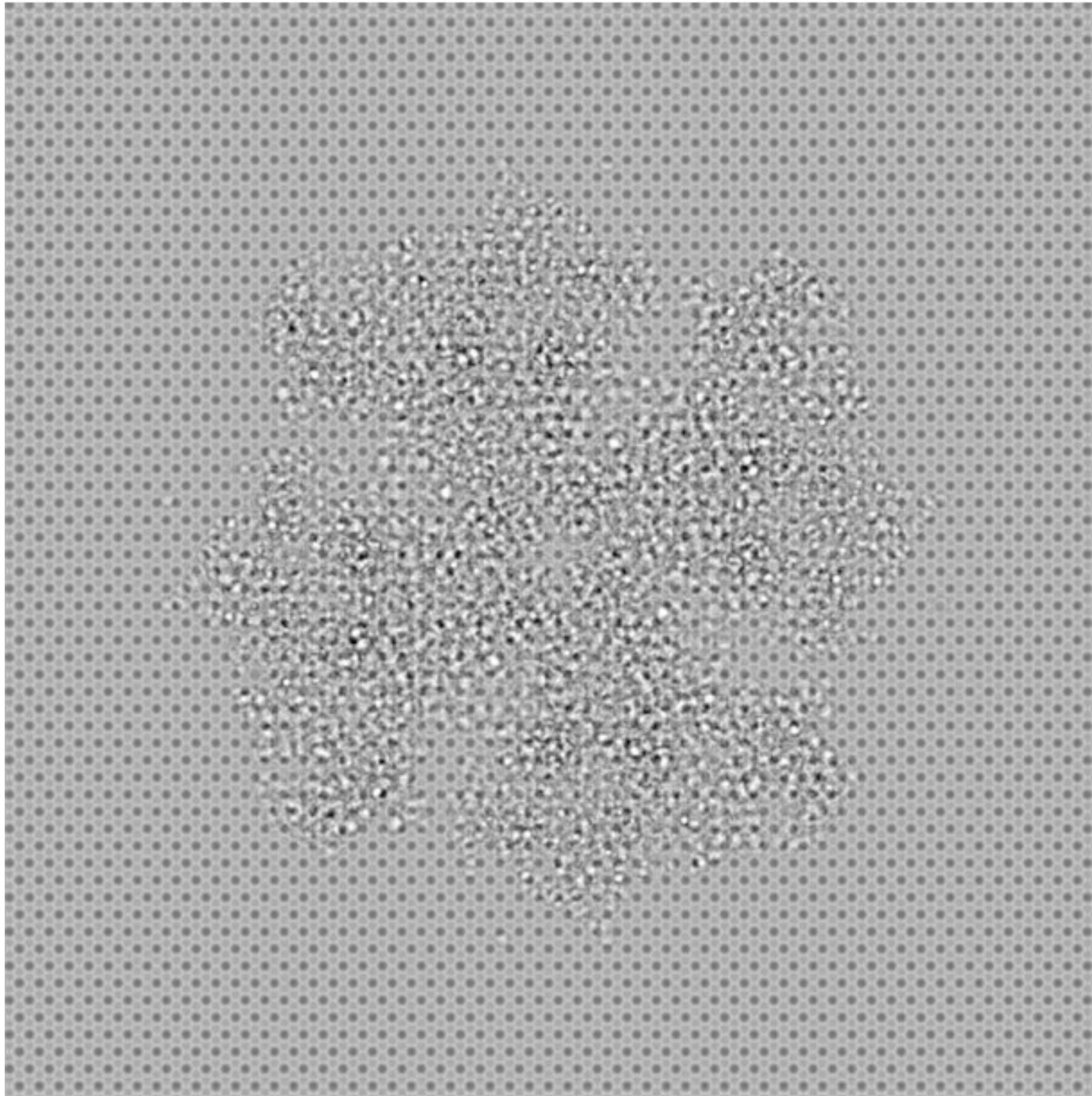
0.14nm



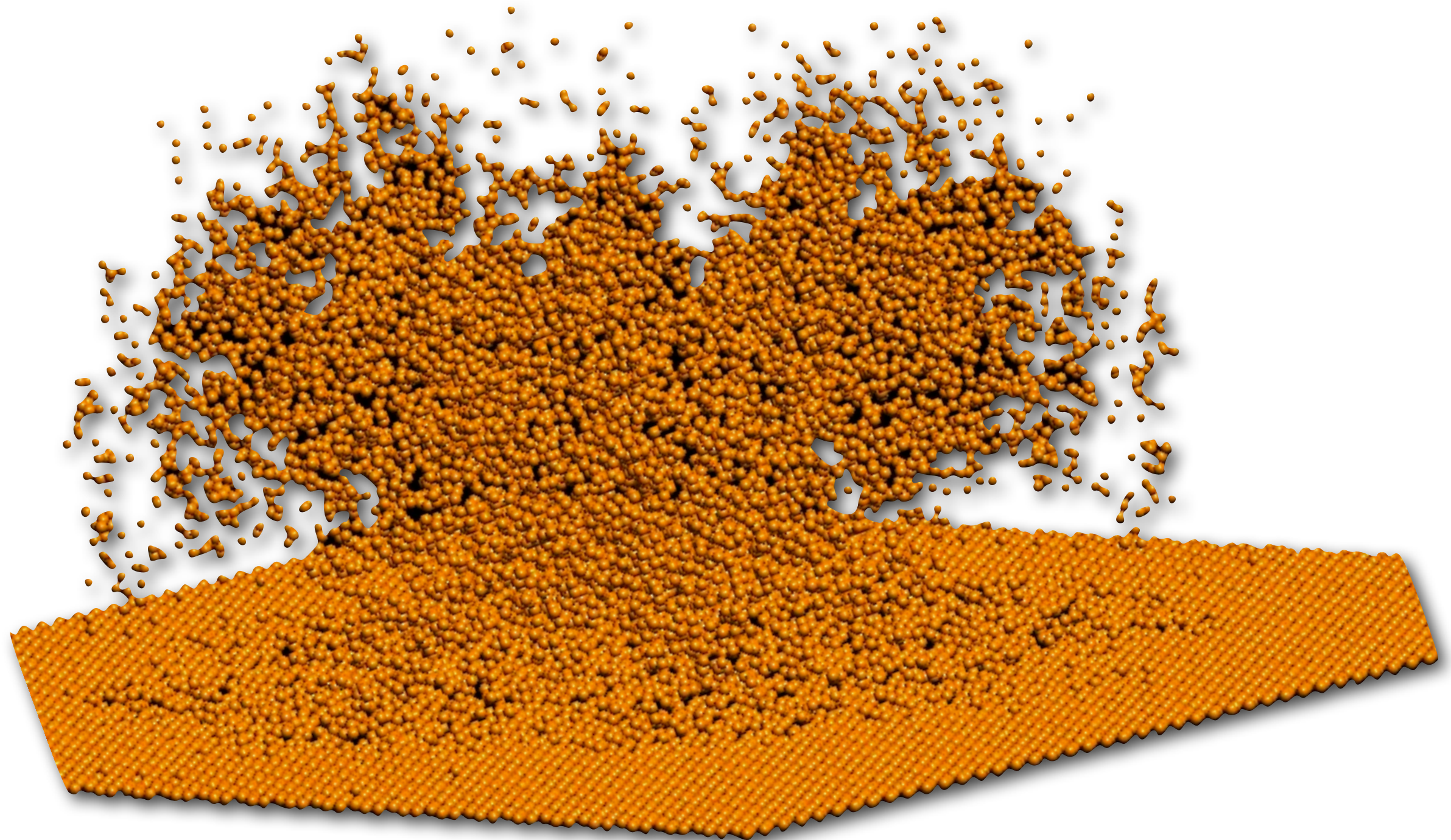
Big-Bang Tomography (NATURE, June 2012)



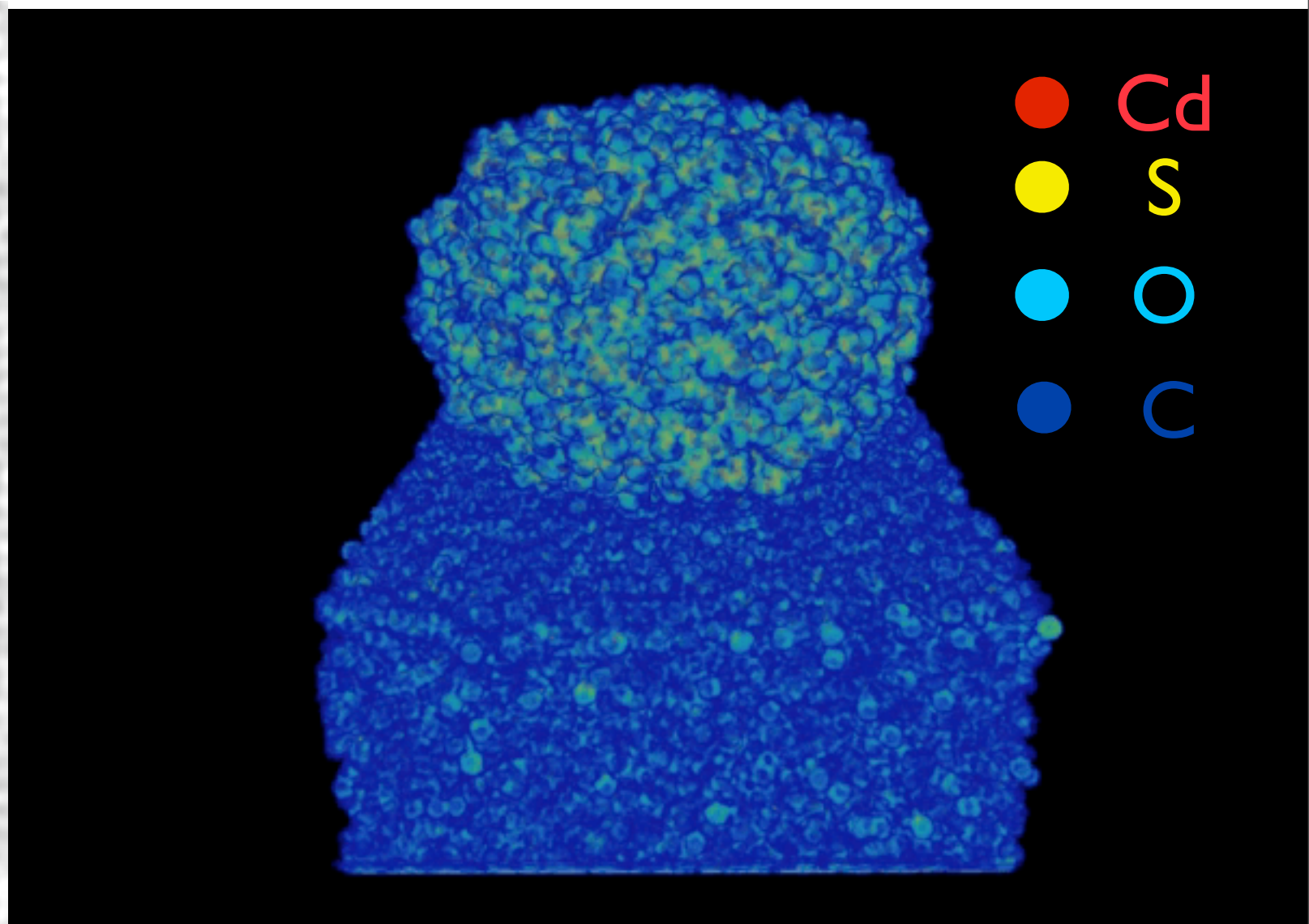
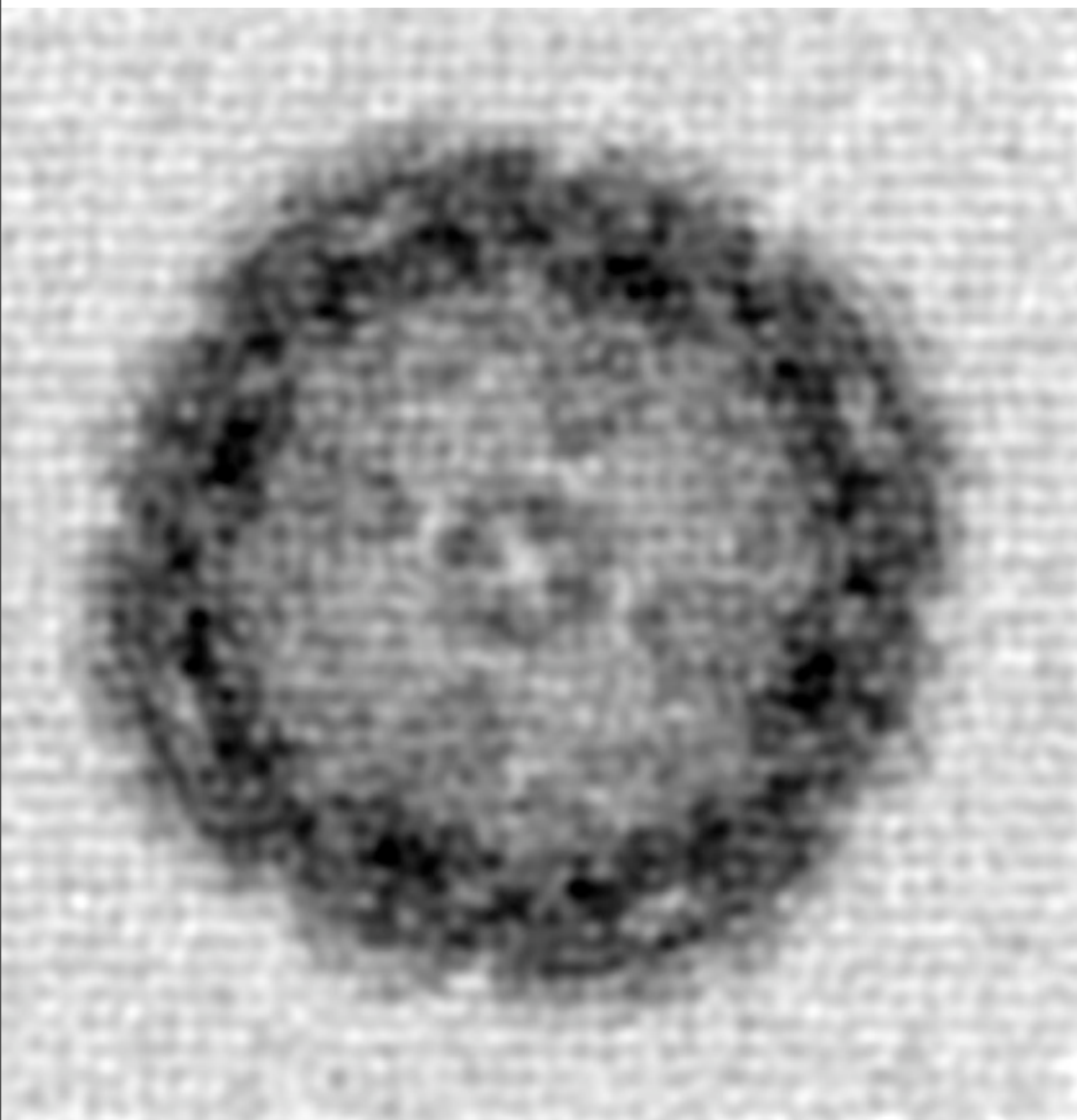
AIHJP Protein on Graphene



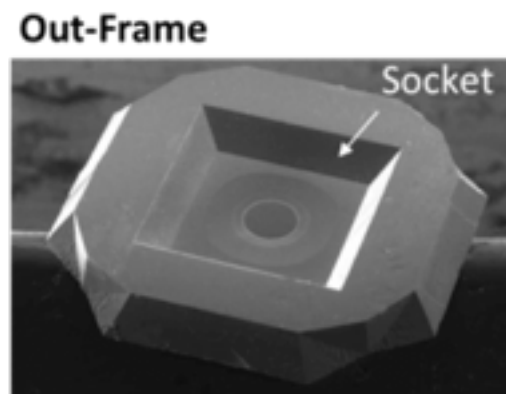
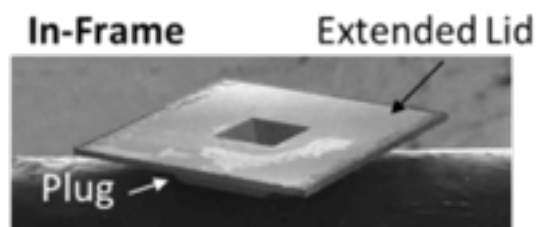
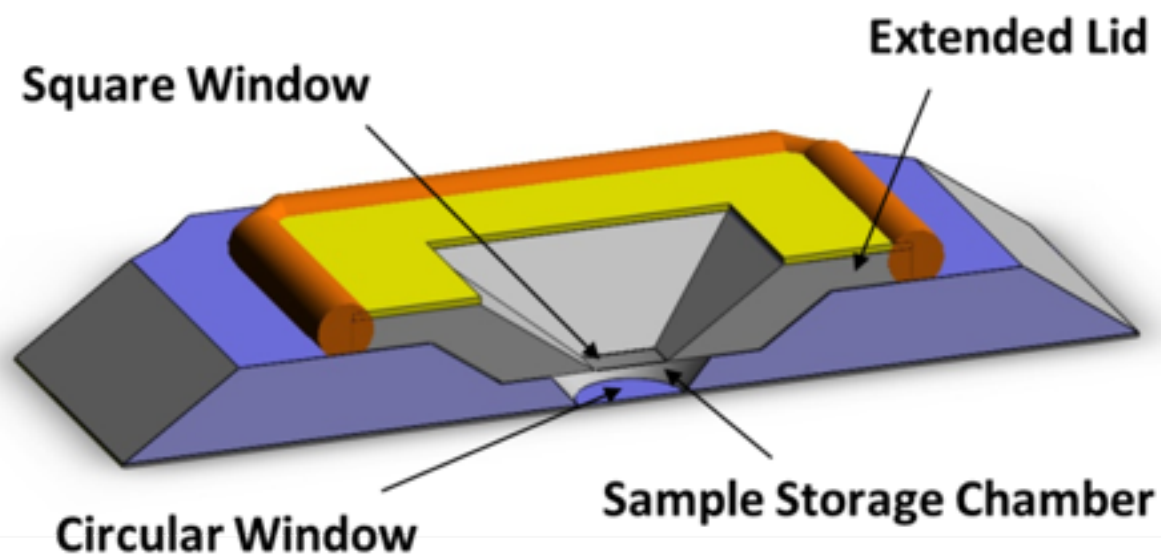
AIHJP Protein on Graphene



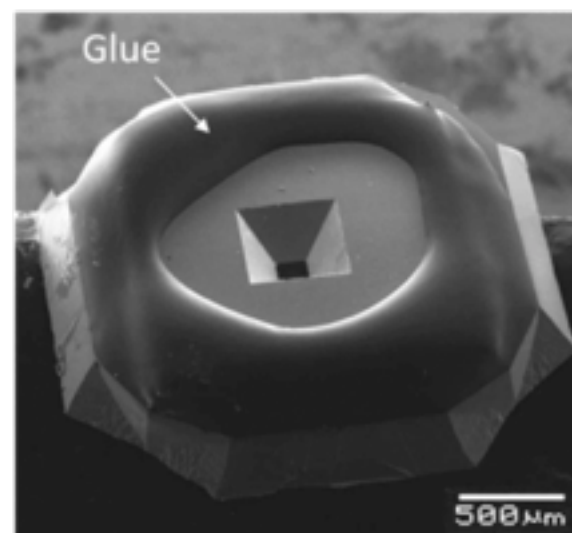
Ferritin on Carbon Film



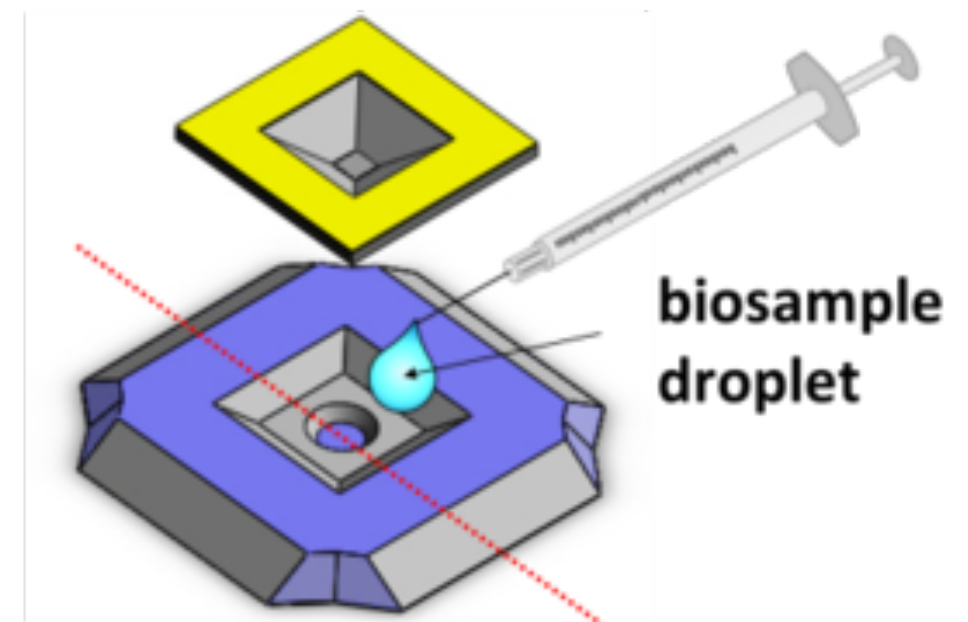
Self Aligned Wet (SAW) Cell



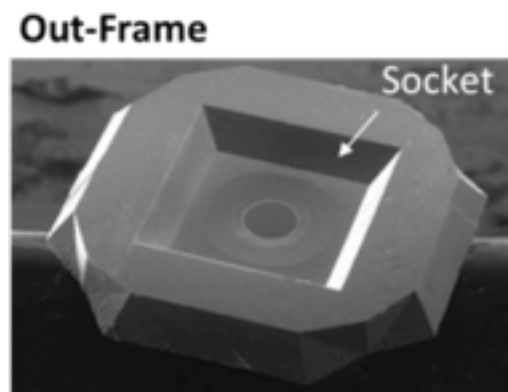
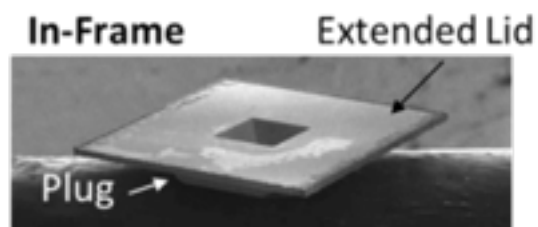
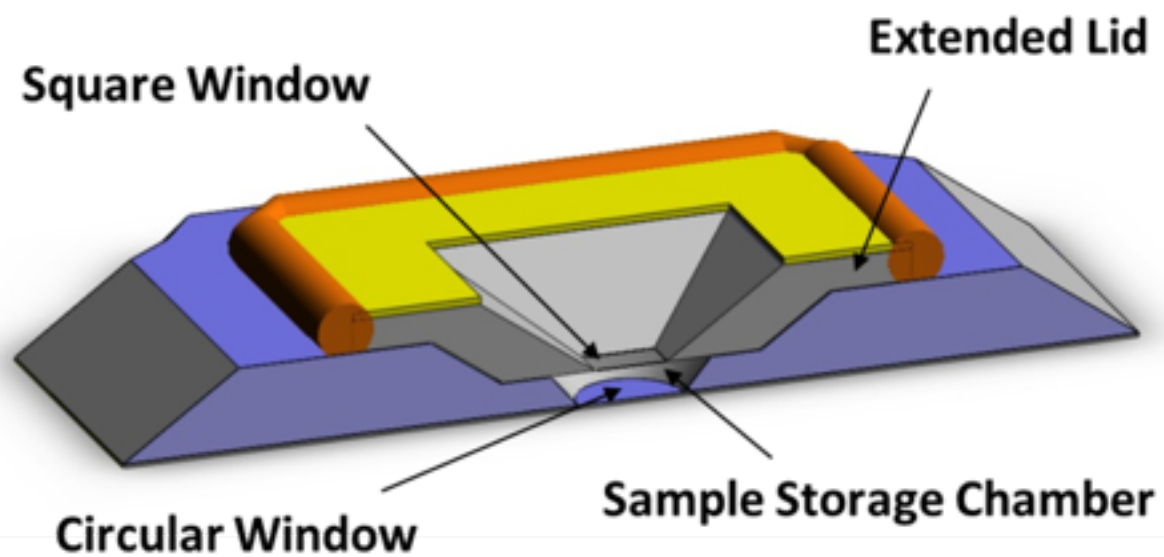
Sealed SAW Cell



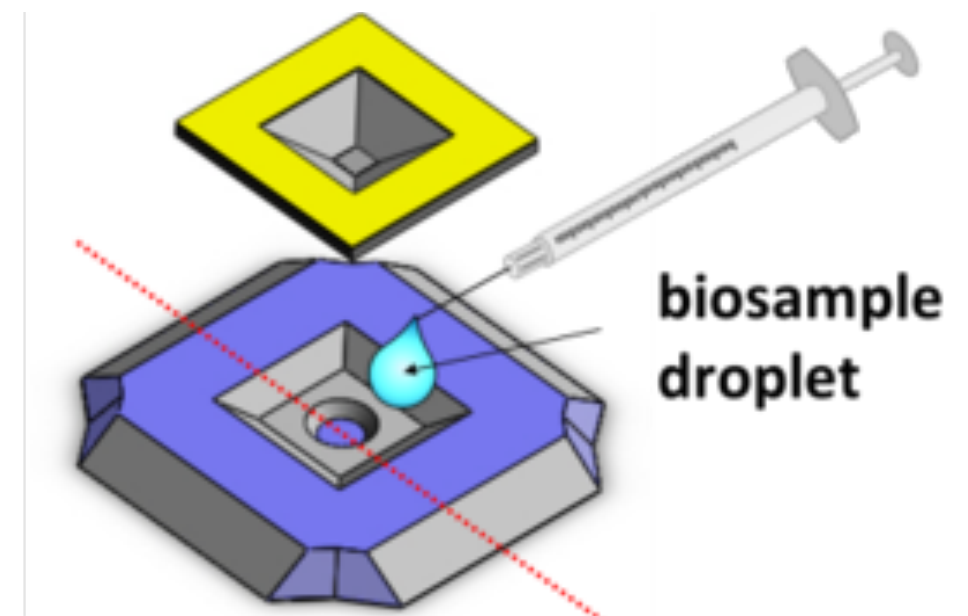
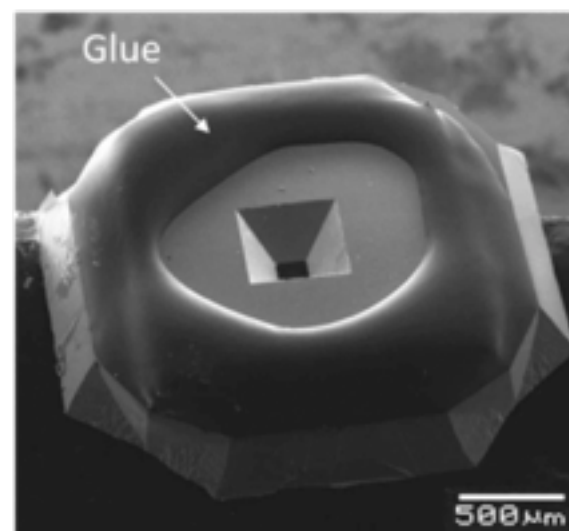
Self-Aligned Wet-Cell



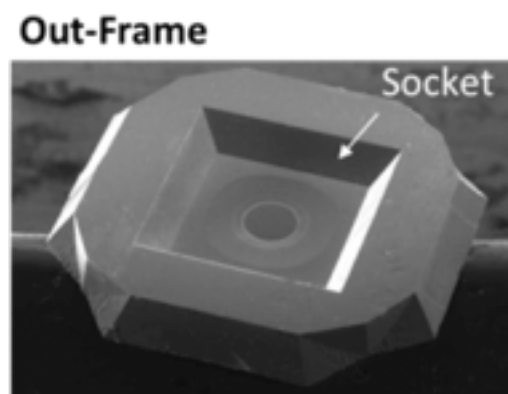
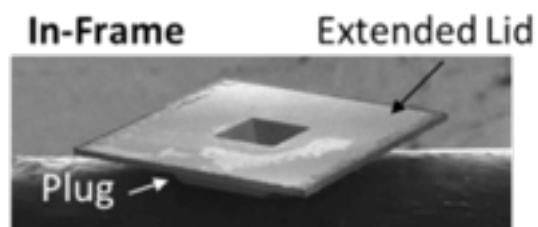
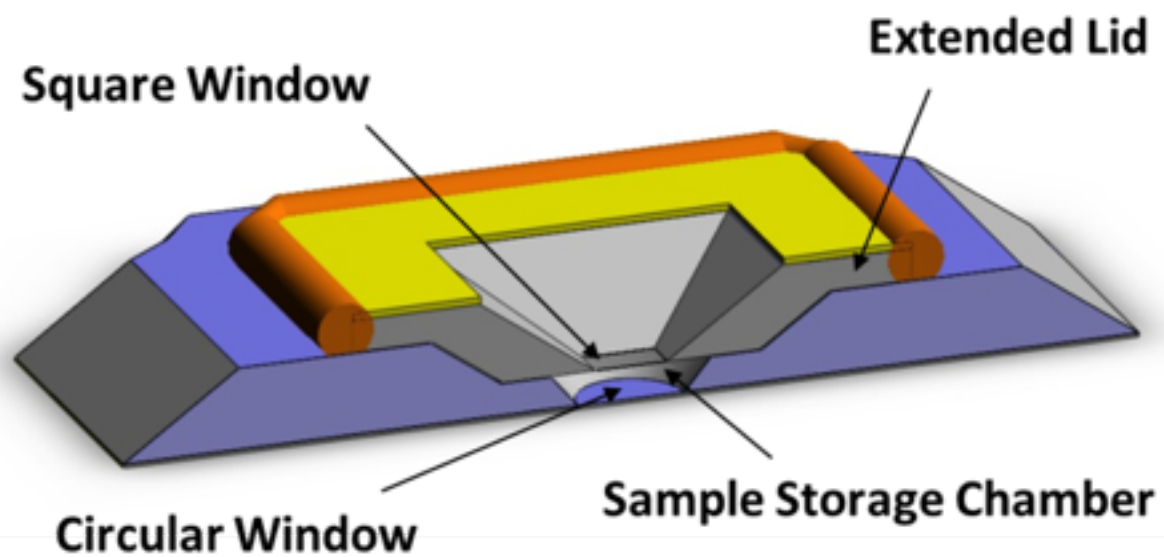
Self Aligned Wet (SAW) Cell



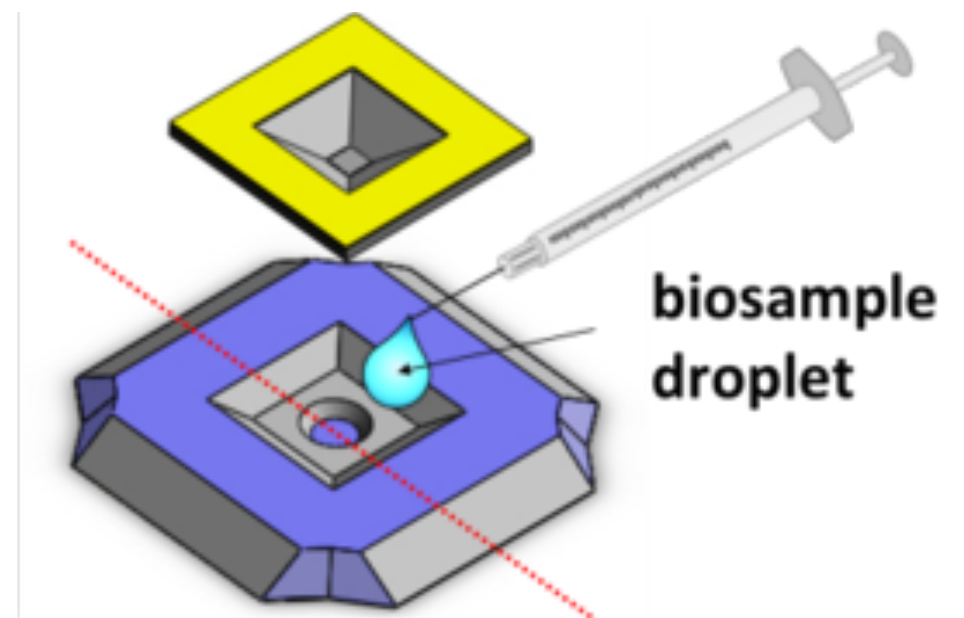
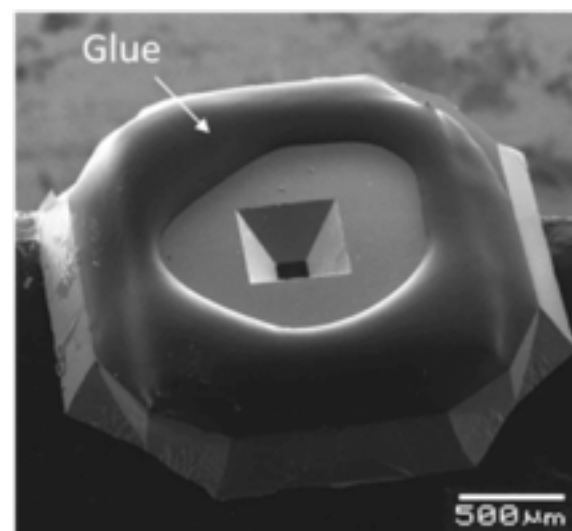
Sealed SAW Cell



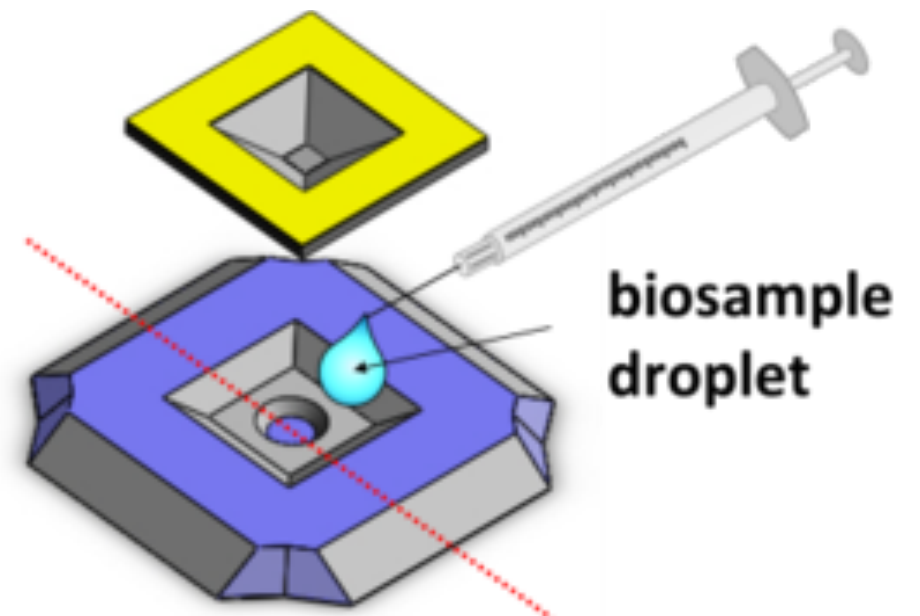
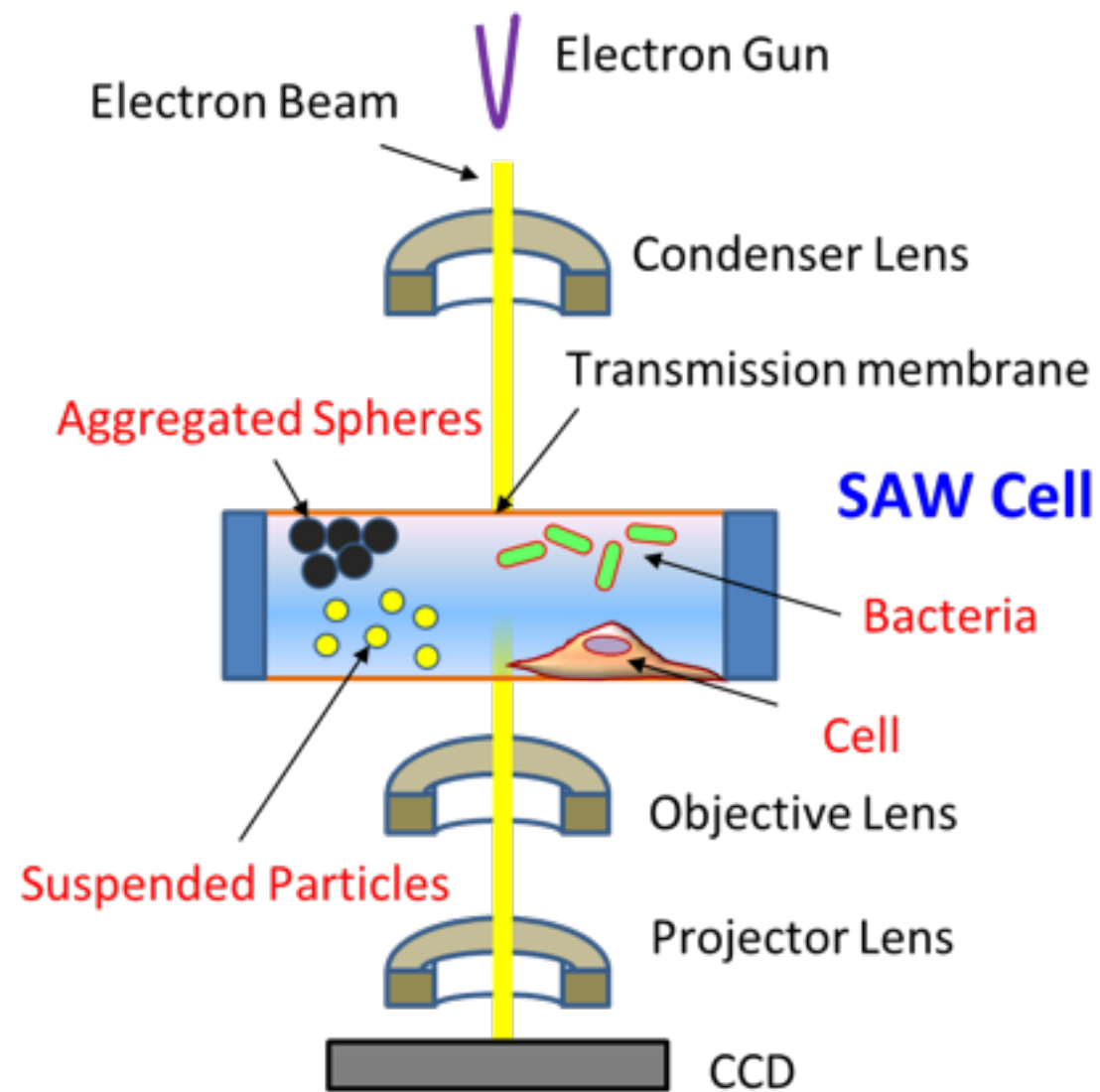
Self Aligned Wet (SAW) Cell



Sealed SAW Cell

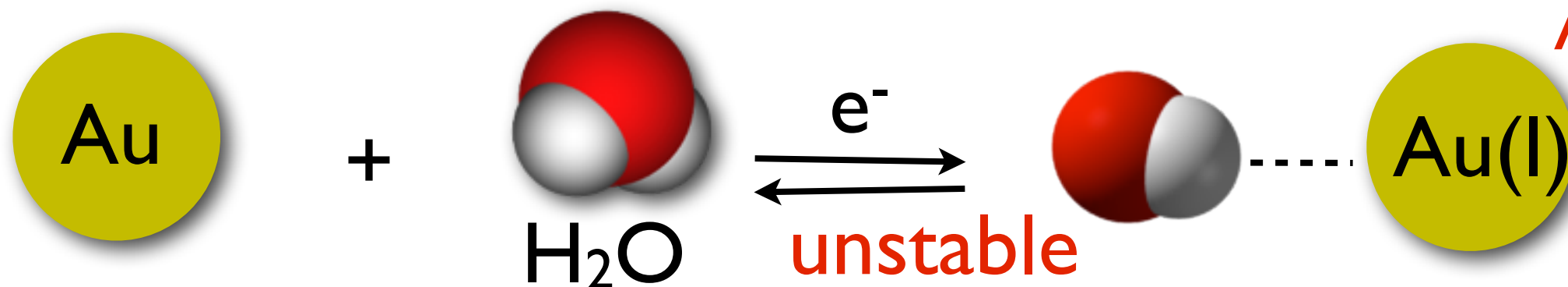
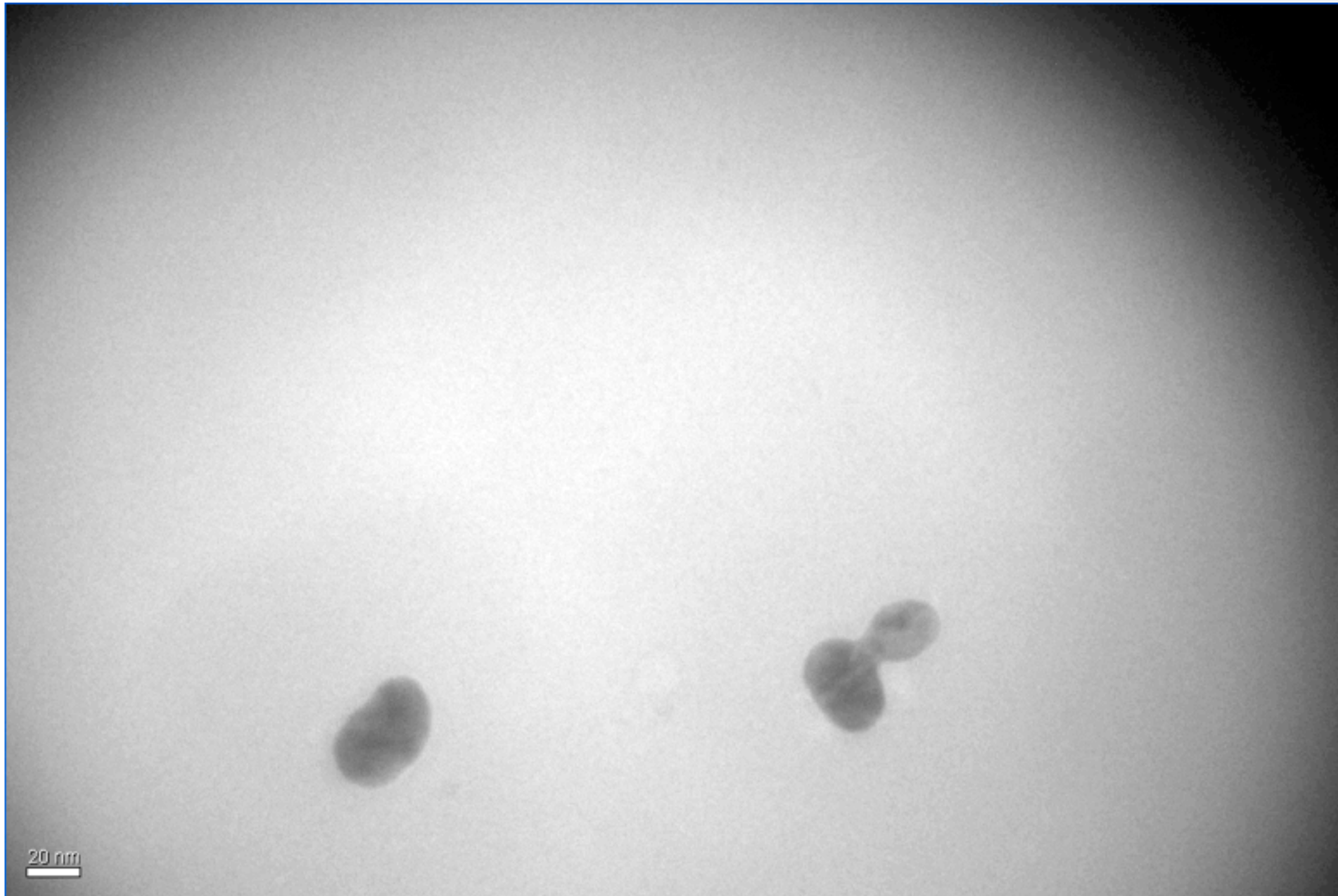


Self Aligned Wet (SAW) Cell



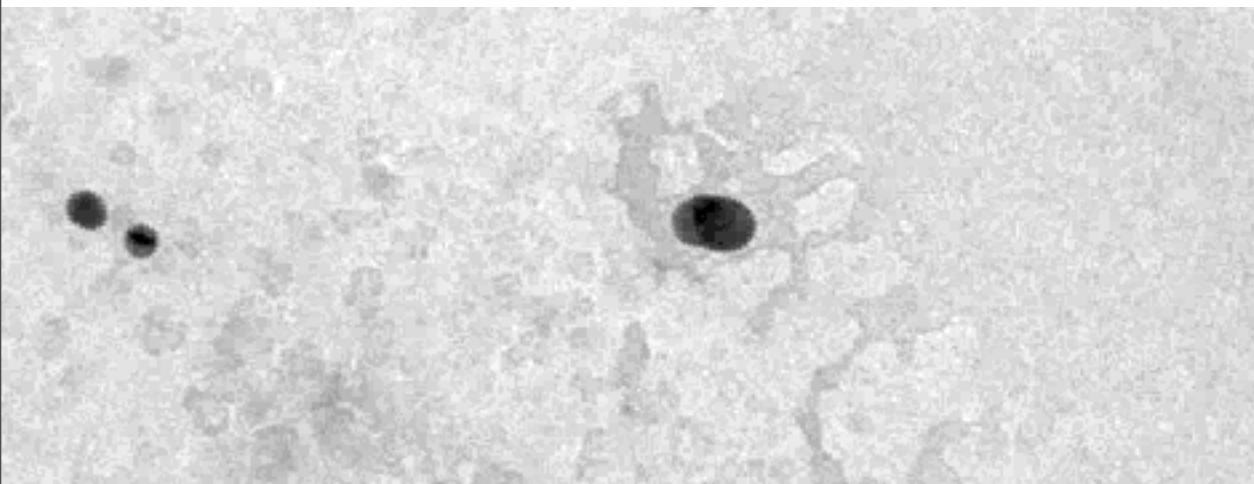
"Self-Aligned Wet-Cell for Hydrated Microbiology Observation in TEM," *Lab on a Chip*, 12(2), pp.340-347, 2012.

dissolution of Au nano-particles

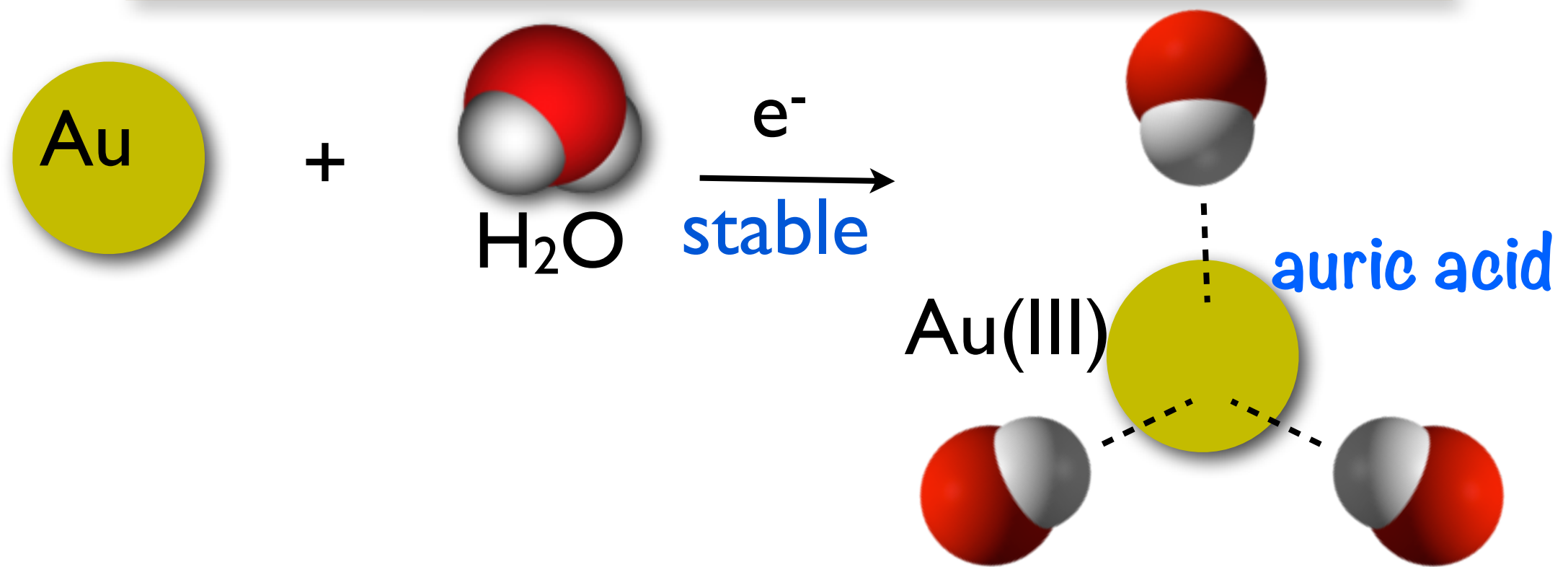


Aurous acid

Dynamics of NW Growth from GNPs



Dynamics of NW Growth from GNPs



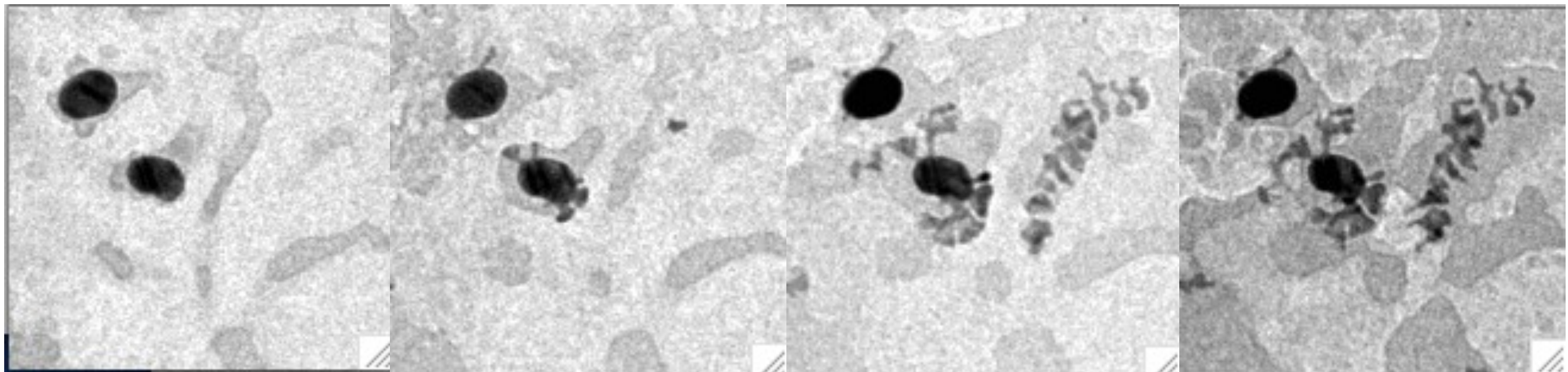
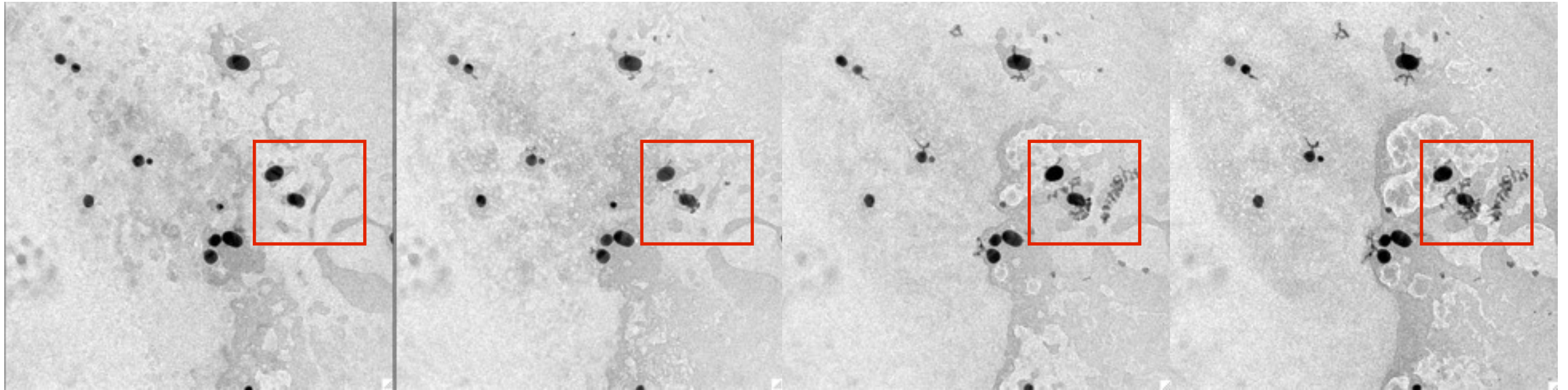
Dynamics of NW Growth from GNPs

15sec

30 sec

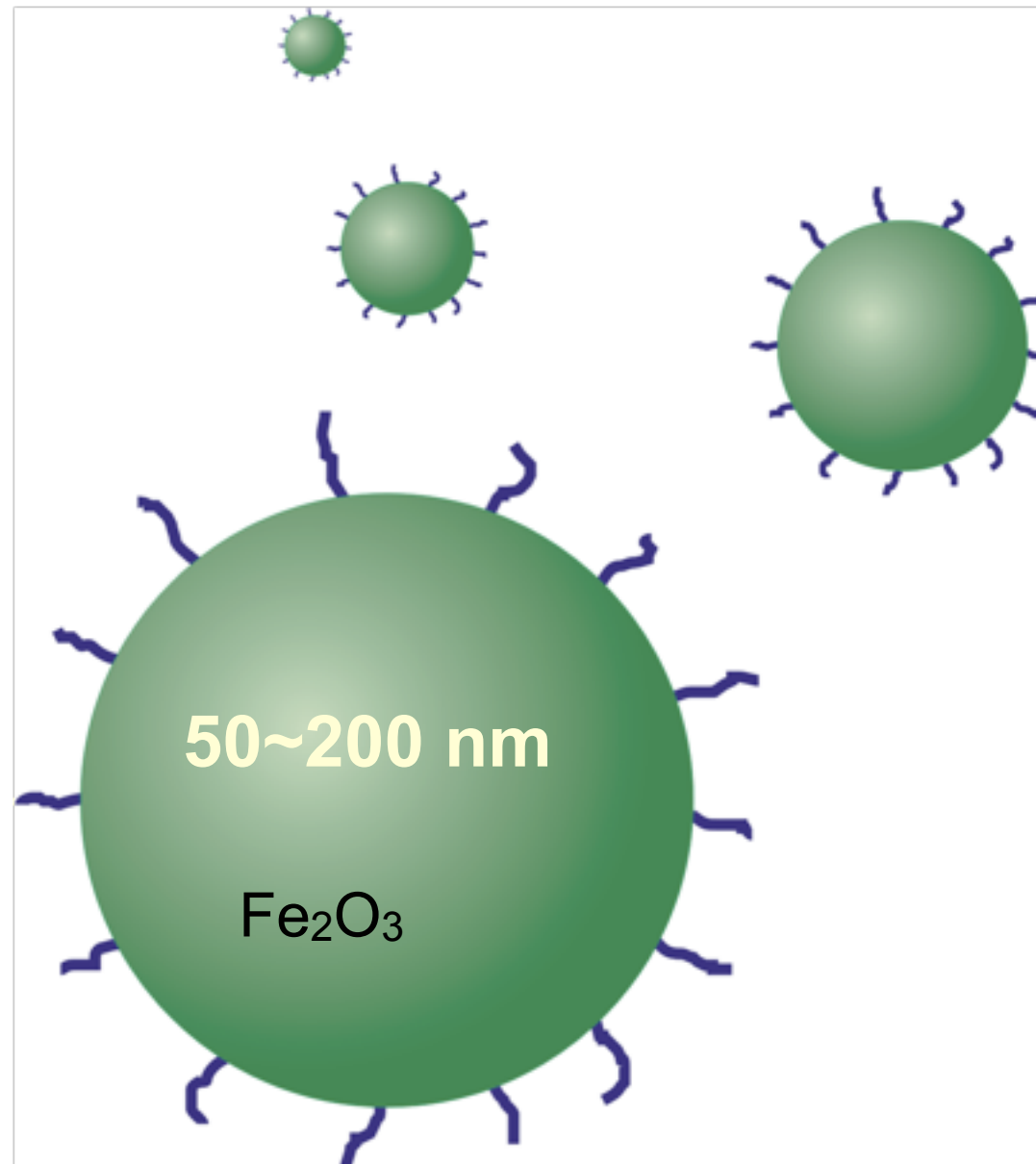
45 sec

60 sec



Magic Bullet: Targeting Drug

~ proposed by Nobel Prize winner (1908) Paul Ehrlich



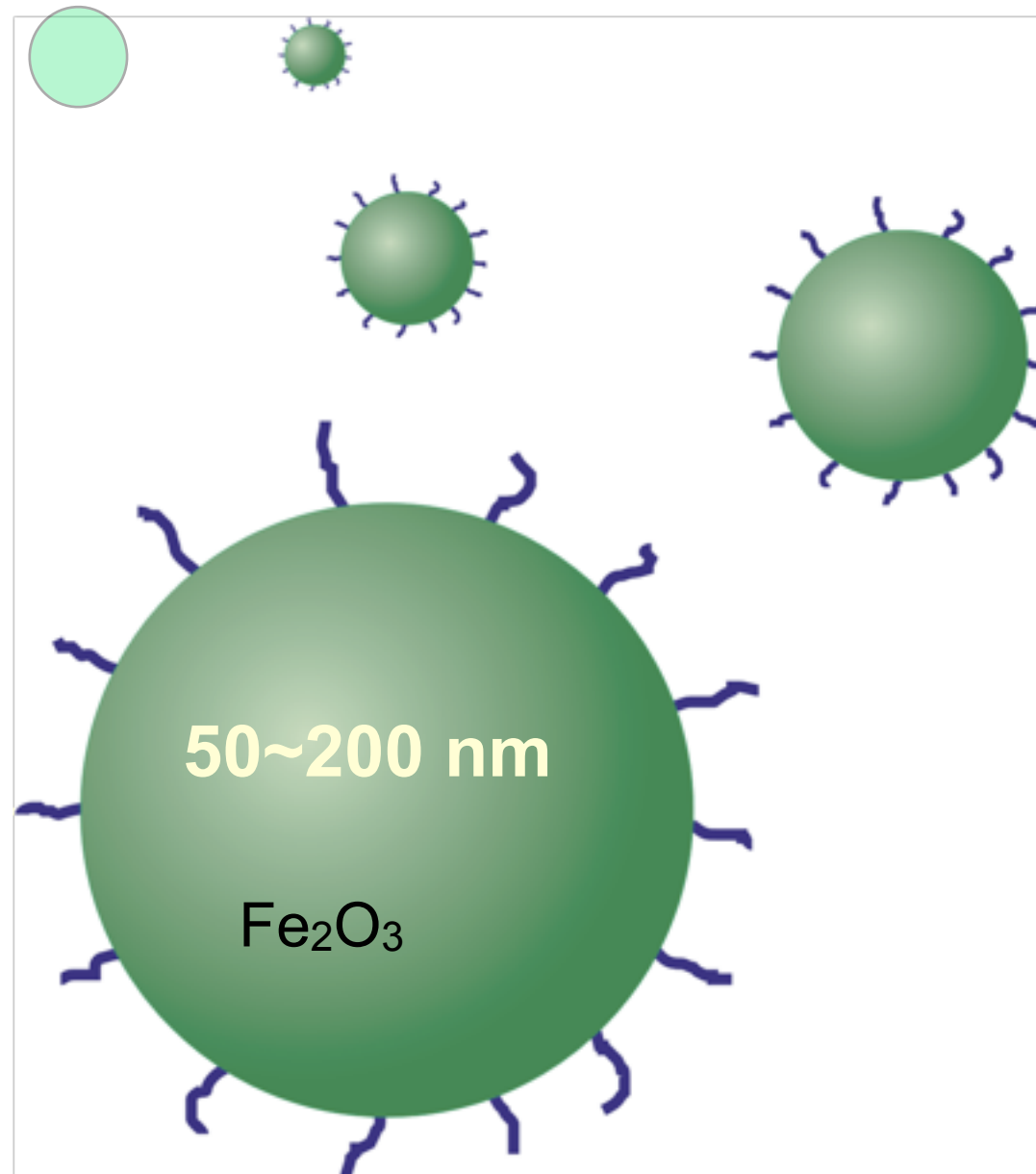
Easy Storage
High Drug Content
Stimuli Responsive
Large Surface Area
Modification

Superparamagnetic iron oxide (SPIO)

Magic Bullet: Targeting Drug

~ proposed by Nobel Prize winner (1908) Paul Ehrlich

Chitosan



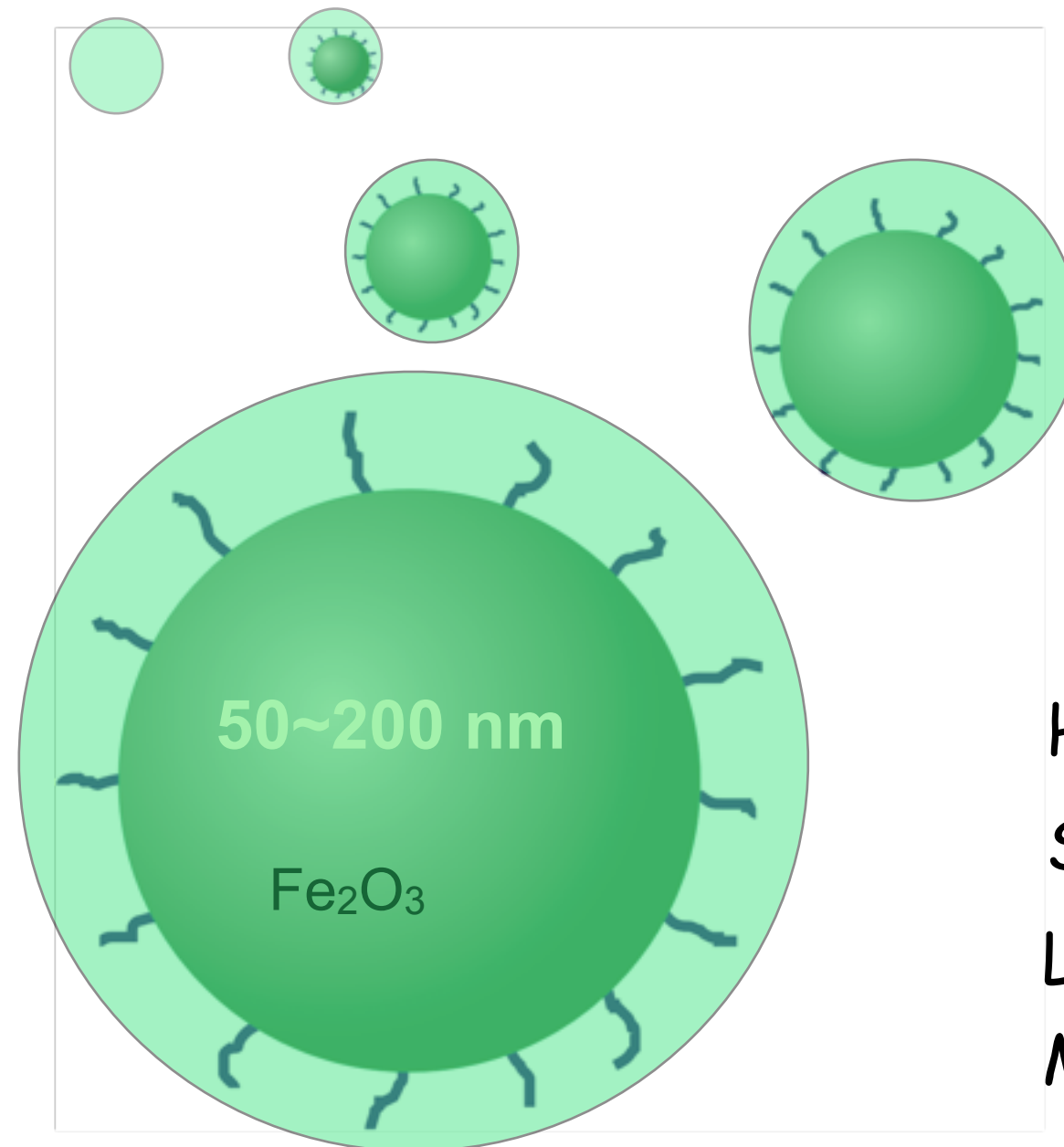
Easy Storage
High Drug Content
Stimuli Responsive
Large Surface Area
Modification

Superparamagnetic iron oxide (SPIO)

Magic Bullet: Targeting Drug

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Chitosan



Easy Storage
High Drug Content
Stimuli Responsive
Large Surface Area
Modification

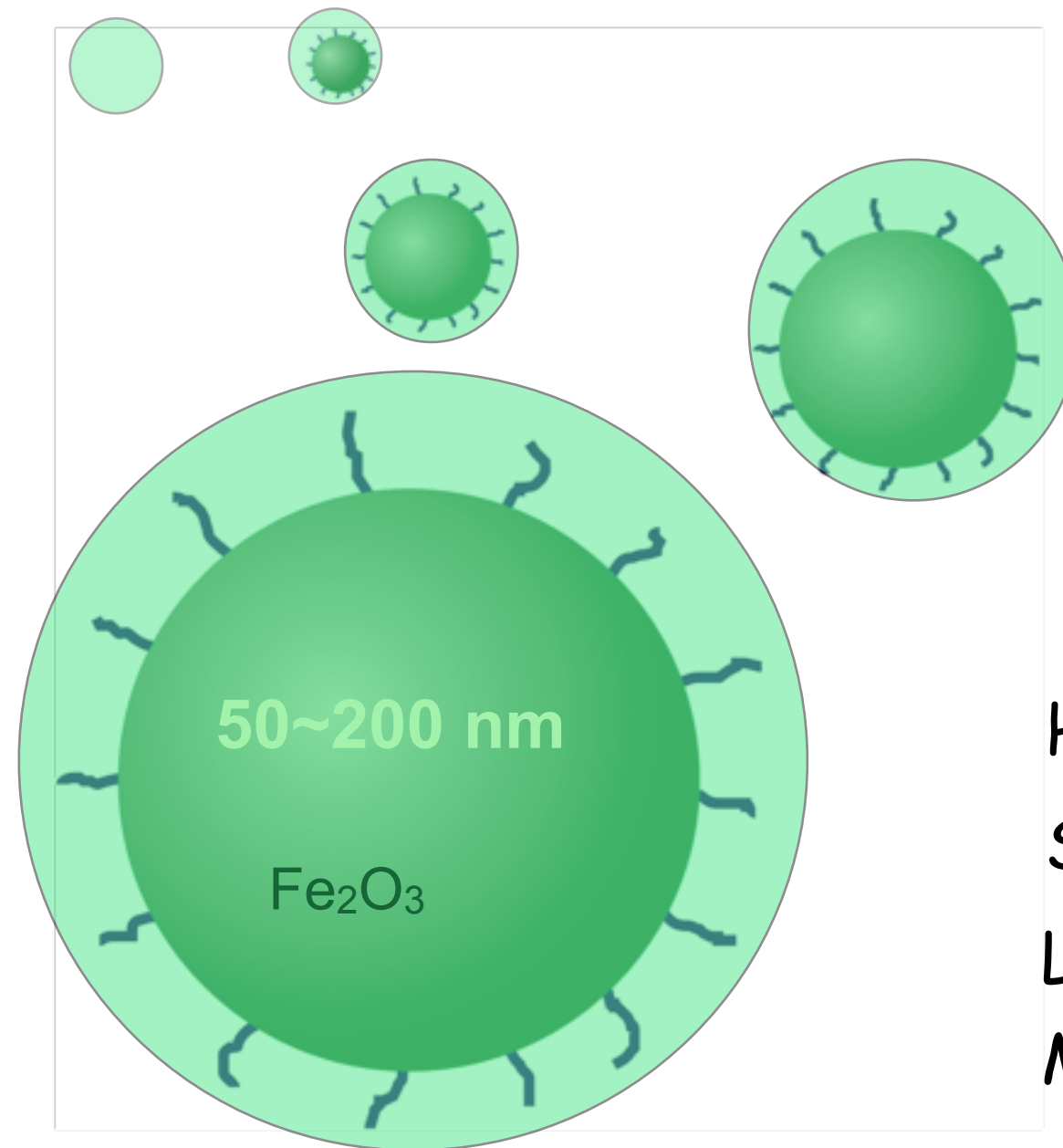
Superparamagnetic iron oxide (SPIO)

Magic Bullet: Targeting Drug

~ proposed by Nobel Prize winner (1908) Paul Ehrlich

Chitosan-SPIO: Targeting drug for MRI contrast agent

Chitosan



Easy Storage
High Drug Content
Stimuli Responsive
Large Surface Area
Modification

Superparamagnetic iron oxide (SPIO)

Mechanisms for core-(single crystal shells) nanospheres under HFMF

Single crsytal

Poly-crsytal

Rupture

Original Name Fe3O4_2A_08
Magnification 0300K

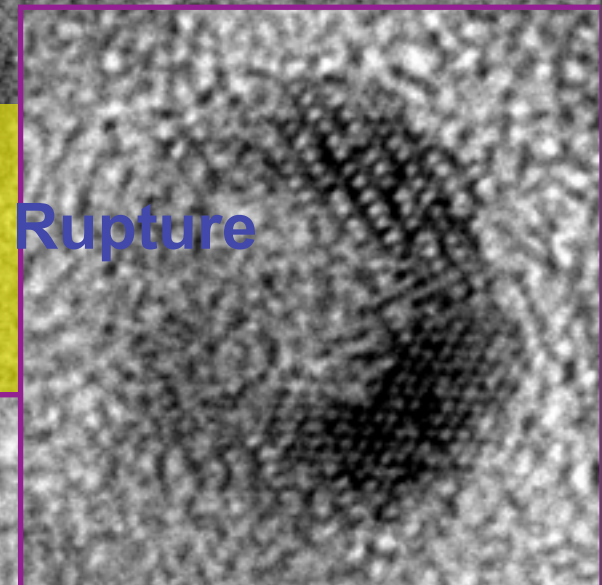
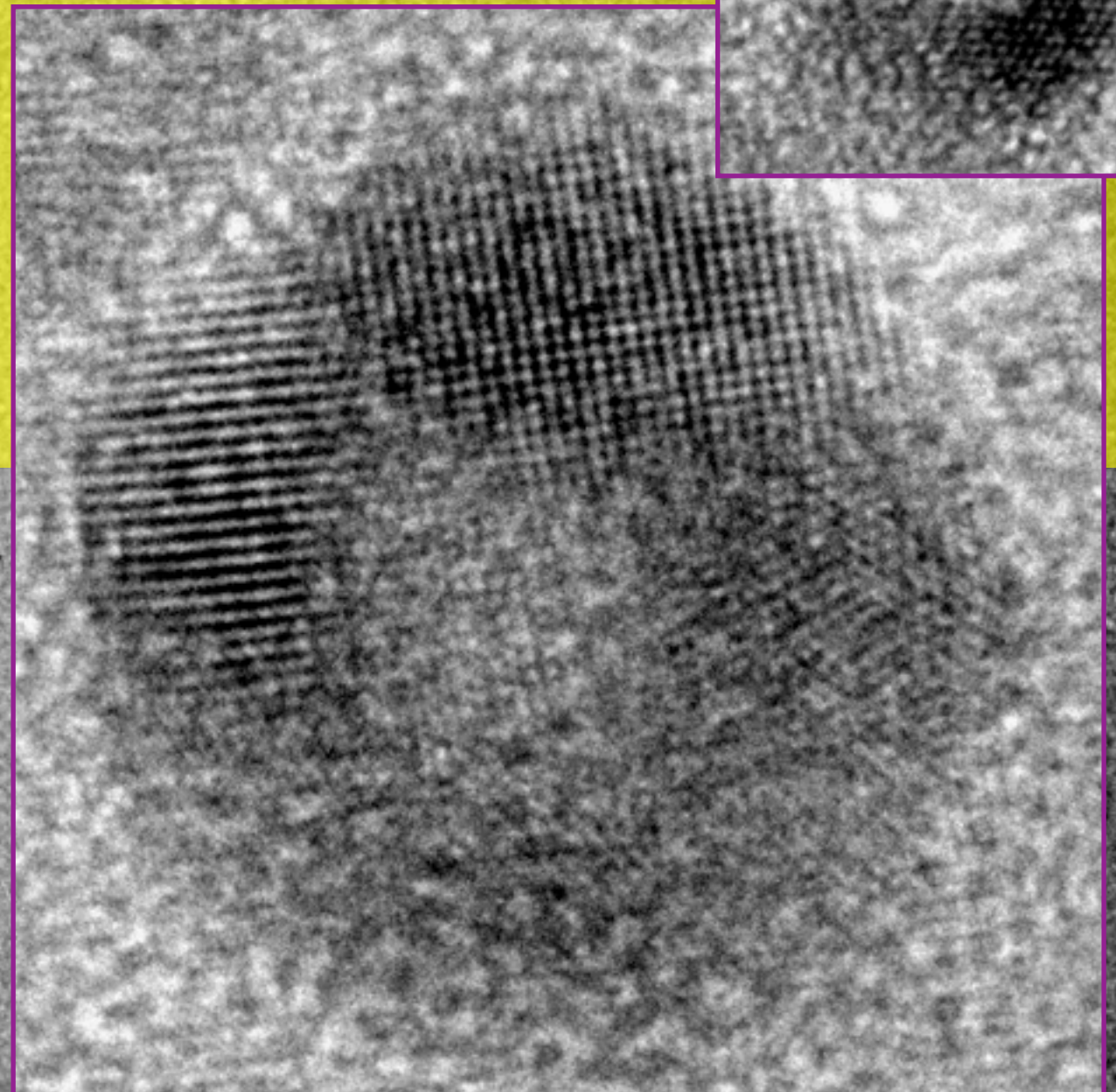
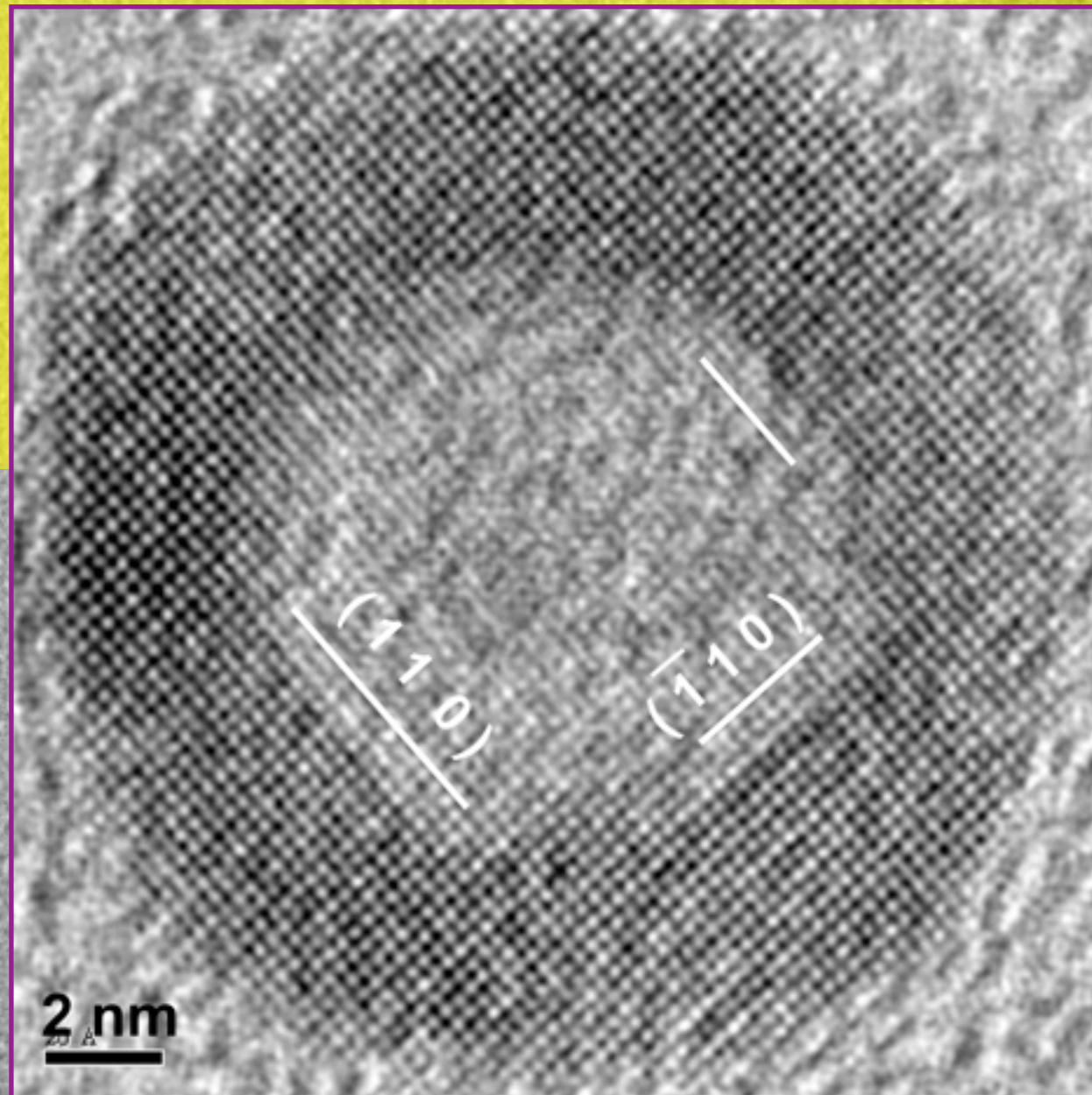
30.0 nm

Mechanisms for core-(single crystal shells) nanospheres under HFMF

Single crsytal

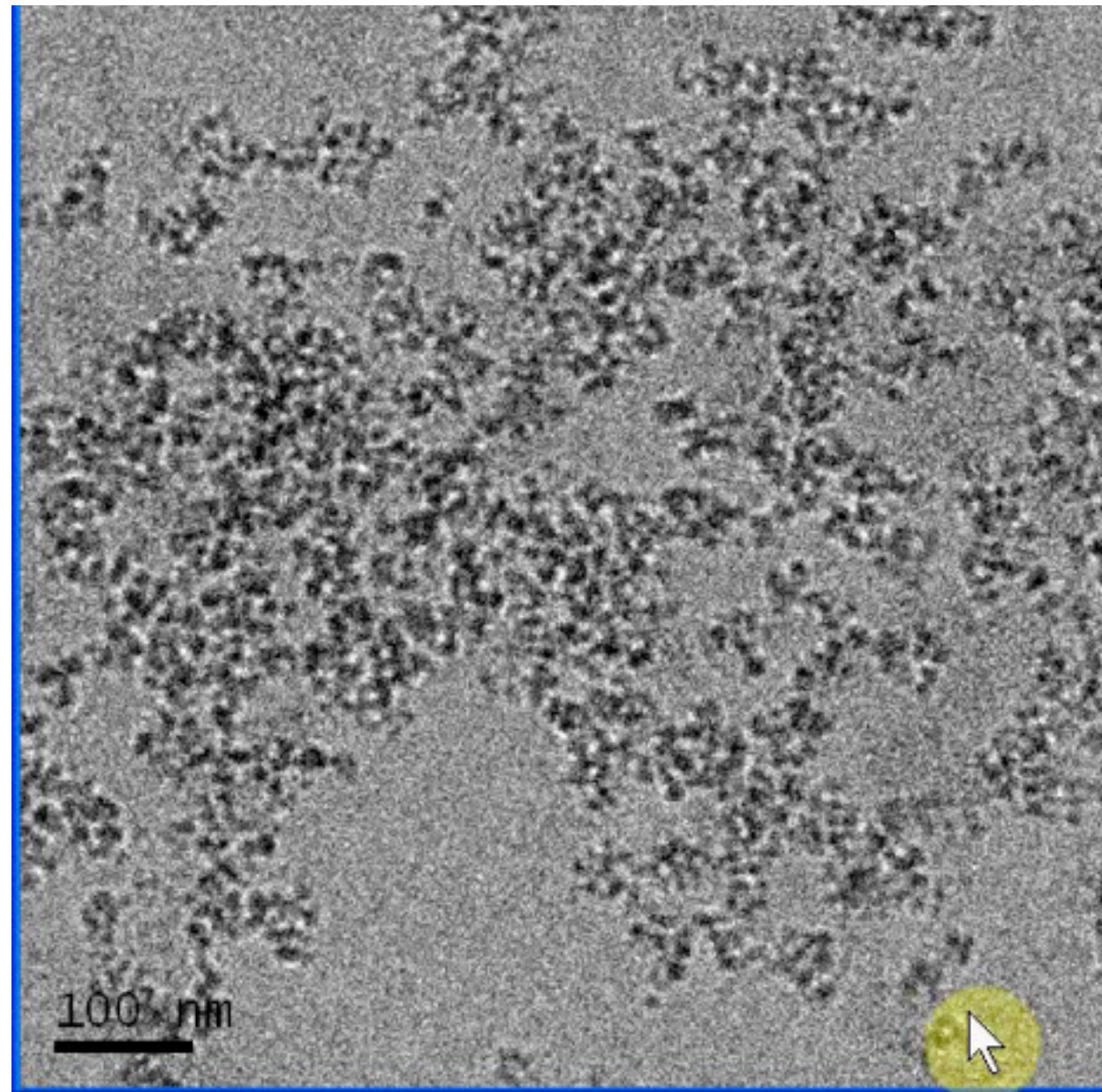
Poly-crsytal

Rupture

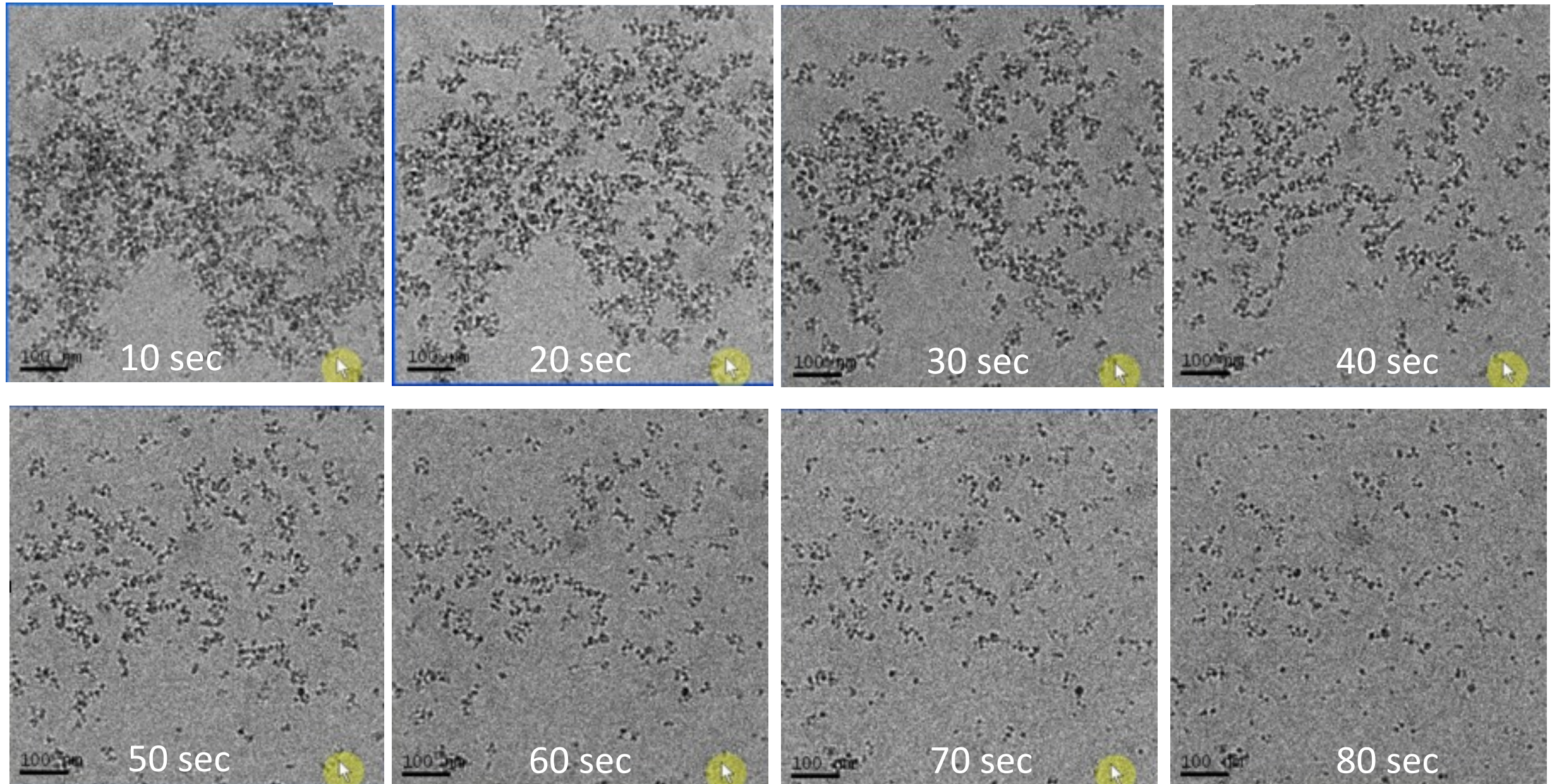


Original Name Fe3O4_2A_08
Magnification 0300K

Reduction of Fe₂O₃ NPs



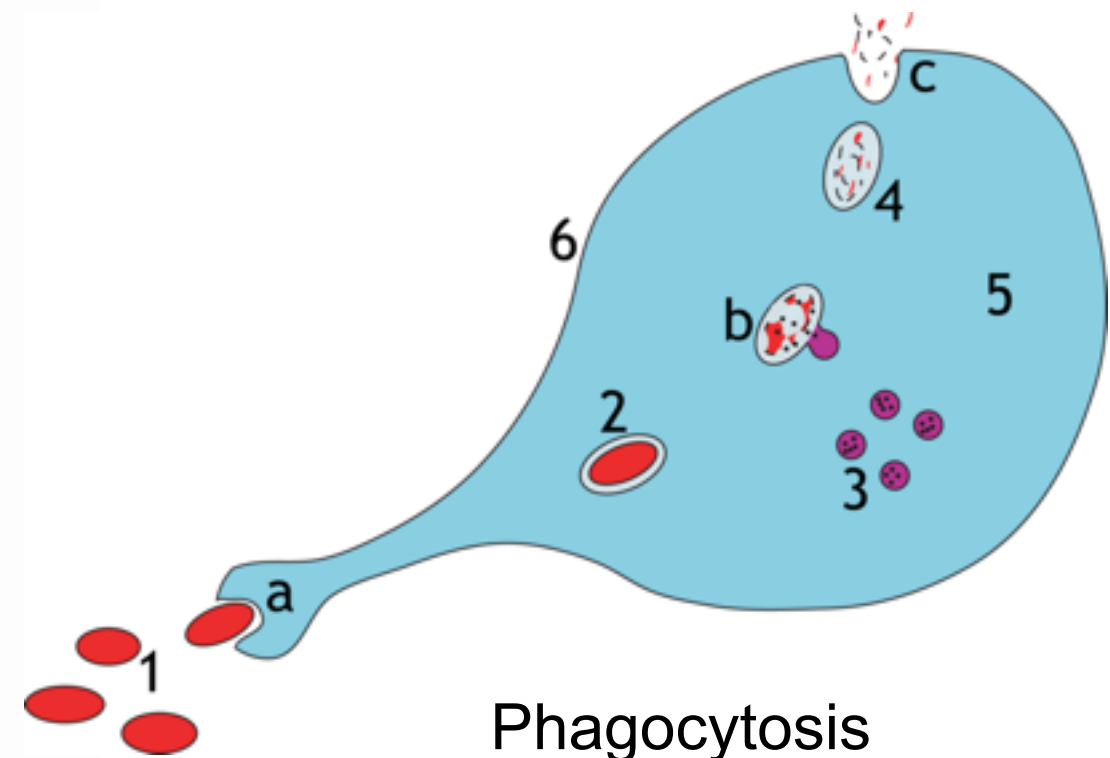
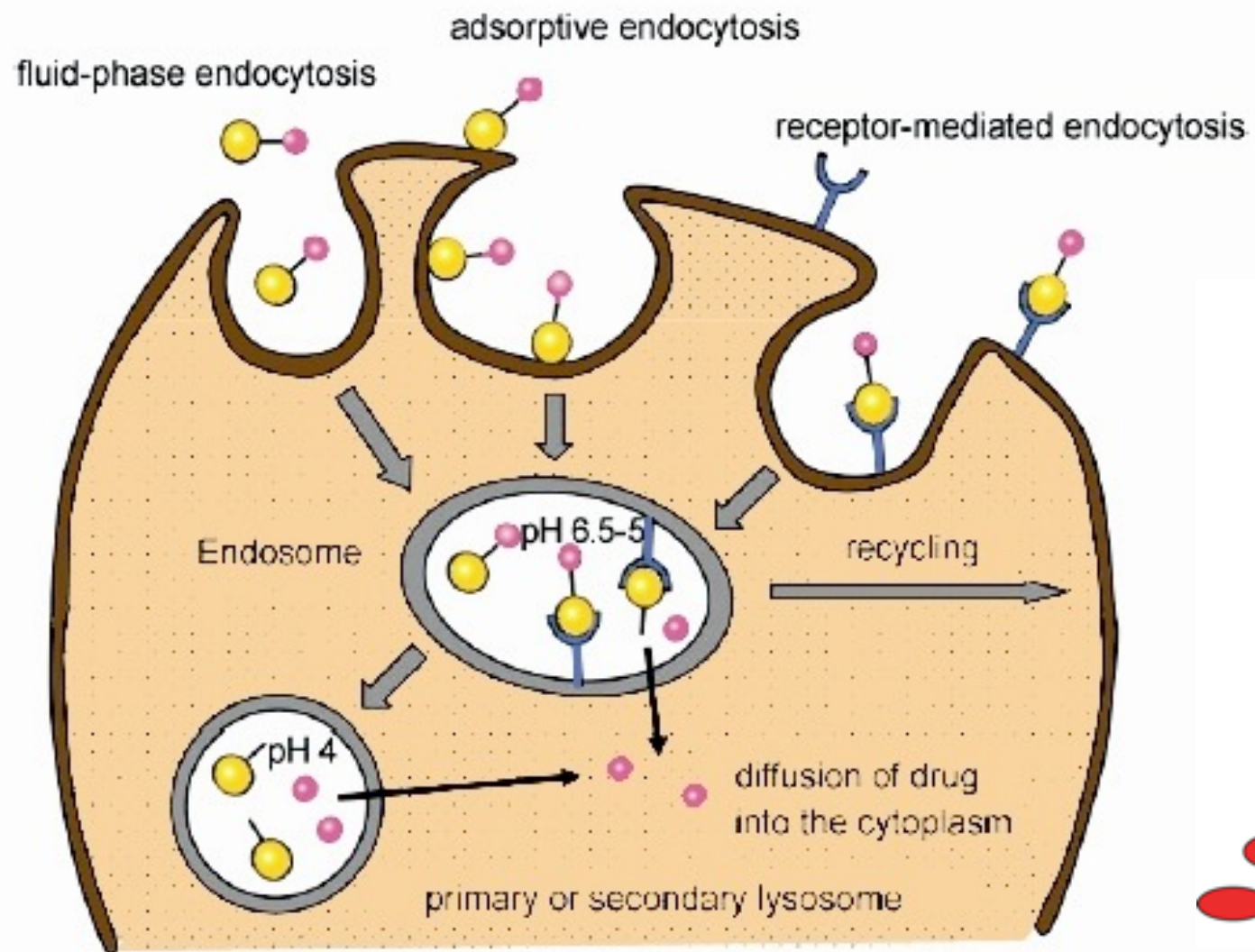
Reduction of Fe_2O_3 NPs



Nano-drug Tumor Targeting

MRI contrast agent

Endocytosis



Nano-Bubbles for Cancer Therapy

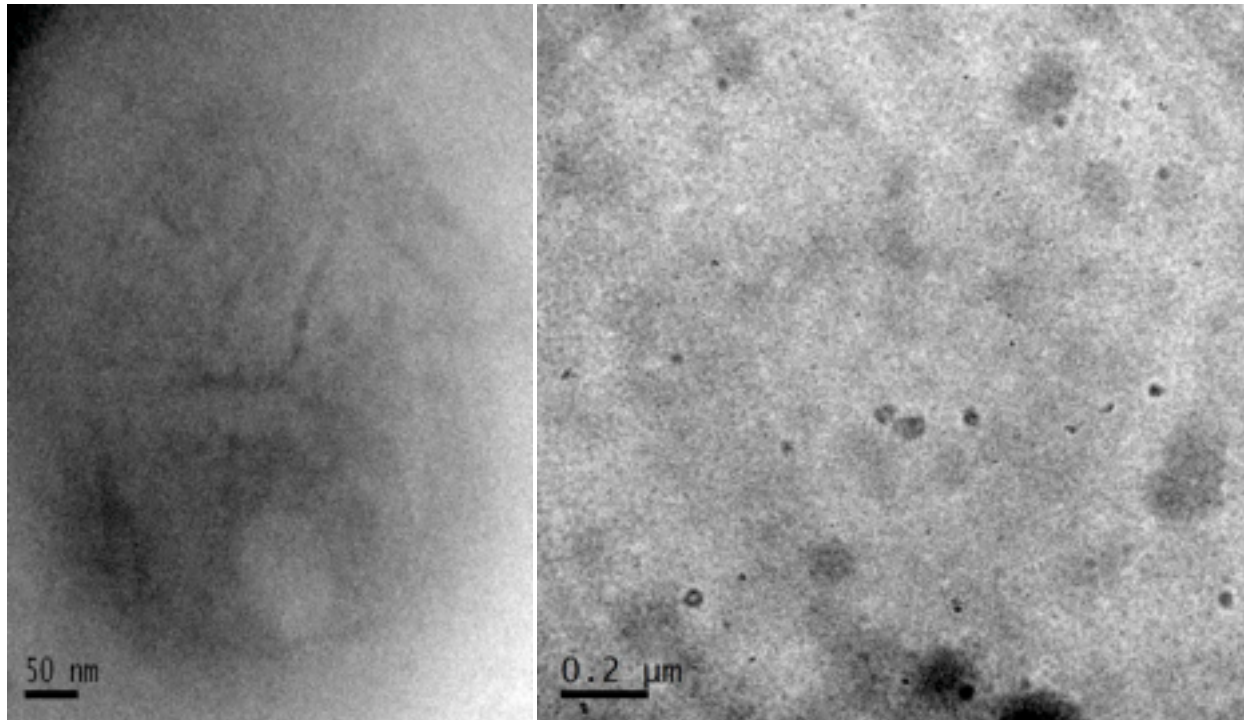


Protein Liquid

Au NPs in Protein Liquid

$$P=2\gamma/R$$

Nano-Bubbles for Cancer Therapy



Protein Liquid

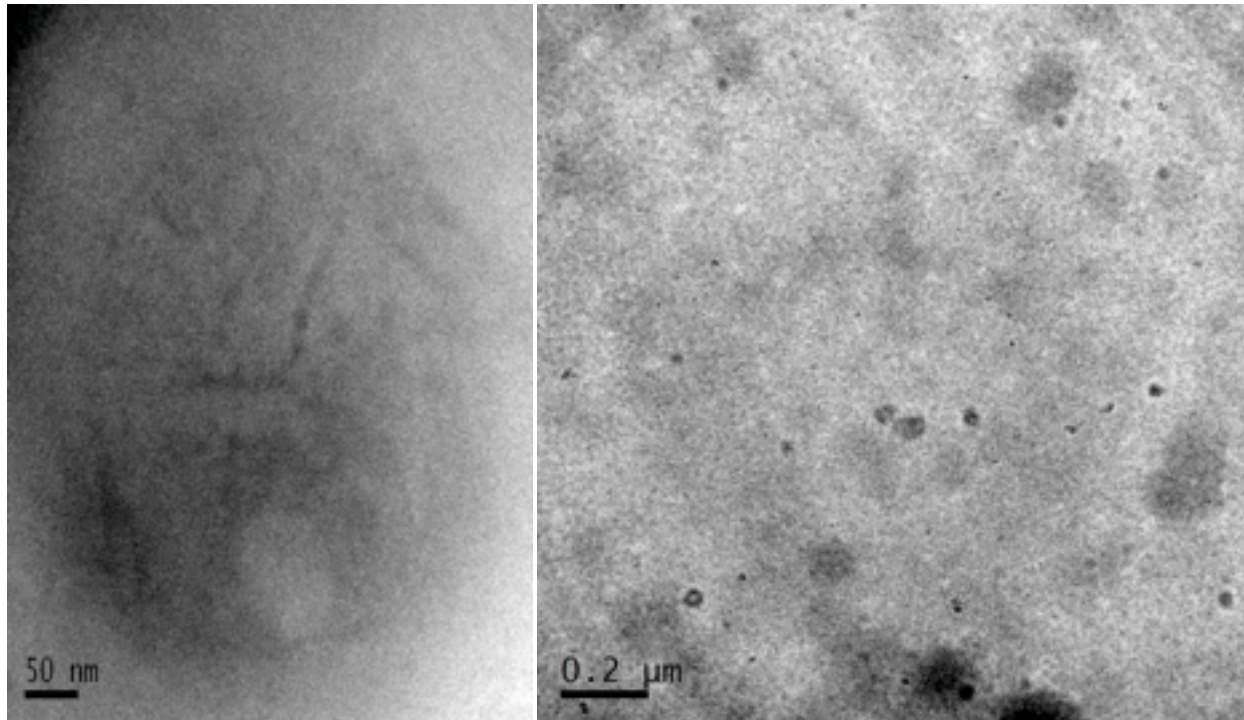
Au NPs in Protein Liquid

$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

Nano-Bubbles for Cancer Therapy



Protein Liquid

Au NPs in Protein Liquid

$$P=2\gamma/R$$

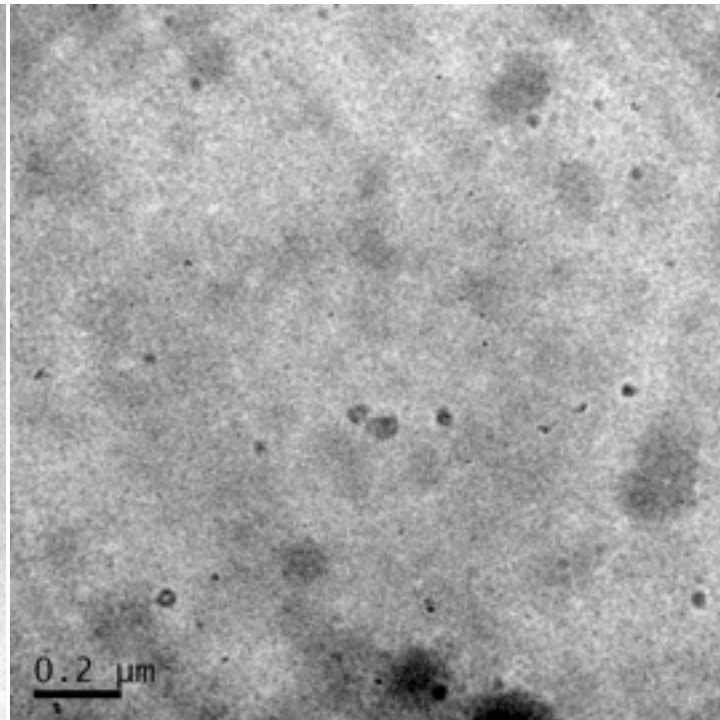
H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

Nano-Bubbles for Cancer Therapy



Protein Liquid



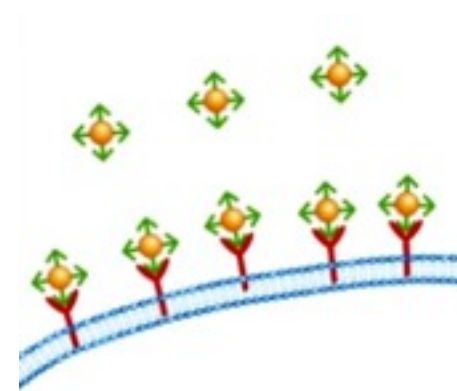
Au NPs in Protein Liquid

$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

Au NPs with antibody

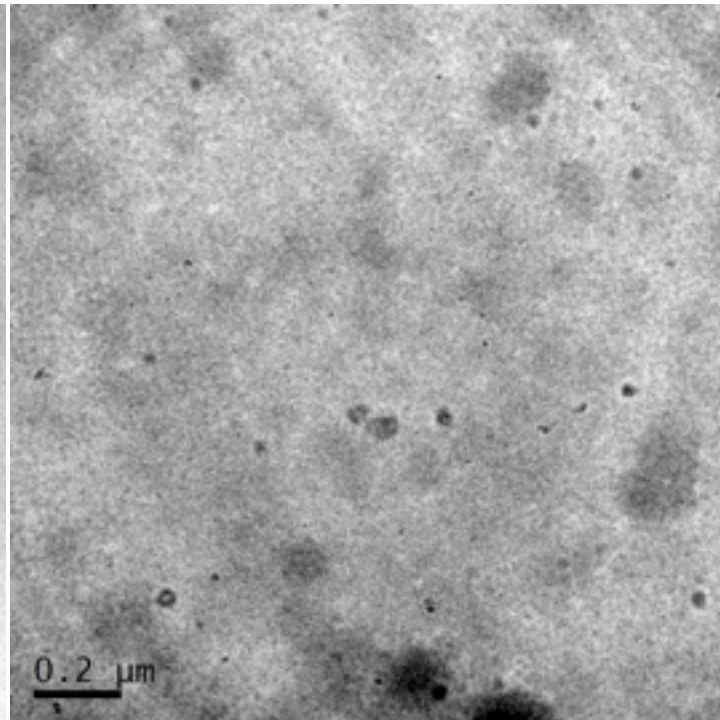


cancer cell

Nano-Bubbles for Cancer Therapy



Protein Liquid



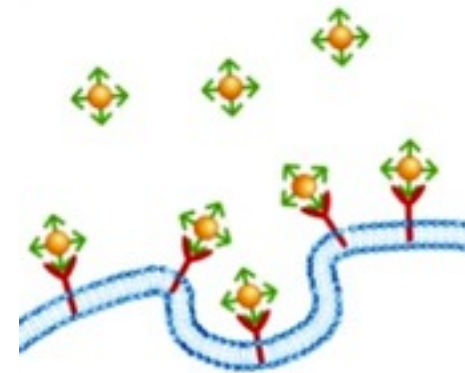
Au NPs in Protein Liquid

$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

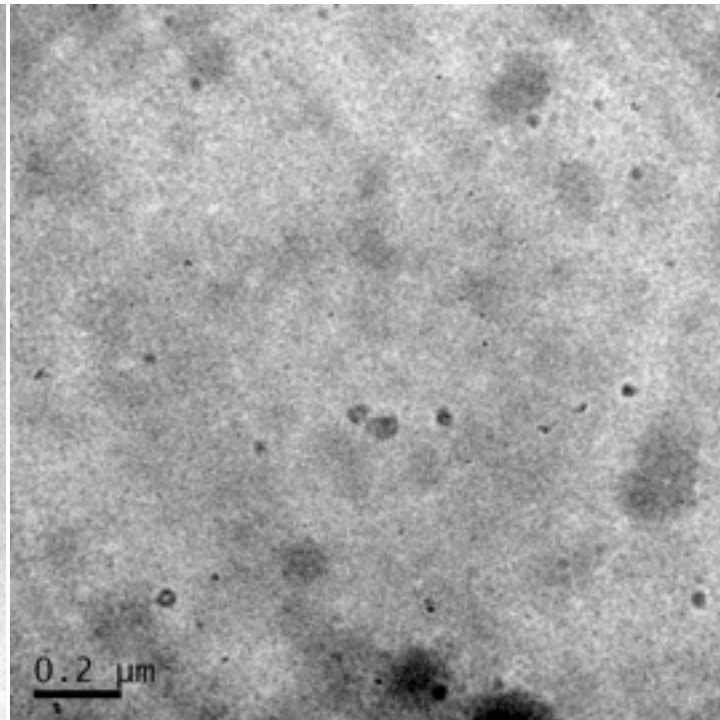
Au NPs with antibody



Nano-Bubbles for Cancer Therapy



Protein Liquid



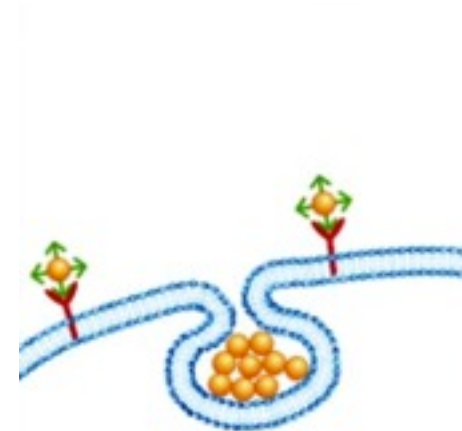
Au NPs in Protein Liquid

$$P=2\gamma/R$$

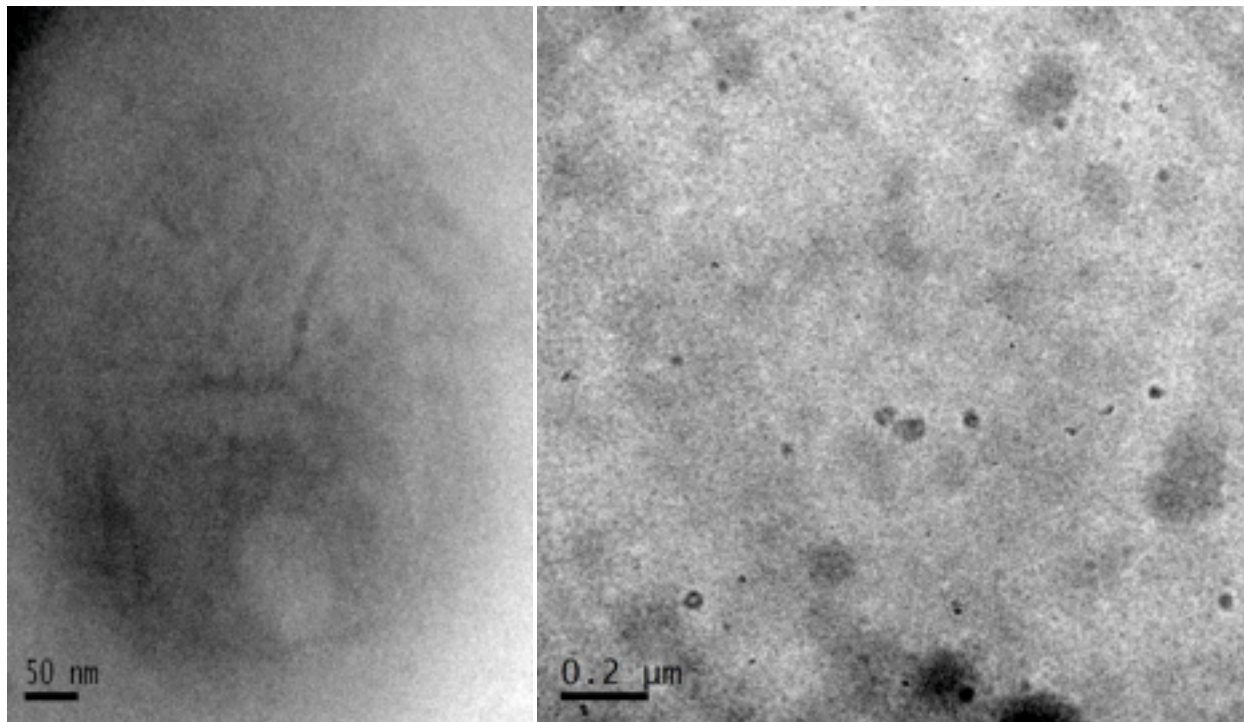
H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

Au NPs with antibody



Nano-Bubbles for Cancer Therapy



Protein Liquid

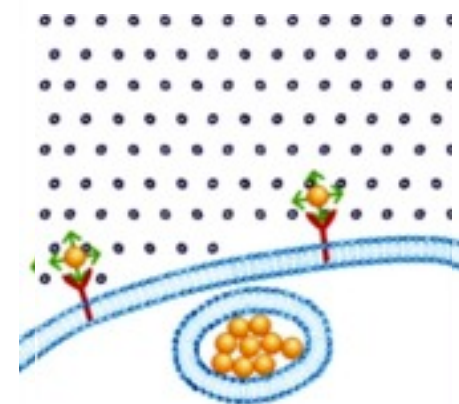
Au NPs in Protein Liquid

$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

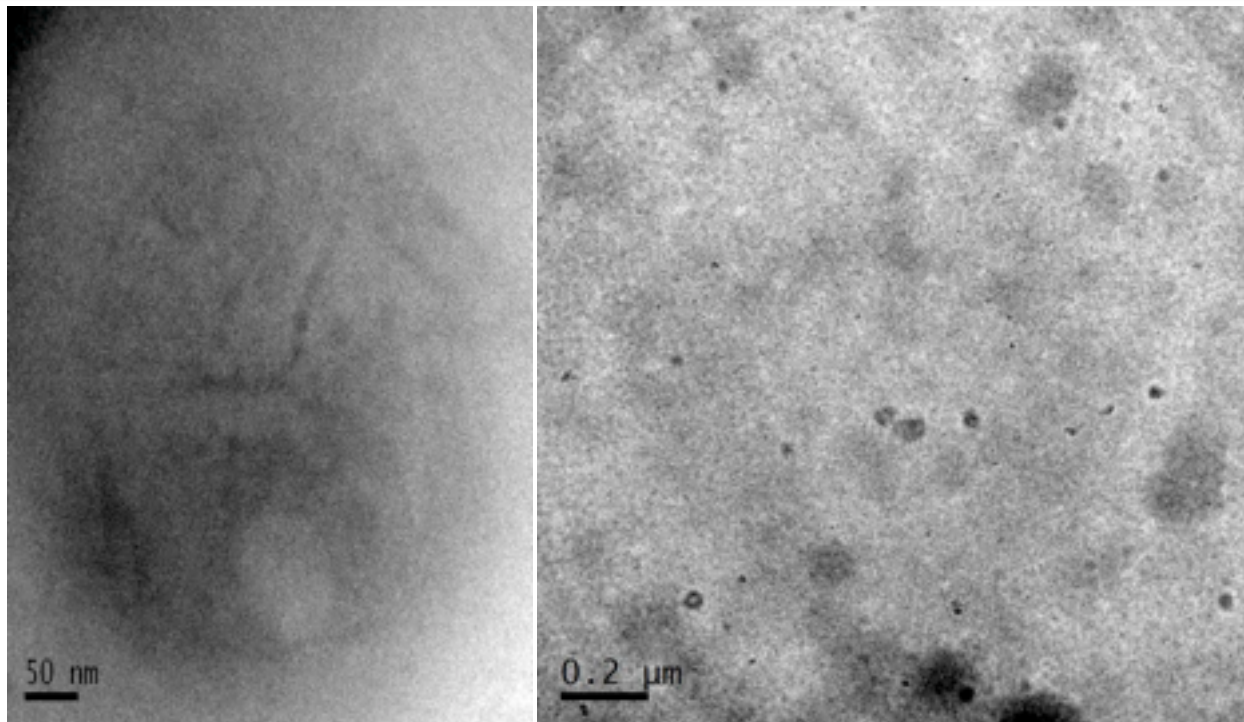
Bose gas (quantum gas: Bose–Einstein condensate)

Au NPs with antibody



cancer drug

Nano-Bubbles for Cancer Therapy



Protein Liquid

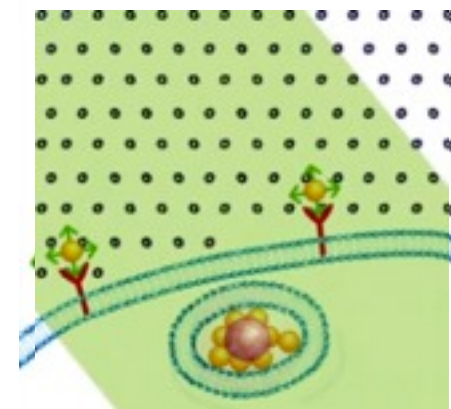
Au NPs in Protein Liquid

$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

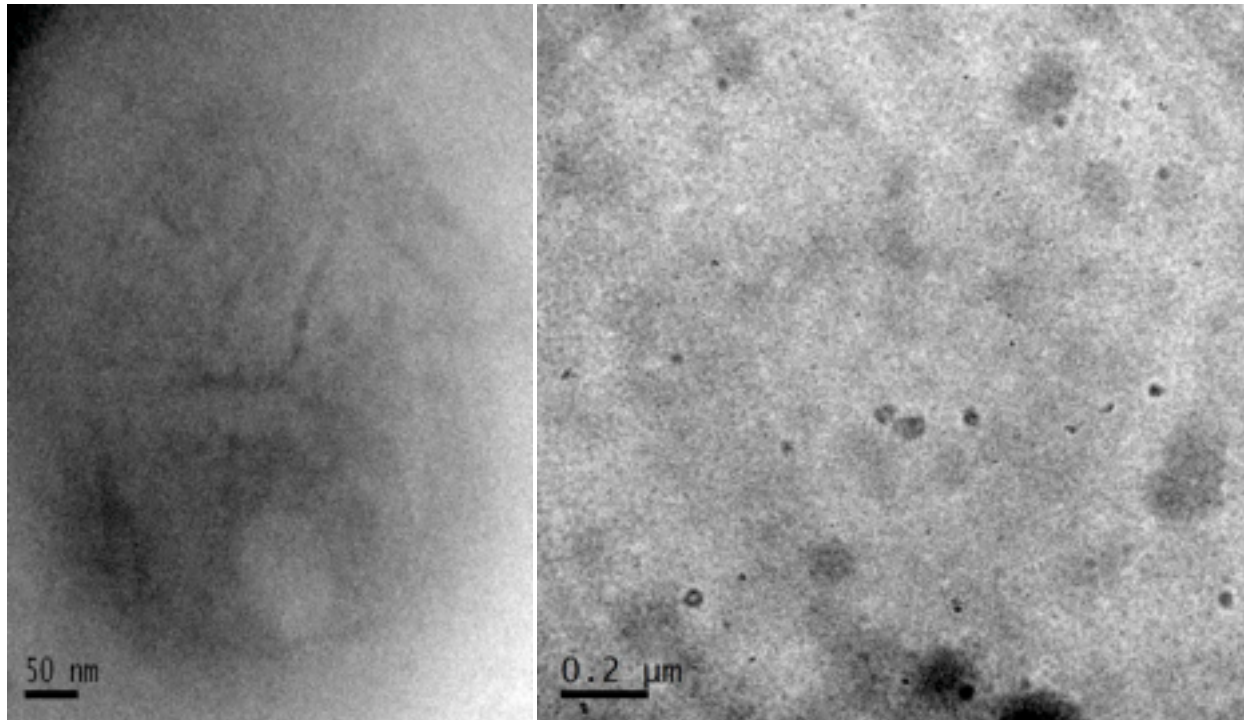
Au NPs with antibody



cancer drug

Laser

Nano-Bubbles for Cancer Therapy



Protein Liquid

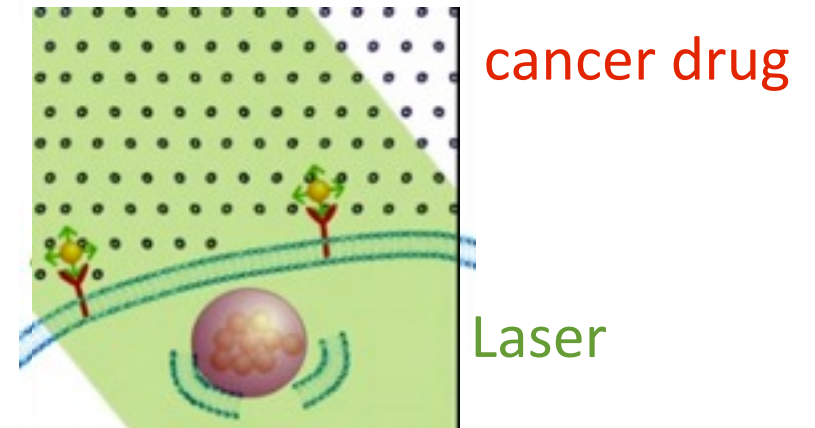
Au NPs in Protein Liquid

$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

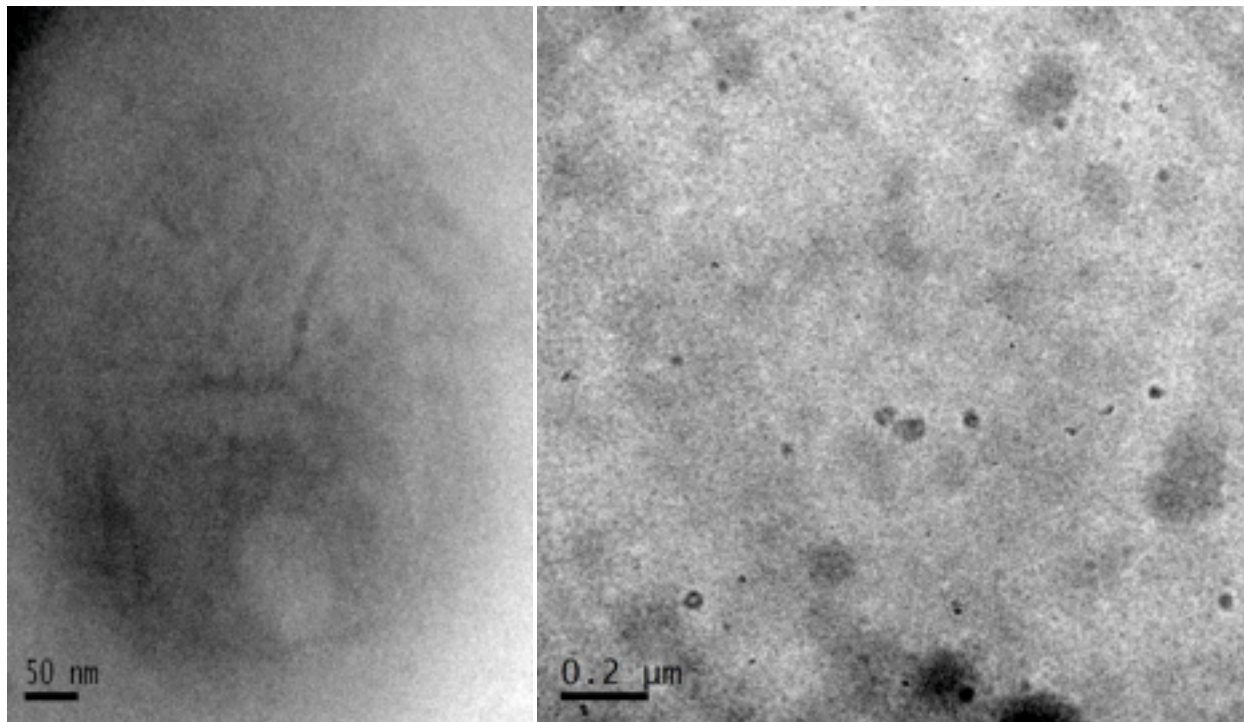
Bose gas (quantum gas: Bose–Einstein condensate)

Au NPs with antibody



Plasmonic Nanobubble

Nano-Bubbles for Cancer Therapy



Protein Liquid

Au NPs in Protein Liquid

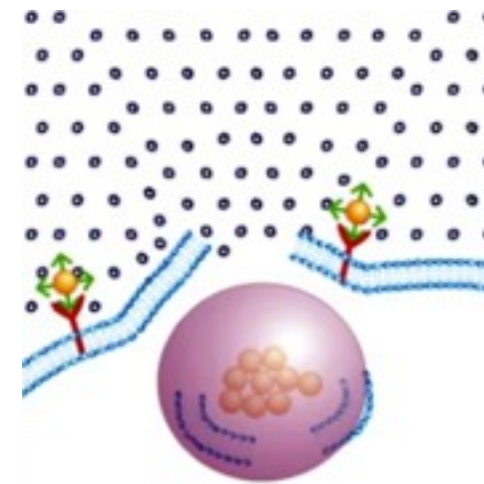
$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

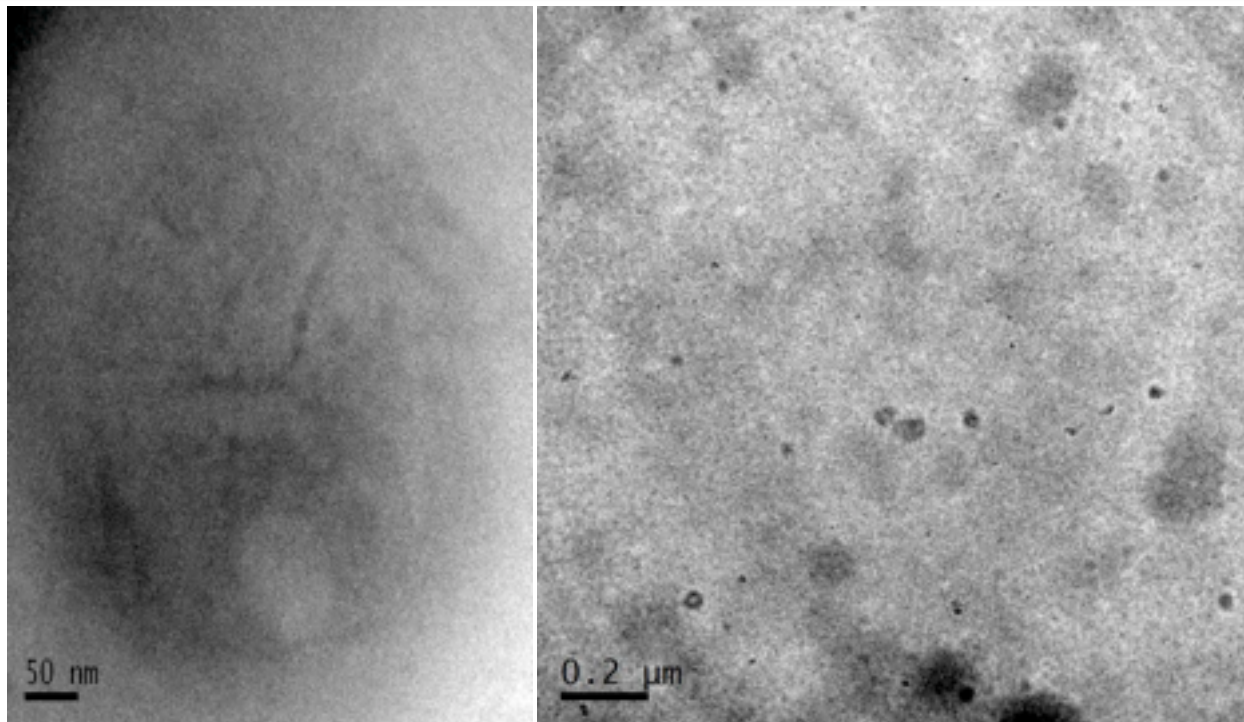
Au NPs with antibody

cancer drug



Plasmonic Nanobubble

Nano-Bubbles for Cancer Therapy



Protein Liquid

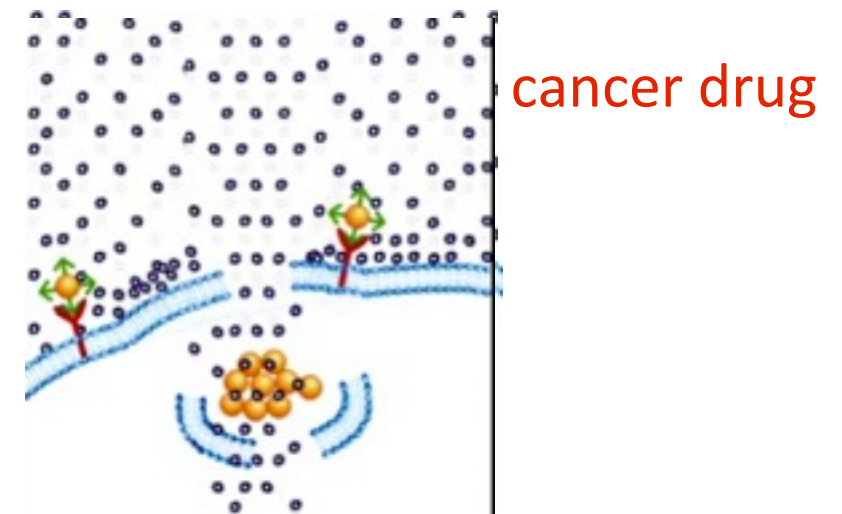
Au NPs in Protein Liquid

$$P=2\gamma/R$$

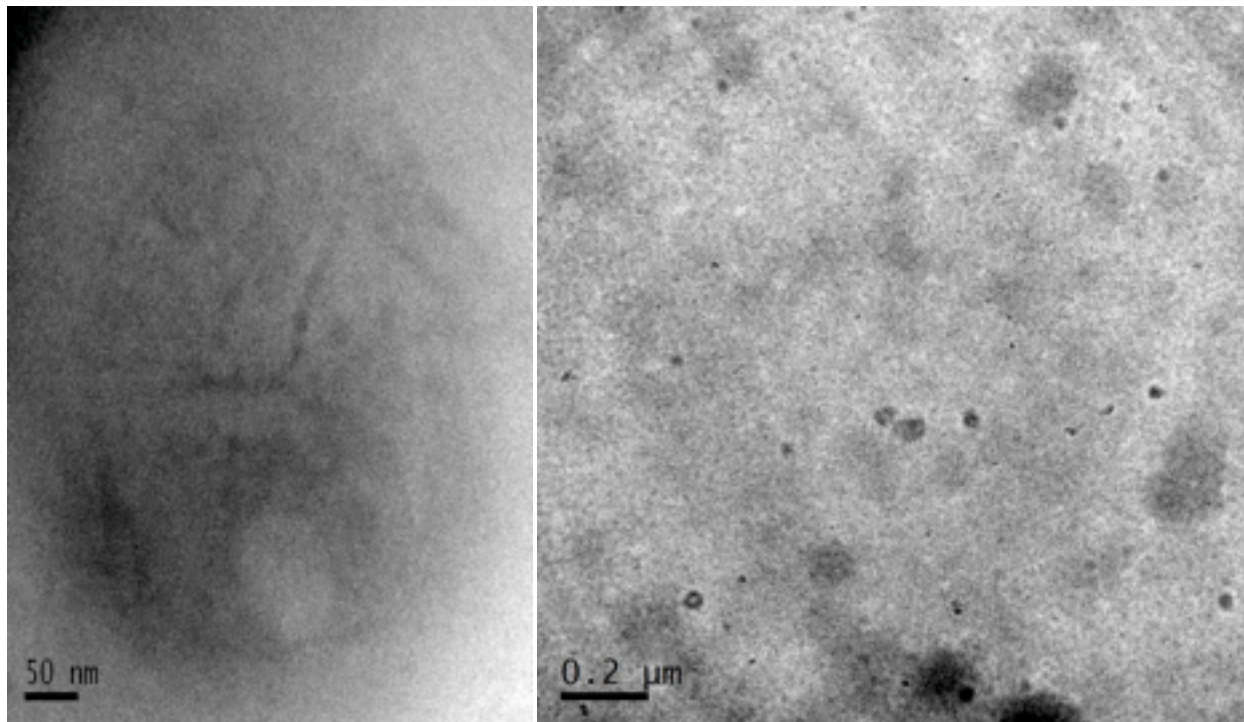
H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

Au NPs with antibody



Nano-Bubbles for Cancer Therapy



Protein Liquid

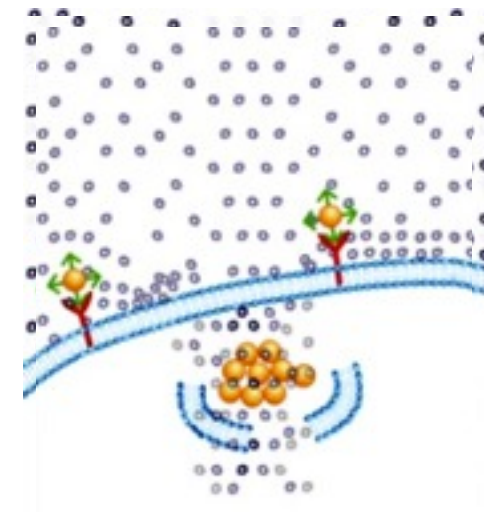
Au NPs in Protein Liquid

$$P=2\gamma/R$$

H₂ Knudsen gas (classical ideal gas)

Bose gas (quantum gas: Bose–Einstein condensate)

Au NPs with antibody

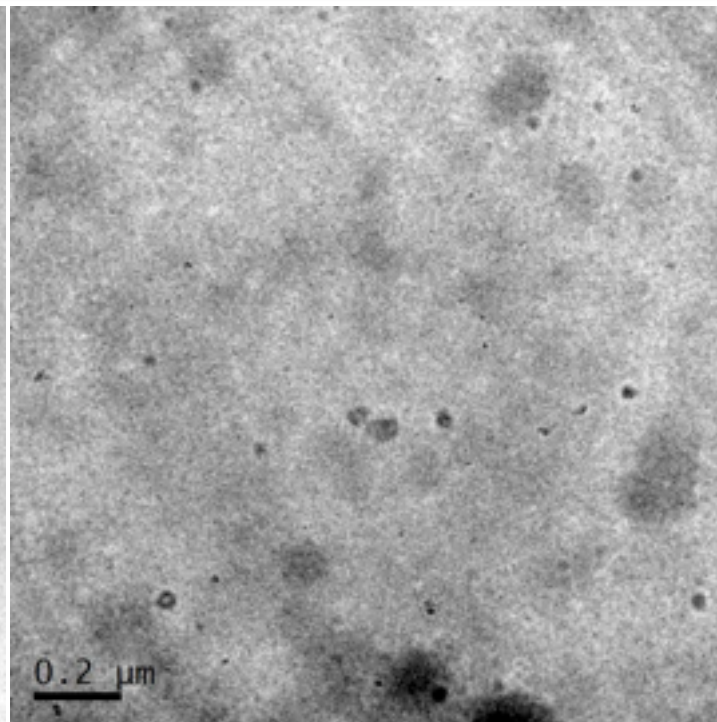


cancer drug

Nano-Bubbles for Cancer Therapy

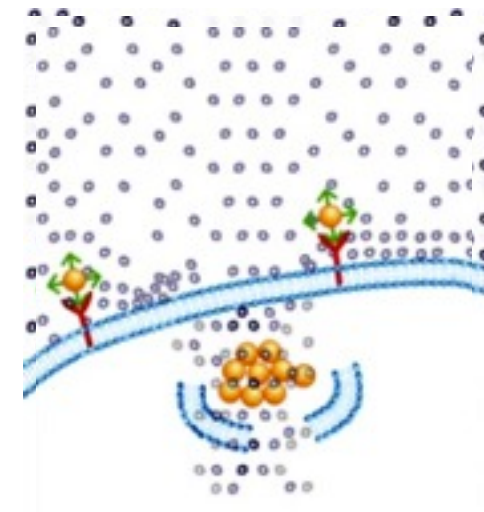


Protein Liquid

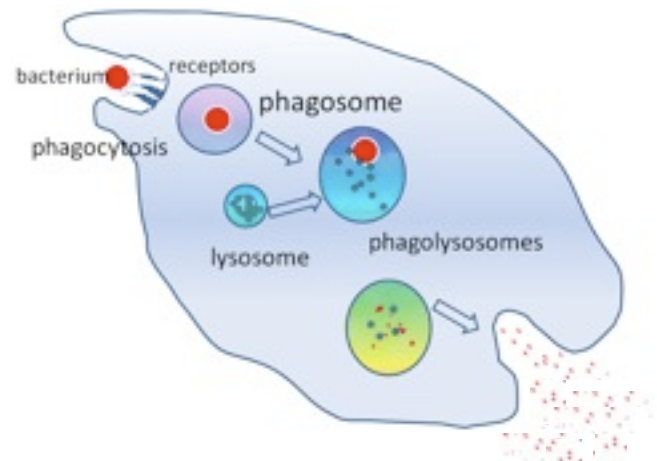


Au NPs in Protein Liquid

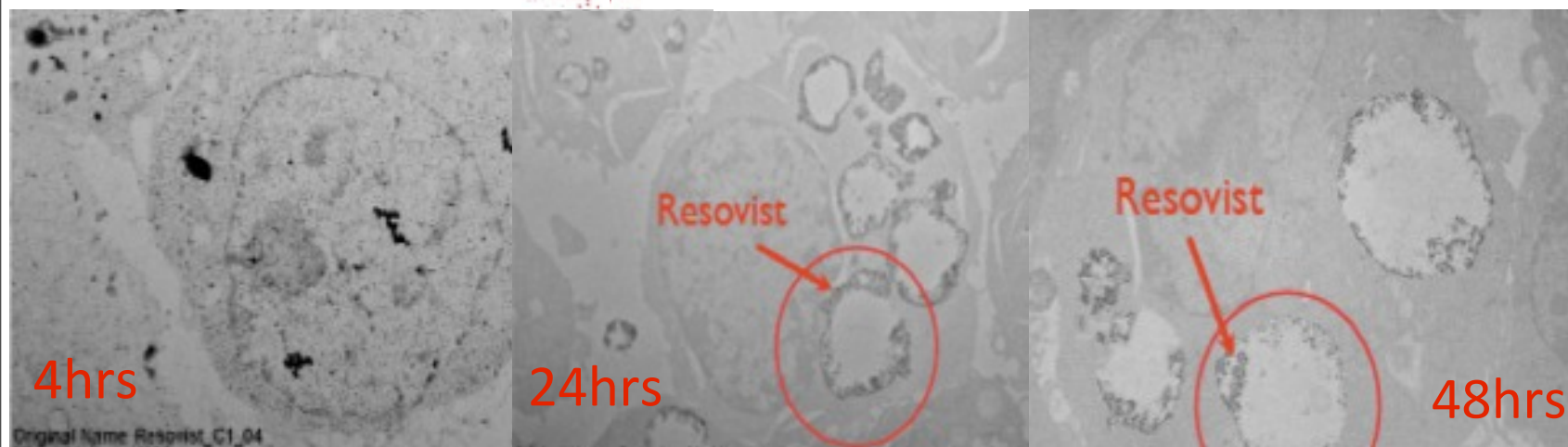
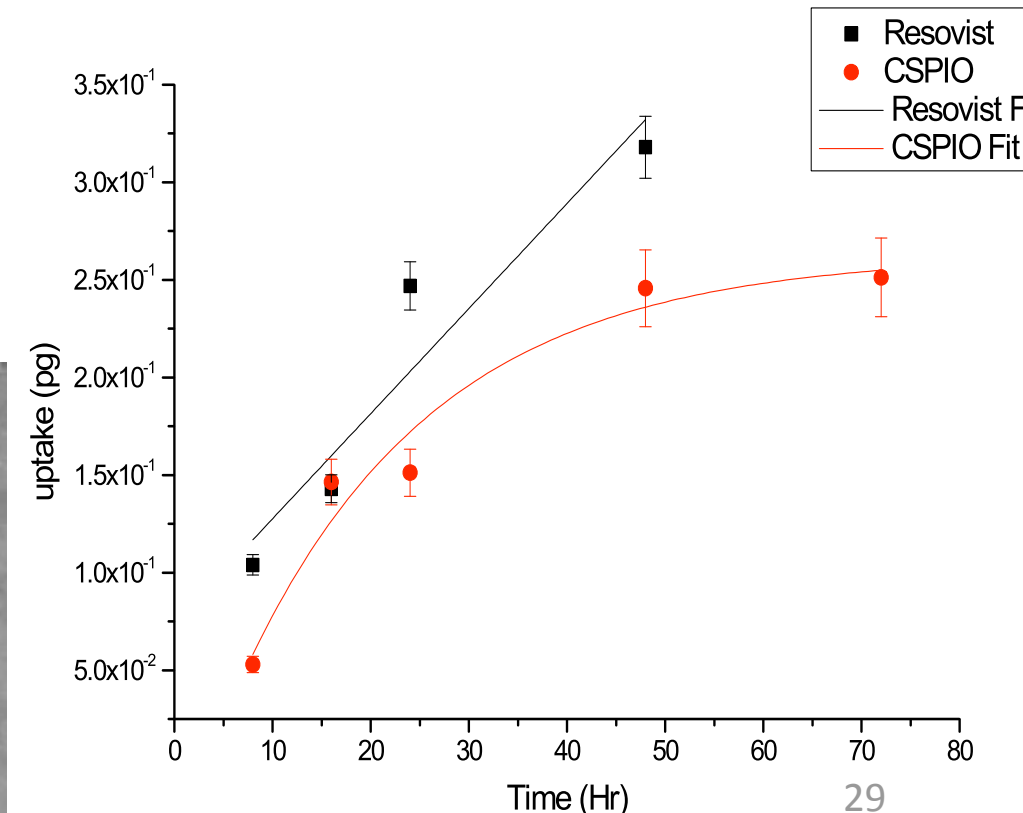
Au NPs with antibody



cancer drug



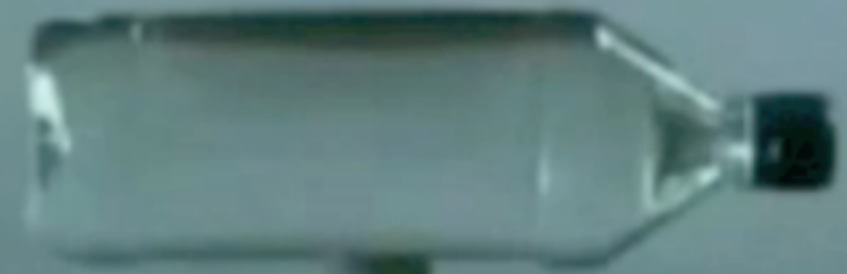
CSPIO (or PLGA+Fe₃O₄)



Ultra-fast Electron Microscope

Ultra-fast Electron Microscope

High Speed Camera



Ultra-fast Electron Microscope

High Speed Camera

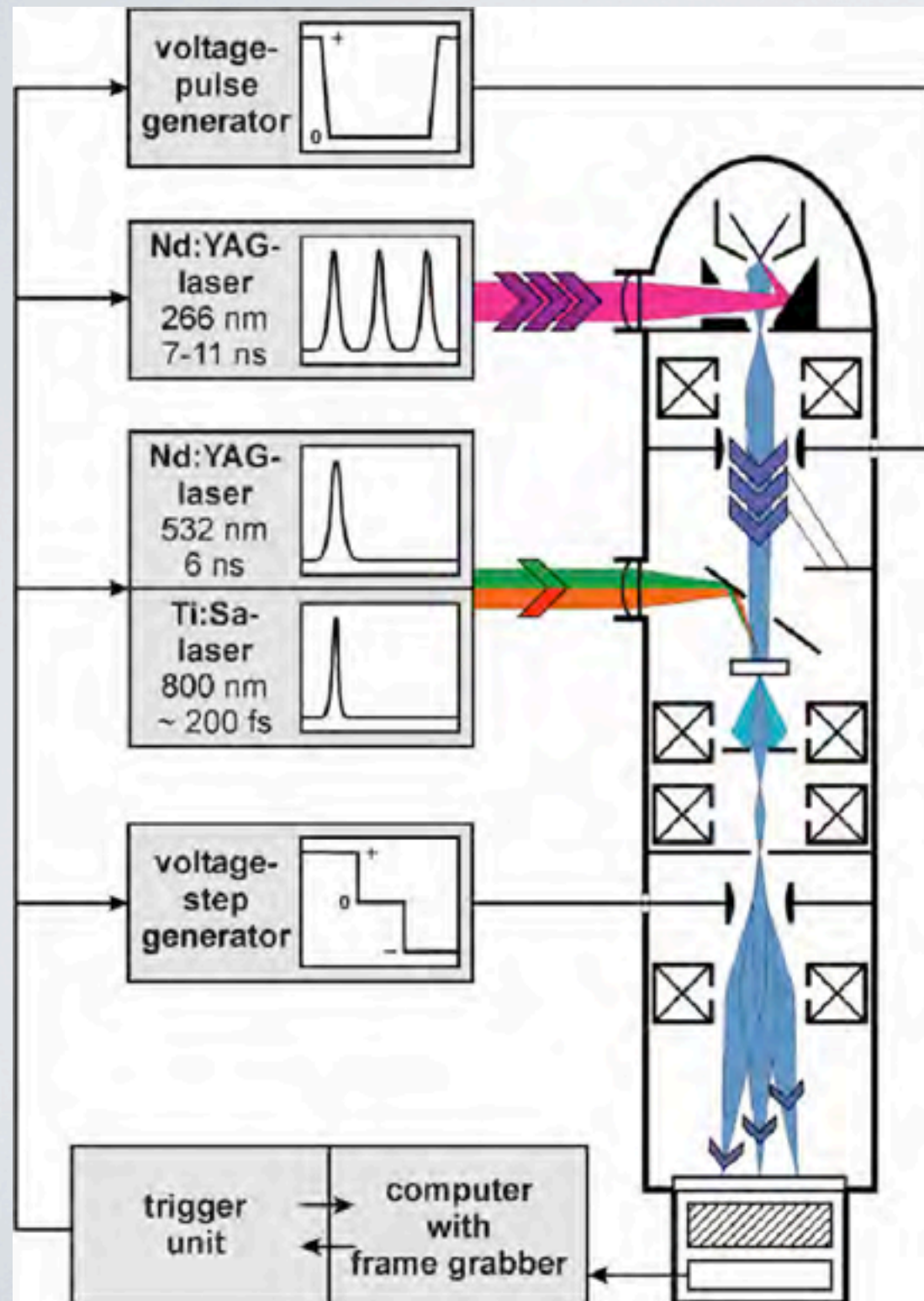


A falling apple photographed by stroboscopic illumination at intervals of $\sim 1/25$ s. The acceleration due to gravity is clear.

Time-resolved TEM was developed at TU-Berlin beginning in the late 1970's

Oleg Bostanjoglo

H. Dömer and O. Bostanjoglo, Rev. Sci. Inst. 74, 4369 (2003)

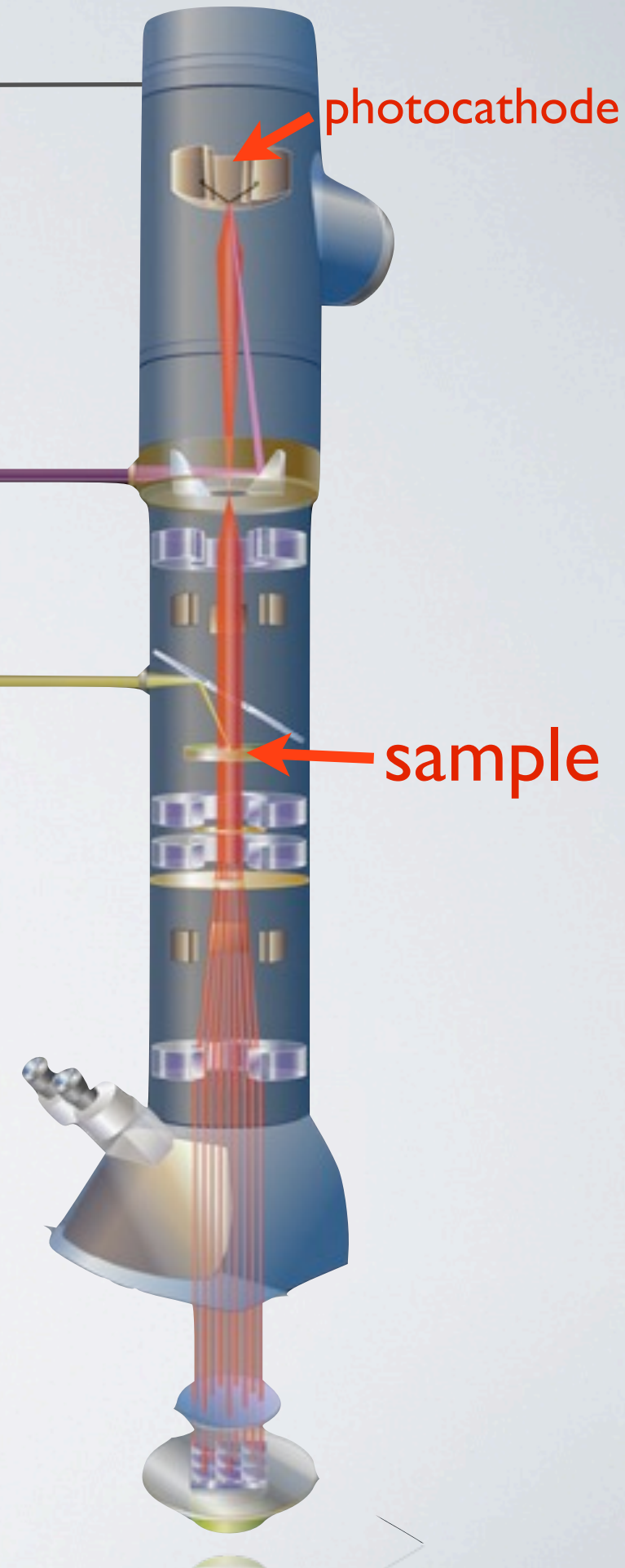


Laser
(probe)

Laser
(pump)

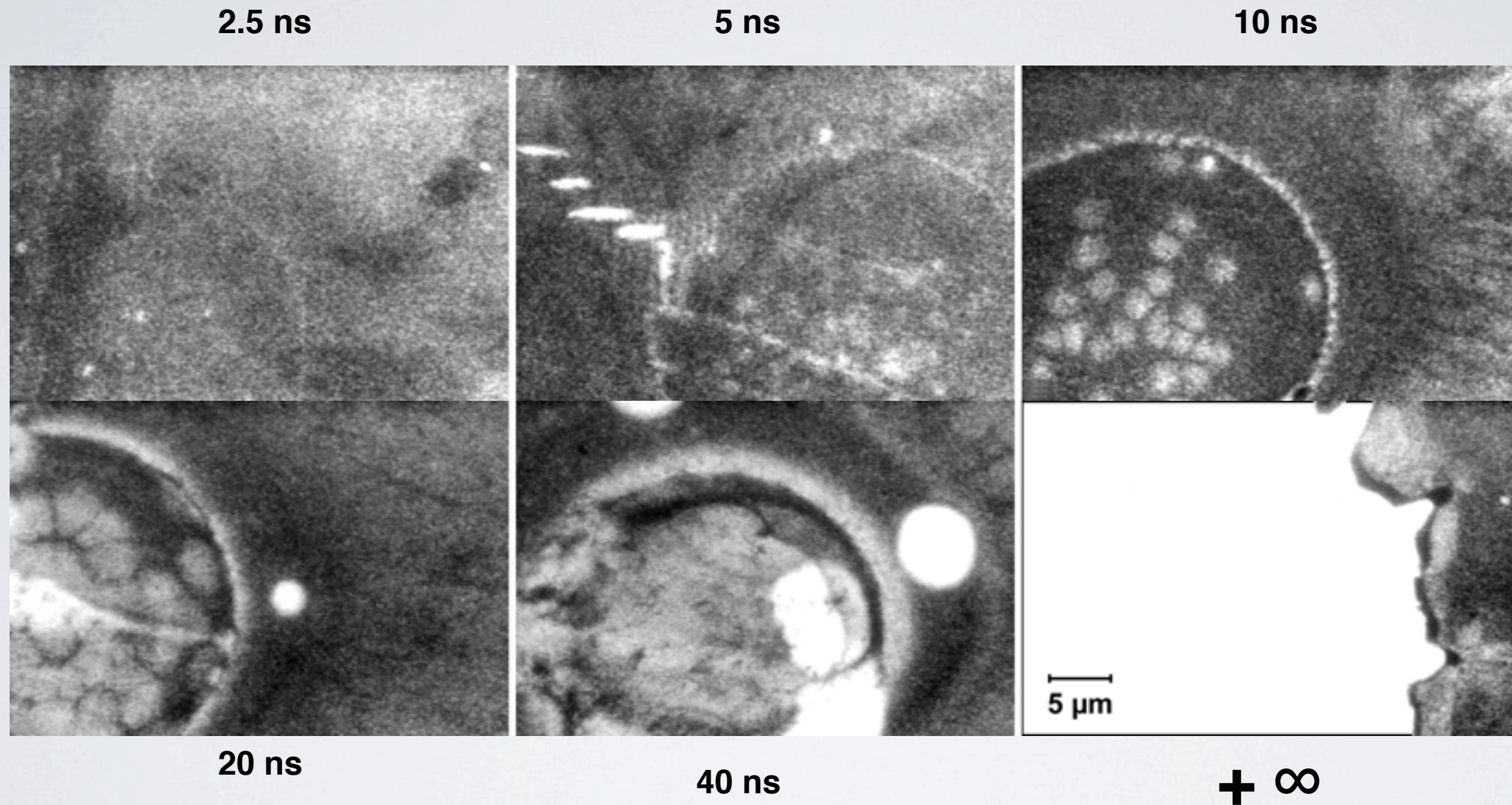
photocathode

sample



Time-resolved TEM

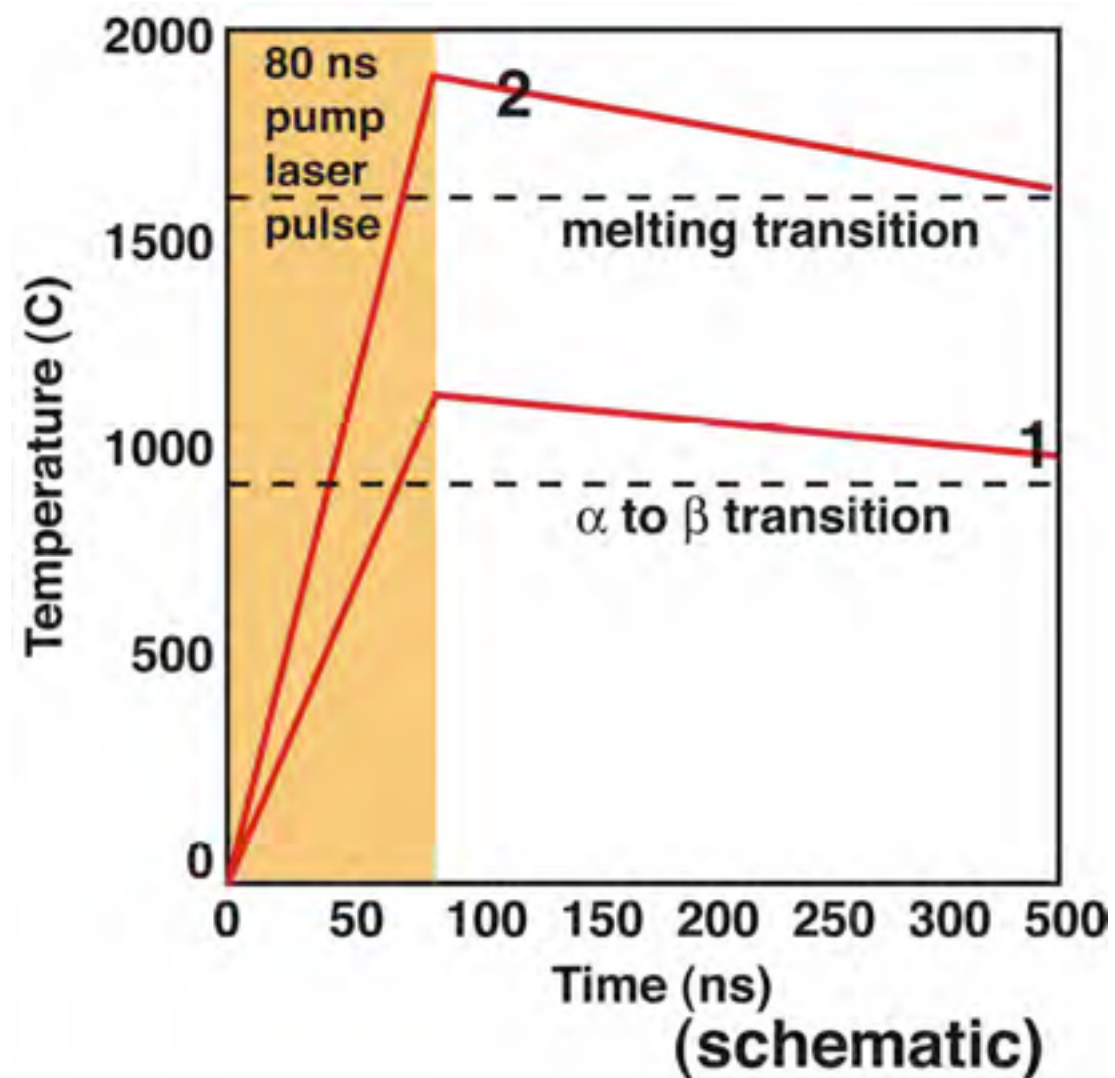
ablation of Ni film by ultrashort laser pulse



H. Dömer and O. Bostanjoglo, Journal of Applied Physics **91**, 5462-5467 (2002).

Time-resolved electron diffraction

α -Ti (hcp) $\rightarrow$ β -Ti (bcc) martensitic transformation

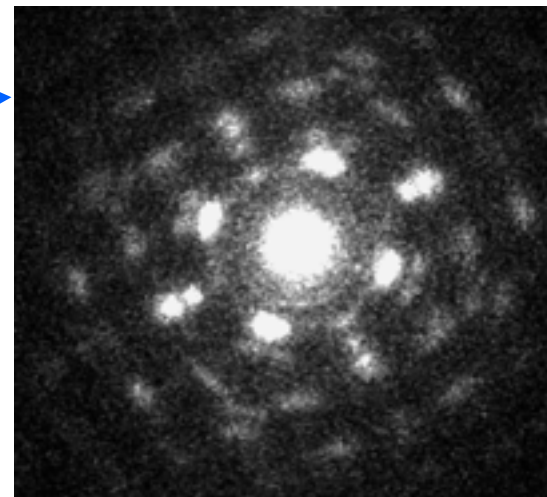
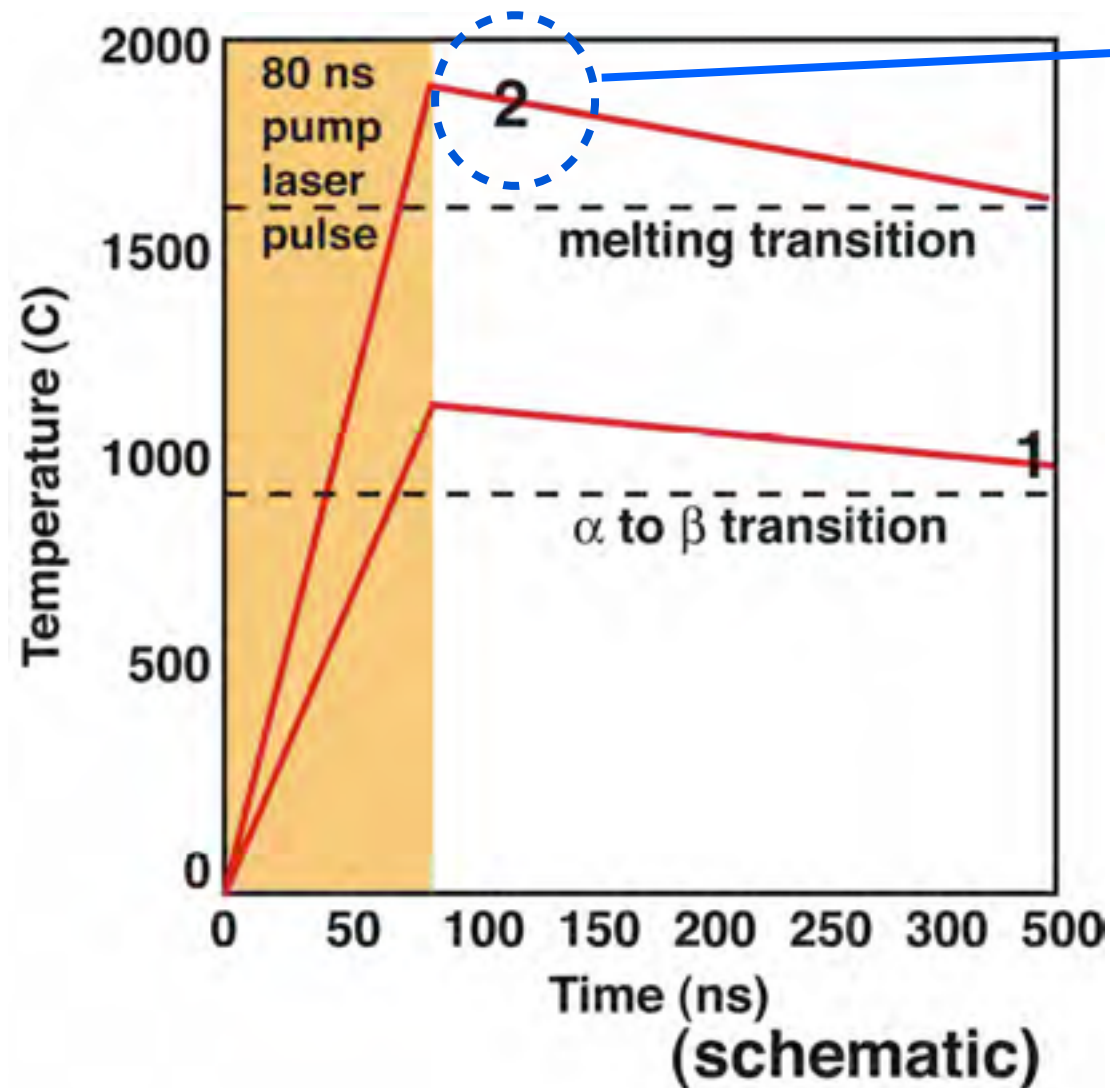


Bryan W. Reed, NLLB, UC, 2008

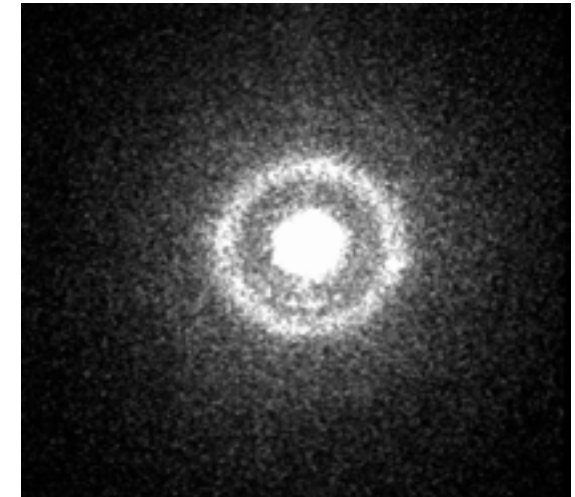
Time-resolved electron diffraction

α -Ti (hcp) $\rightarrow$ β -Ti (bcc) martensitic transformation

Melting transition



hcp Ti, α -phase



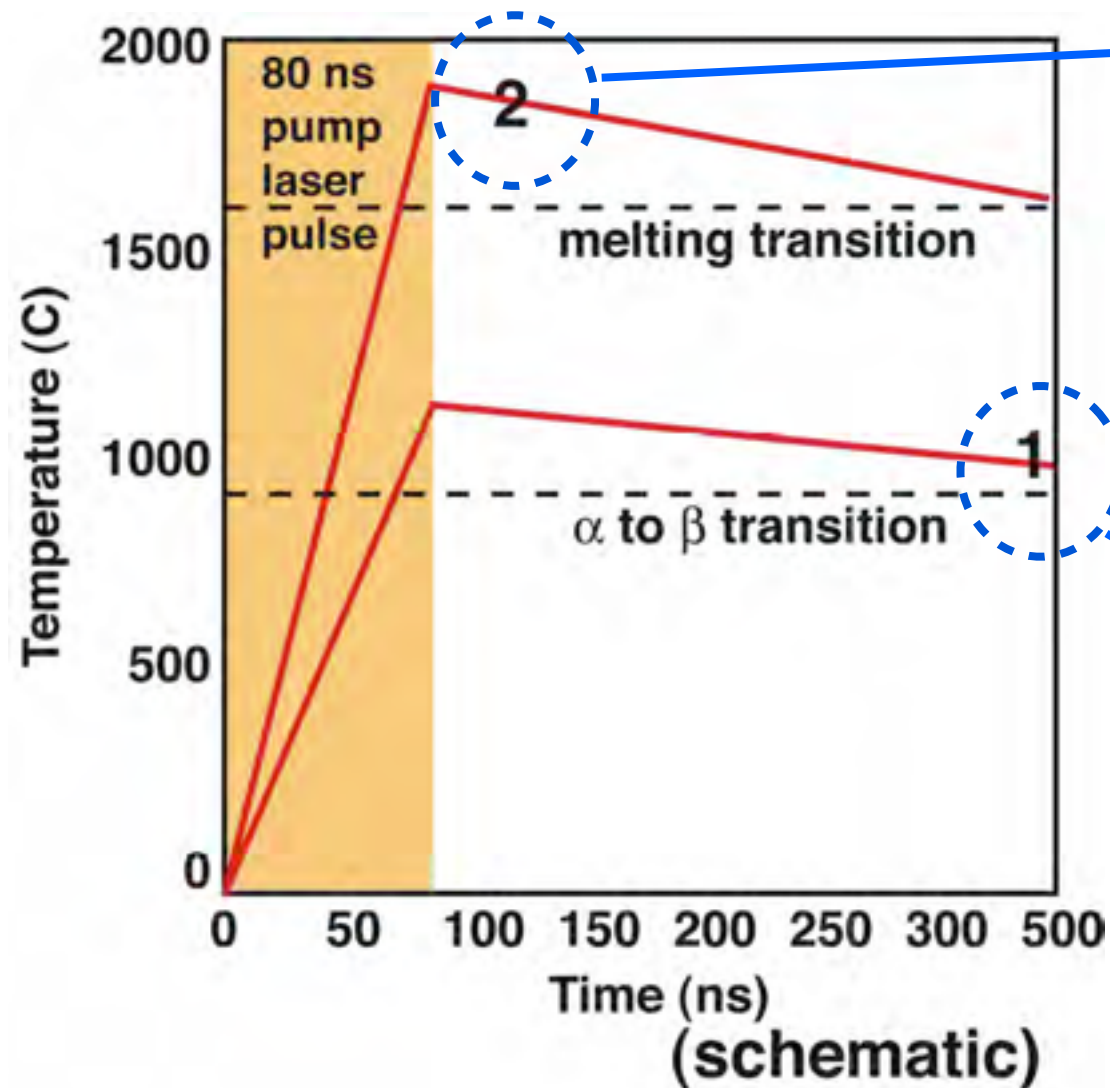
Melt structure

Bryan W. Reed, NLLB, UC, 2008

Time-resolved electron diffraction

α -Ti (hcp) $\rightarrow$ β -Ti (bcc) martensitic transformation

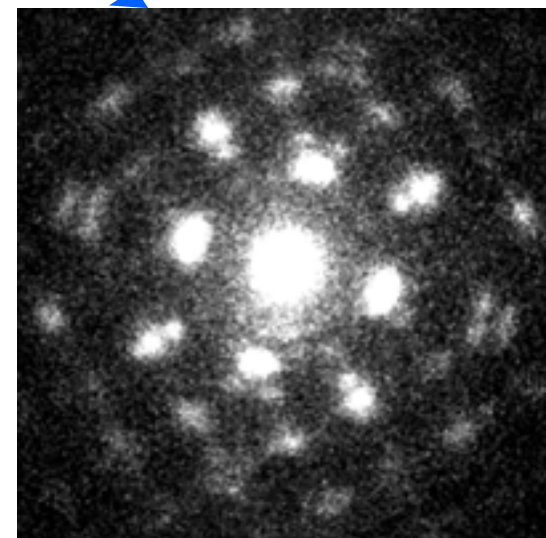
Melting transition



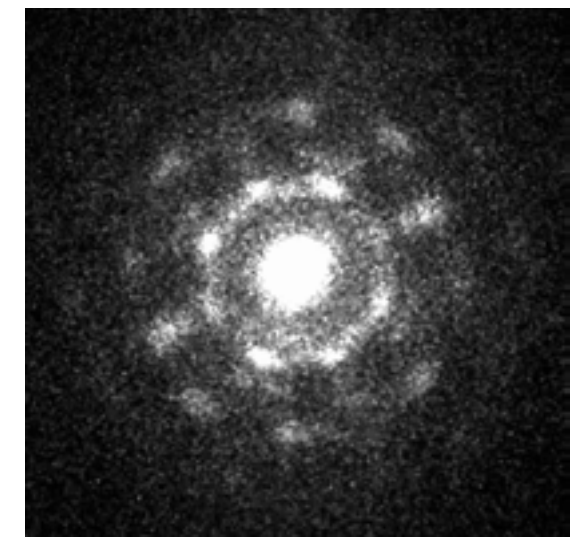
hcp Ti, α -phase

Melt structure

Phase transformation (α to β)



hcp Ti, α -phase



bcc Ti, β -phase

Bryan W. Reed, NLLB, UC, 2008

The

END