Electron Diffraction Techniques

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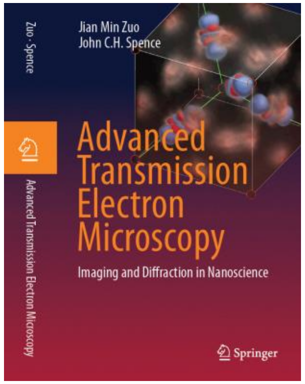
Reference

Advanced Transmission Electron Microscopy, Imaging and Diffraction In Nanoscience, Zuo and Spence, Springer, 2017



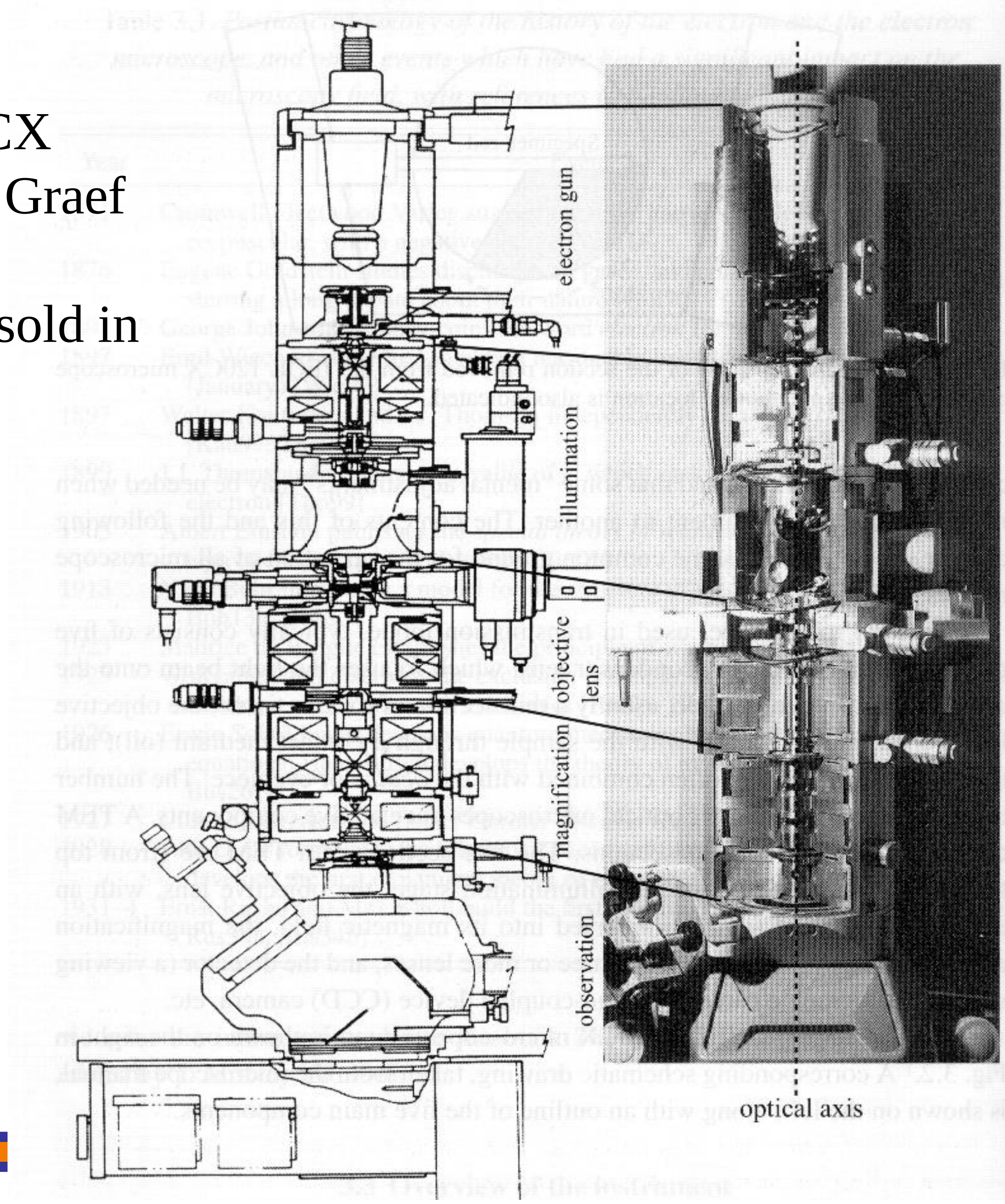
Electron Diffraction Workshop, Trondheim, Norway, 2017

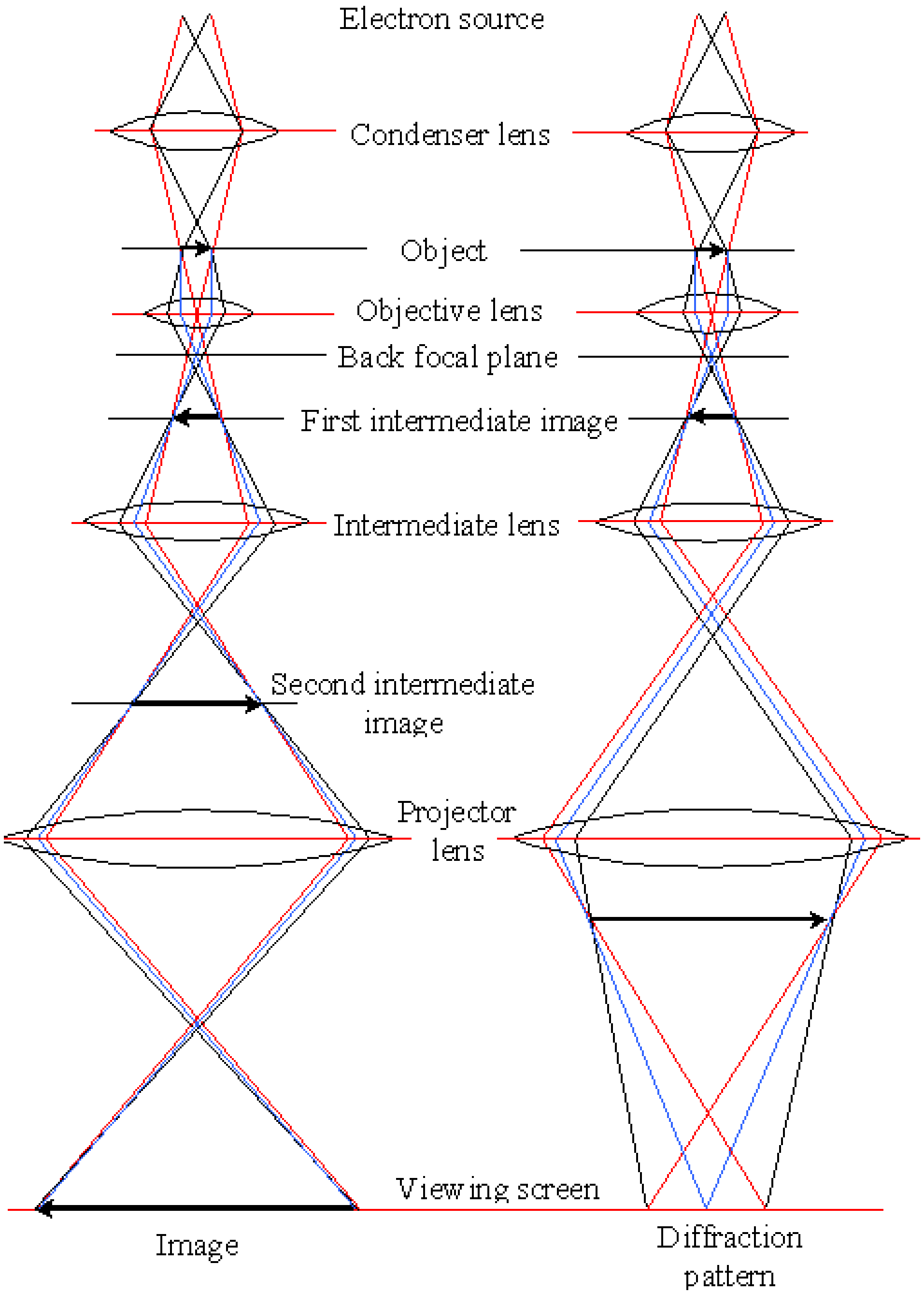
Covered Topics

- Electron diffraction techniques
  - Selected area electron diffraction
  - Convergent beam electron diffraction
  - Large angle methods
  - Precession electron diffraction
  - Nanoarea electron diffraction
  - Scanning electron nanodiffraction
- Energy-filtering
- STEM, Ronchigram, Position averaged CBED
- Electron illumination
  - Beam current
  - Probe size
  - Coherence
- Electron detectors and radiation effects

JEOL 120 CX
From Marc De Graef

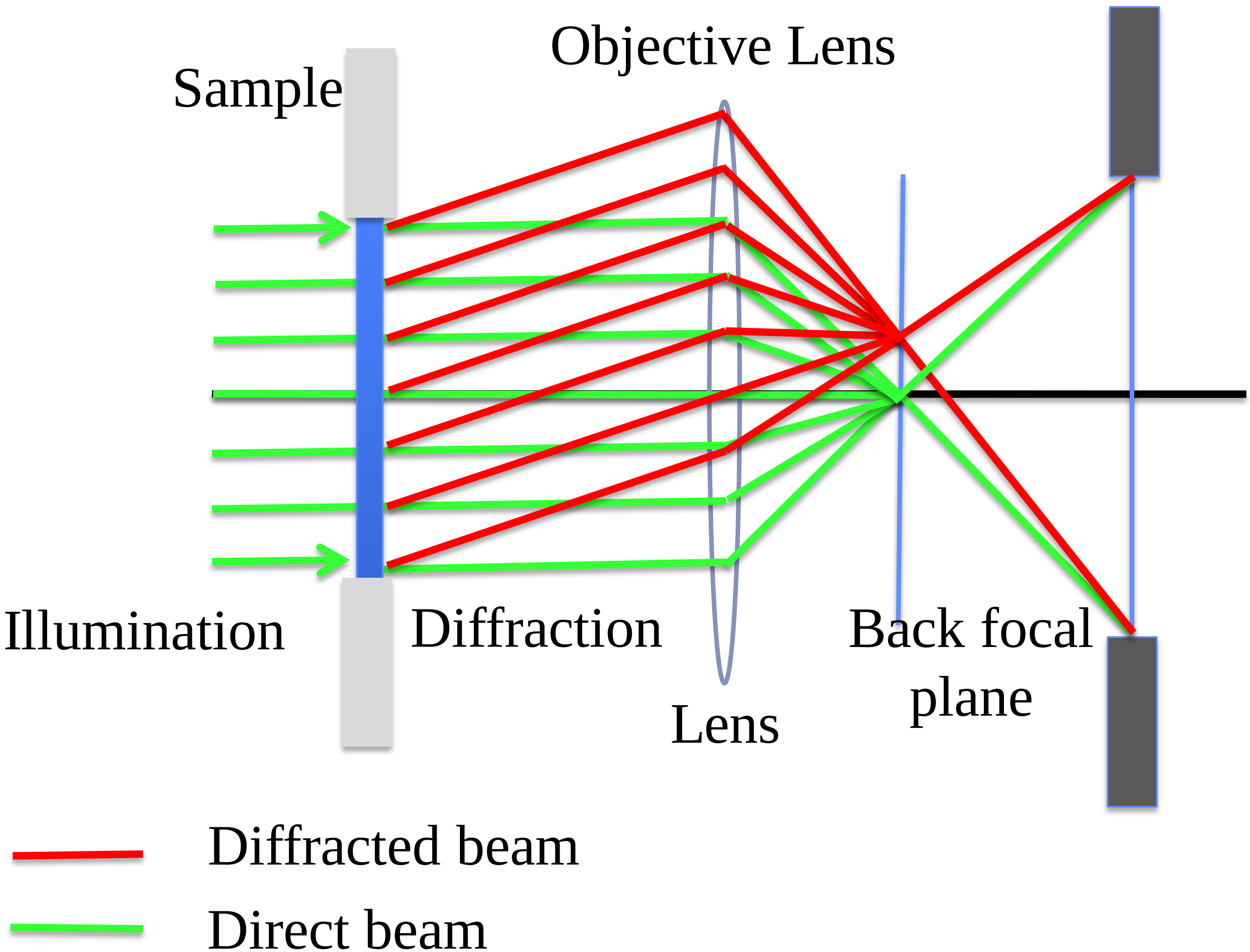
Developed and sold in early 80s



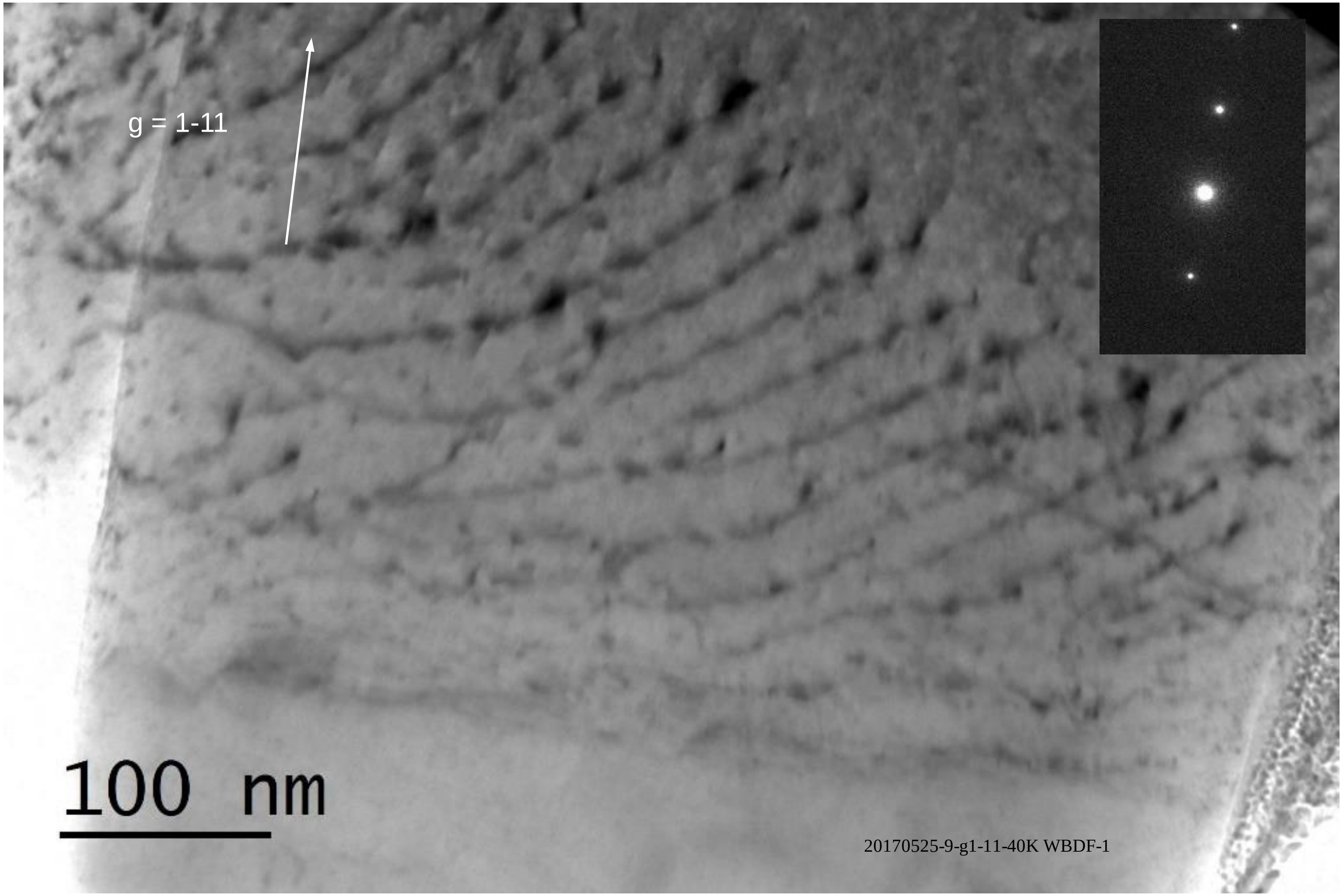


Electron source
Condenser lens
Object
Objective lens
Back focal plane
First intermediate image
Intermediate lens
Second intermediate image
Projector lens
Viewing screen
Image
Diffraction pattern

Formation of Diffraction Patterns

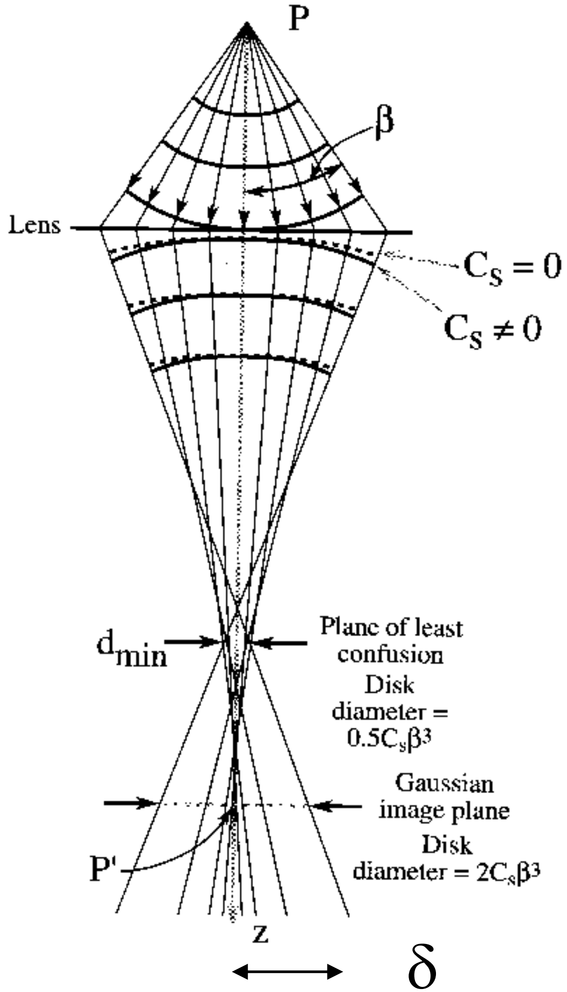


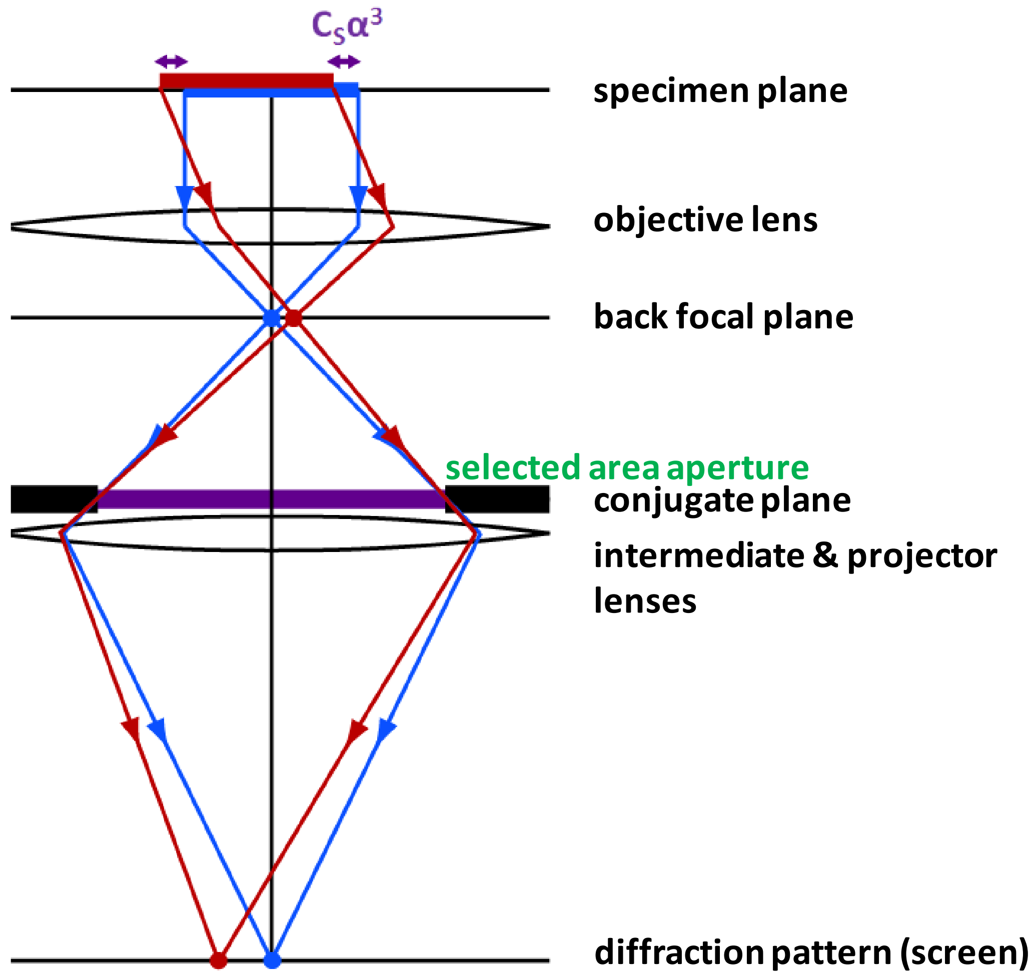
Diffraction Contrast Imaging



From Qun Yang, UIUC

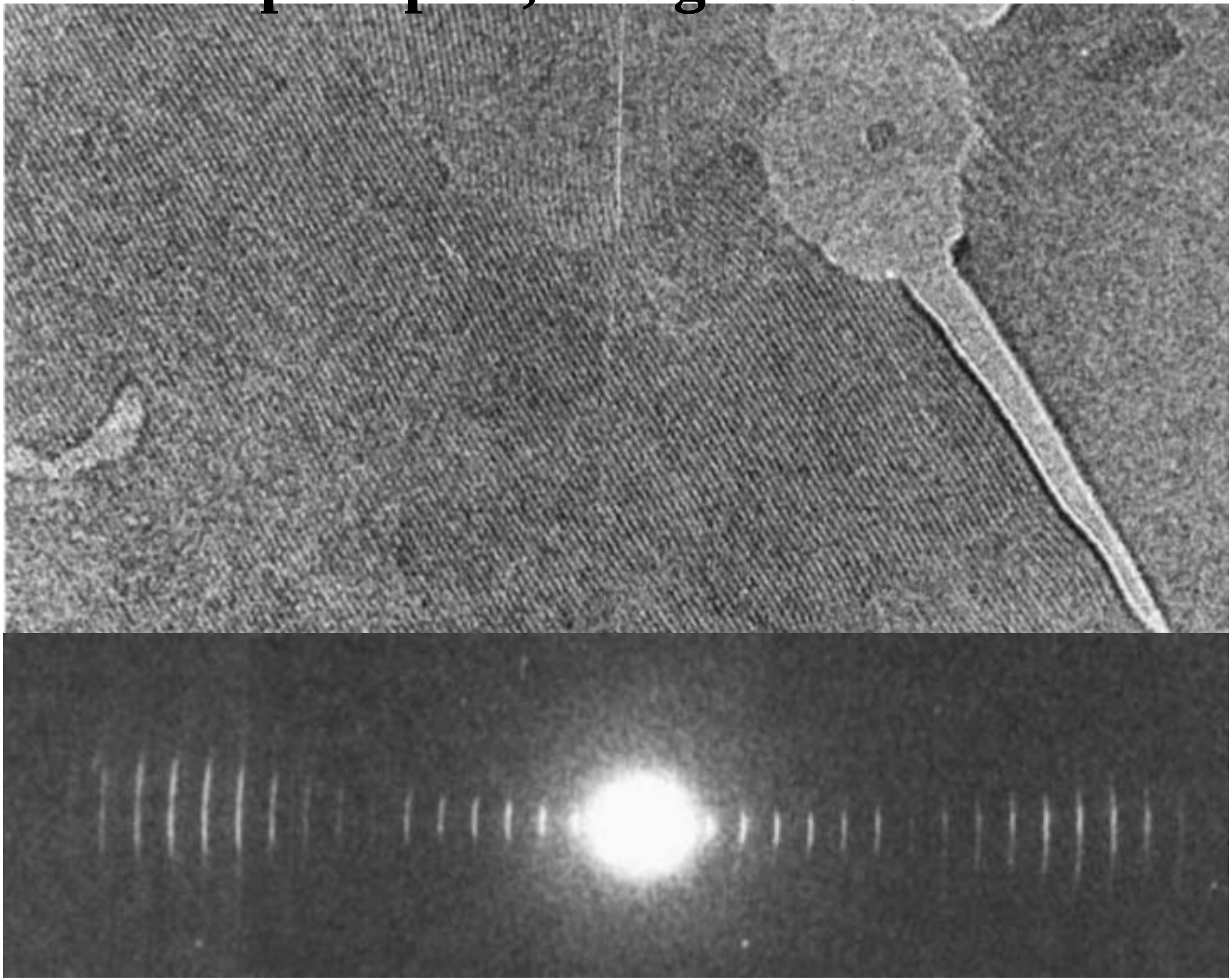
Effects of Spherical Aberration





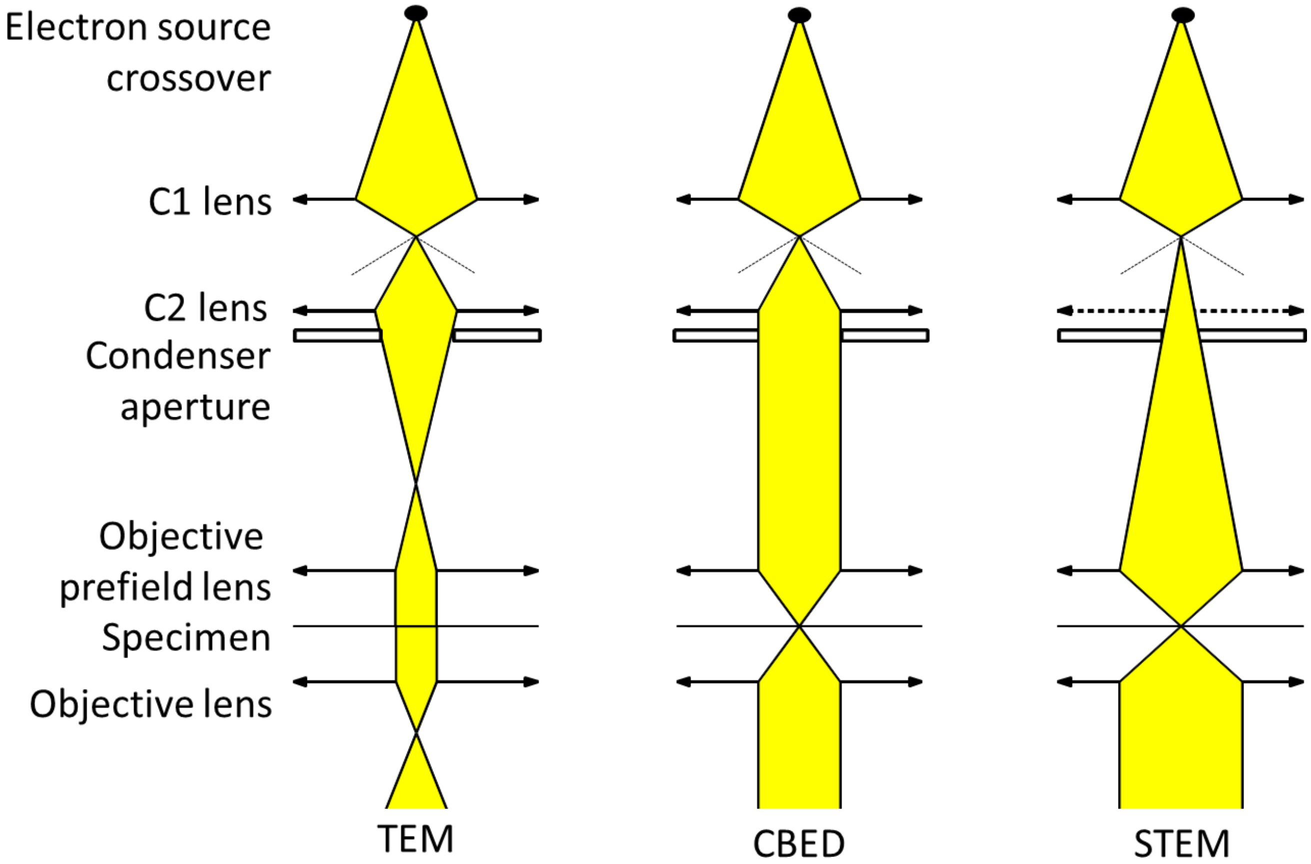
Provided by Jun Yamasaki of Nagoya University, Japan

Phospholipids, Douglas L. Dorsett

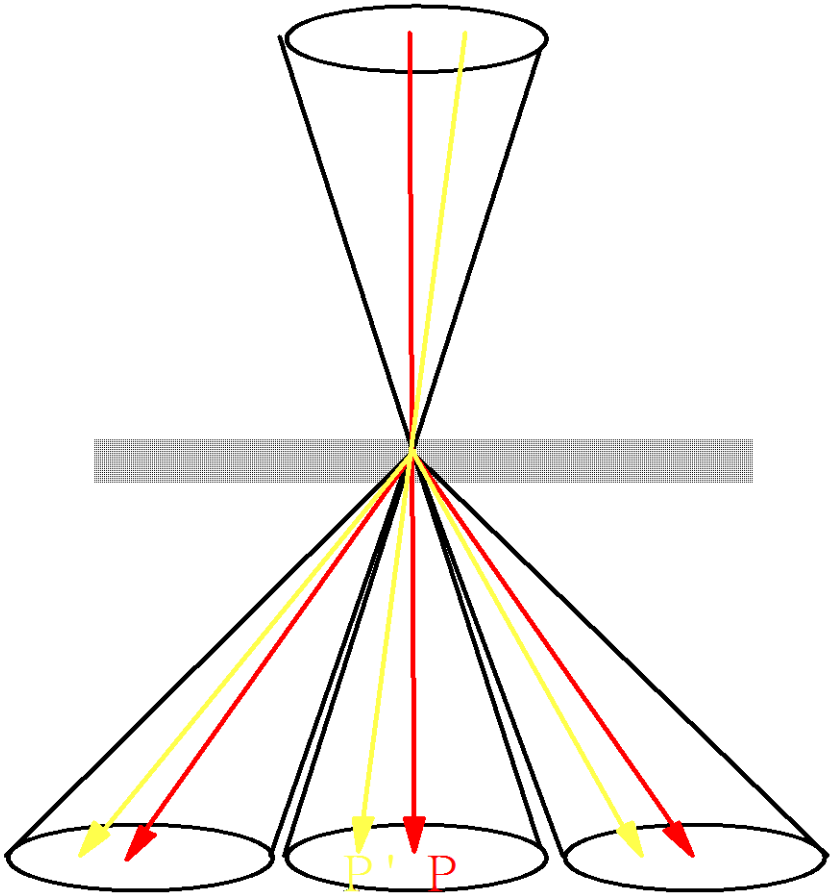


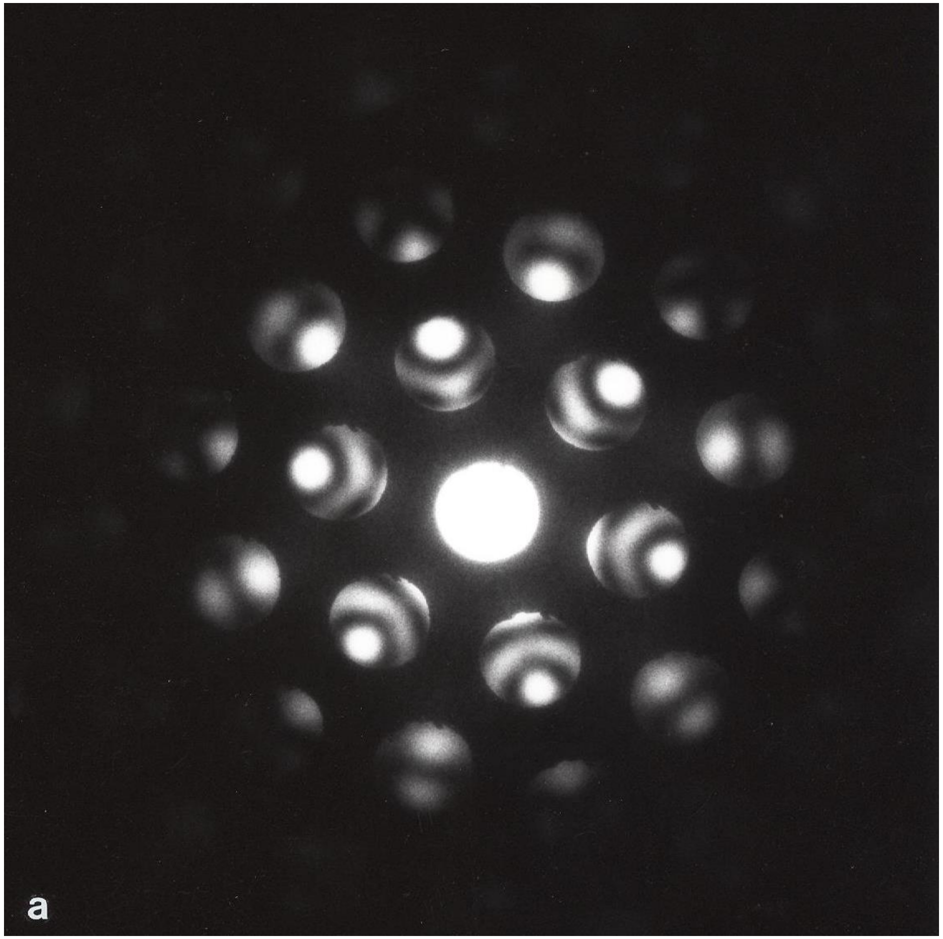
The use of condenser-objective lens

Philips EM400T



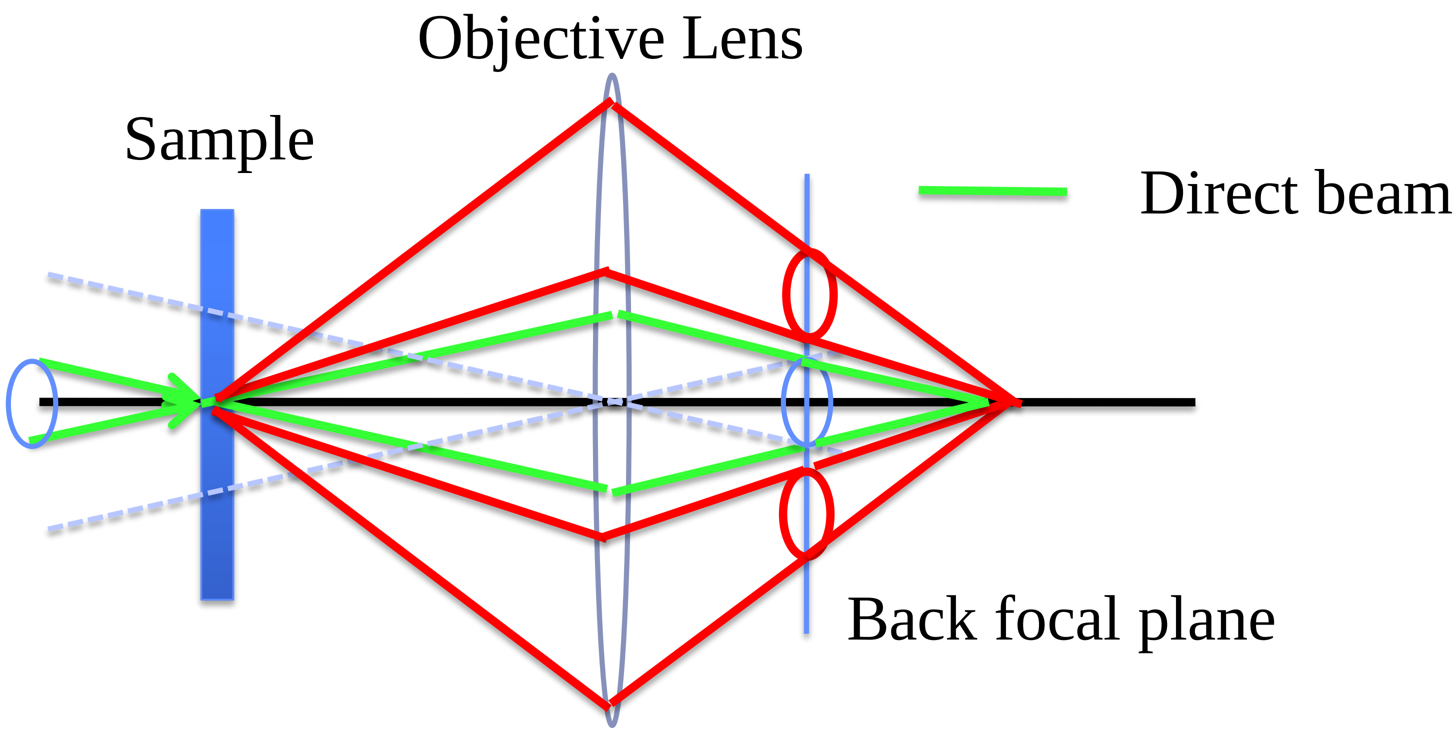
Convergent-beam electron diffraction



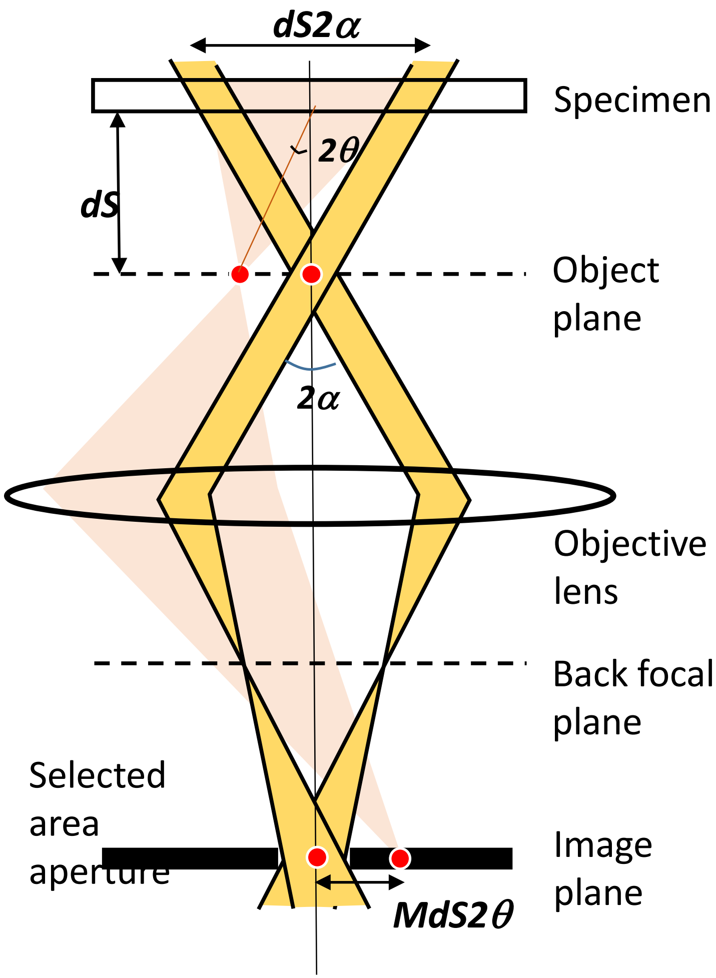


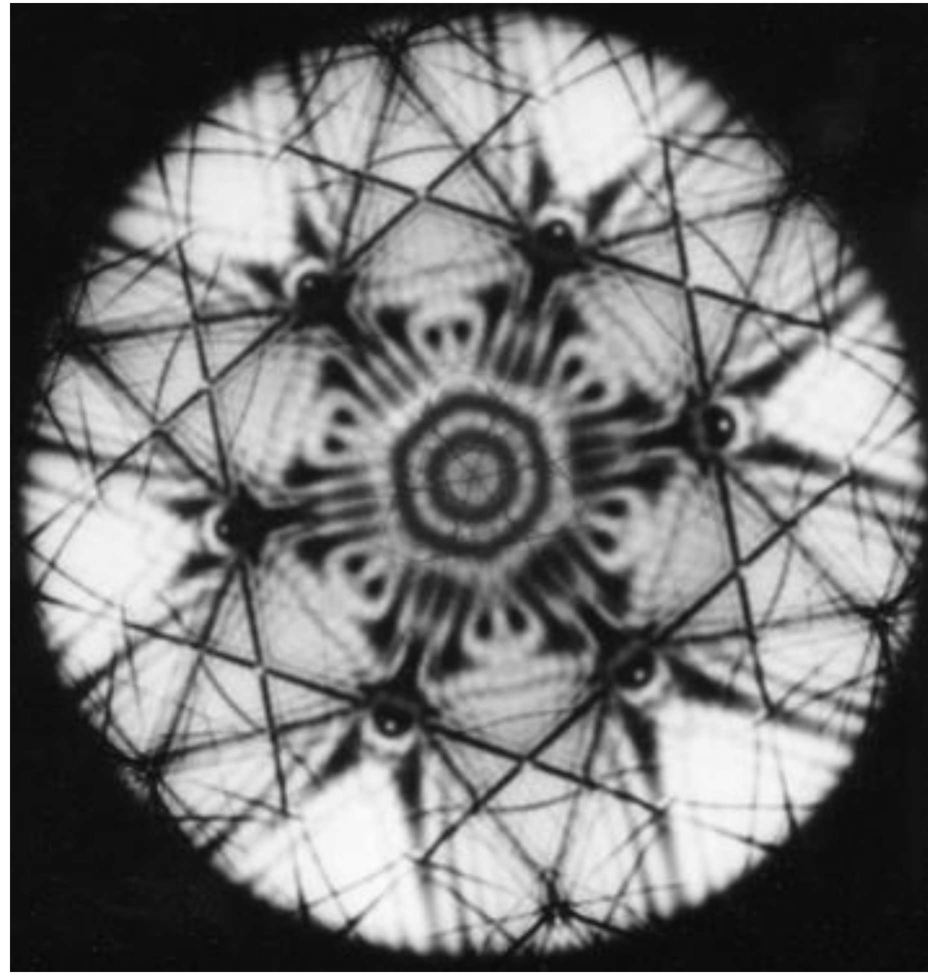
BeO CBED pattern recorded at 100 kV

Lens Action for CBED



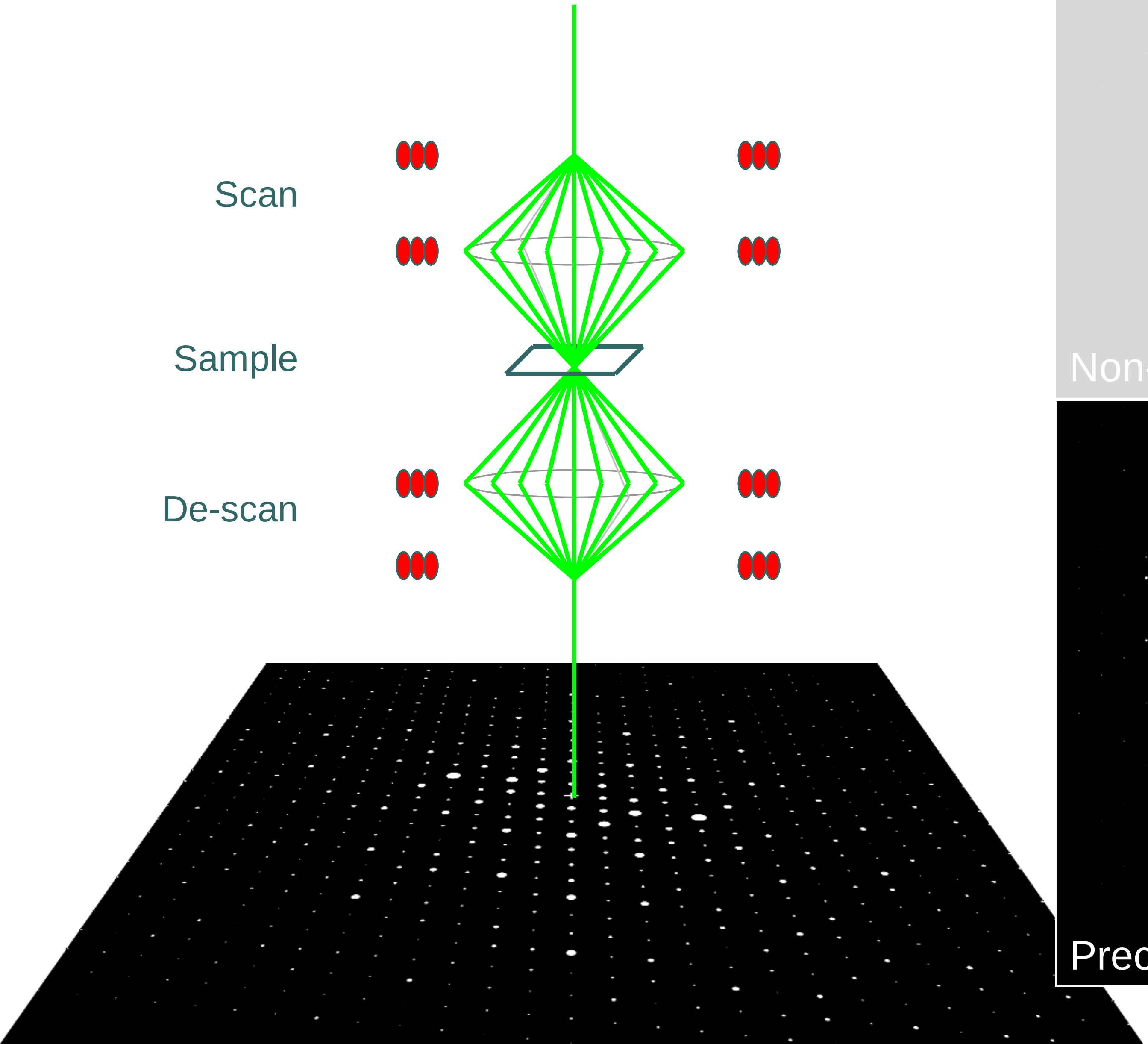
Large Angle CBED

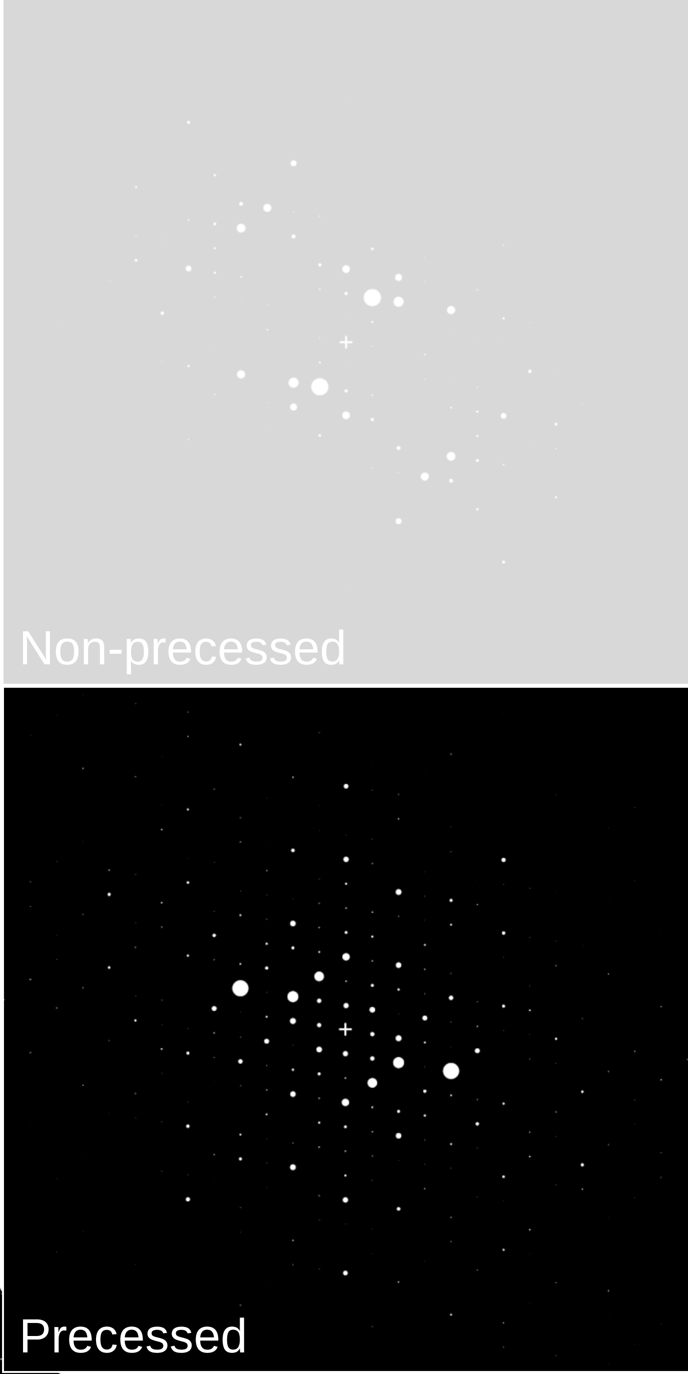




Si <111> 120keV, From John Steeds, Bristol

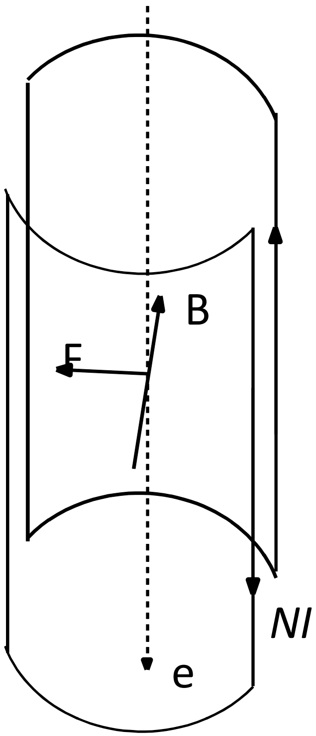
Vincent-Midgley Precession Method

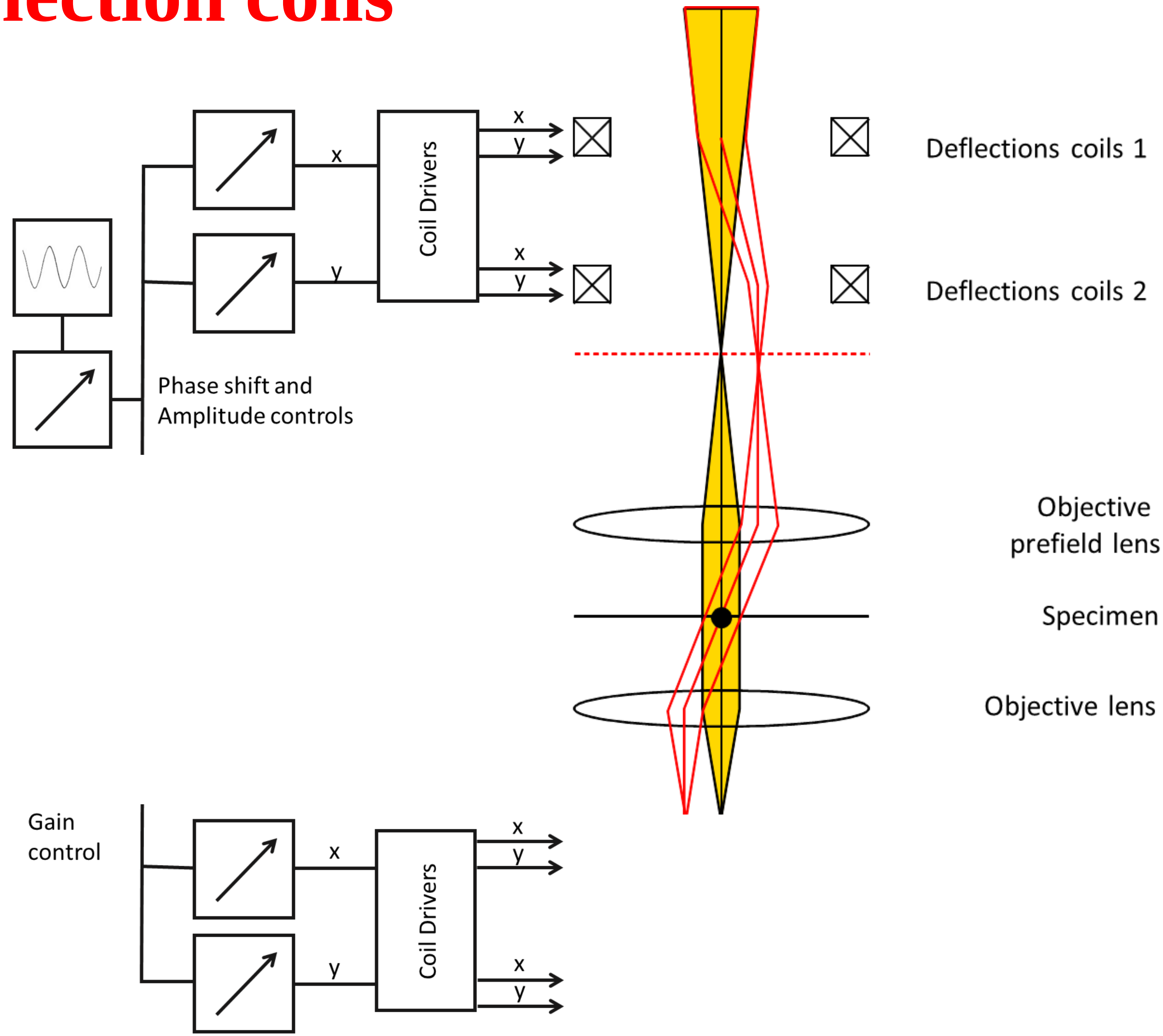




From Chris Own

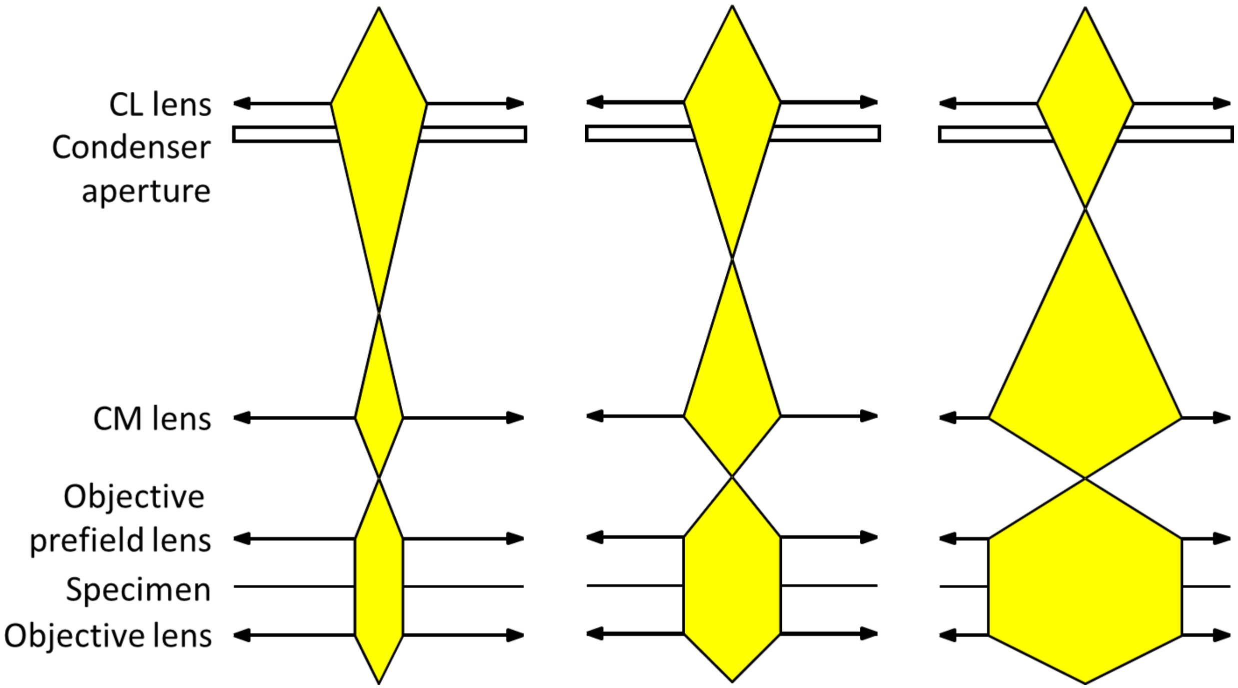
Deflection coils

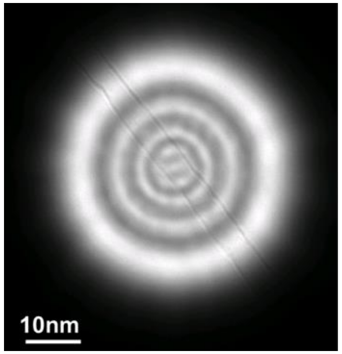




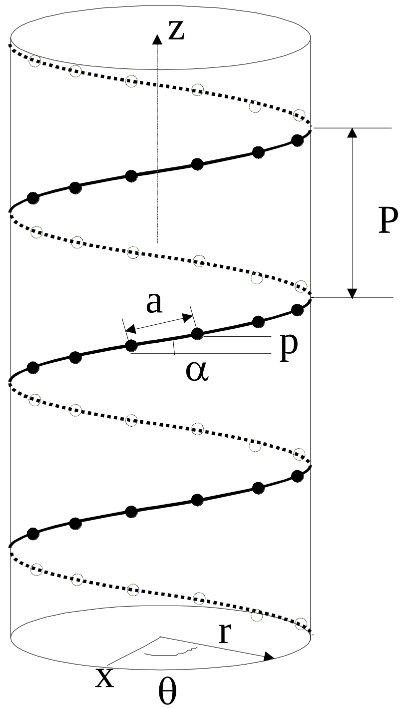
Nanoarea electron diffraction

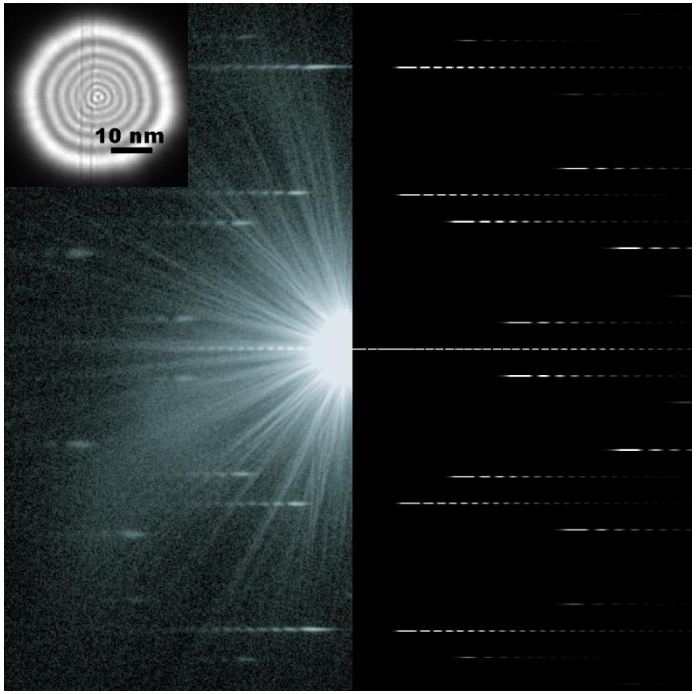
JEOL 2010F

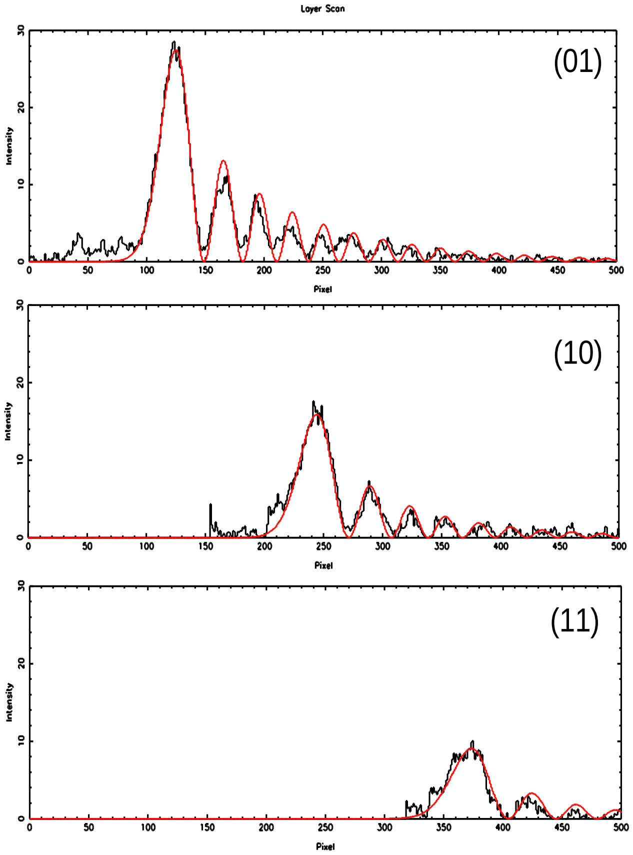




Diffraction of A Single Carbon Nanotube







D =34.4 Å and α=19.53°

Gao et al, APL, 2003 and Zuo et al. Z. Krist. 2008

Au trisoctahedra

Table 1. Direction and Angles of Diffraction Streaks for the Small TOH in Figure 3

reflection	points to reflection	facet direction	angle between planes	angle from image/ calculation	facets direction from image
$(3\ \overline{3}\ \overline{1})$	$(\overline{1}19)$	$[\overline{4}410]$	65.0°		
	$(\overline{6}6\overline{2})$	$[\overline{9}9\overline{1}]$			
$(3\ \overline{3}\ \overline{1})$	$(\overline{1}19)$	$[\overline{4}410]$	54.7°	55.4° (54.7°)	$[\overline{4}410]$
	$(4\overline{4}2)$	$[1\overline{1}3]$			$[1\overline{1}3]$
$(0\ 0\ \overline{4})$	$(1\overline{1}\overline{1})$	$[1\overline{1}3]$	53.2°		
	$(\overline{6}612)$	$[\overline{6}616]$			
$(2\ \overline{2}\ \overline{4})$	$(\overline{3}3\overline{5})$	$[\overline{5}5\overline{1}]$	24.9°		
	$(\overline{5}5\overline{1})$	$[\overline{7}73]$			
				61° (64.8°)	$[1\overline{1}0]$
					$[1\overline{1}3]$
				40° (43°)	$[1\overline{1}\overline{3}]$
					$[\overline{2}2\overline{8}]$

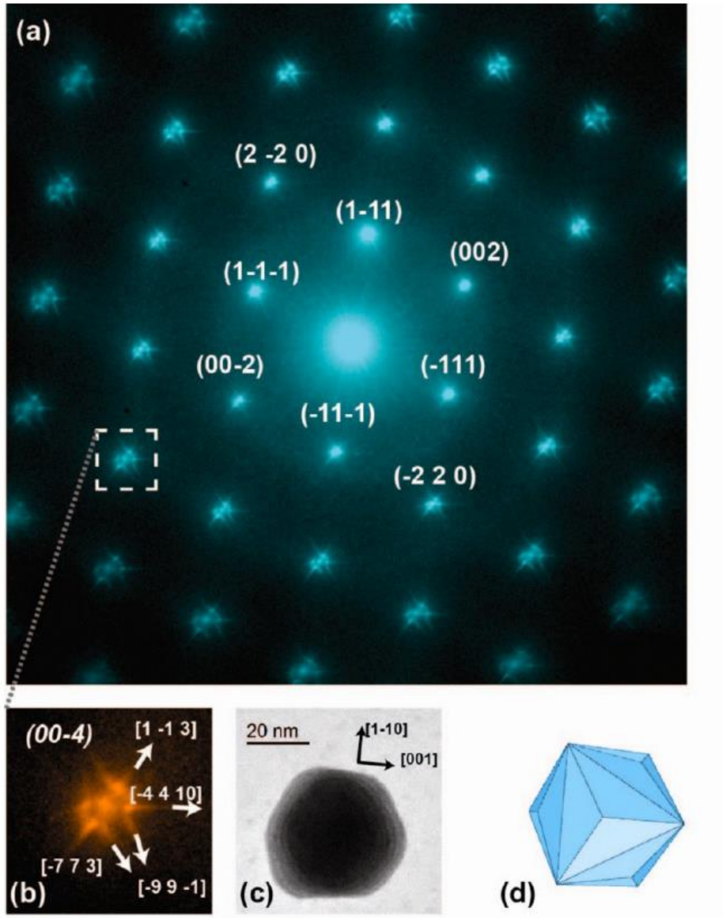
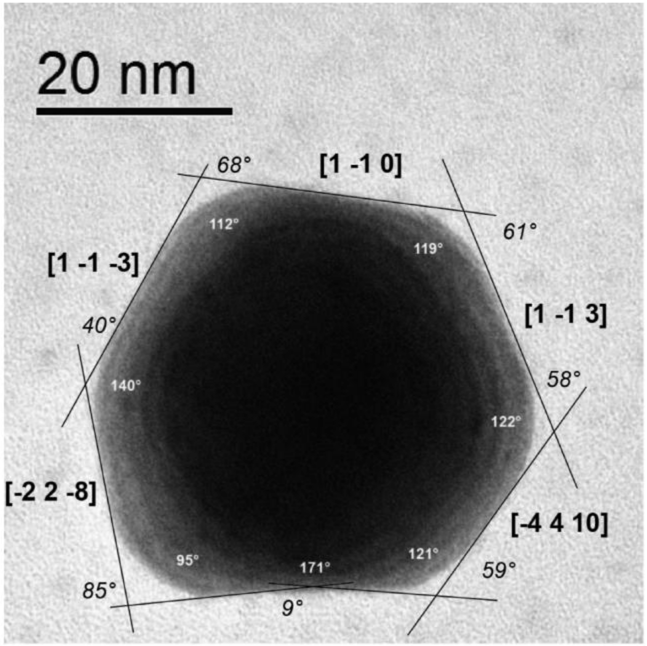
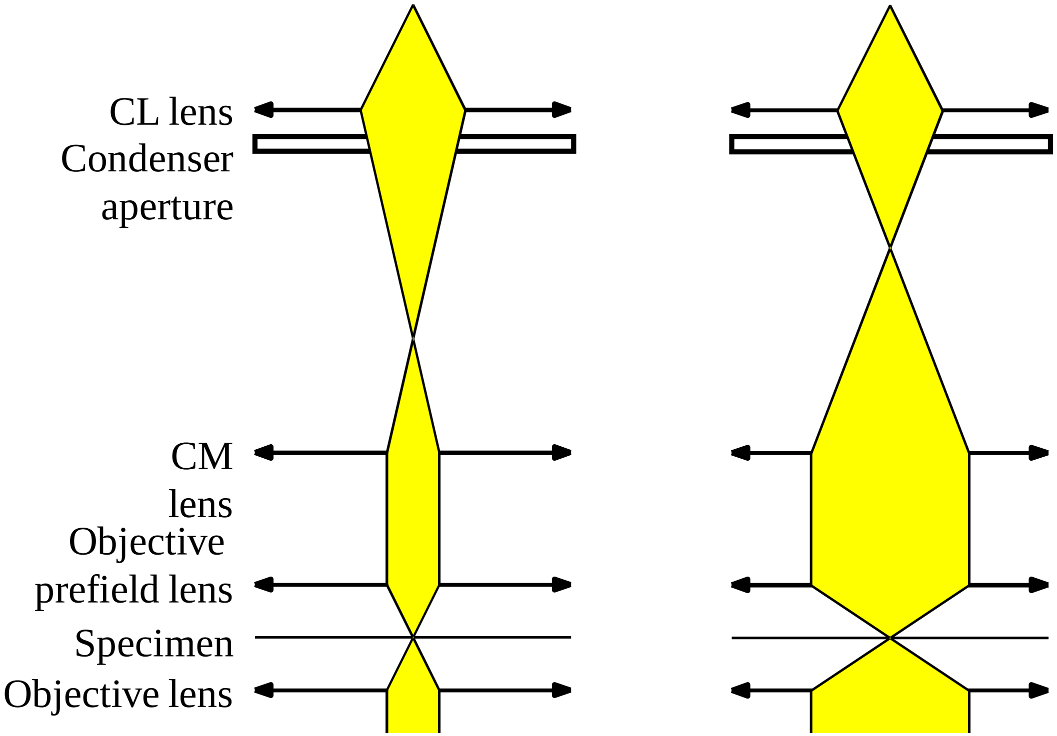
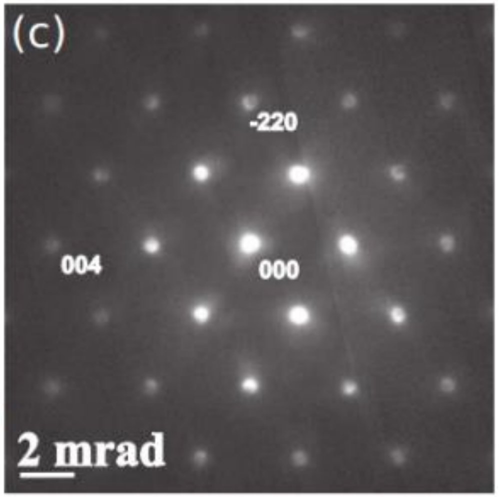


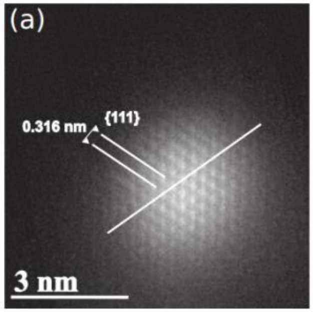
Figure 3. (a) NED pattern of a 44 nm TOH crystal oriented on the [110] zone axis. The streaks are normal to the planes. (b) Magnified view of $(00\overline{4})$ reflection showing streaks measured from this and other reflections. (c) TEM image of the TOH crystal. (d) Computer generated image of the TOH crystal.



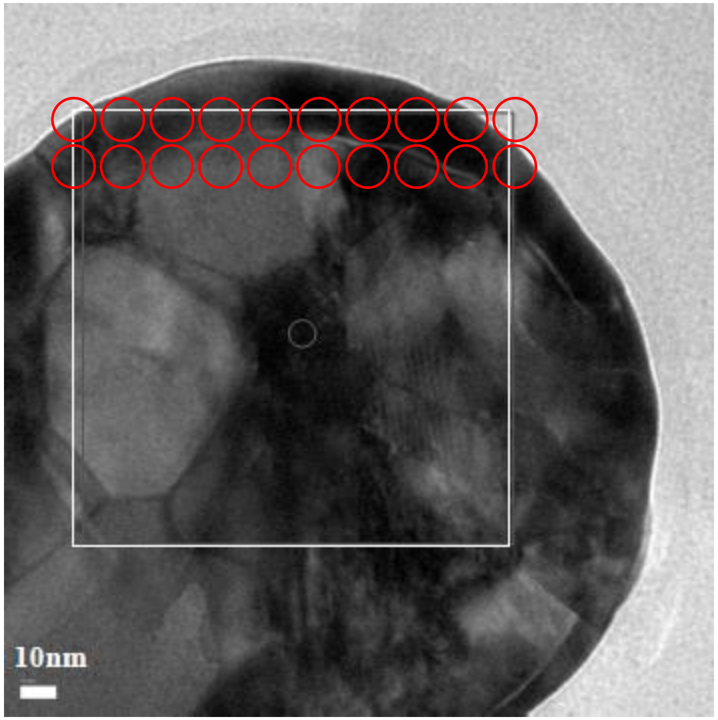
Electron Nanodiffraction



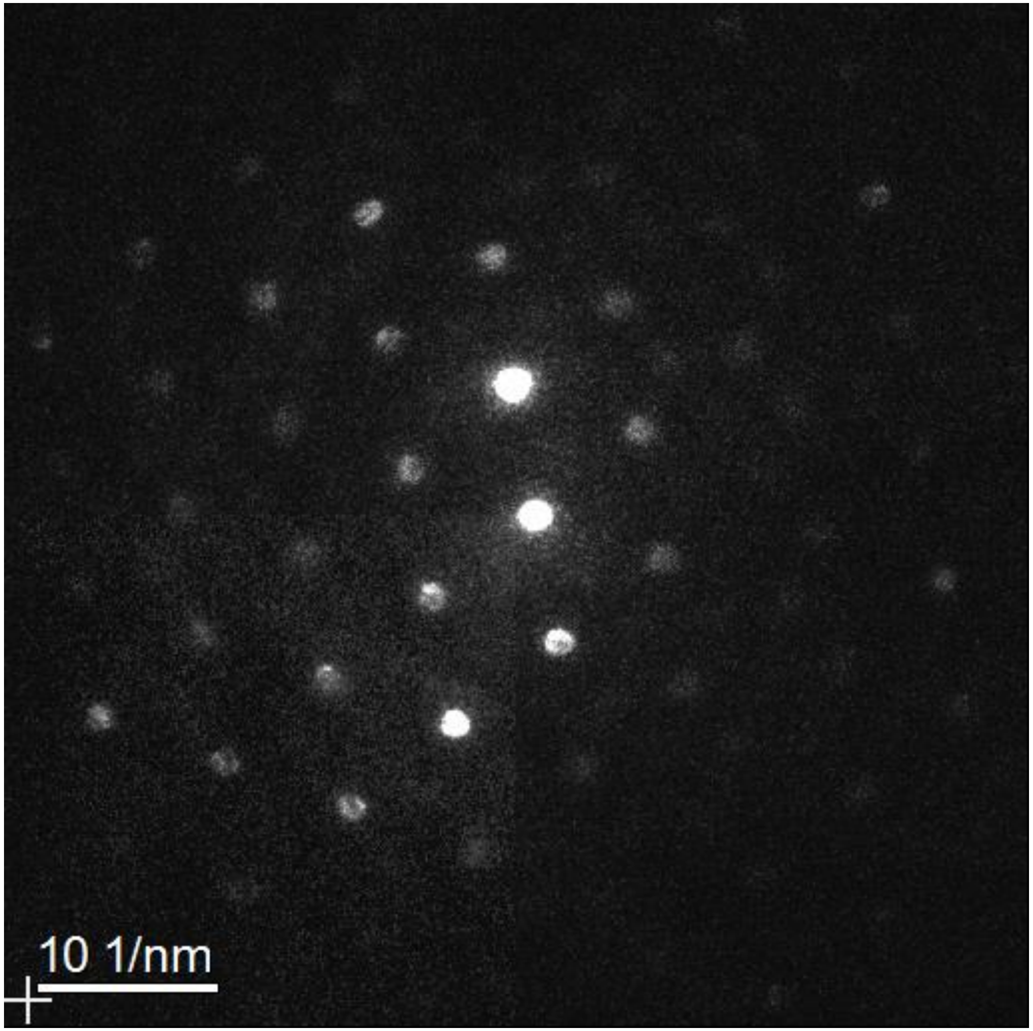




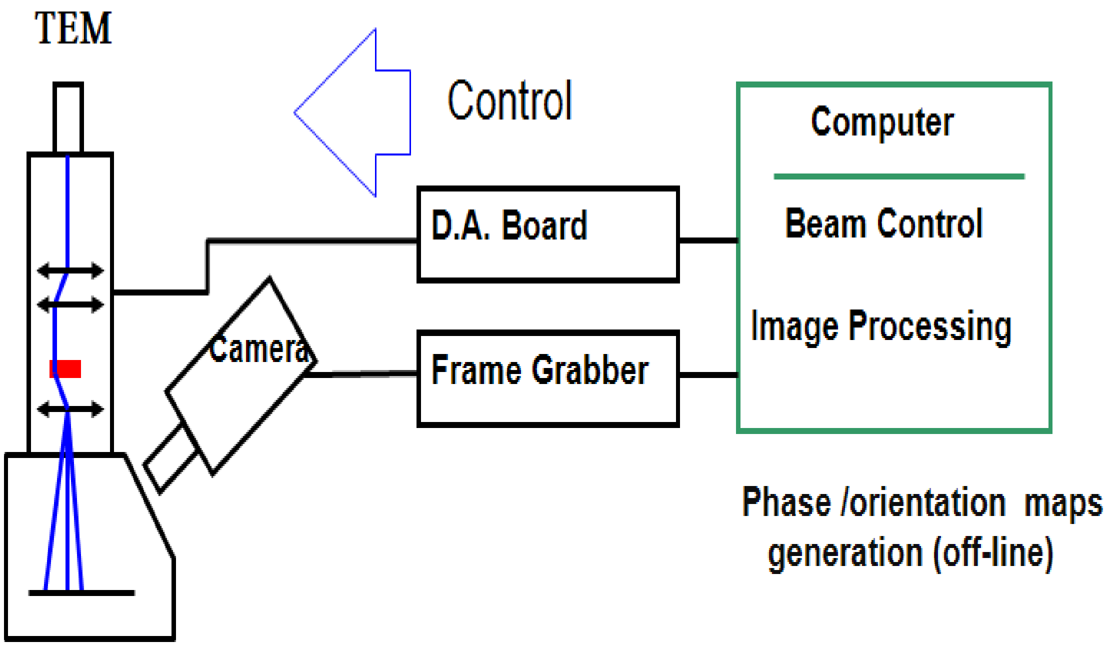
Scanning Electron Nanodiffraction

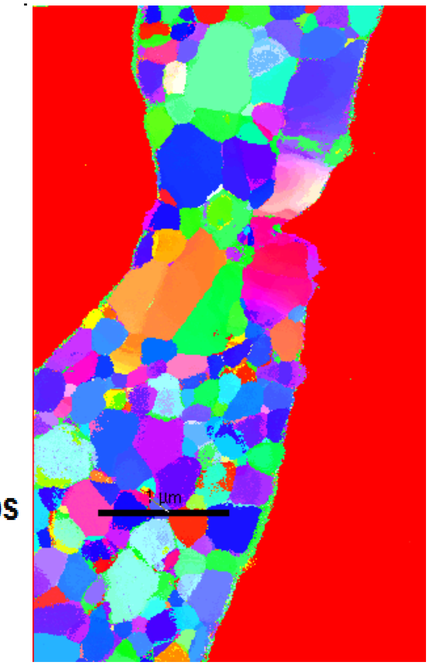


Cross-section of fabricated Au wire



ASTAR (EBSD like capability in TEM)



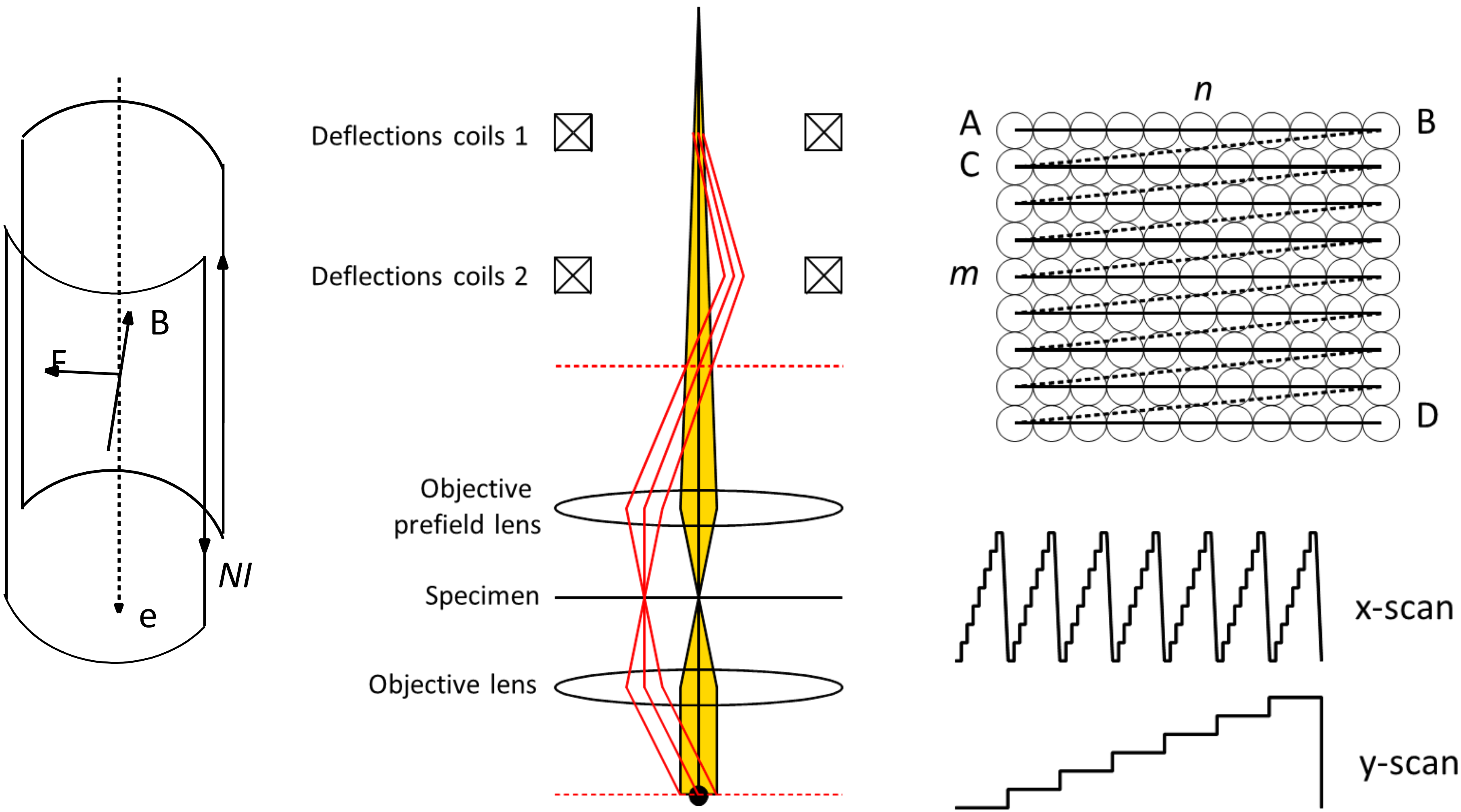


CM20 UTwin LaB6
Prof . S. Godet
ULB Brussels

Demonstrated Resolution: ***15 nm for LaB6***
~1 nm for FEG

NanoMEGAS
Advanced Tools for electron diffraction

nanocrystallography.research.pdx.edu/media/cms.../BerlinASTARII.ppt



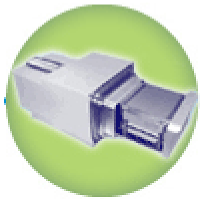
B
F
NI
e
Deflections coils 1
Deflections coils 2
Objective
prefield lens
Specimen
Objective lens
n
A
B
C
m
D
x-scan
y-scan

Low Dose Scanning Electron Nanodiffraction (SEND)

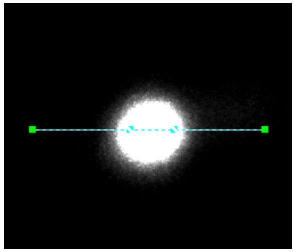
JEOL 2100LaB_6 80-200 kV

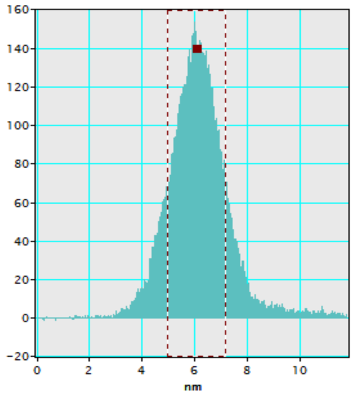
Gatan Ultrascan
2kx2k CCD
Camera

+



Fiber-optic coupled
14 µm square pixels



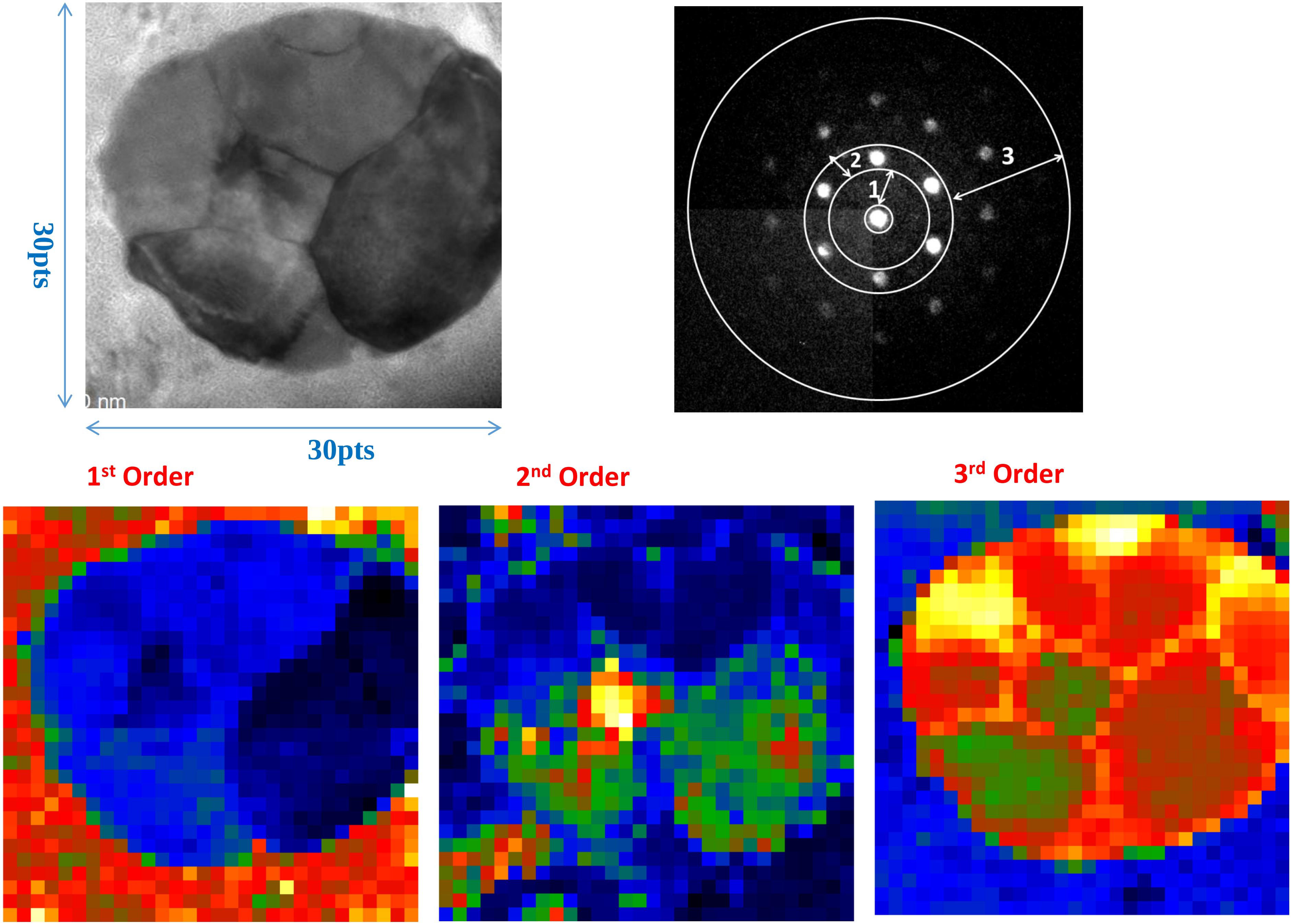


Demonstrated Resolution: ***2 nm for LaB6***

Advantage: Low Dose

I=0.1 pA or less

Kim, Kyou-Hyun; Xing, Hui; Zuo, Jian-Min; et al.
MICRON 71, 39-45, 2015



30pts
30pts
1
2
3
1st Order
2nd Order
3rd Order

Covered Topics

- Electron diffraction techniques
  - Selected area electron diffraction
  - Convergent beam electron diffraction
  - Large angle methods
  - Precession electron diffraction
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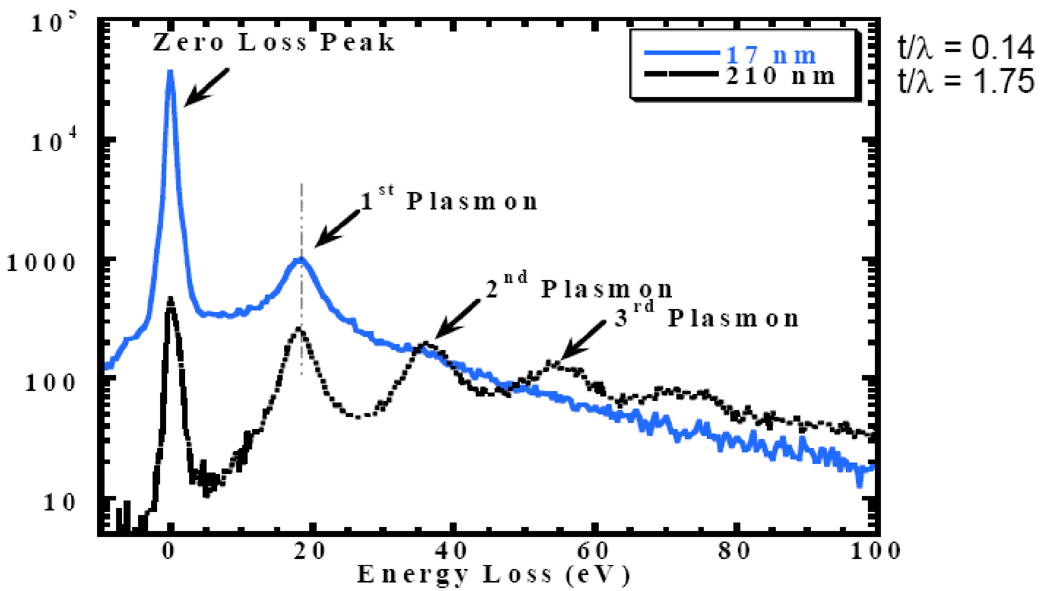
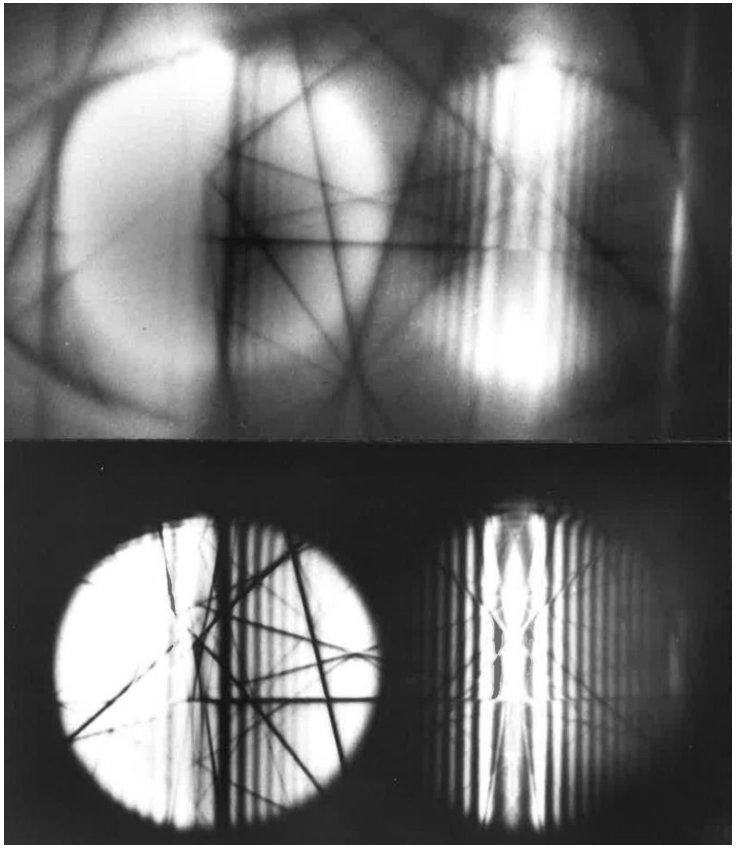
- Energy-filtering
- STEM, Ronchigram, Position averaged CBED
- Electron illumination
  - Beam current
  - Probe size
  - Coherence
- Electron detectors and radiation effects

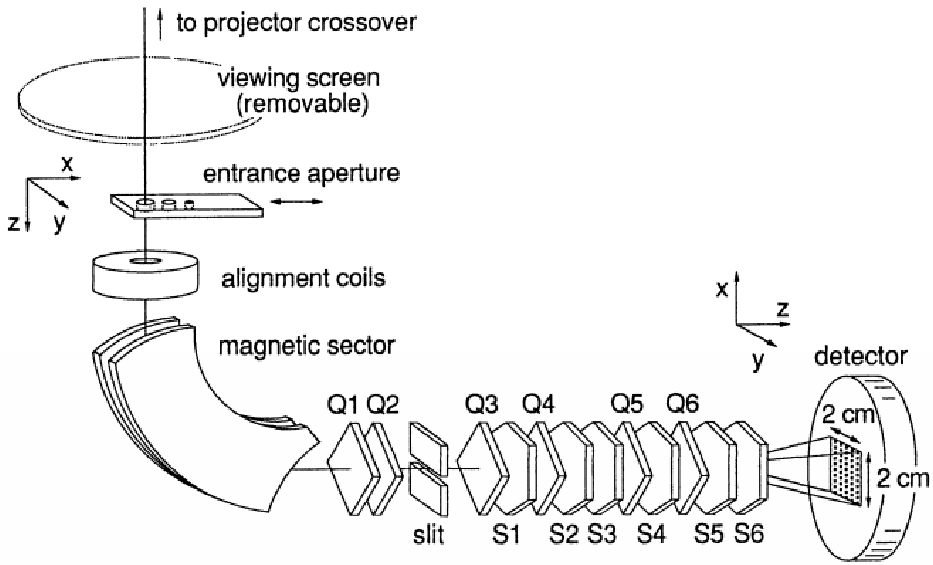
Energy Filtering



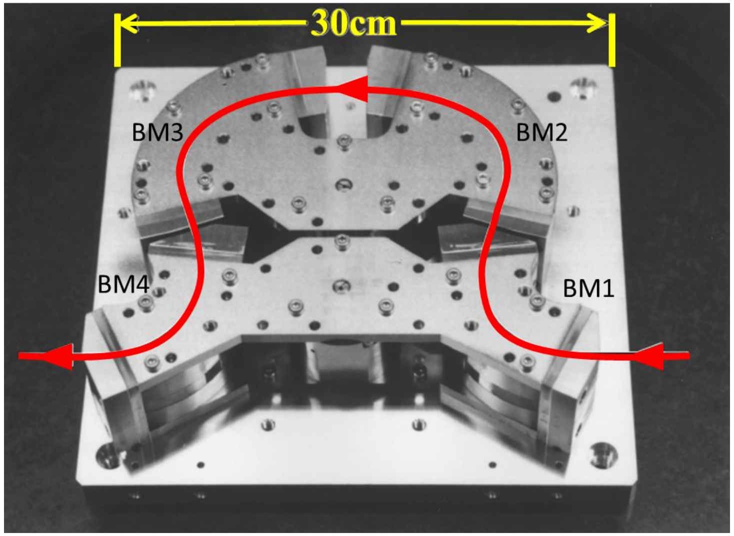
Silicon 100 kV



Top: Unfiltered
Bottom: Filtered

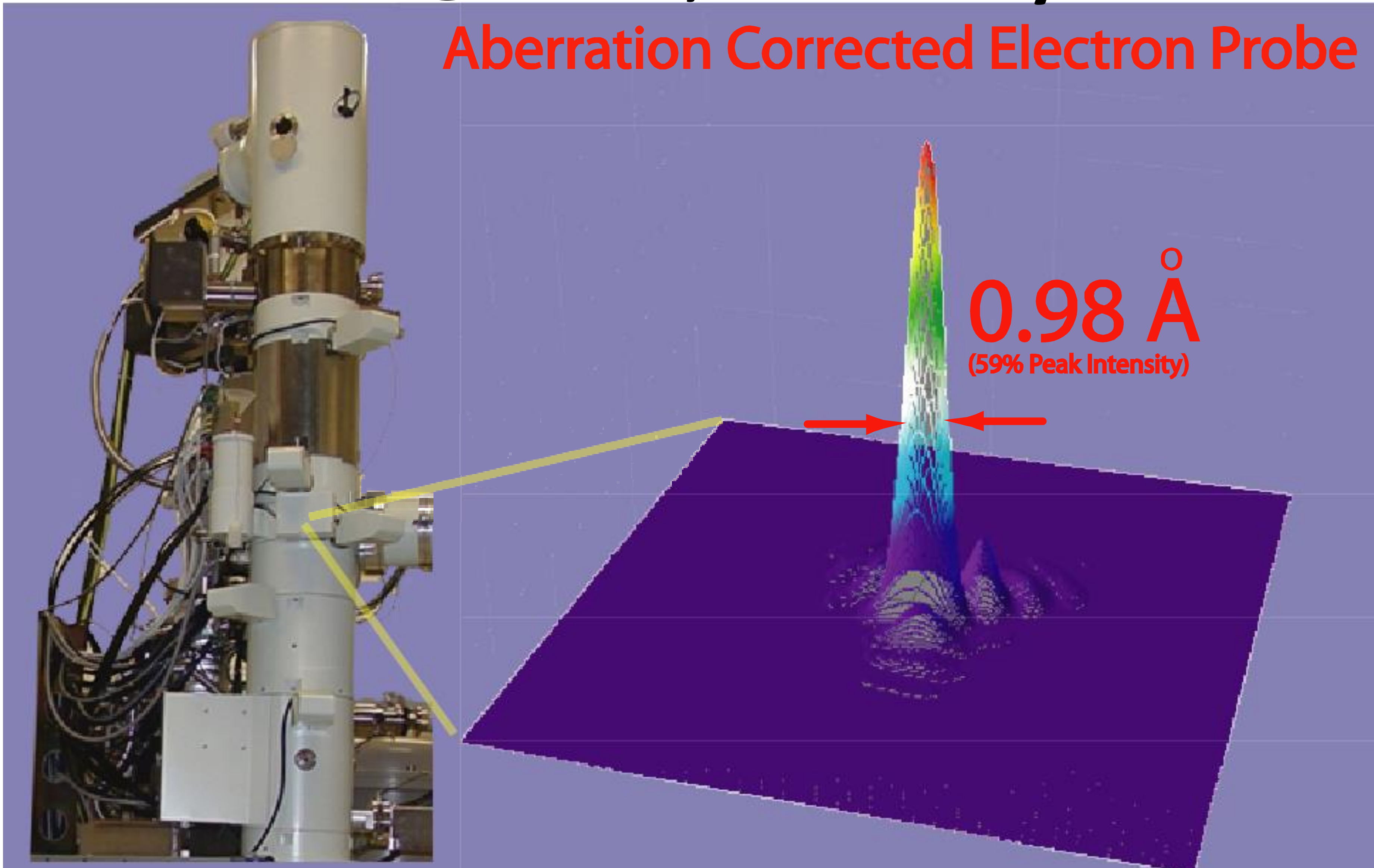


to projector crossover
viewing screen
(removable)
x
z
y
entrance aperture
alignment coils
magnetic sector
Q1 Q2
slit
Q3 Q4
Q5 Q6
S1
S2 S3
S4
S5 S6
x
z
y
detector
2 cm
2 cm

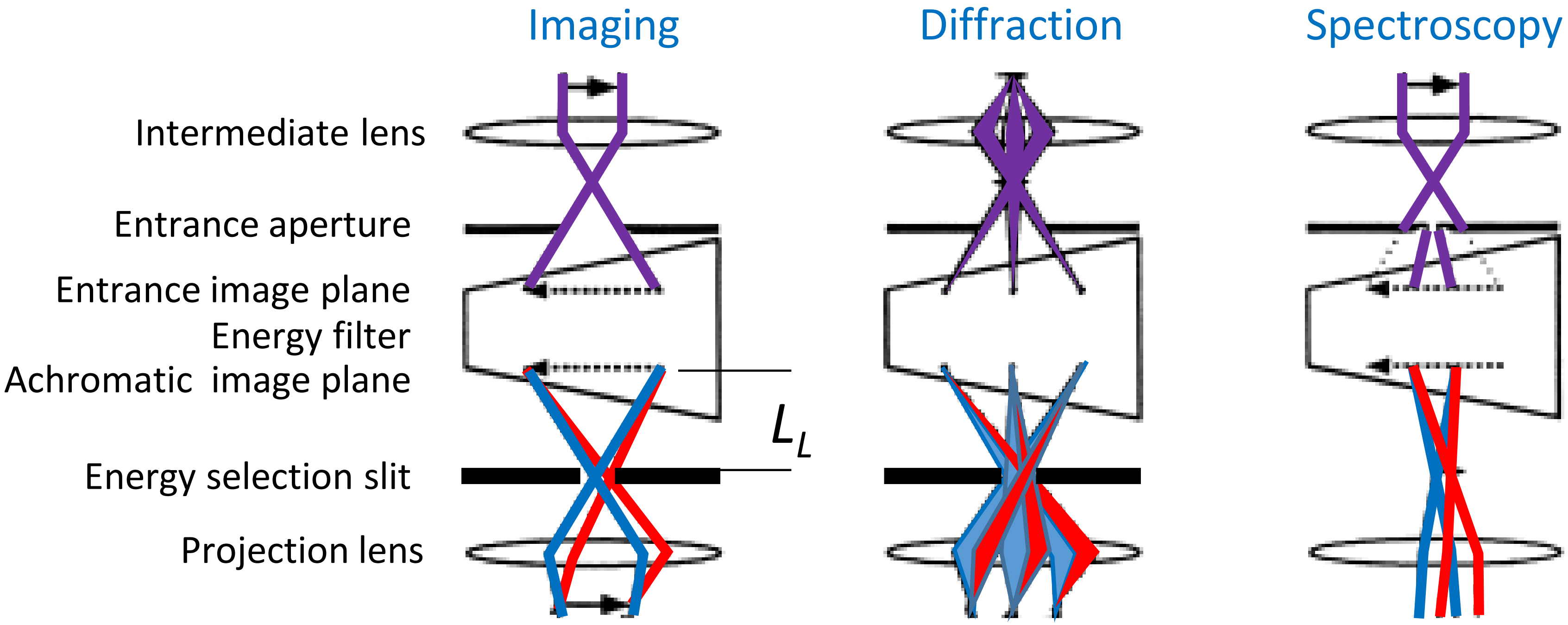


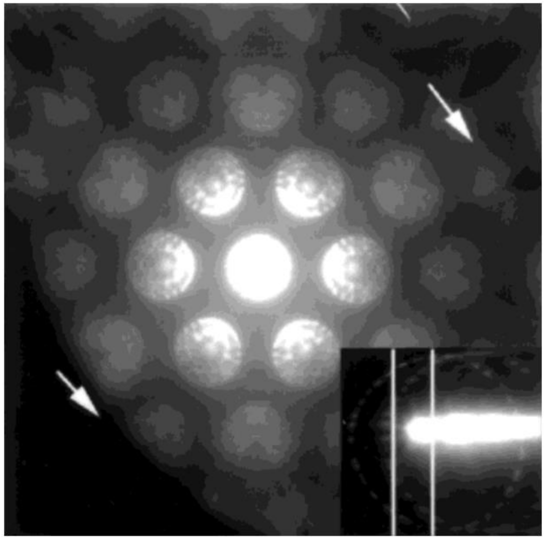
30cm
BM3
BM2
BM4
BM1

JEOL 2200FS @ CMM, University of Illinois

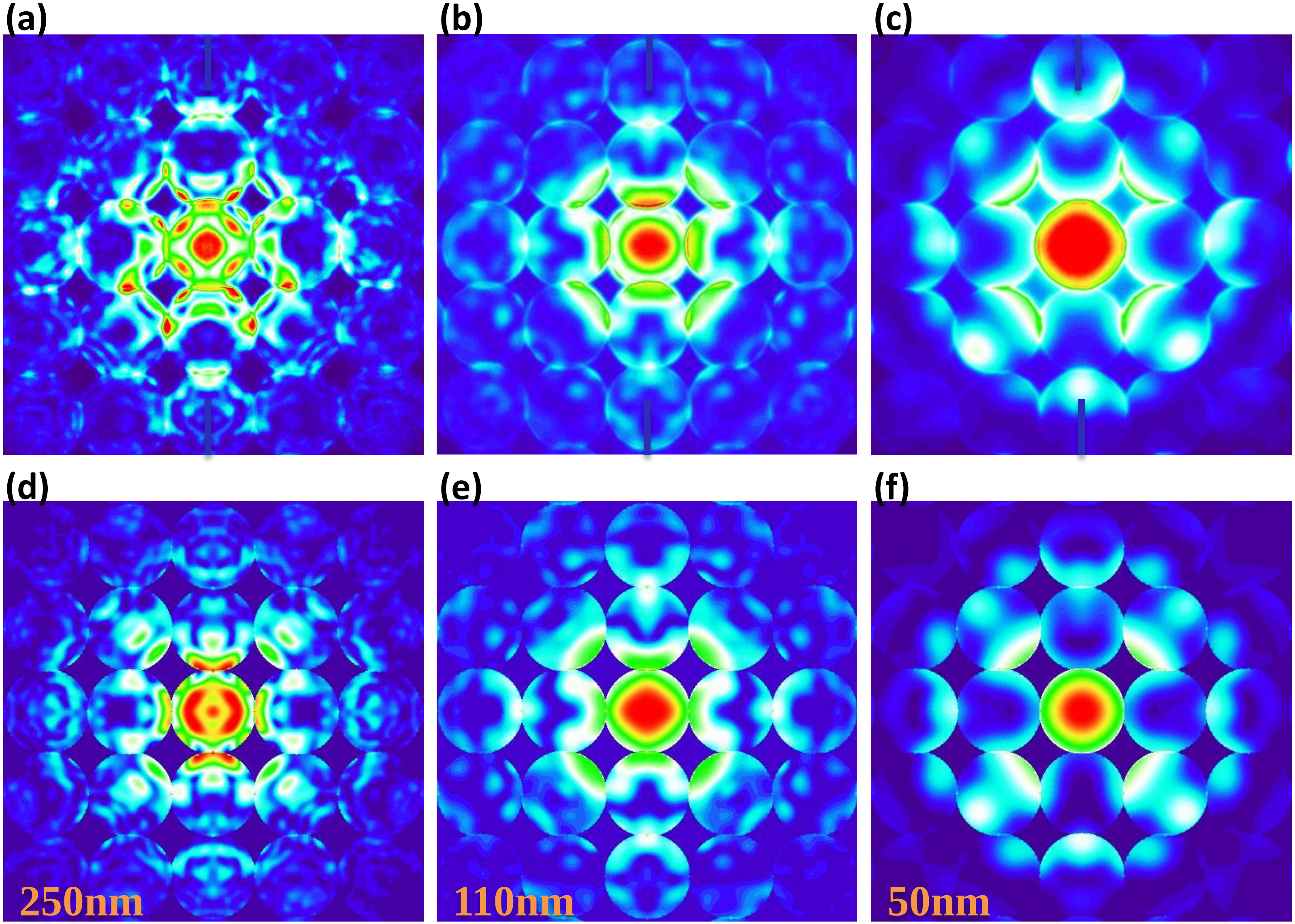


Installed since 2008


Imaging
Diffraction
Spectroscopy
Intermediate lens
Entrance aperture
Entrance image plane
Energy filter
Achromatic image plane
L_L
Energy selection slit
Projection lens



Energy-Filtered CBED of Ferroelectric $BaTiO_3$



Experiment

Atomic Model

Covered Topics

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  - **- Selected area electron diffraction**
  - **- Convergent beam electron diffraction**
  - **- Large angle methods**
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  - **- Nanoarea electron diffraction**
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- **Energy-filtering**
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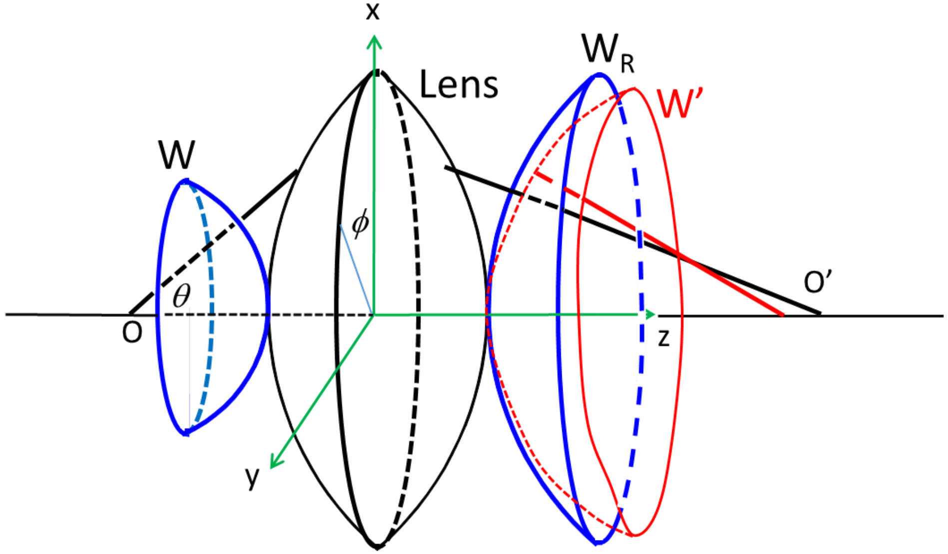
- **Electron detectors and radiation effects**

JEOL 2200FS @ CMM, University of Illinois

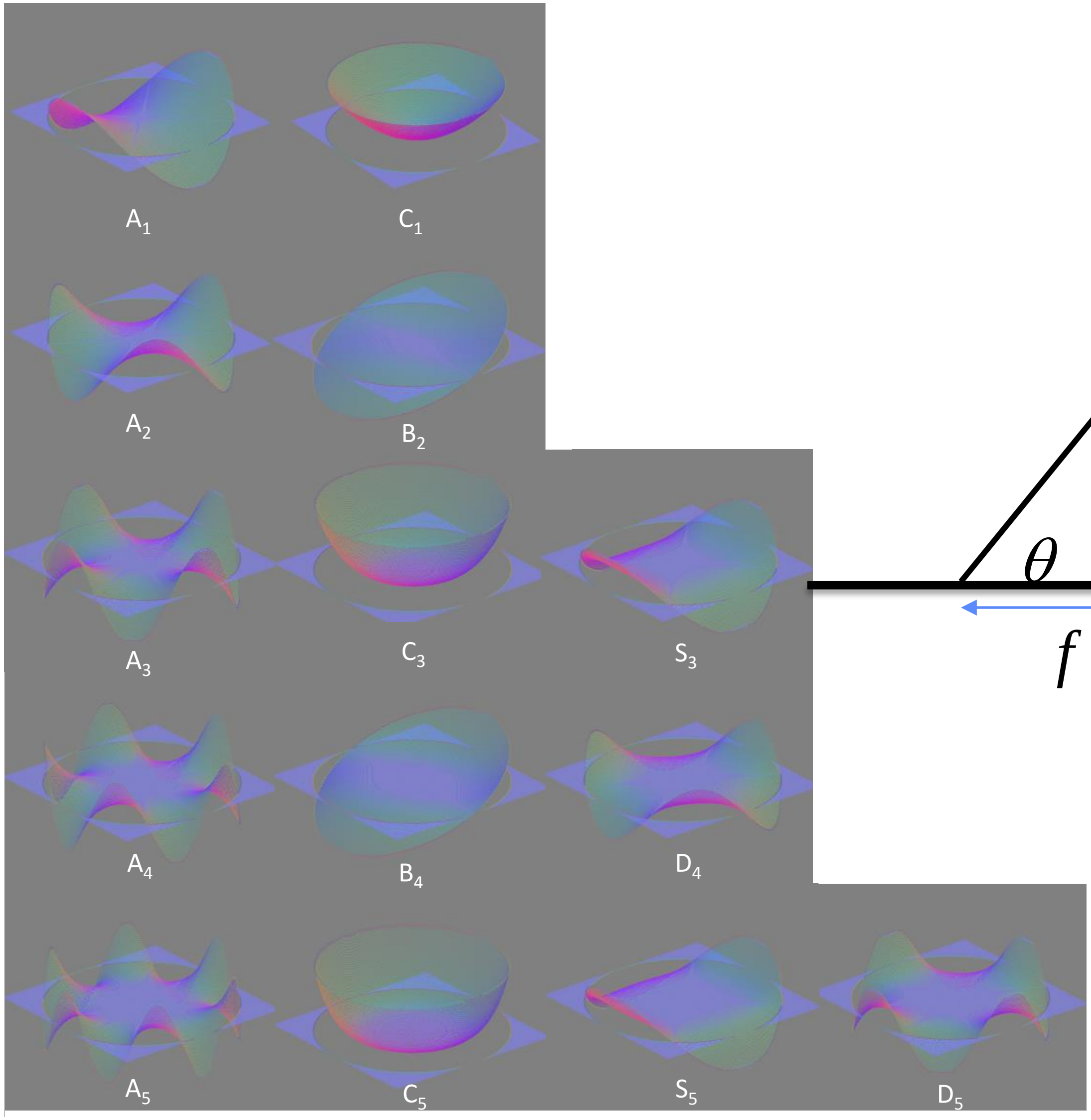
Aberration Corrected Electron Probe



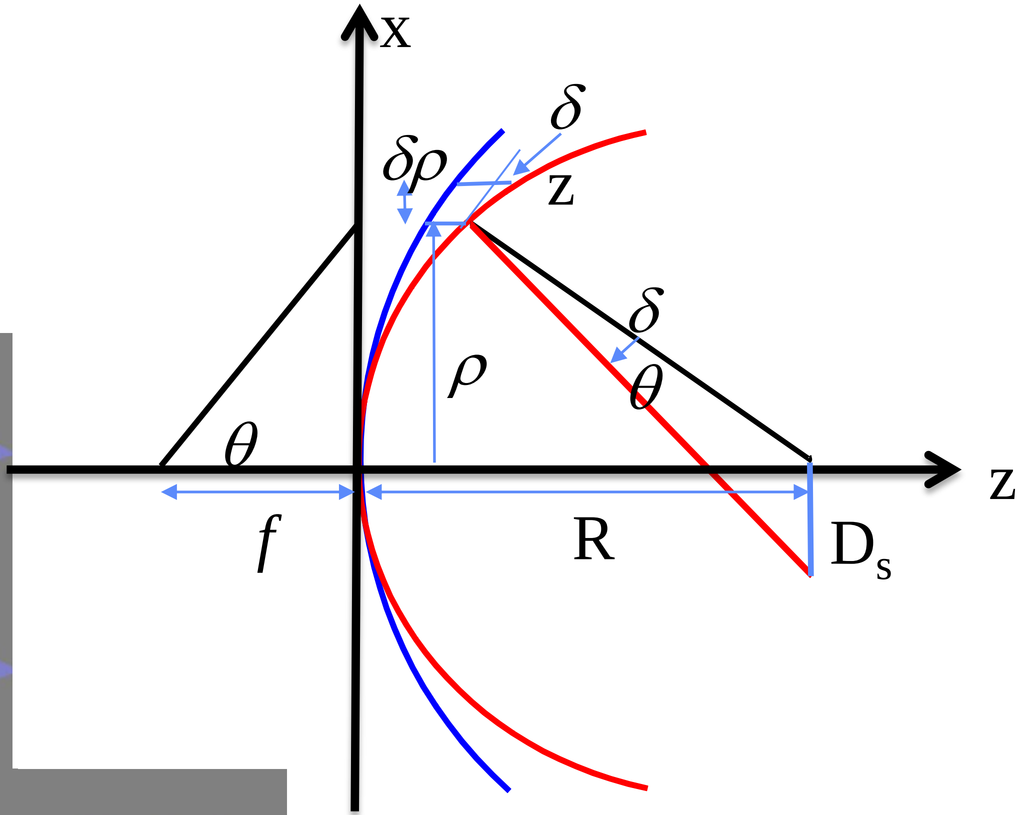
$$\chi(\omega,E)=\frac{2\pi}{\lambda}\mathrm{Re}\left\{\frac{1}{2}\varpi^2A_1+\frac{1}{2}\omega\varpi C_1+\frac{1}{3}\varpi^3A_2+\omega^2\varpi B_2+\frac{1}{4}\varpi^4A_3+\frac{1}{4}(\omega\varpi)^2C_3+\omega^3\varpi S_3\right.$$
$$\left.+\frac{1}{5}\varpi^5A_4+\omega^3\varpi B_4+\omega^4\varpi D_4+\frac{1}{6}\varpi^6A_5+\frac{1}{6}(\omega\varpi)^3C_5+\cdots\right\}$$

Aberrations = Phase

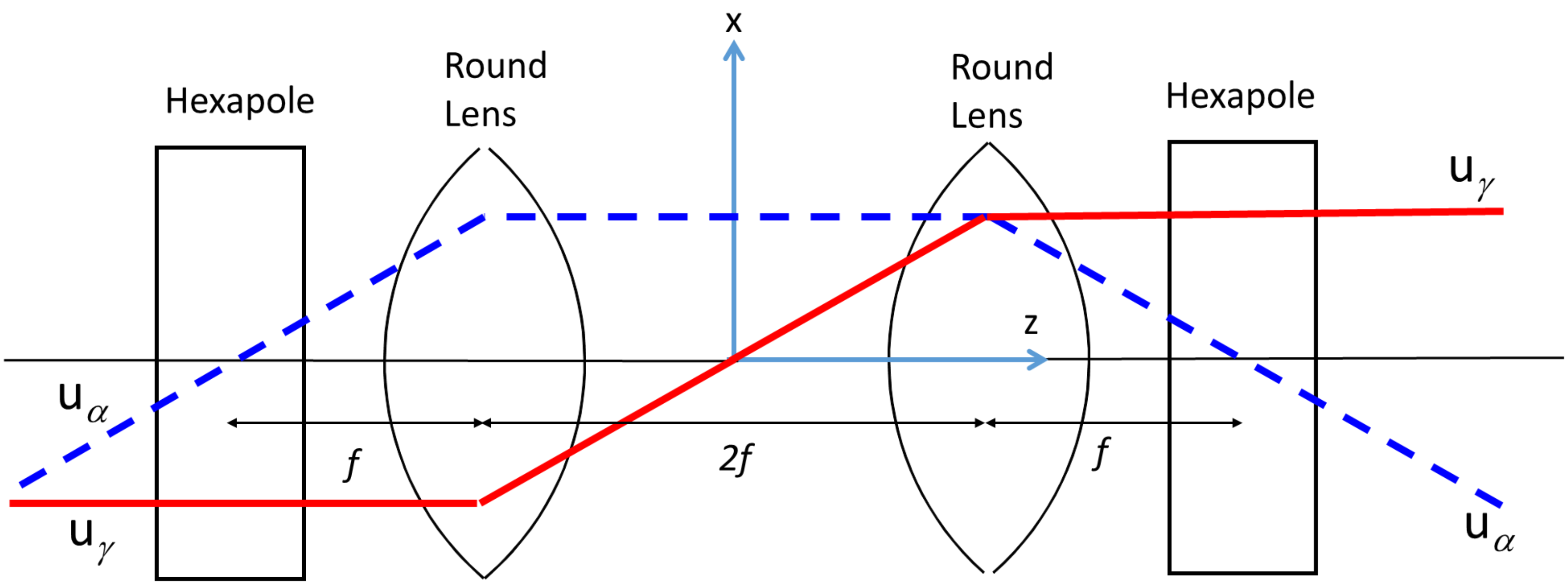
Aberration Coefficients



$$\chi(\theta) = 2\pi(C_s\,\theta^4 / 4)/\lambda$$

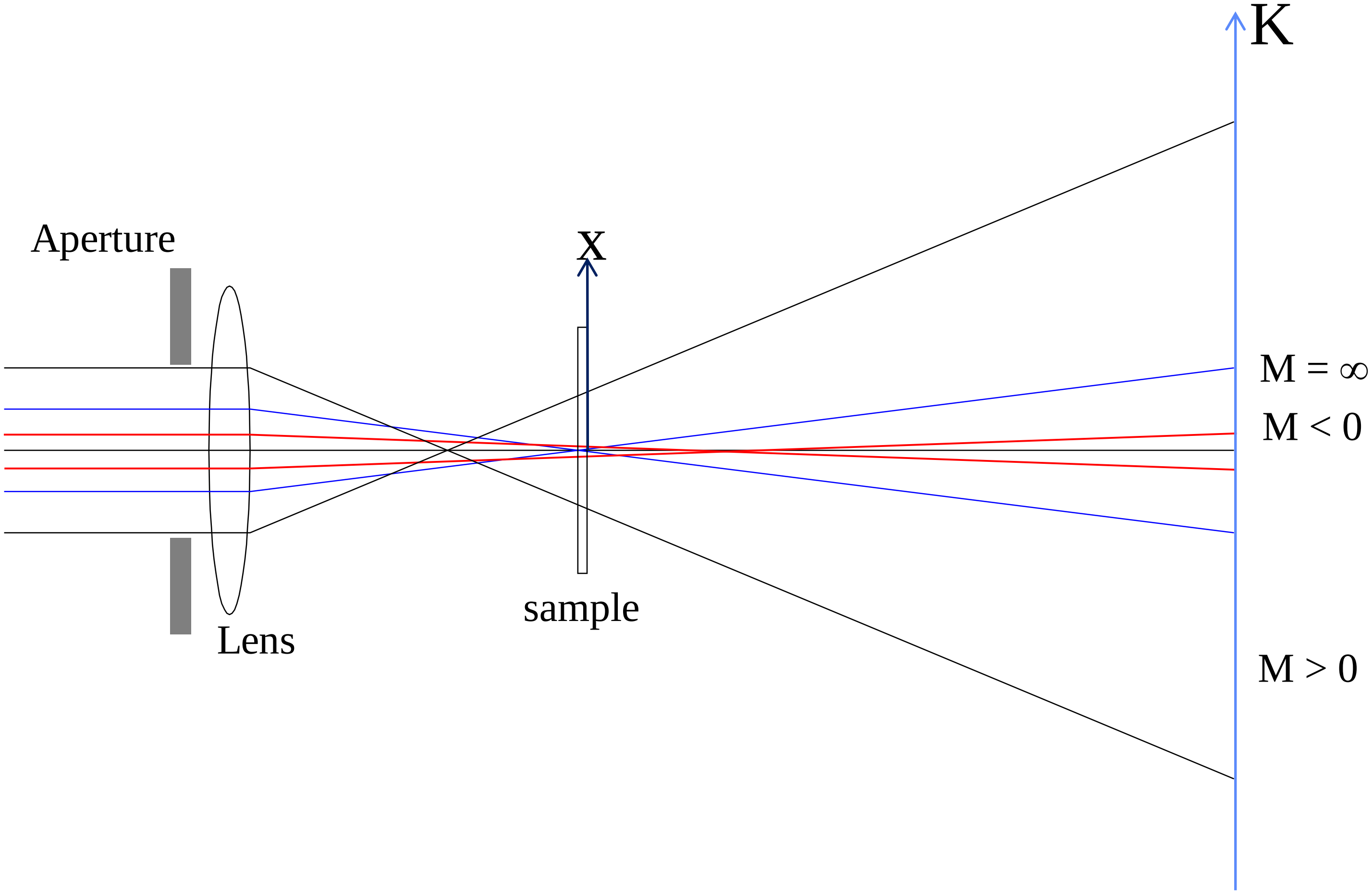


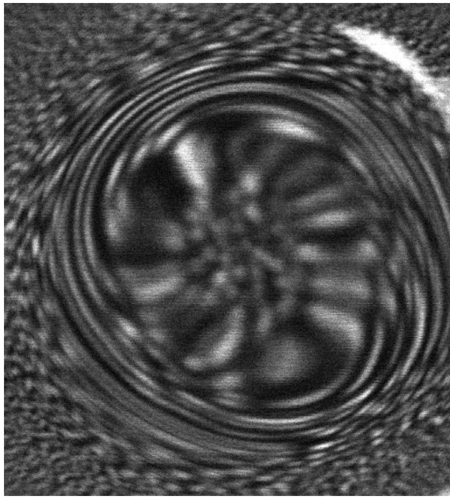
Aberration Corrector



$$C_S = -3\left|\eta\Psi_{3s}\right|^2 f^4 L^3 < 0$$

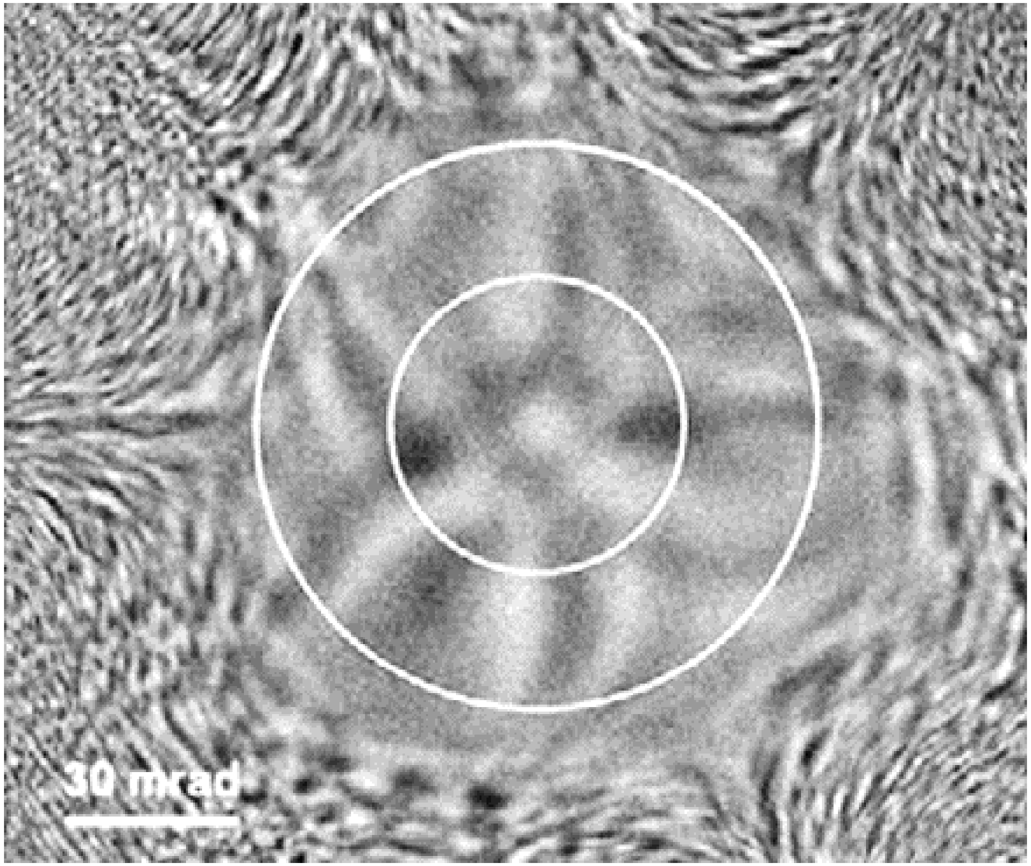
Ronchigram



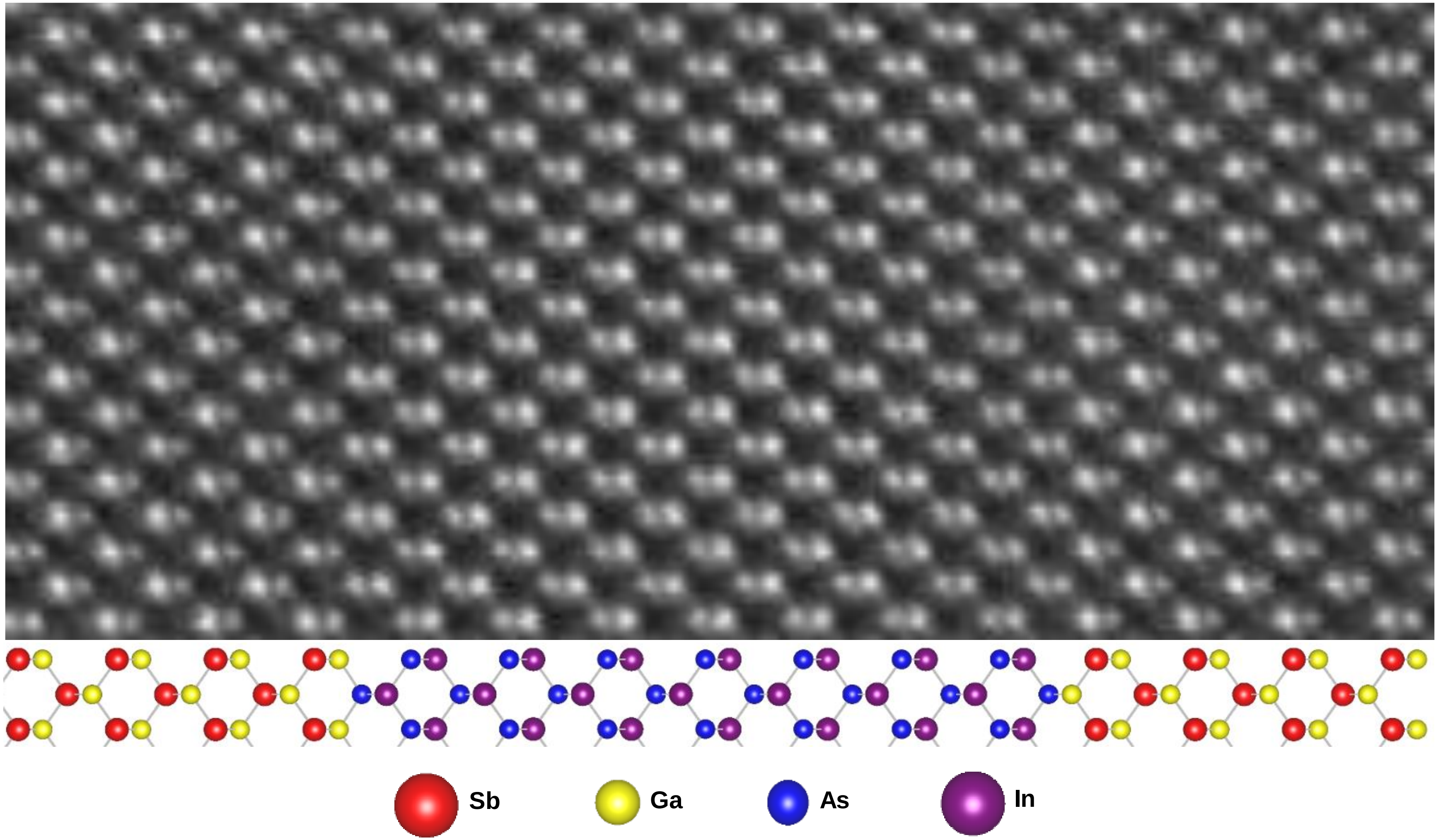


Higgs & Krivanek,
EMSA 1981

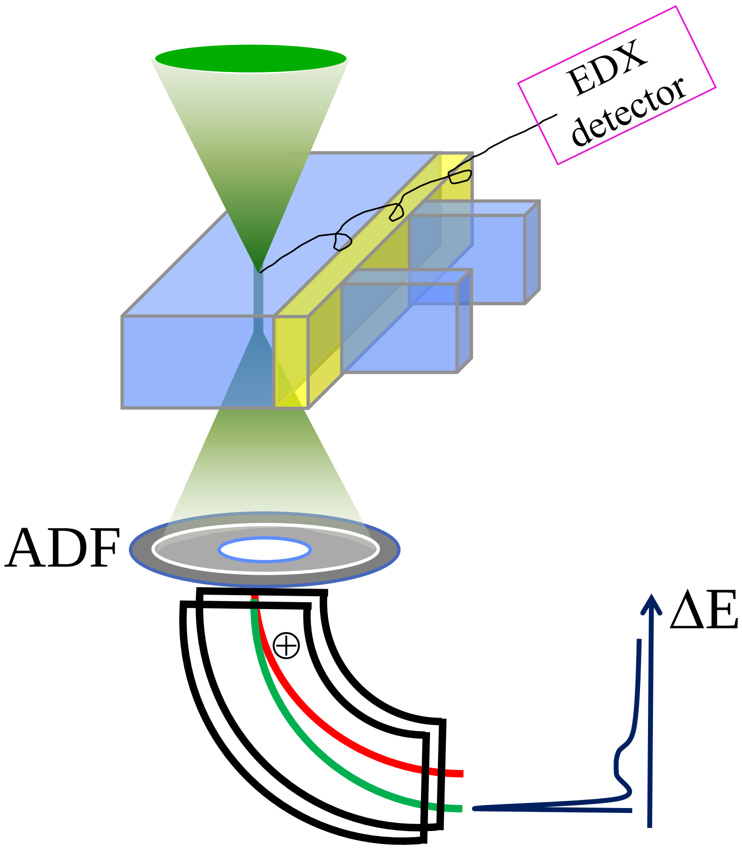
Ronchigram

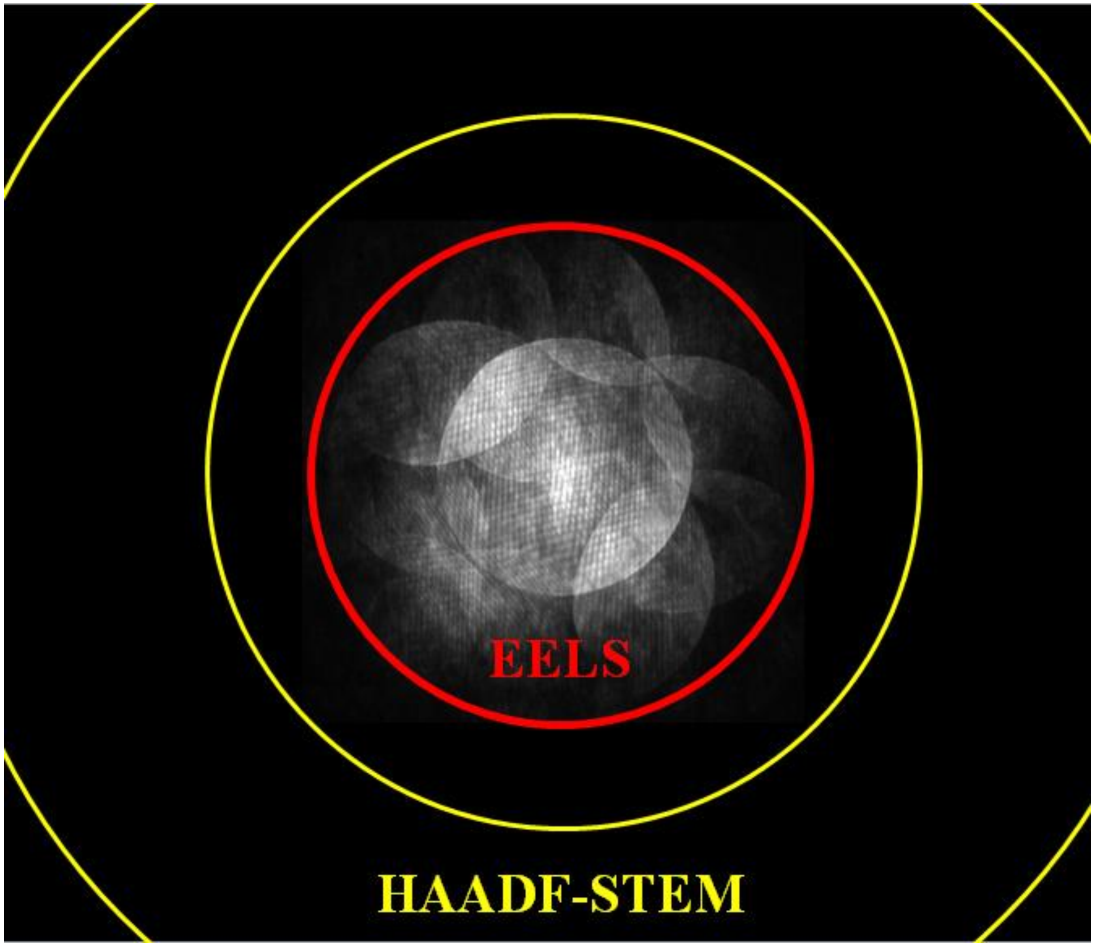


From JEOL2200F

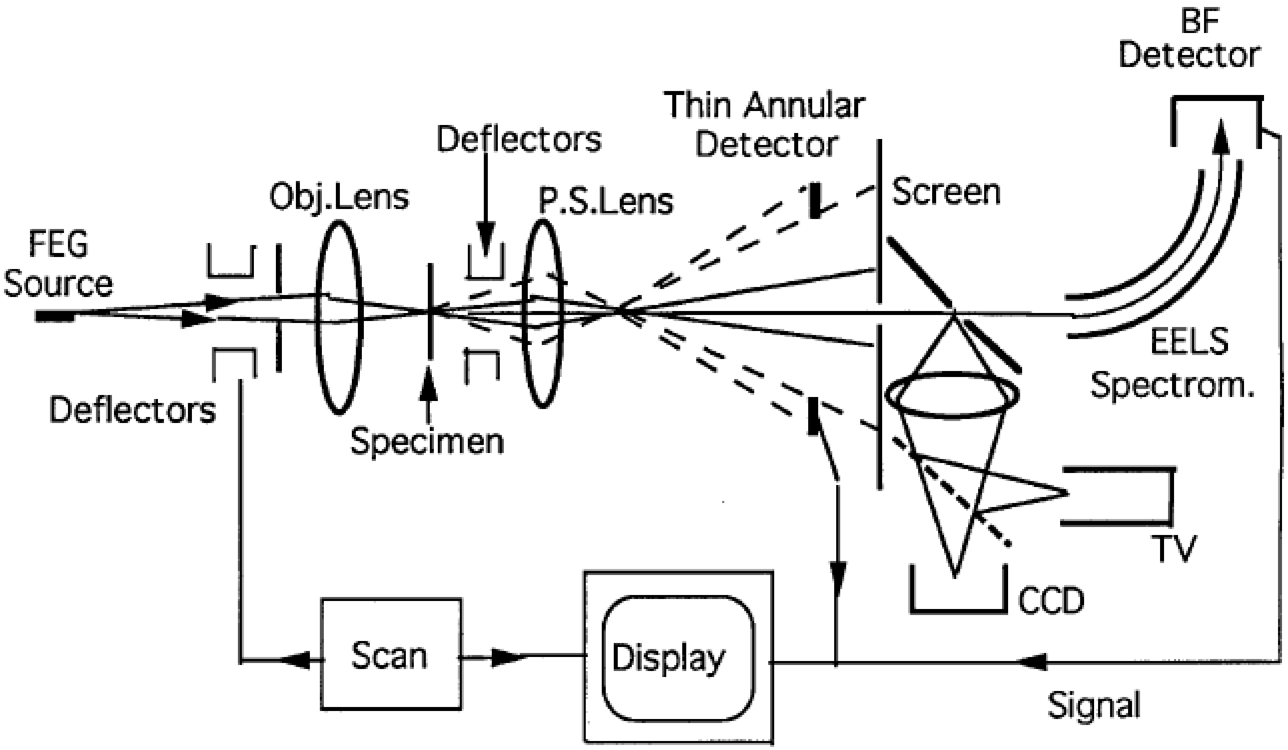

Sb
Ga
As
In

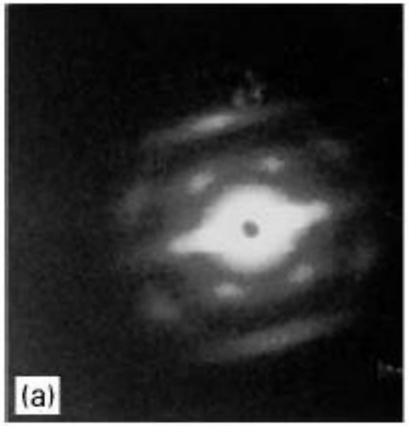
STEM

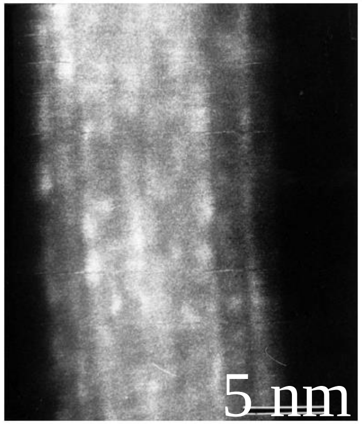




Electron Nanodiffraction (END) in STEM



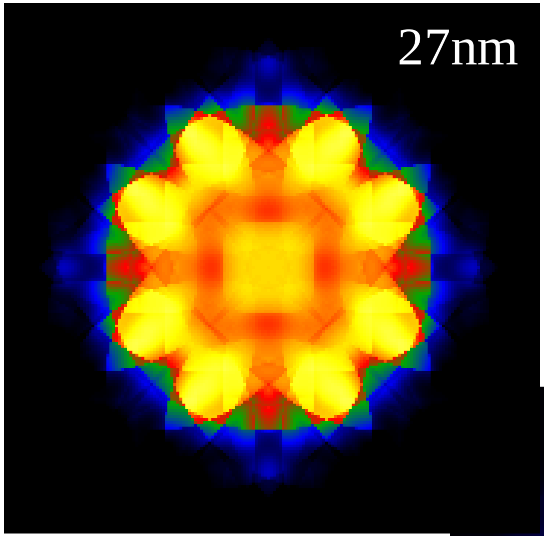


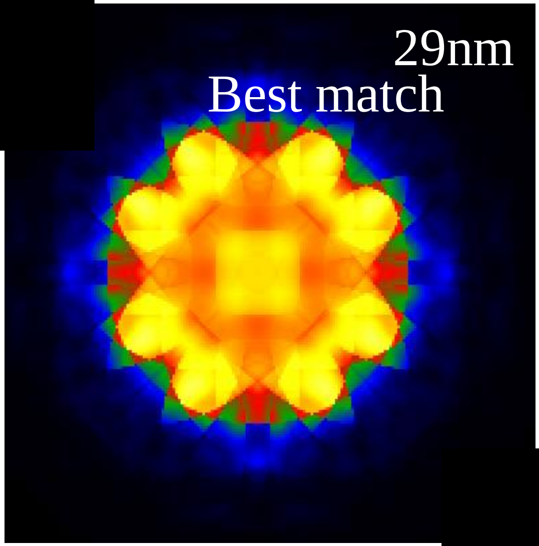


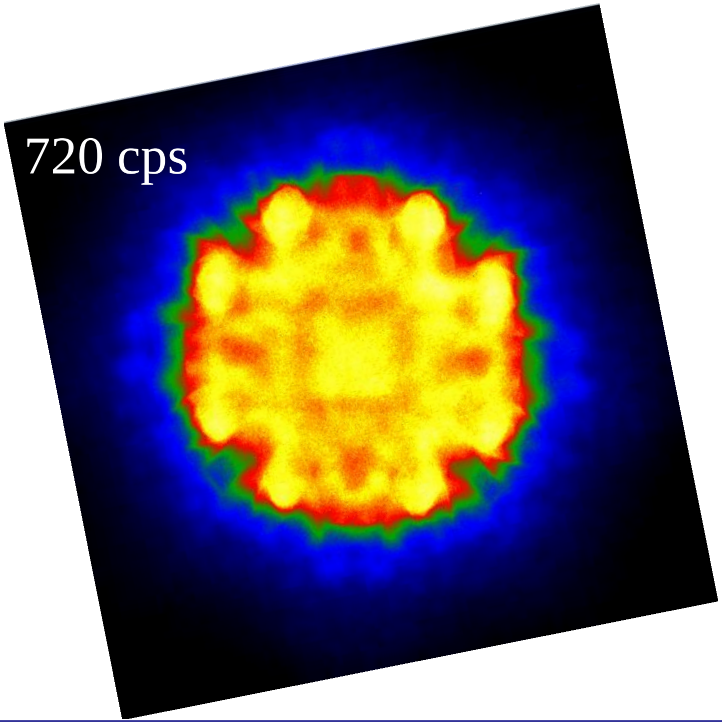
Probe focused to 0.7 nm at the specimen level; Nanodiffraction patterns recorded from: 1) any spot, 2) A scan line, or 3) over a small scan area.

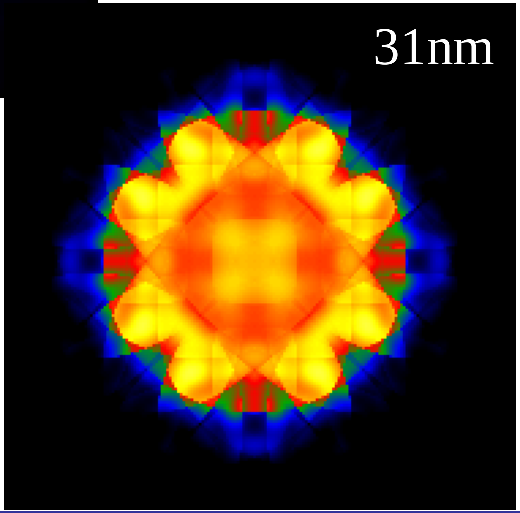
John Cowley, Ultramicroscopy 68 (1997) 1-12

Position Averaged CBED

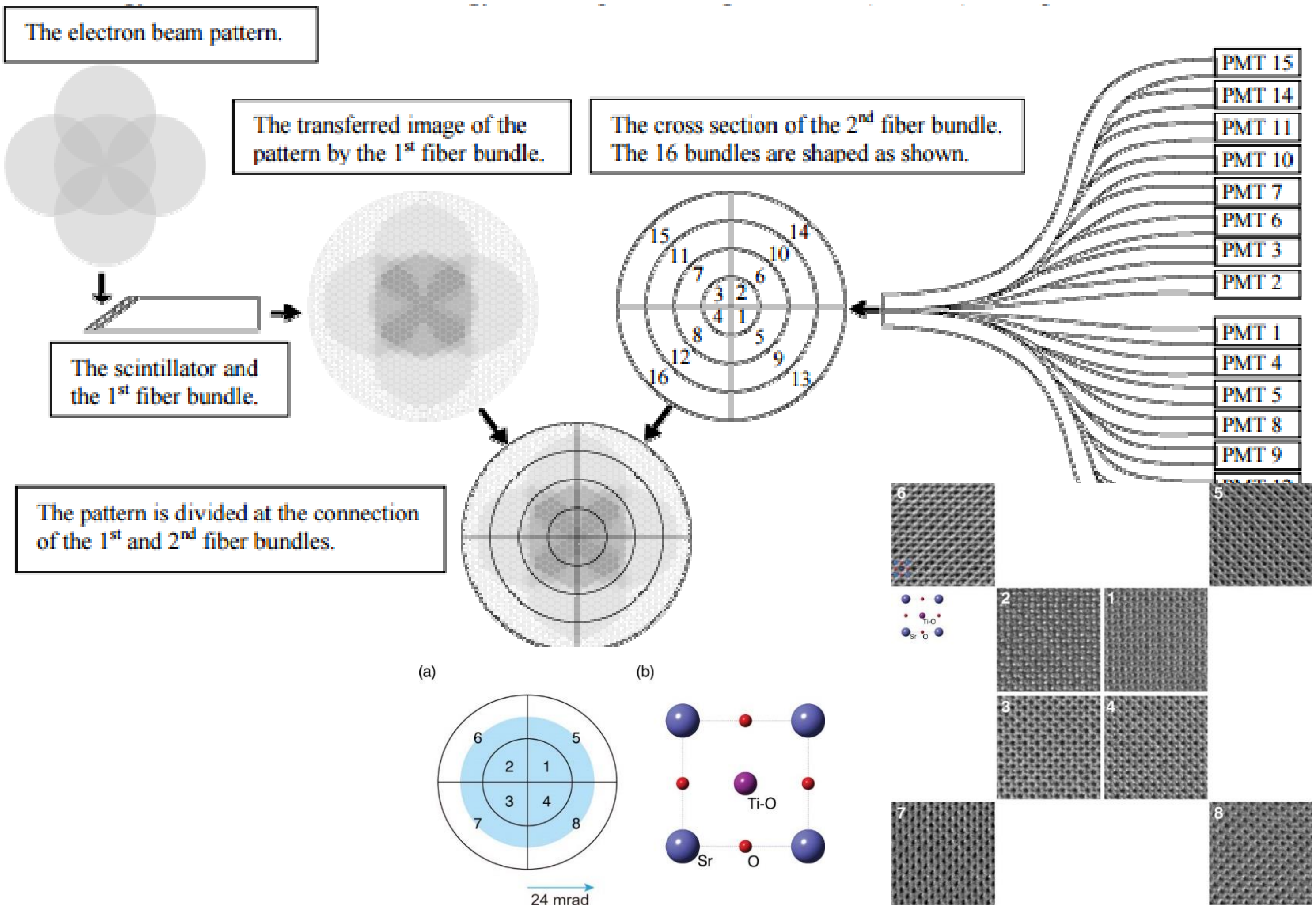






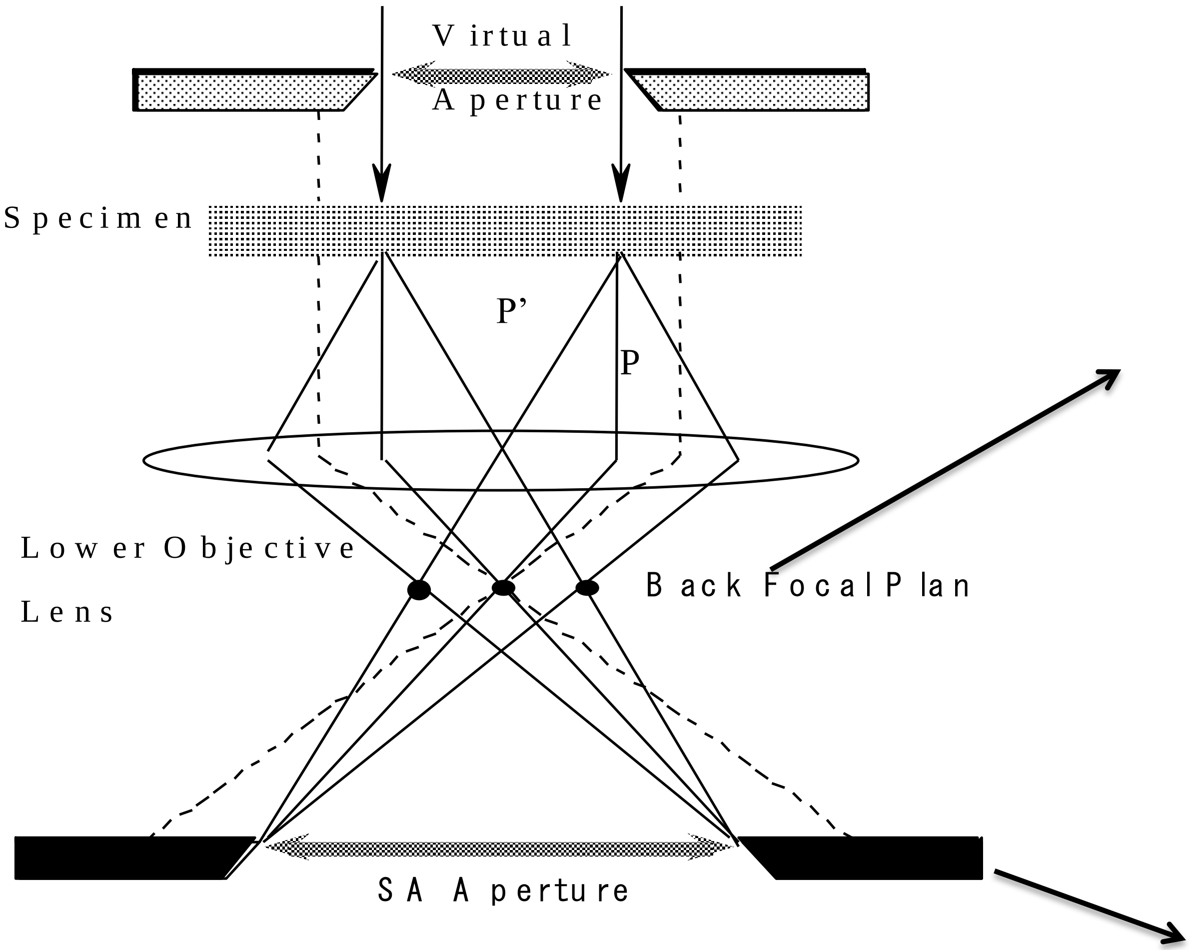


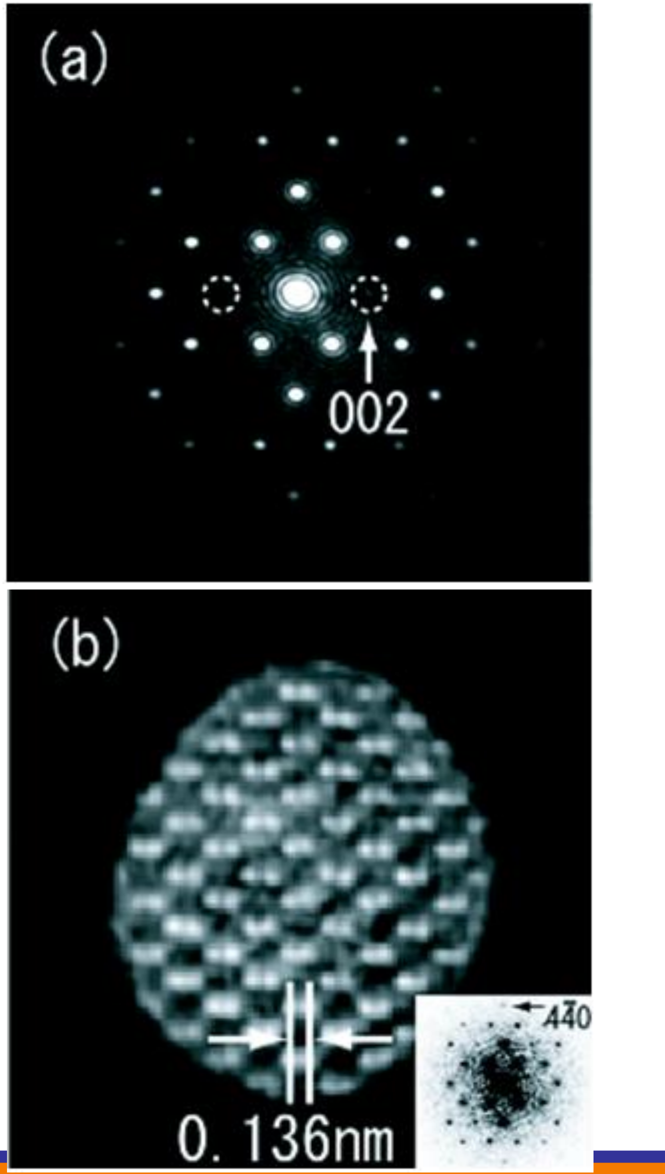
Segmented Detector



Kohno et al., Microsc. Microanal. 16 (Suppl 2), 2010

Selected Area Electron Diffraction with Cs Correction





Covered Topics

- Electron diffraction techniques
  - Selected area electron diffraction
  - Convergent beam electron diffraction
  - Large angle methods
  - Precession electron diffraction
  - Nanoarea electron diffraction
  - Scanning electron nanodiffraction
- Energy-filtering
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- Electron illumination
  - Beam current
  - Probe size
  - Coherence
- Electron detectors and radiation effects

Beam Current

- Source brightness: The emission current density J per unit solid angle, $\beta_s = J/\pi\alpha^2$.
- The brightness β_s of a source is conserved and constant even if aberrations and aperture stops are permitted.
- The beam current after the condenser aperture is given by

$$I_b = \beta_s \pi^2 \alpha_c^2 (d_s/2)^2$$

The probe radius plotted against the probe convergence angle for brightness 10^{12} A/m^2/sr

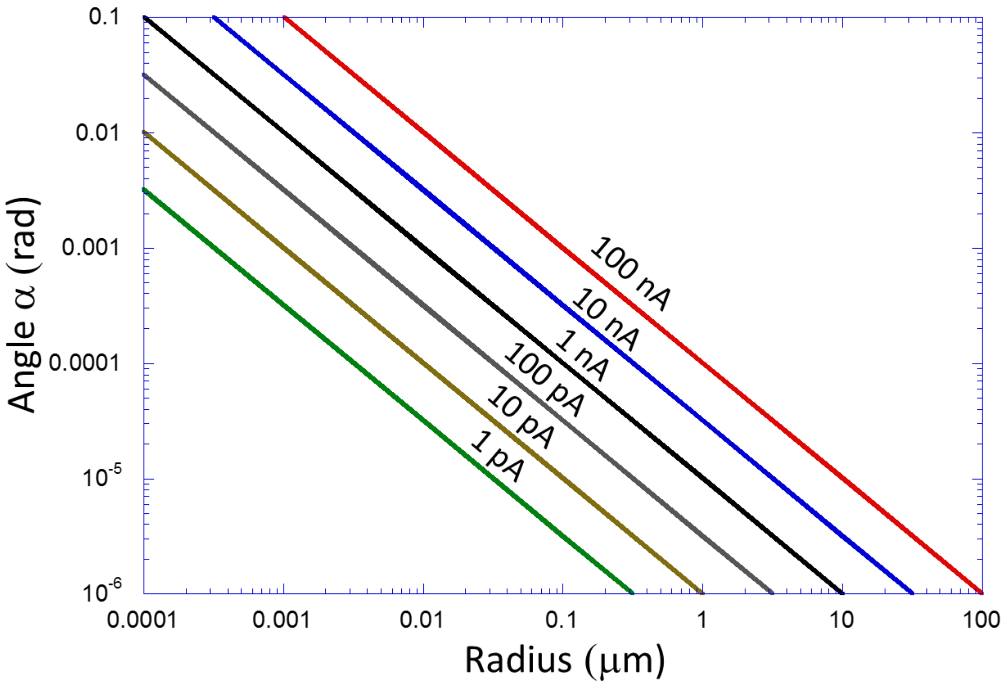


Table 1. Probe Conditions 1 and 2 Used for High Spatial Resolution STEM Imaging*

Conditions	1 HR-STEM	2 HR-STEM	3 STEM EELS	4 STEM HR-EELS
Emission current (μA)	160	160	160	80
Energy resolution (eV)	0.8	0.8	0.9	0.6
CL aperture	30 (μm)	20 (μm)	30 (μm)	40 (μm)
	26.5 (mrad)	17 (mrad)	26.5 (mrad)	34.5 (mrad)
Probe size setting	8	8	3	1
Probe current (pA)	30	13	160	120
Spatial resolution (nm)[a]	<0.078	<0.096	0.11	0.13

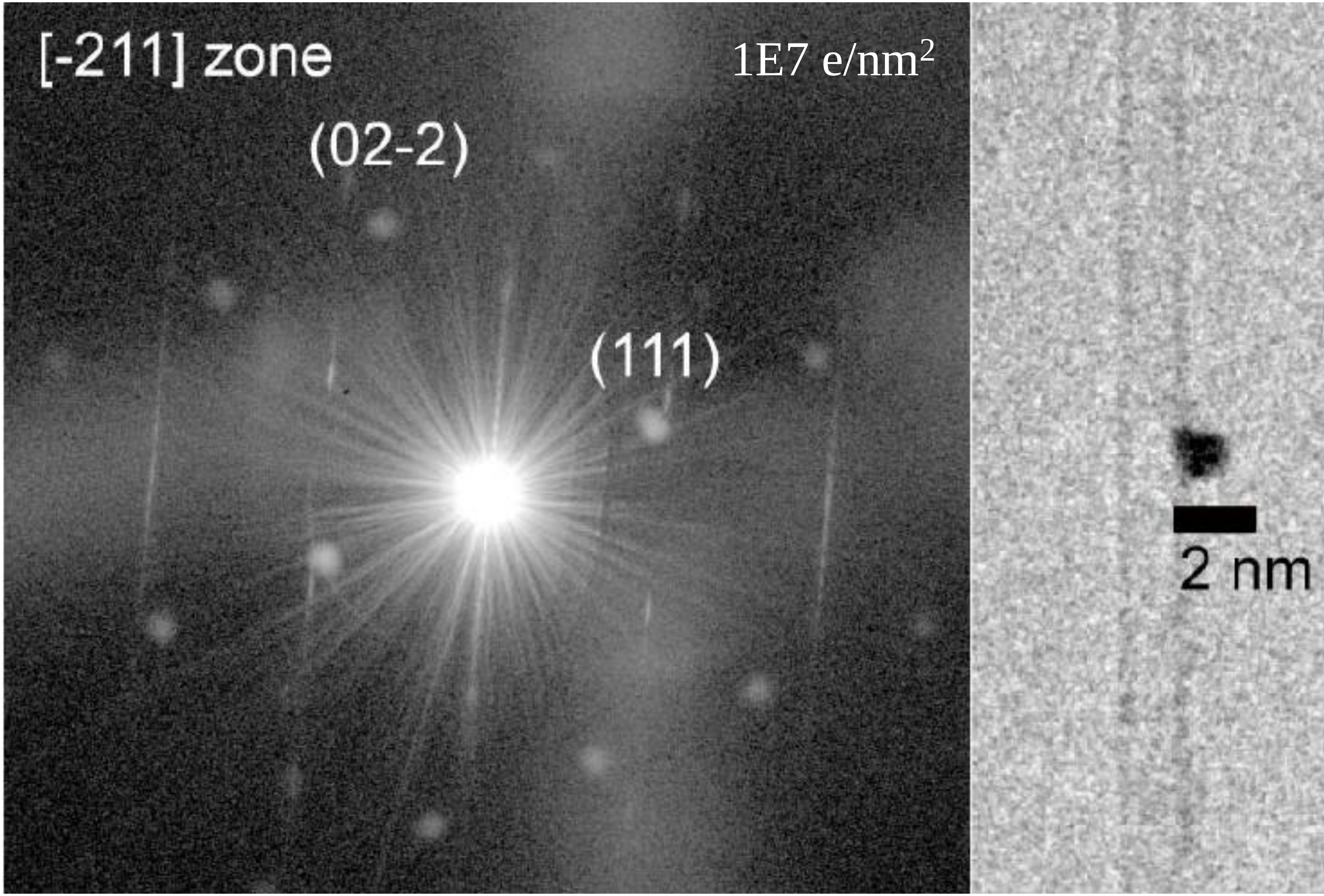
*Probe condition 3 for STEM imaging and EELS, and 4 STEM and high-resolution EELS. The energy resolution is measured using FWHM of ZLP using half collection angle (17 mrad).
[a]The spatial resolution is measured using FFT of Si along [110].

In STEM, for a 30 pA probe

$30\text{x}10^{-12}/1.6\text{x}10^{-19}*10\text{x}10^{-6}*25$ ← 5x5 points/Å^2 sampling
= 50k electrons

Recording time (s)

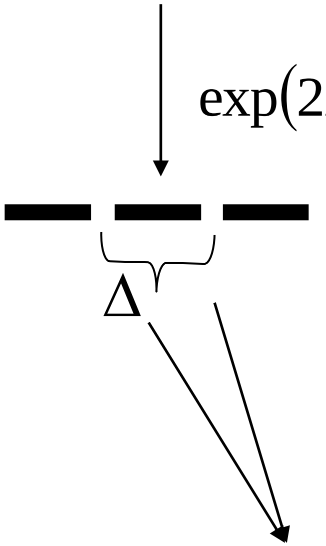
Wen et al., Microsc. microanal. 16, 183-193 (2010)



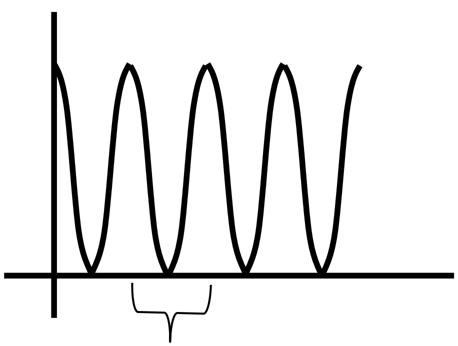
Weijie Huang, Ph.D. Thesis, UIUC

Coherence Length and Coherent Current

$$\exp(2\pi i k z)$$

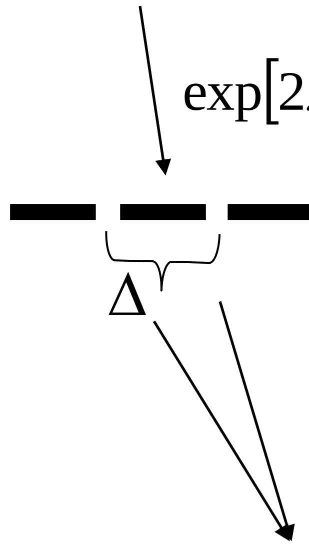


$$\left|\cos(\pi k_x \Delta)\right|^2$$

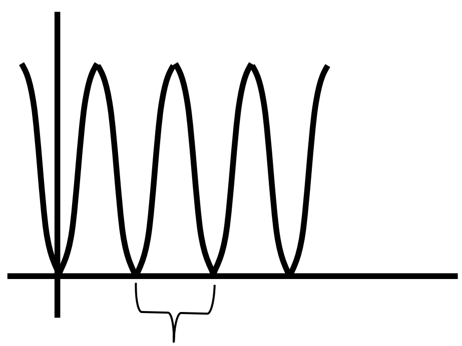


$1/\Delta$

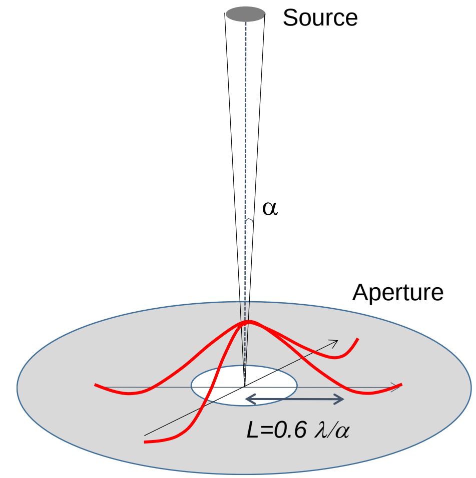
$$\exp[2\pi i(kz + k_o x)]$$



$$\left|\cos(\pi k_x \Delta + \pi k_o \Delta)\right|^2$$



$1/\Delta$

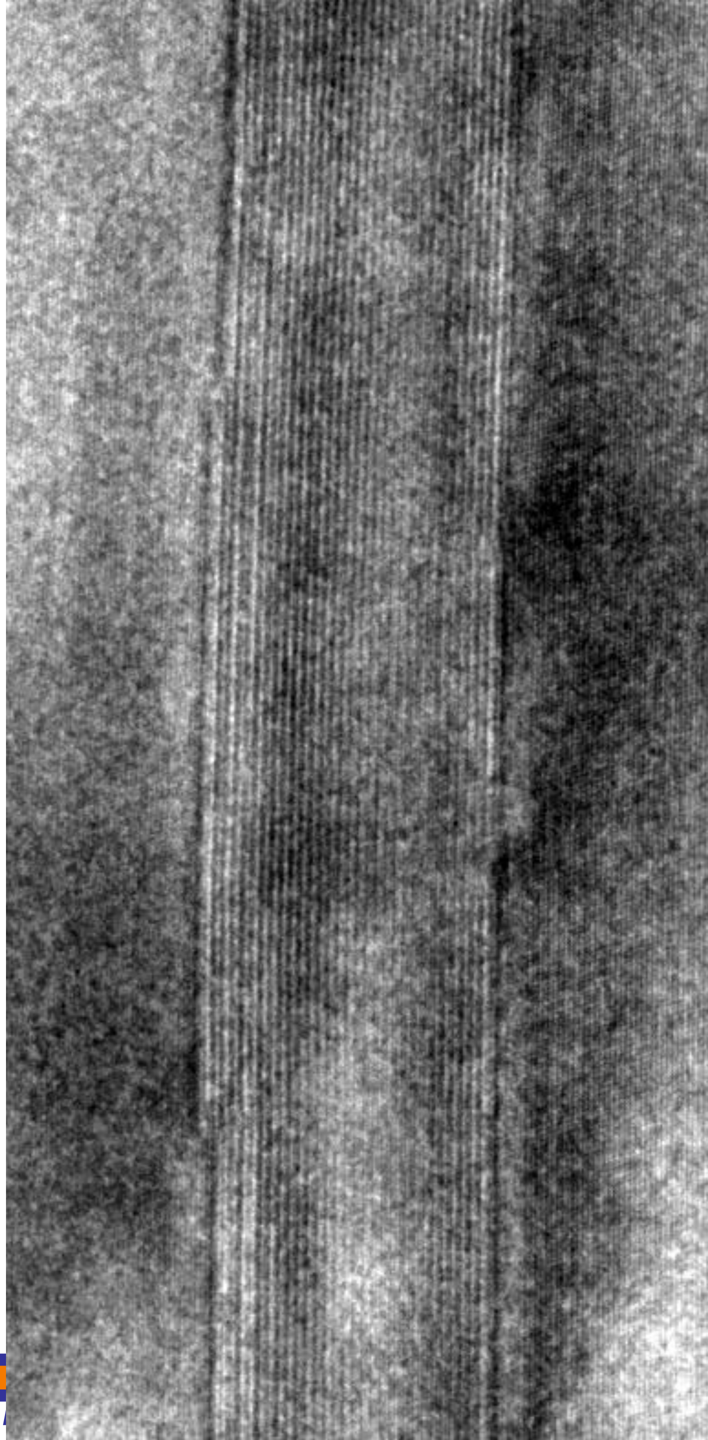


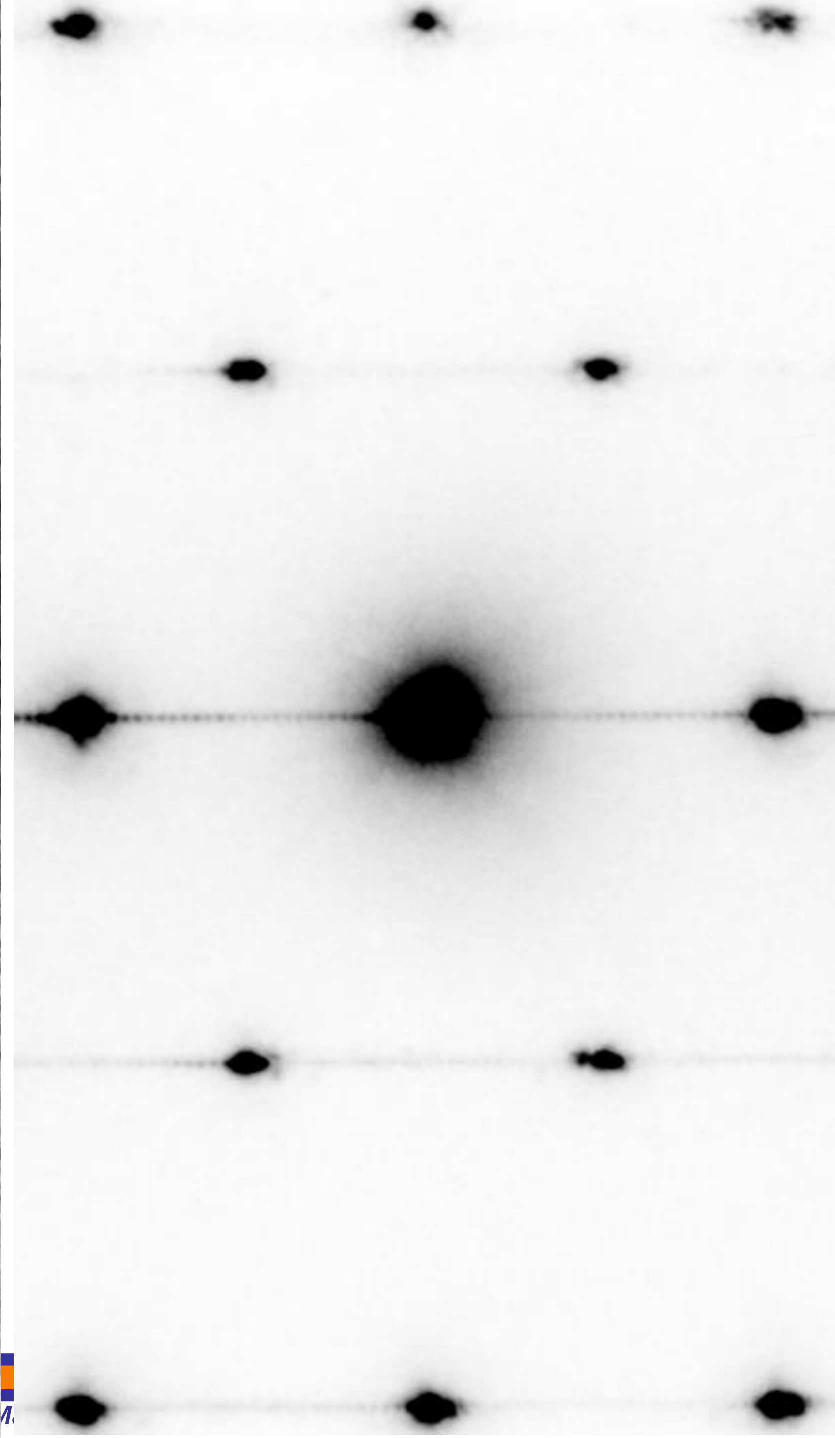
$$L = 0.6\lambda / \alpha$$

$$I_{coh}(\theta) = -\ln\left[\gamma(\theta,0)\right]\beta_s \lambda^2$$

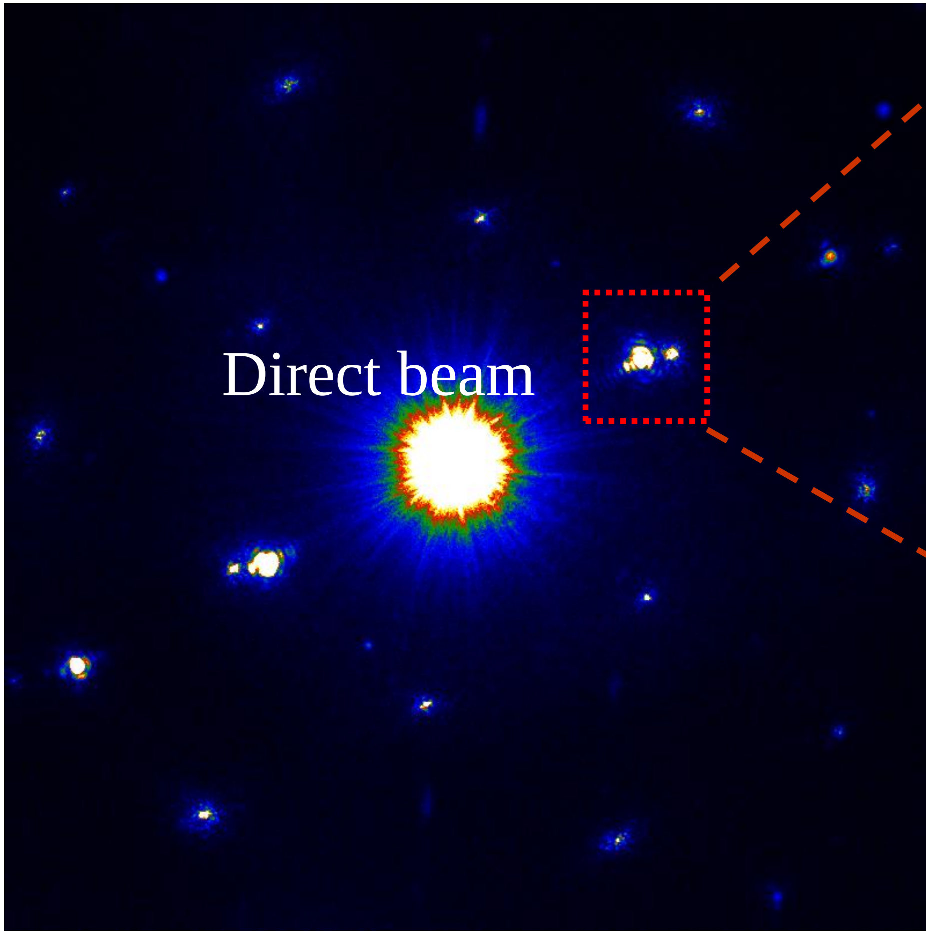
$$\gamma(\theta,0) = \exp\left[-(\pi\theta r_s/\lambda)^2\right]$$

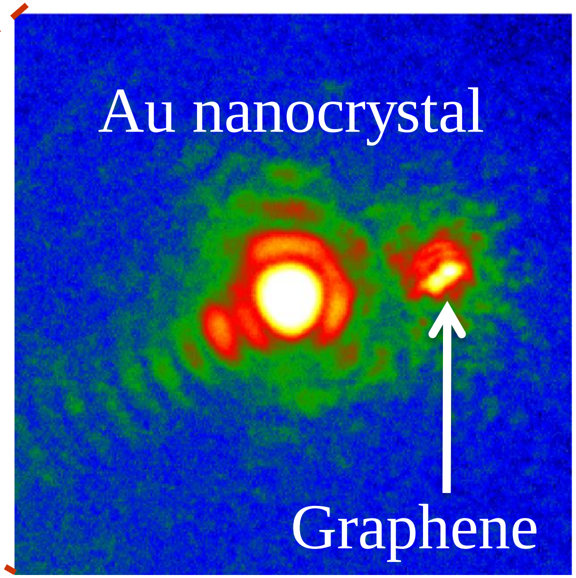
θ is the semi acceptance angle of the CA

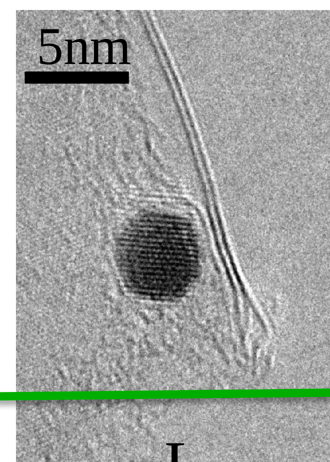




Coherent Electron Diffraction of Au Nanocrystal





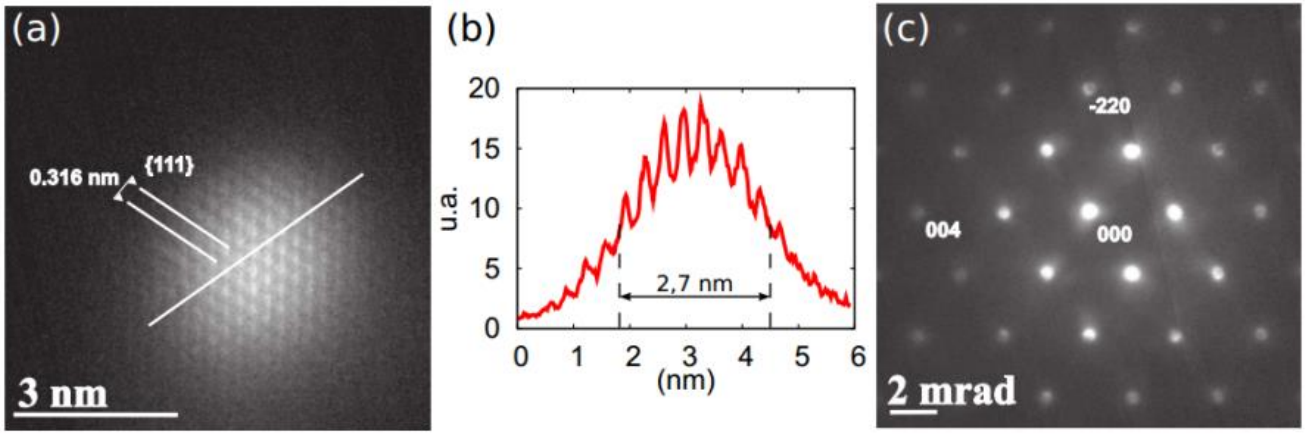


L

+ Low background

Probe Size

Coherent and aberration free $$d_{FWHM} = 0.52\frac{\lambda}{\theta}$$



Coherent with aberrations

$$\phi_P(x,y) = FT\left(\frac{A\sqrt{\pi}}{a}\exp\left[-k_t^{\,2}/(a/M\pi)^2\right]A(\vec{k}_t)\exp\left[i\chi(\vec{k}_t)\right]\right)$$

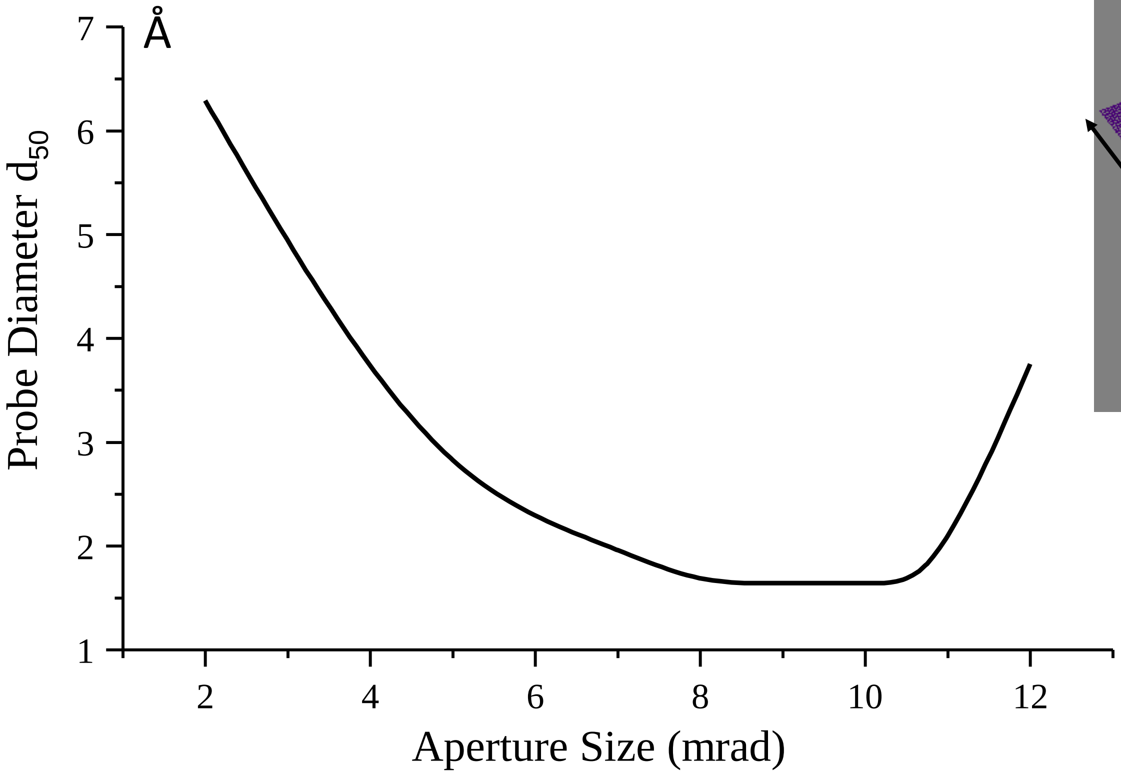
Incoherent

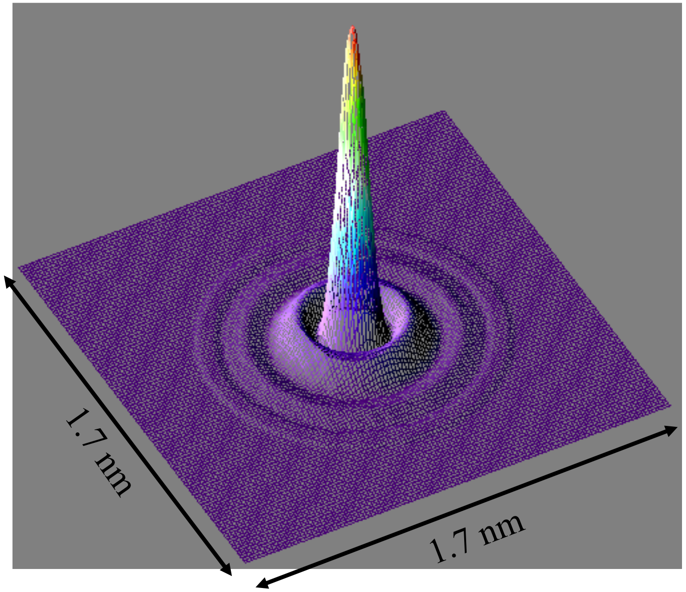
$$d_o^2 = d_s^2 + d_d^2 + d_{sa}^2 + d_c^2 + d_f^2$$

Source image diameter, diffraction broadening, lens aberrations, Cc and focusing error

Electron Probe Formation

C_s=1 mm, C_c=1.2 mm, ΔE=0.7 eV





@Scherzer focus

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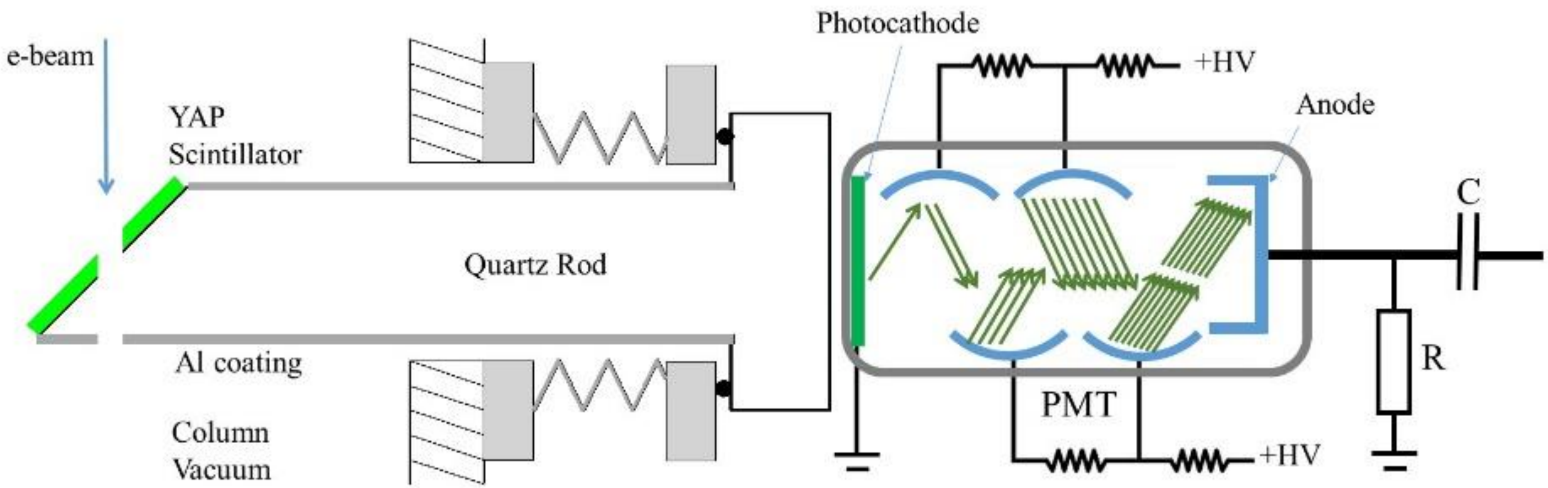
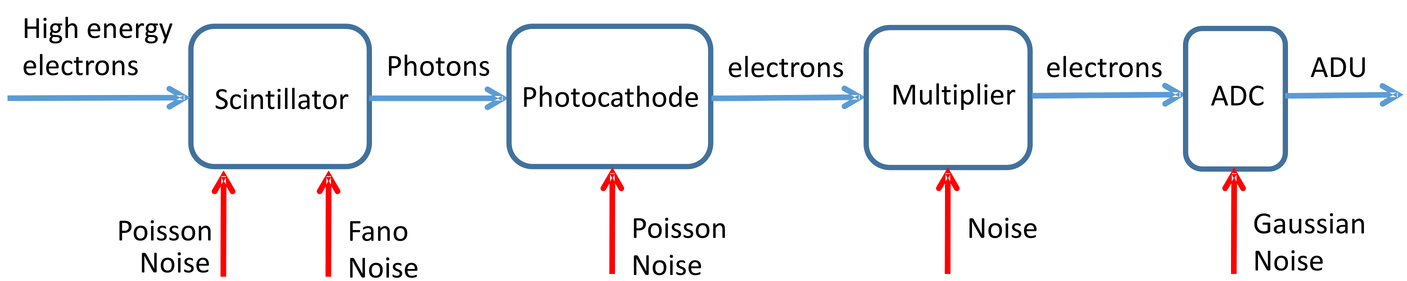
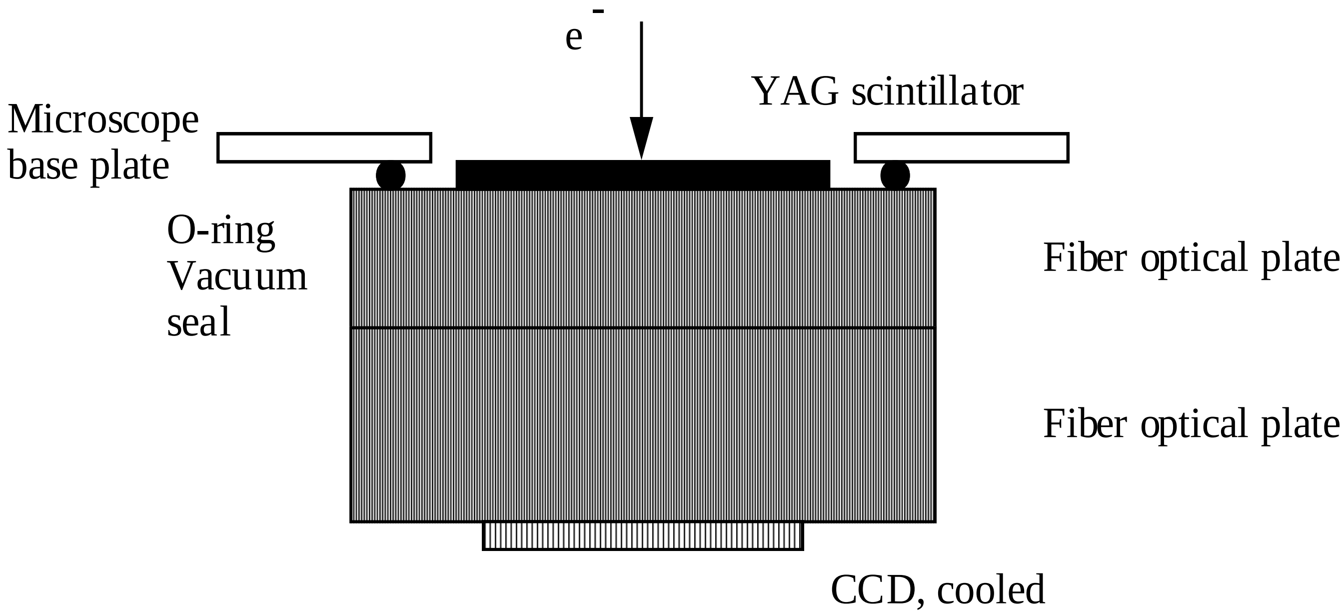
Electron Detection





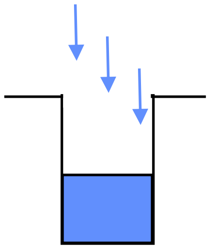
$$DQE = \left(SNR_{out} \,/\, SNR_{in} \right)^2$$

Electron Recording: CCD cameras



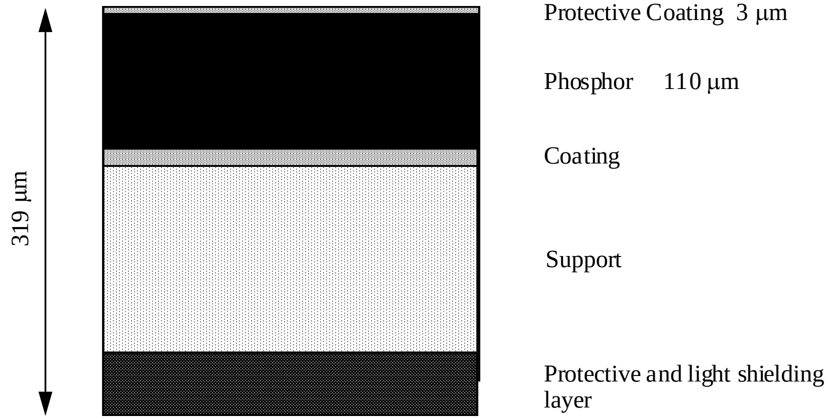
Basics characteristics:

Number of pixels : 512x512, 1024x1024, 2048x2048, 4096x4096
Pixel size : ~24 μm, or smaller
Dynamic range : 8, 12, 14 or 16 bits
Scintillator : YAG or phosphors (thickness is important)
Optical couplings : optical fiber or lens
CCD : pixel size, dark current and readout noise



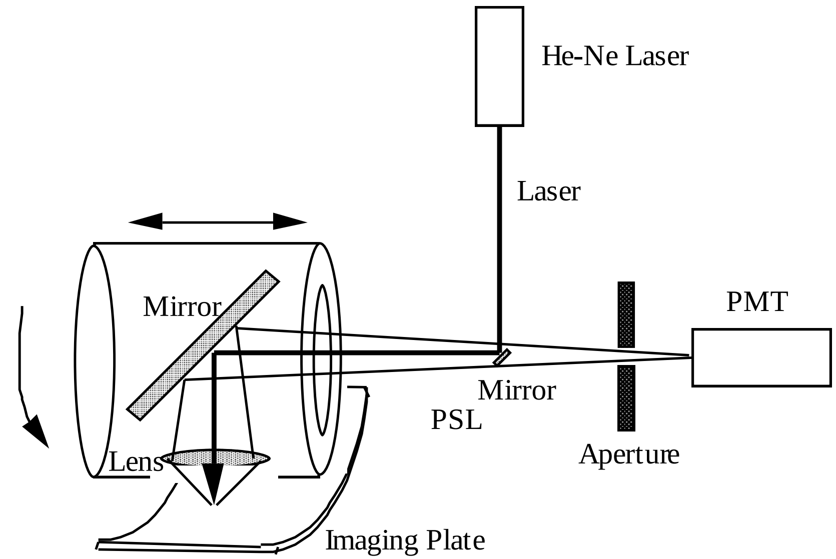
Imaging Plates:

Record like a film



Usable area : 75x94 mm^2, 3000x3750 pixels (25 μm)
Dynamic range : $10^4 \sim 10^5$

Readout like a digitizer

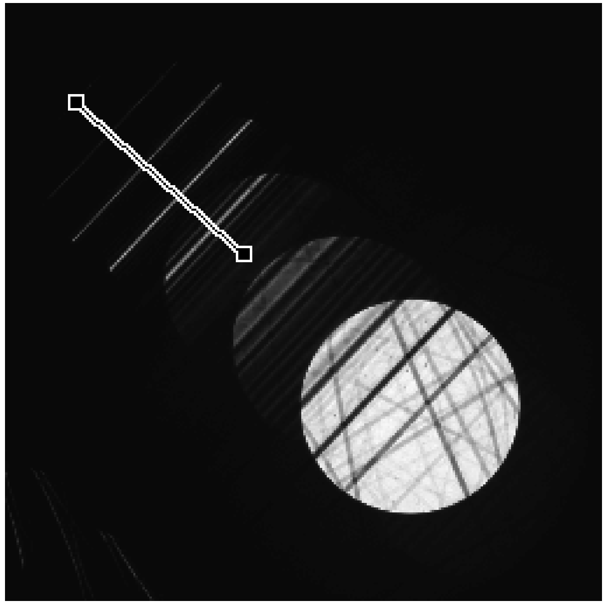


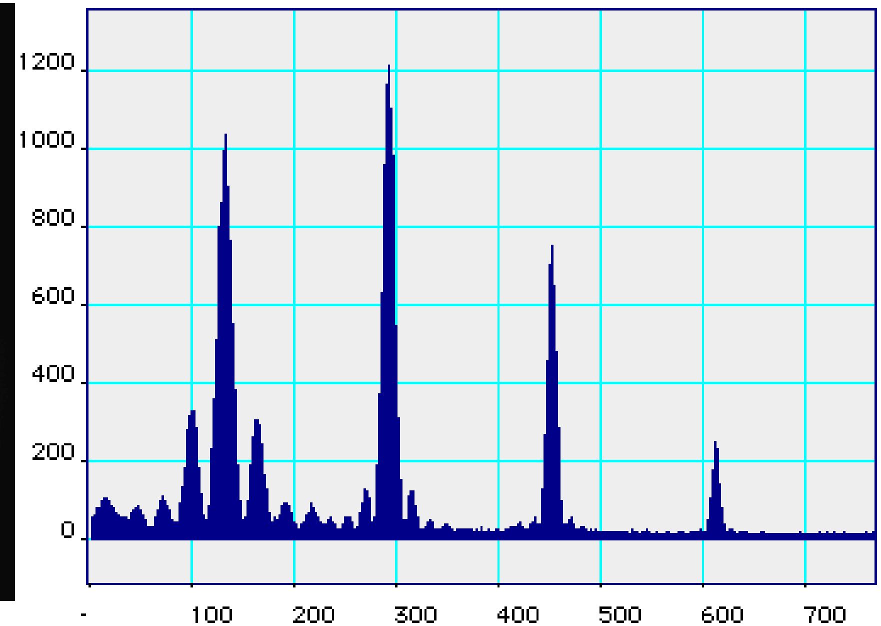
Reader :

- 19 μm laser spot
- Serial scan
- Photomultiplier for detecting photons

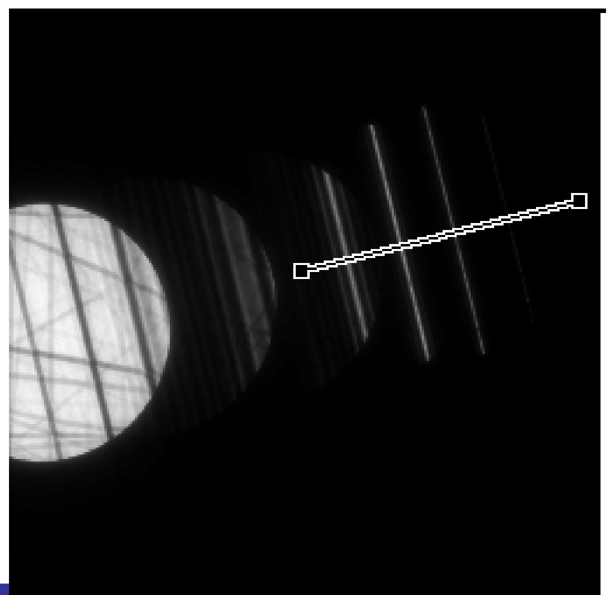
As-recorded CBED patterns by the SSC and IP. The large background in the CCD is due the long tail in the point spread function (PSF).

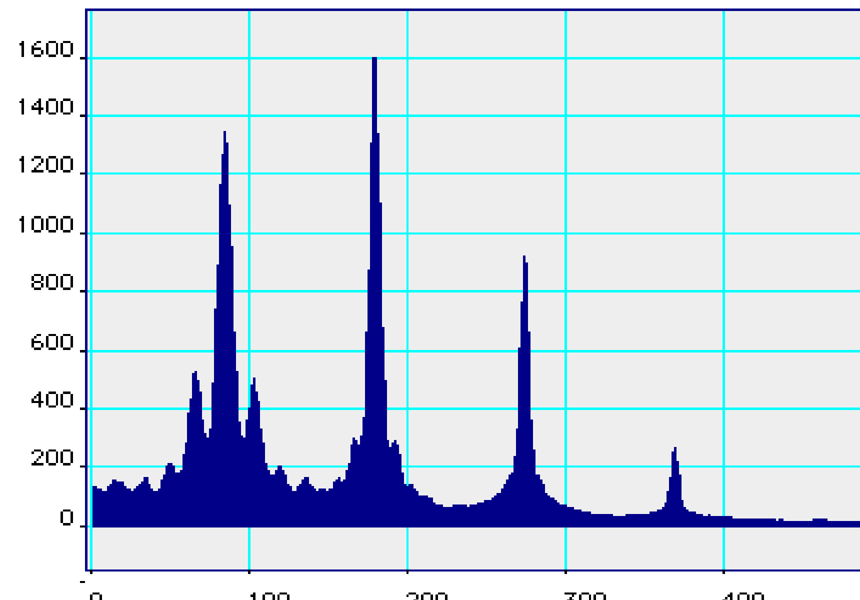
IP
(2048x
2048)





CCD
(1024x
1024



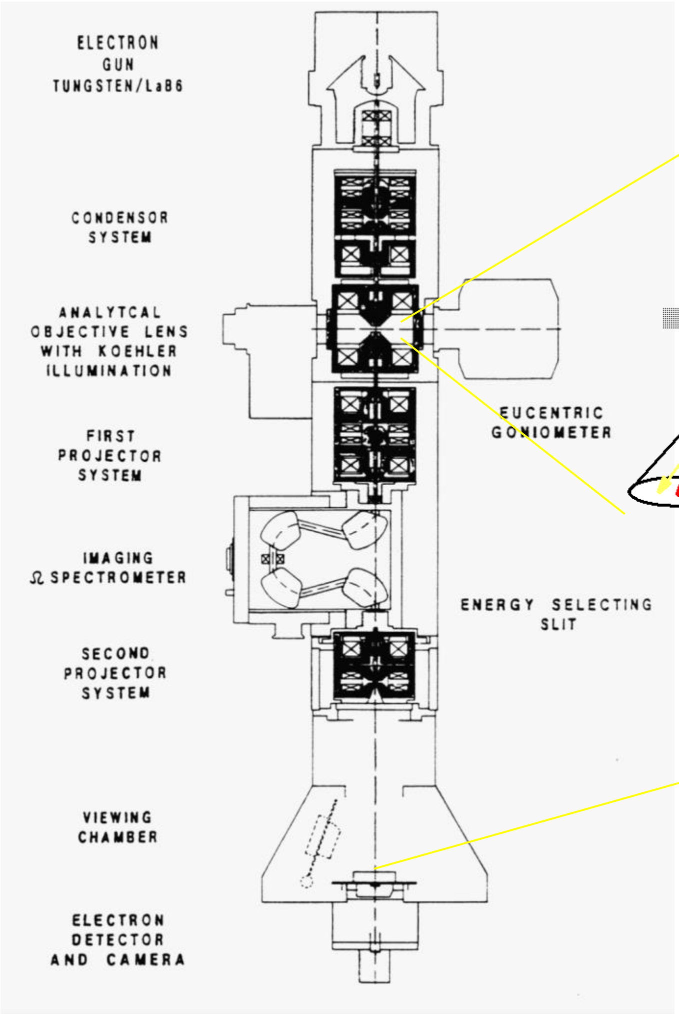


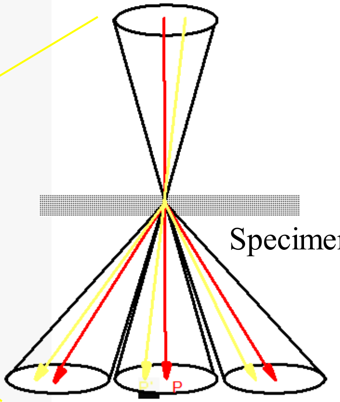
Electron Diffraction Refinement

$$\chi^2 = \frac{1}{n-p} \sum_{i=1,\dots,n} \frac{1}{\sigma_i^2} \left(I_i^{\exp} - I_i^{Theory}\left[p_1, p_2, \dots, p_m\right] \right)^2$$

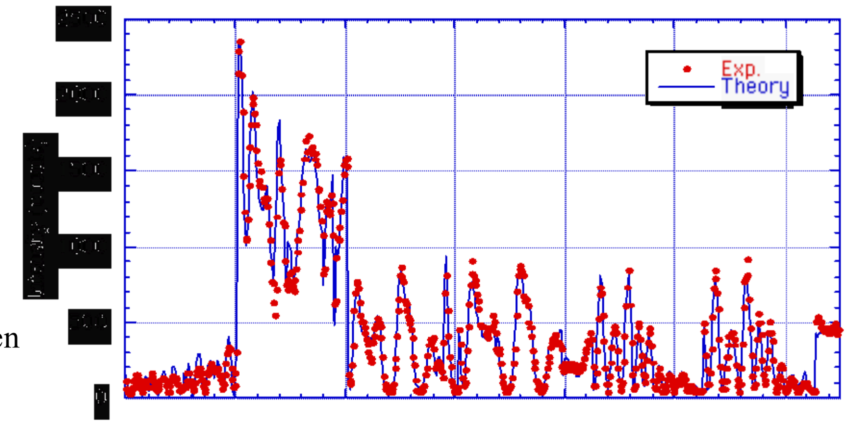
i for sampled points inside CBED disks

Structure factor measurement using electron diffraction

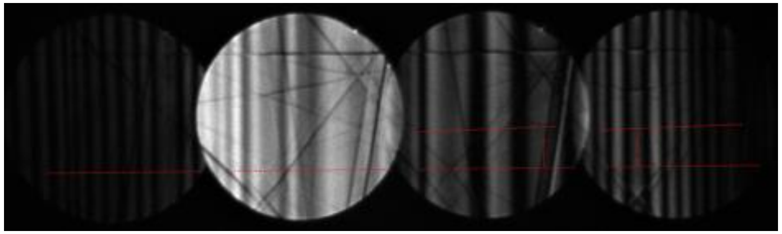




Structure Factor Refinement



Silicon



(000) (111) (222)

	U(111)	U'(111)	U(222)	GOF
χ2	0.04735	0.00086	0.00093	4.2
R-factor	0.04731	0.00084	0.00092	0.096
x-ray	0.04736(4)		0.000943(5)	
Pendelusung				

Pixel Array Detector

Diode Detection Layer

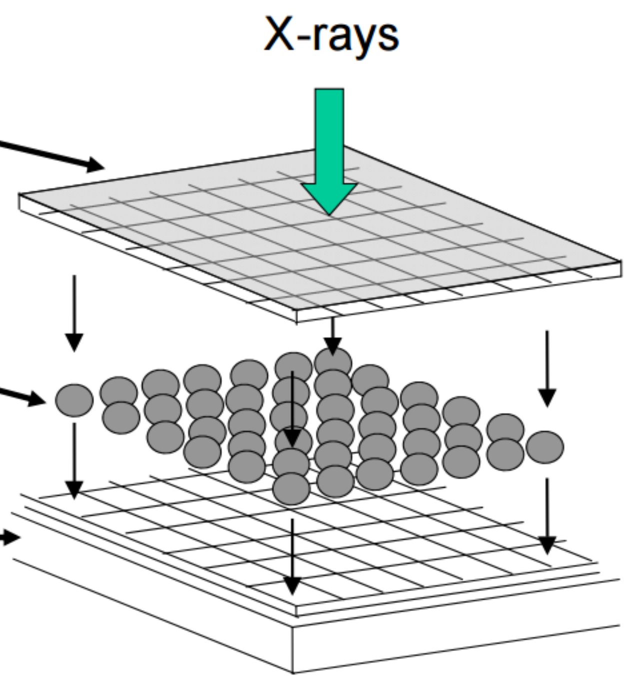
- Fully depleted, high resistivity
- Direct x-ray conversion in Si

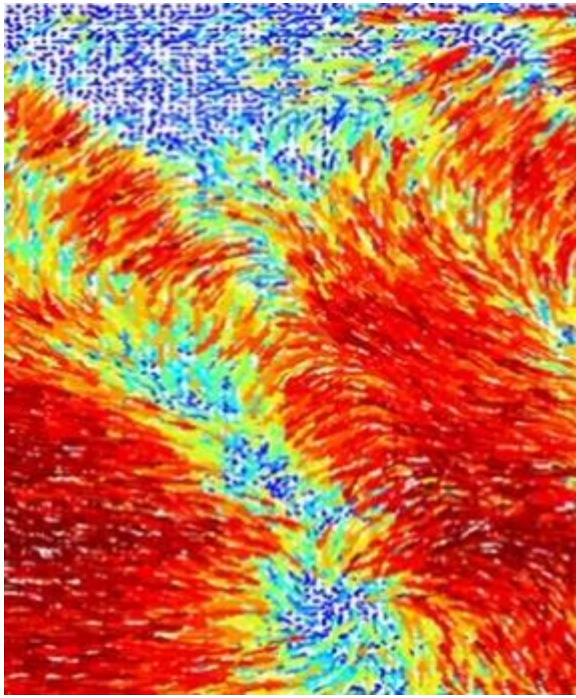
Hybridized with Connecting Bumps

- Solder, 1 per pixel

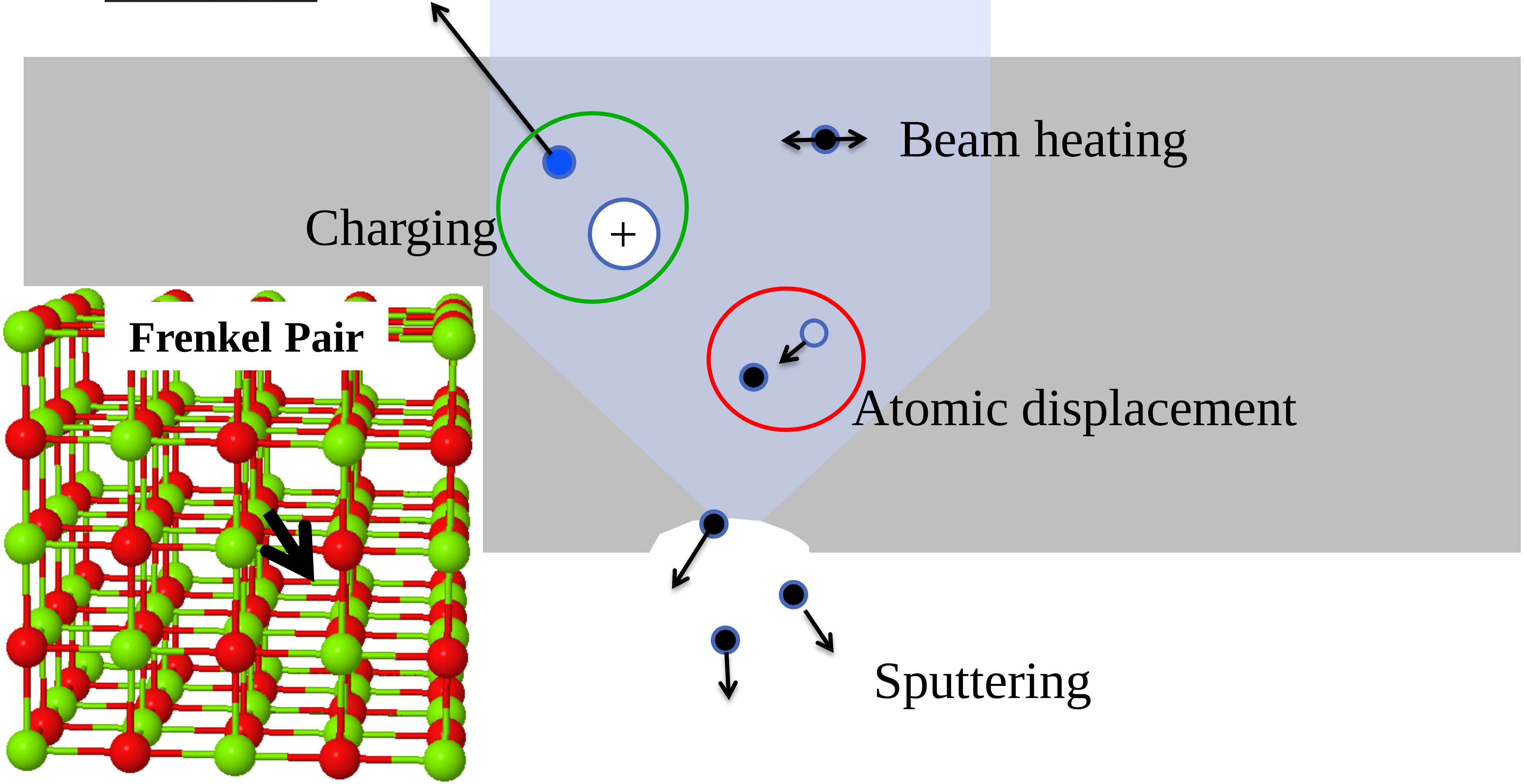
CMOS Layer

- Signal processing
- Signal storage & output



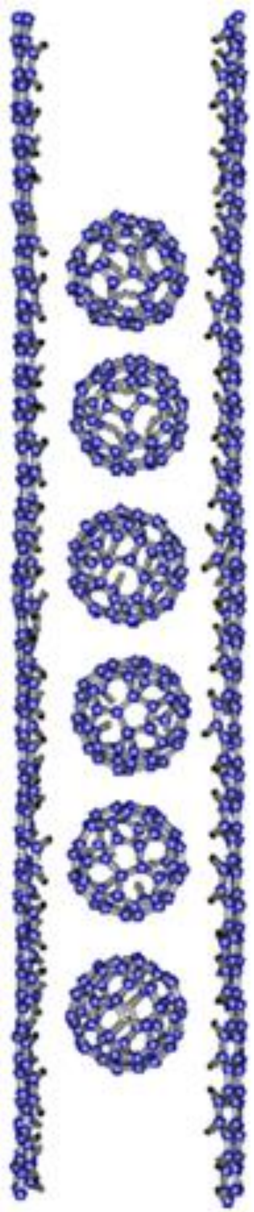


From Cornell, Sol Gruner



R.F. Egerton et al., Micron 35 (2004) 399–409

Carbon “Peapods” C60@CNT



K. Ran, J. M. Zuo, Q. Chen, and Z. J. Shi, "Electron Beam Stimulated Molecular Motions," Acs Nano 5 (4), 3367-3372 (2011)