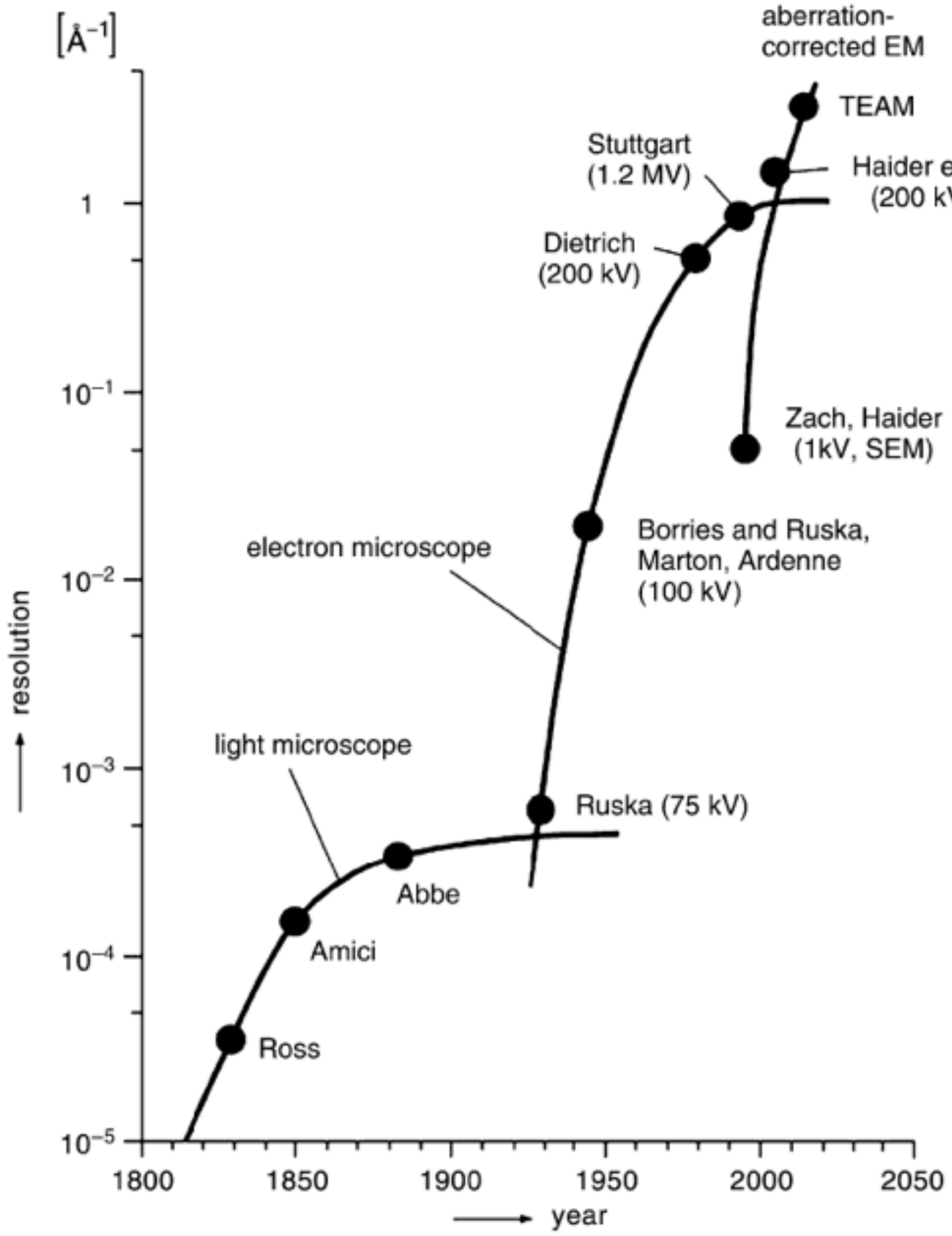
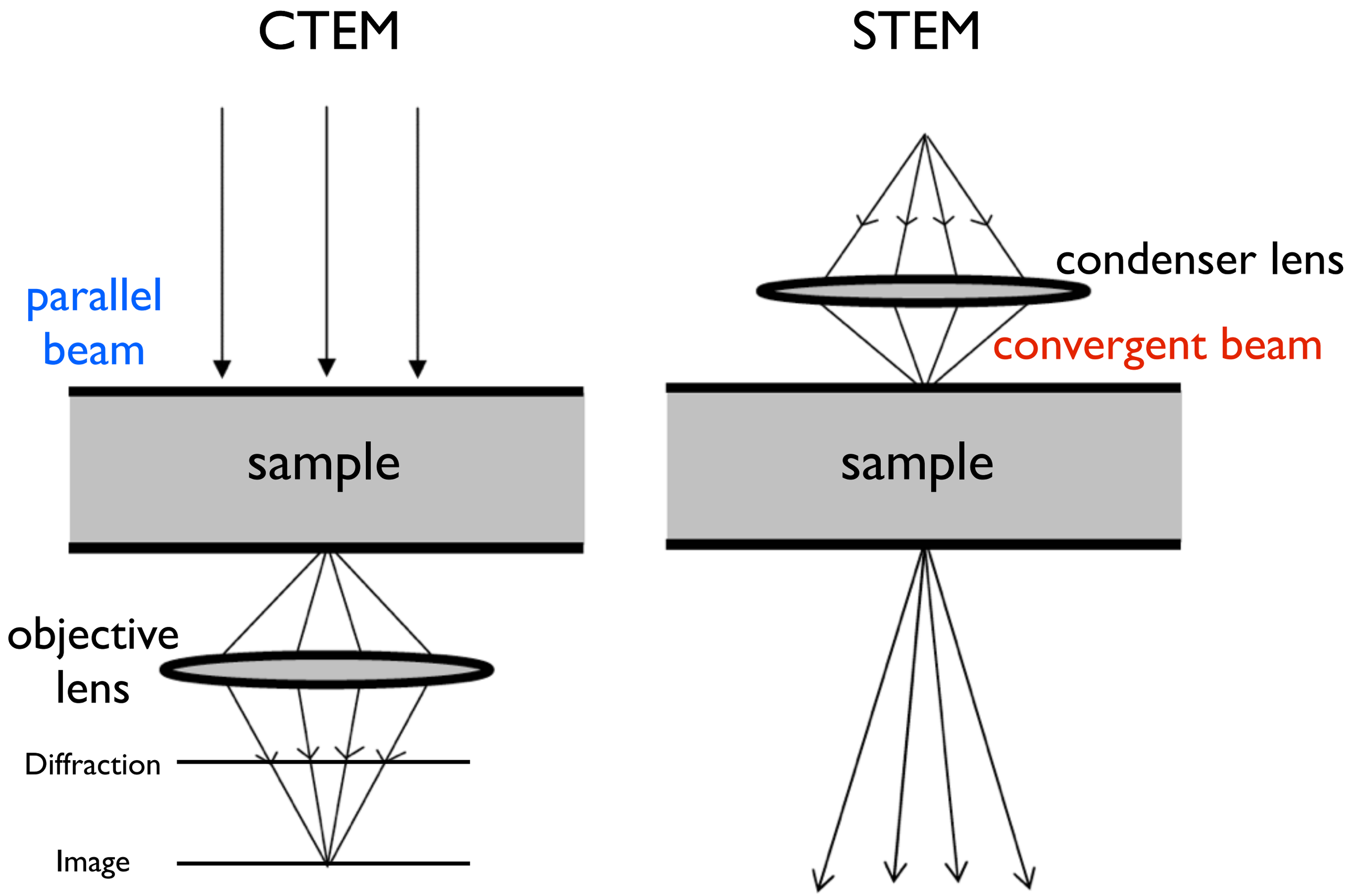
Chapter 8

Atomic Resolution
-Scanning Transmission Electron Microscopy (STEM)

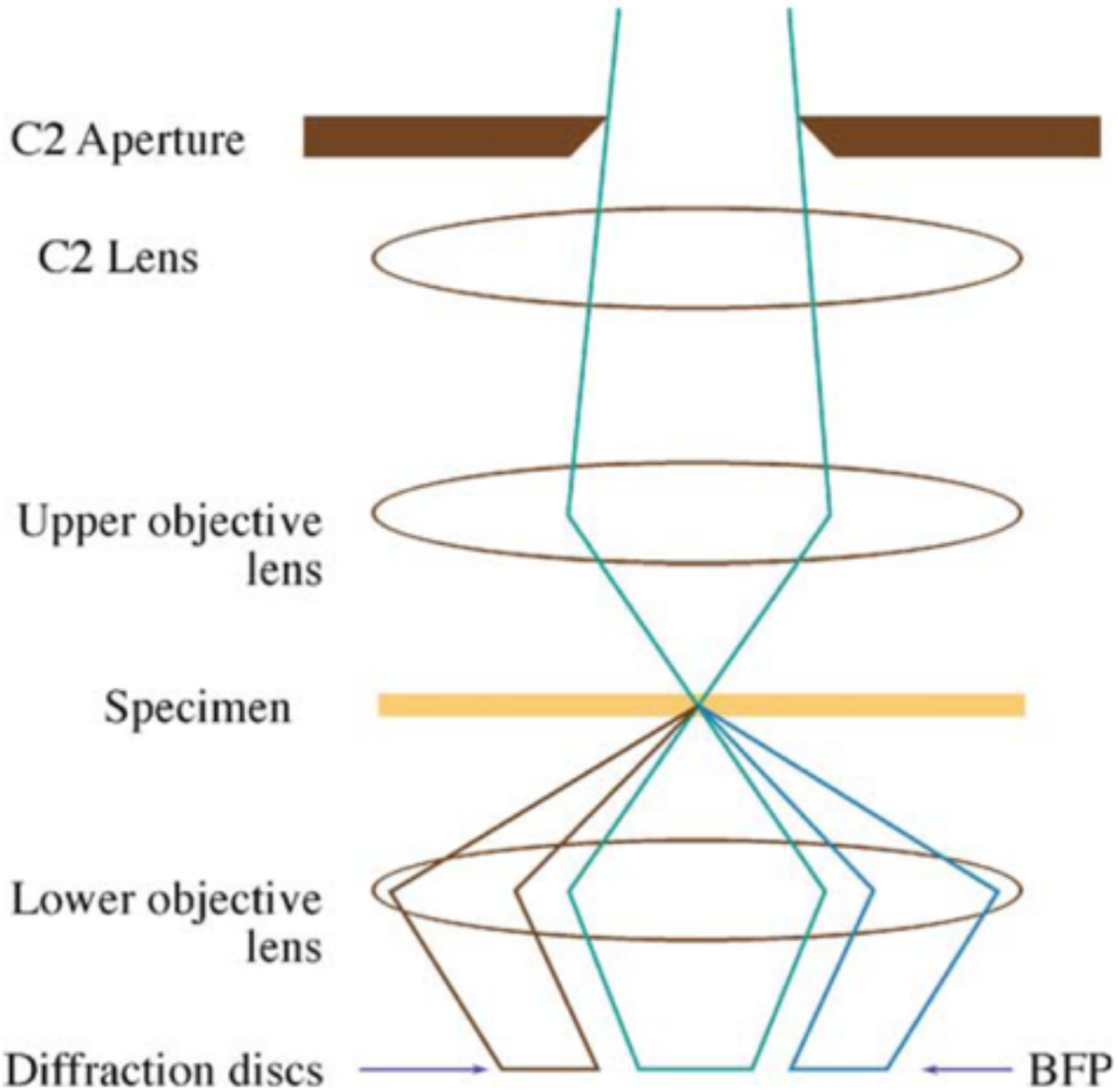
Evolution of resolution in EM

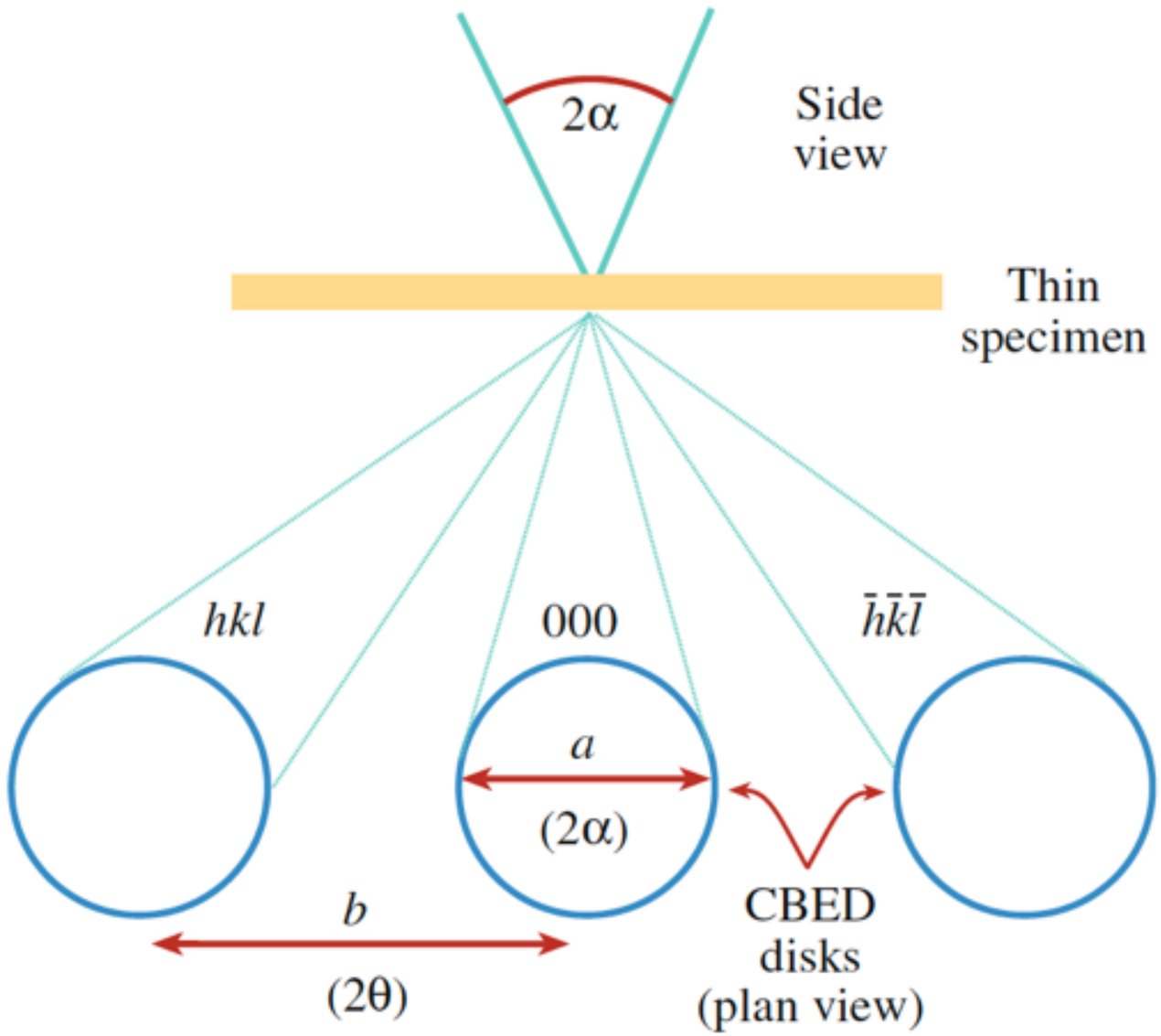


8.1 Geometry of CTEM and STEM

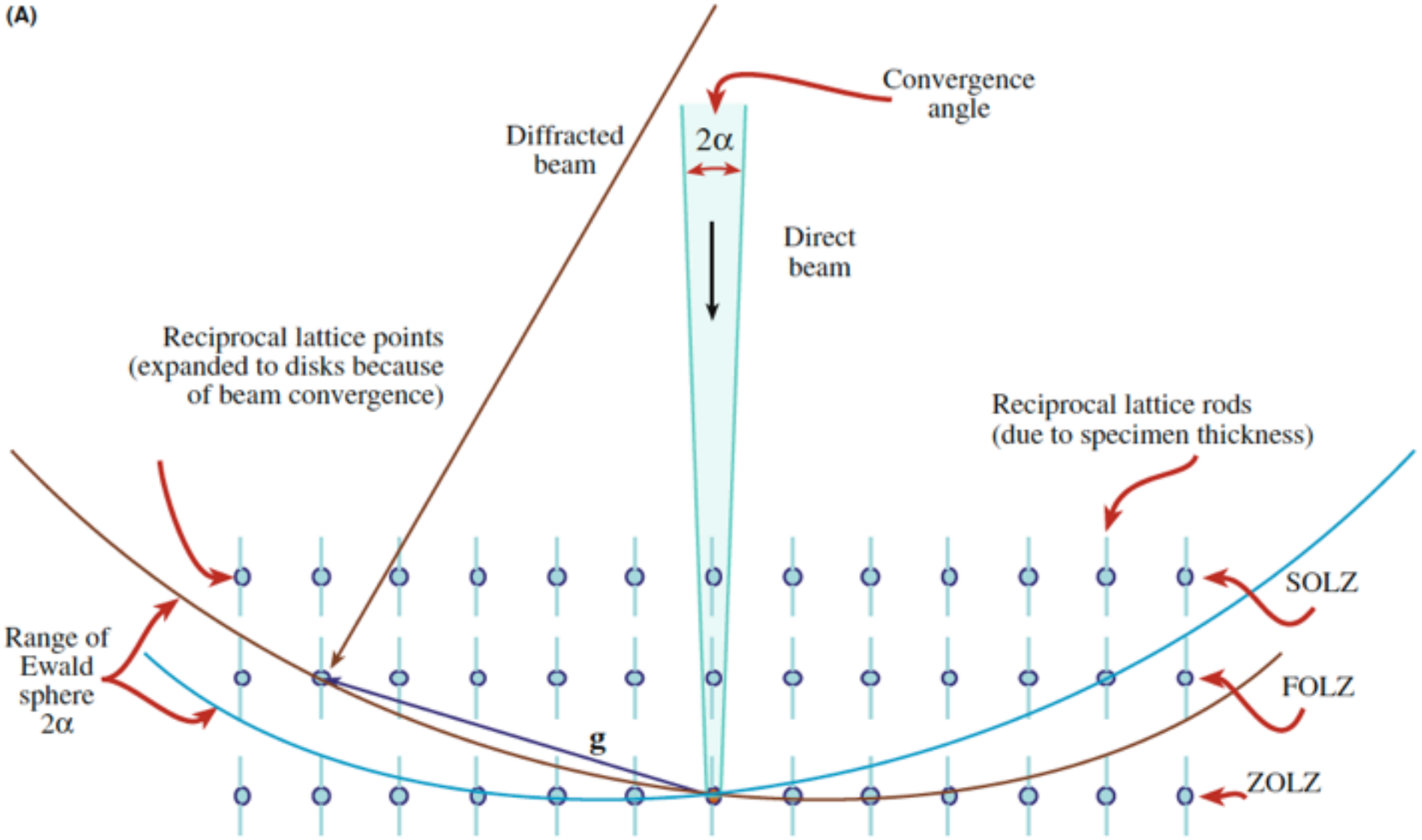


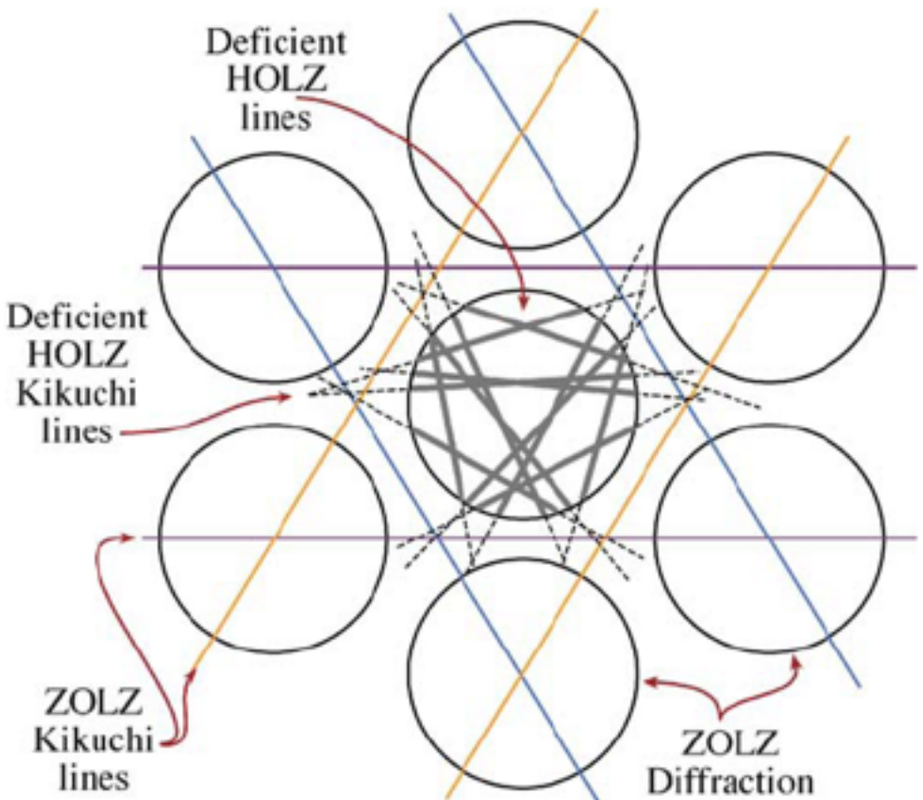
8.2 Convergent Beam Diffraction





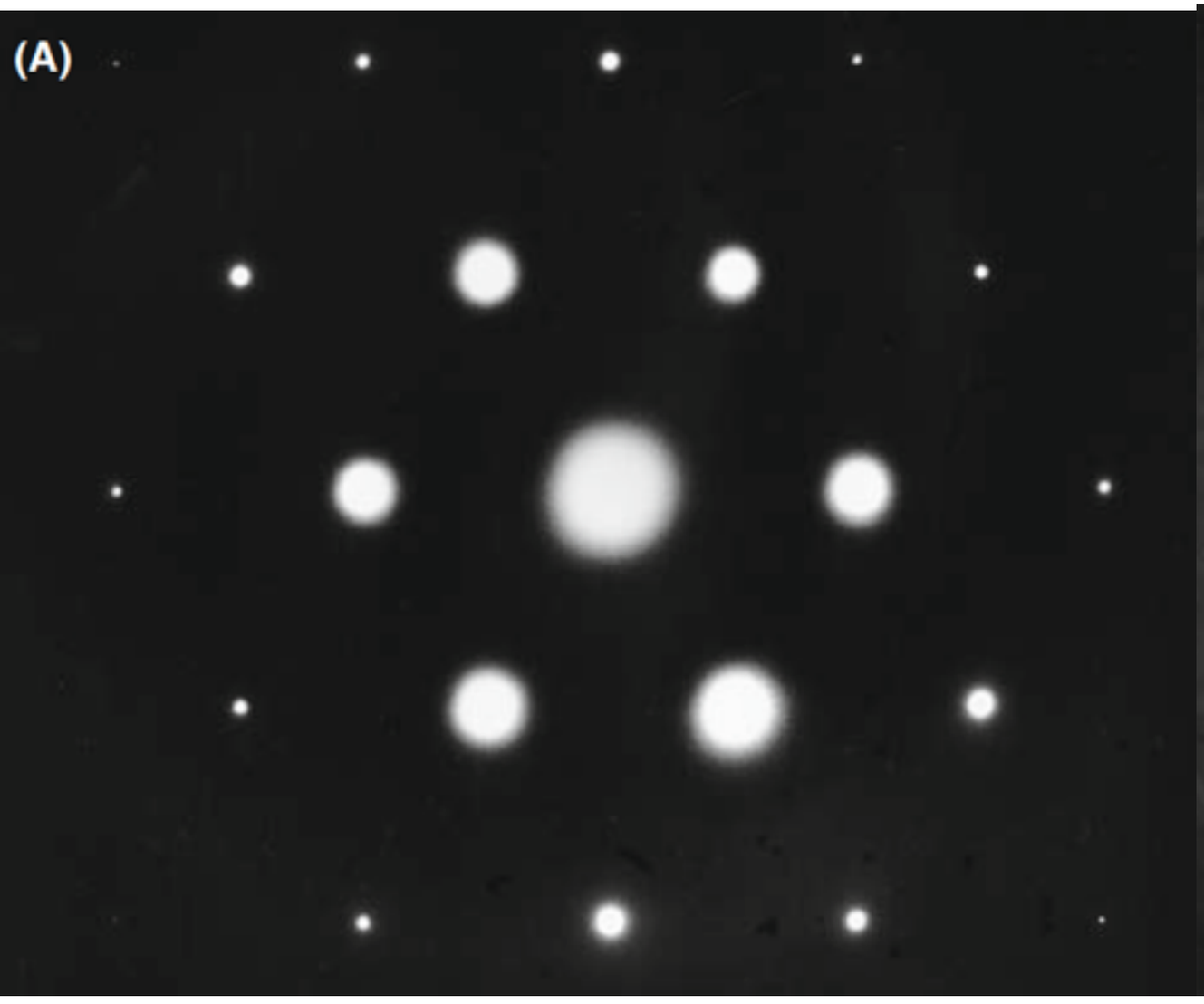
(A)

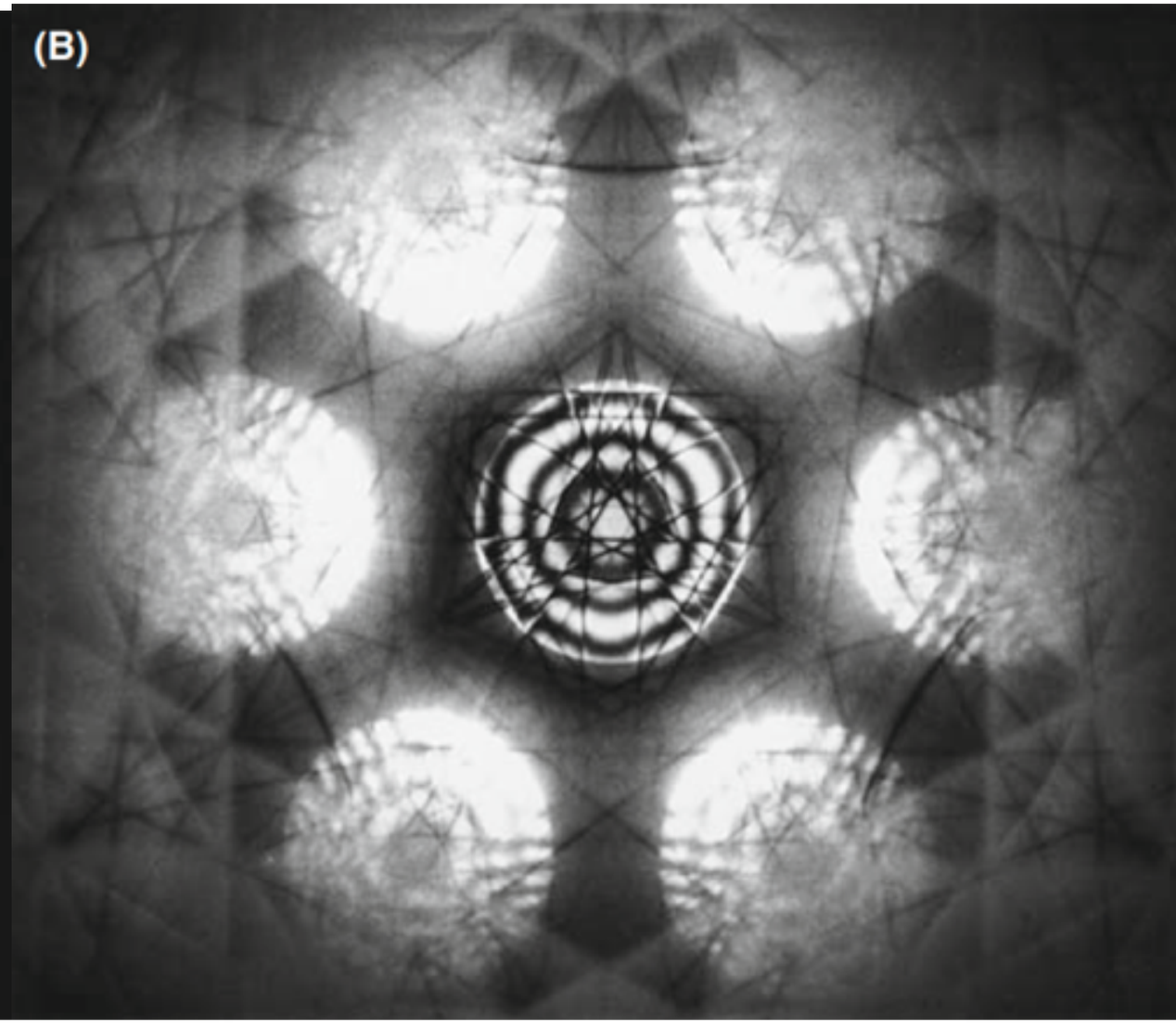

Convergence angle
Diffracted beam
2α
Direct beam
Reciprocal lattice points (expanded to disks because of beam convergence)
Reciprocal lattice rods (due to specimen thickness)
SOLZ
Range of Ewald sphere 2α
FOLZ
g
ZOLZ

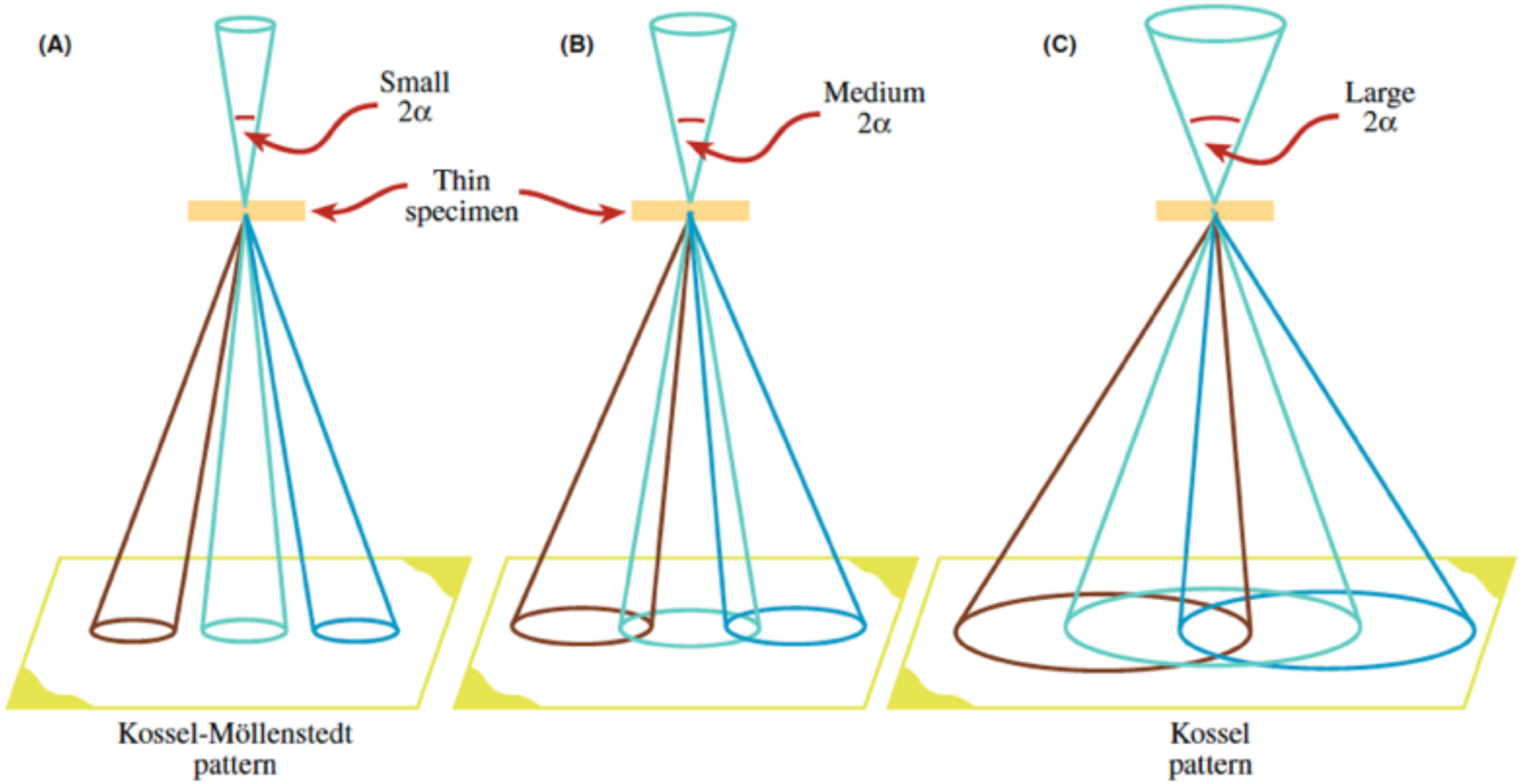

Deficient HOLZ lines
Deficient HOLZ Kikuchi lines
ZOLZ Kikuchi lines
ZOLZ Diffraction disks

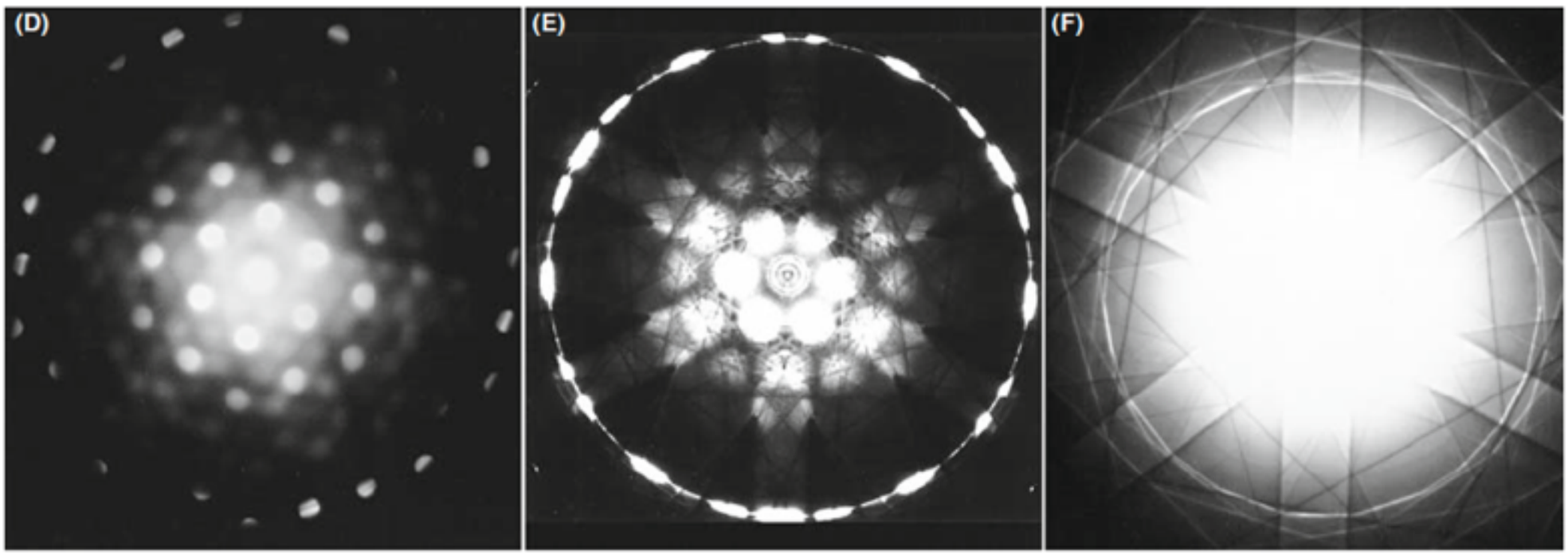
parallel beam electron diffraction (ED)

convergent beam electron diffraction (CBED)

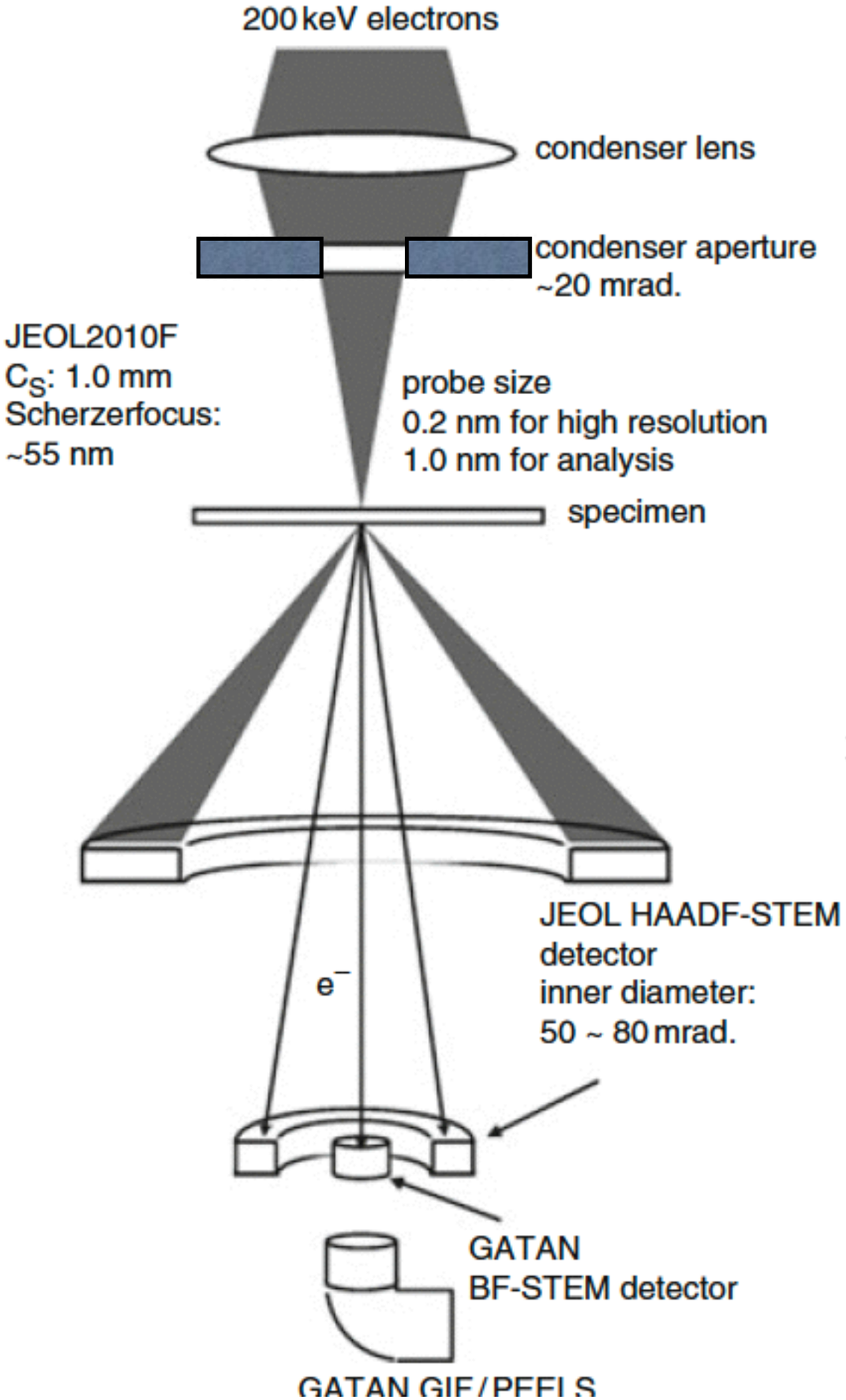


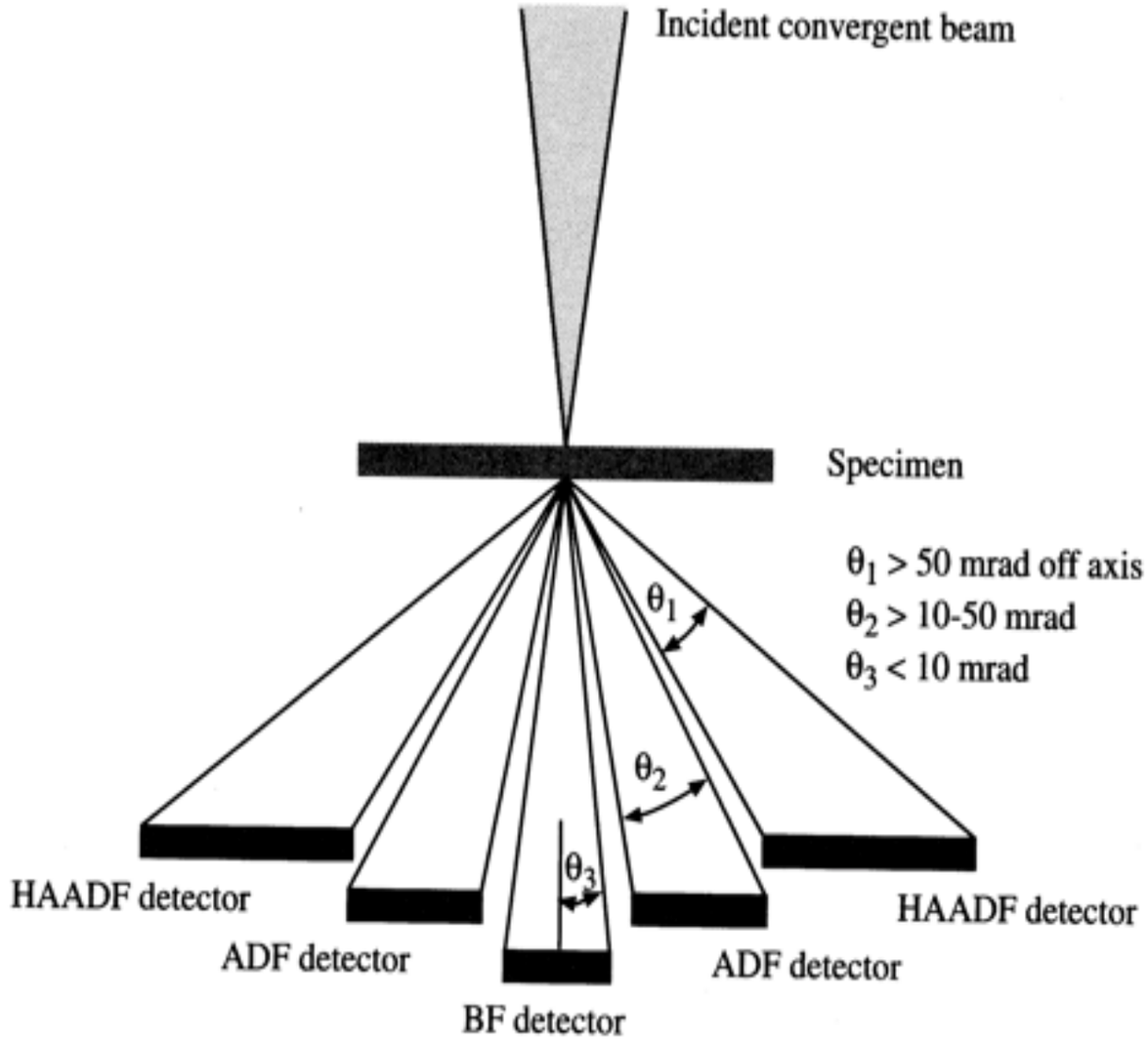



(A)
Small
2α
Thin
specimen
(B)
Medium
2α
(C)
Large
2α
Kossel-Möllenstedt
pattern
Kossel
pattern

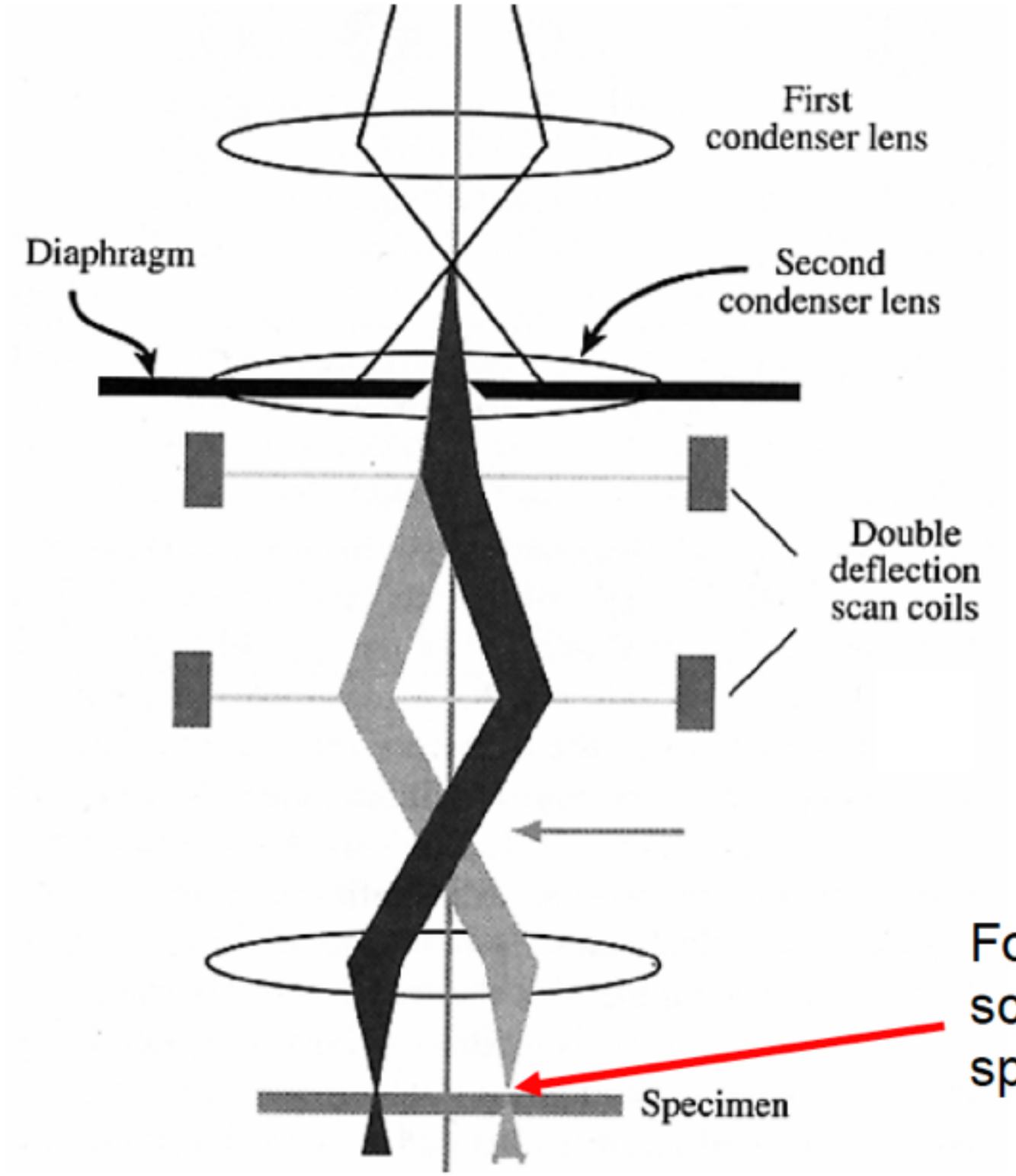

(D)
(E)
(F)

8.3 HAADF- STEM (High Angle Annual Dark Field)





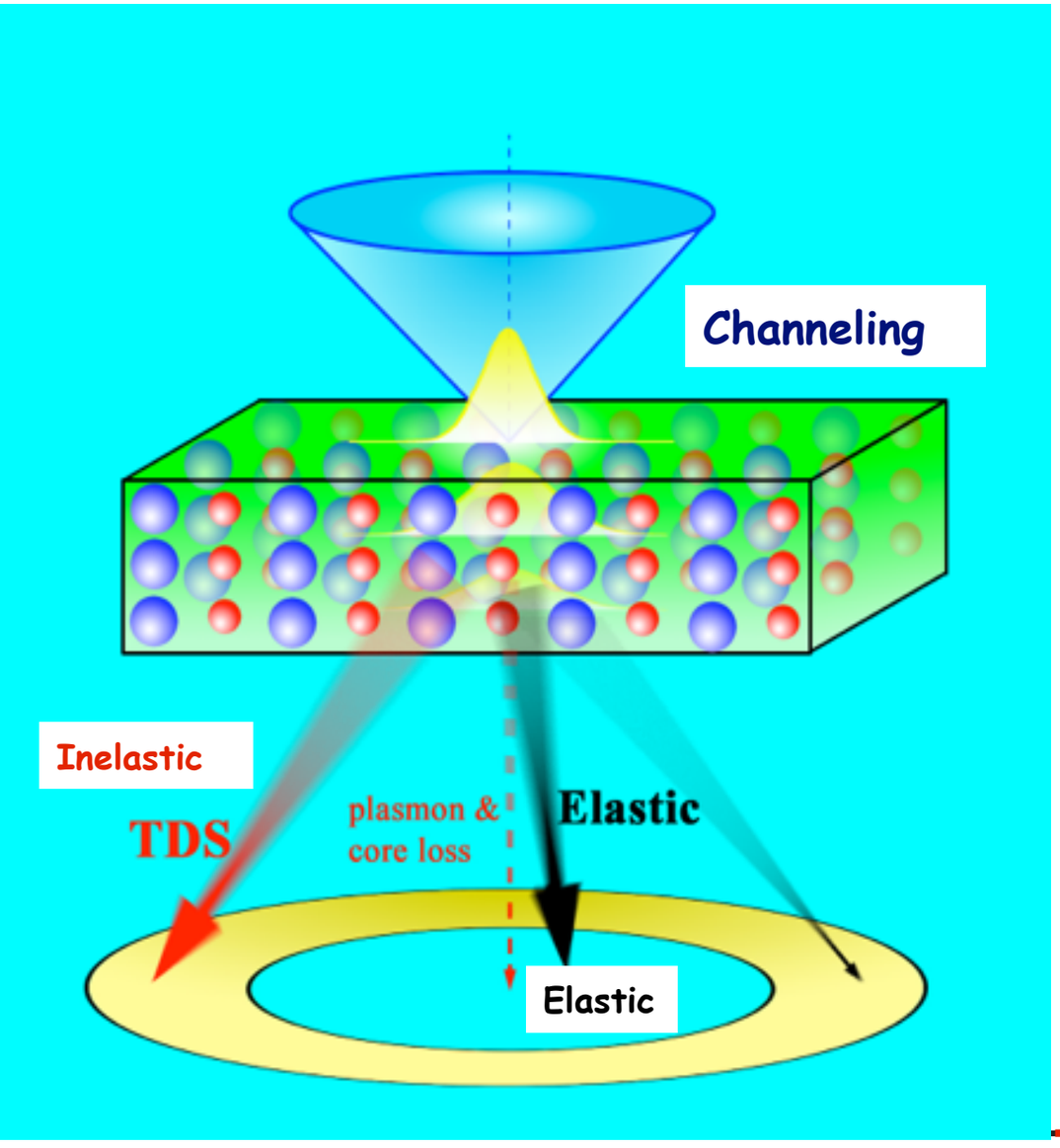
STEM: Scanning a focused spot across the sample

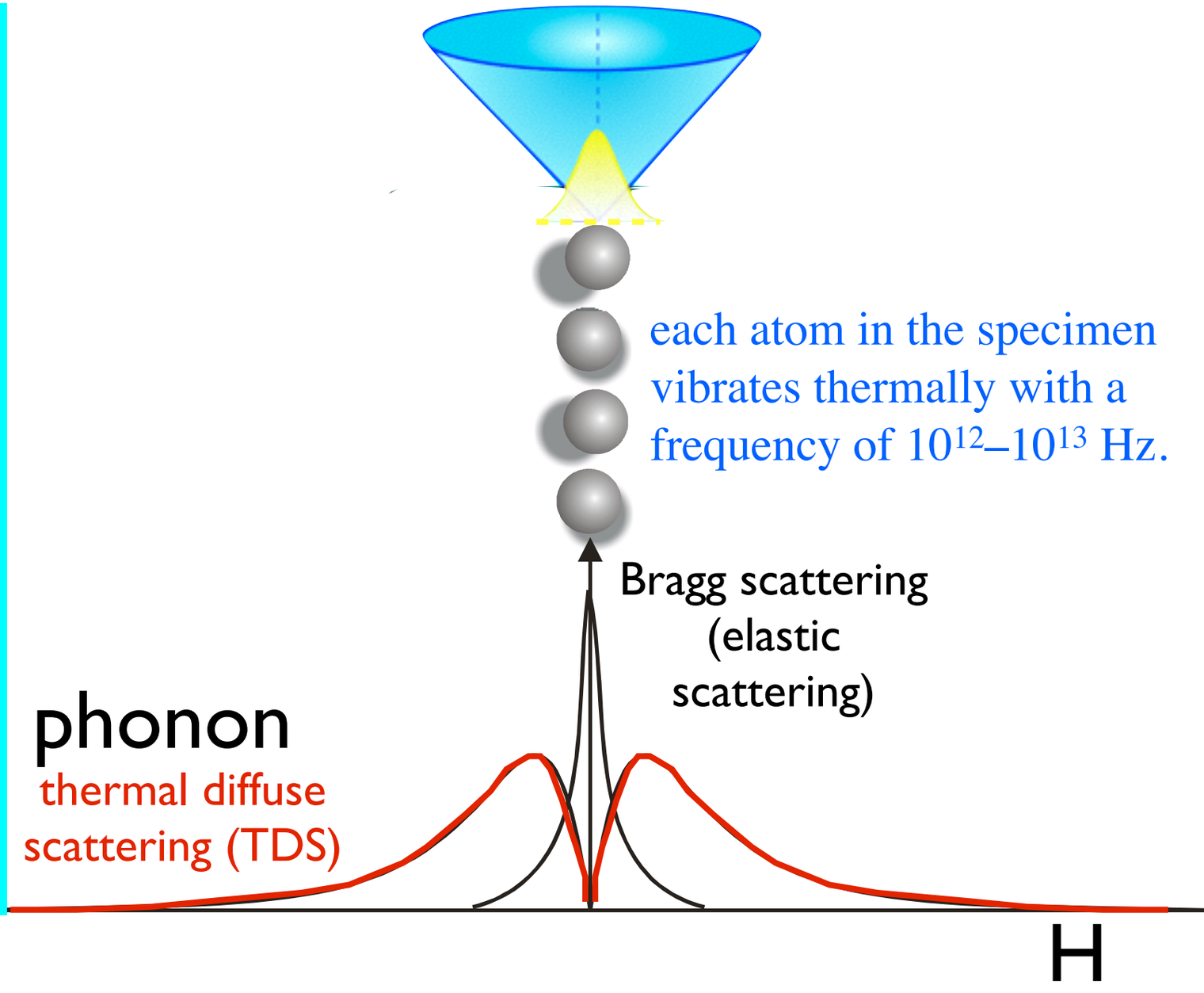


s

Focused spot scanning across specimen

8.4 thermal diffuse scattering (TDS)





Thermal diffuse scattering (TDS), which is a signal used to form the image in HAADF-STEM and which was previously considered as “background intensity,” became a powerful source of information by using an HAADF detector.

8.5 Quantitative interpretation of HAADF-STEM imaging

Intensity $I_S \propto CZ^2Nt$

O(R) * **P^2(R)** = **I(R)**

$1s_{Ga}$ $1s_{As}$ * =

Z=31 Z=33

[110]

[1$\bar{1}$0]

[100]

$I \propto z^{\alpha}$

Ga | | As

1.4Å

STEM Images

- Elastic BF-STEM images are equivalent to TEM bright-field images (reciprocity principle). They are mainly produced Bragg disks hitting the detector. They contain diffraction contrast and are therefore very sensitive to strain/diffraction conditions in the material.

- High-angle annular dark-field STEM (HAADF-STEM) images are mainly produced by thermal diffuse scattering (TDS), because at high scattering angles TDS has the highest scattering cross section.

- (medium angle) ADF-STEM images contain both, Bragg diffraction and TDS contributions.

STEM-Image

The intensity of atom columns in HAADF-STEM imaging depends on the average atomic number Z of individual atom columns (app. proportional to the square). Atom columns with higher average atomic number Z exhibit higher intensity. STEM transfer function has no reversals!

- **Qualitative interpretation of HAADF-STEM images is relatively straightforward.**
- **Quantitative interpretation of HAADF-STEM images, i.e. determination of the chemical composition of atom columns based on intensities, requires extensive image calculations and image matching.**

STEM image signal

- STEM images are acquired **sequentially**, i.e. pixel by pixel. The precision of the image therefore depends on the reproducibility of the beam positioning.

- The emission current of some FEG sources (cold FEGs) may fluctuate. This leads to a **fluctuation** of counts between pixels.

- The signal recorded from the detector is usually amplified with an adjustable bias (threshold) and gain (amplification). This makes it often **impossible to quantify** the number of electrons per pixel in a given STEM image.

- Less than 10% of the incident electrons scatter to the HAADF detector (depends on detector geometry, of course). This makes HAADF images **very noisy**. However, the very high contrast of HAADF images compensates for some of the noise.

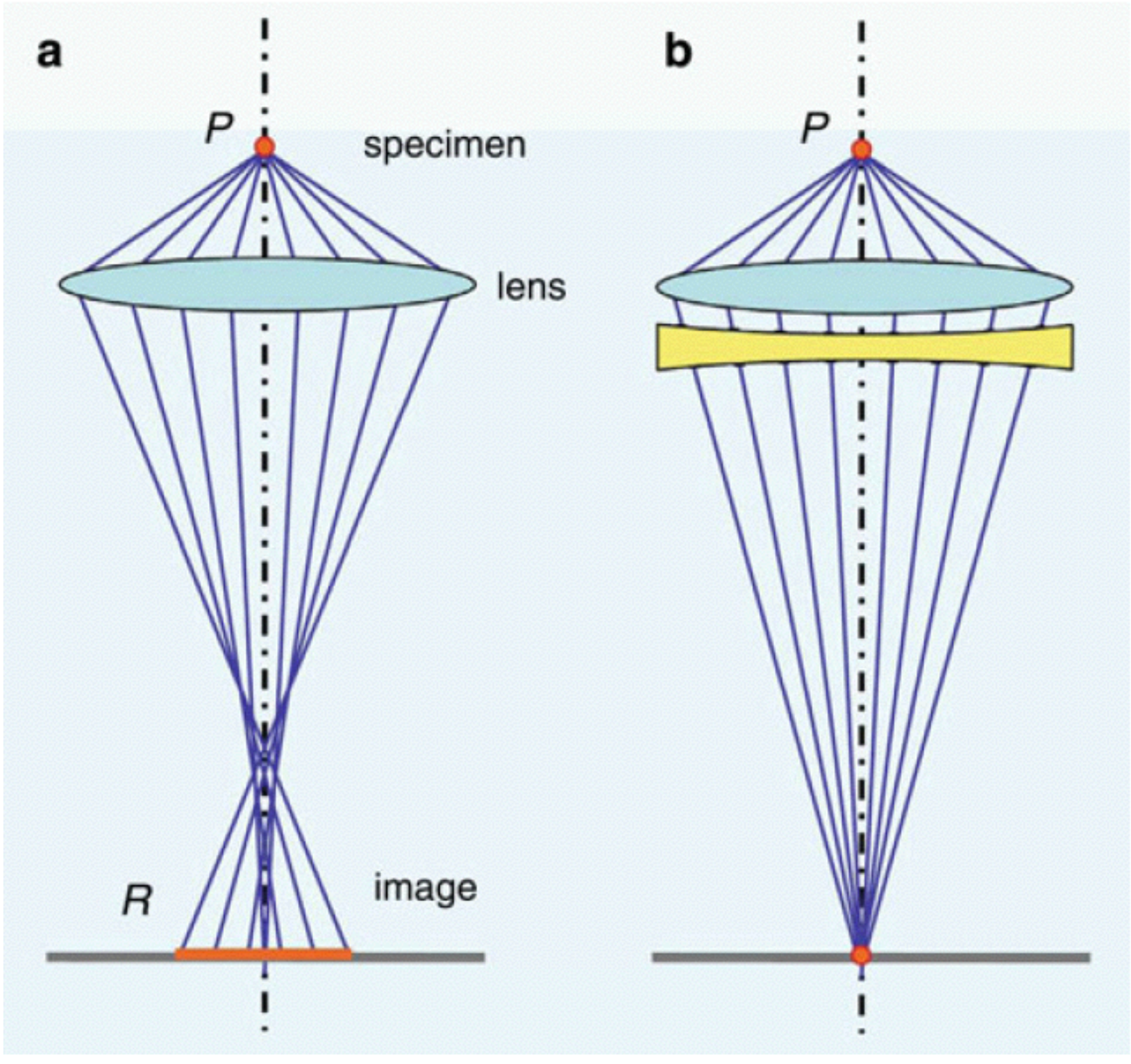
8.6 Comparison of HRTEM and HAADF

HRTEM		HR HAADF-STEM
Parallel illumination	⟷	Convergent beam
Reversals in contrast transfer function	⟷	No reversal in contrast transfer function
No image distortions	⟷	Possible image distortions
Point-to point resolution < 0.1nm	⟷	Point-to-point resolution ~ 0.13nm (0.1 nm)
Limited chemical information	⟷	Limited structural information
Well established simulation procedures	⟷	Few available simulation programs

Phase Contrast

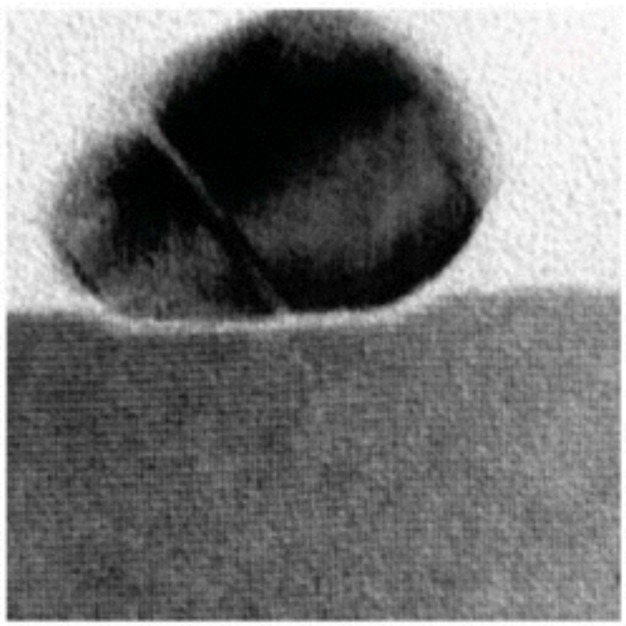
Amplitude Contrast

Aberration corrected TEM

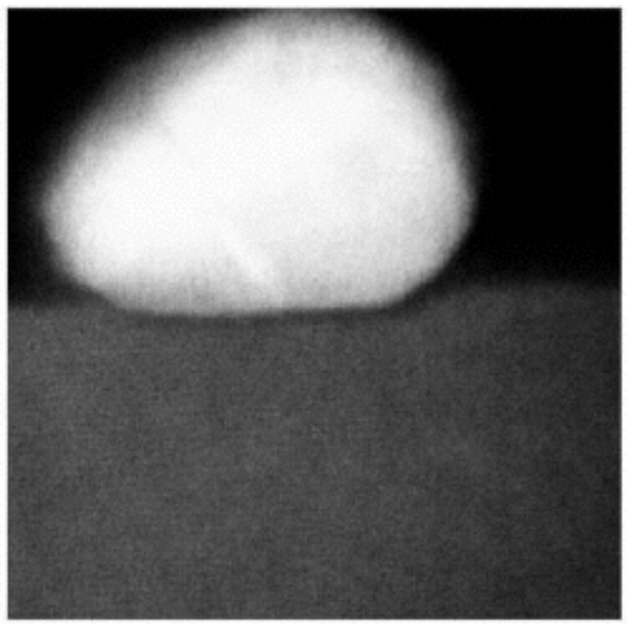


Examples of STEM images

Example Pd particle on SrTiO3

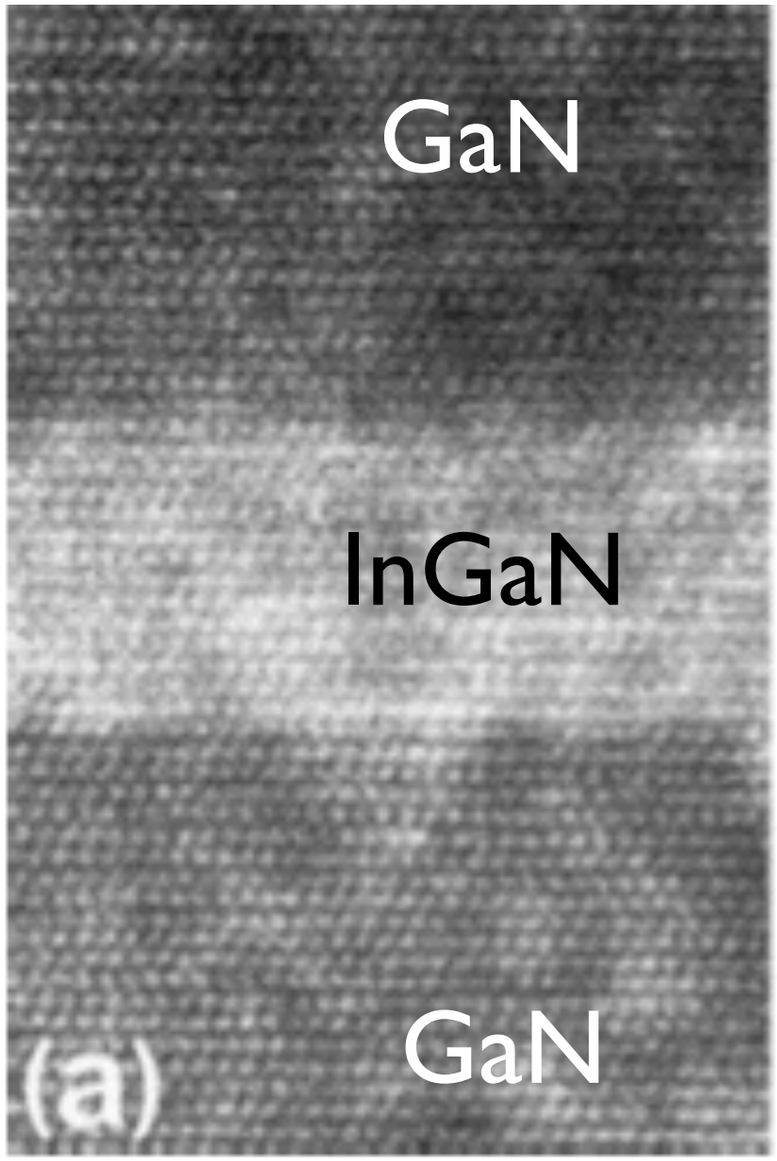


BF-STEM image
(contains diffraction contrast)



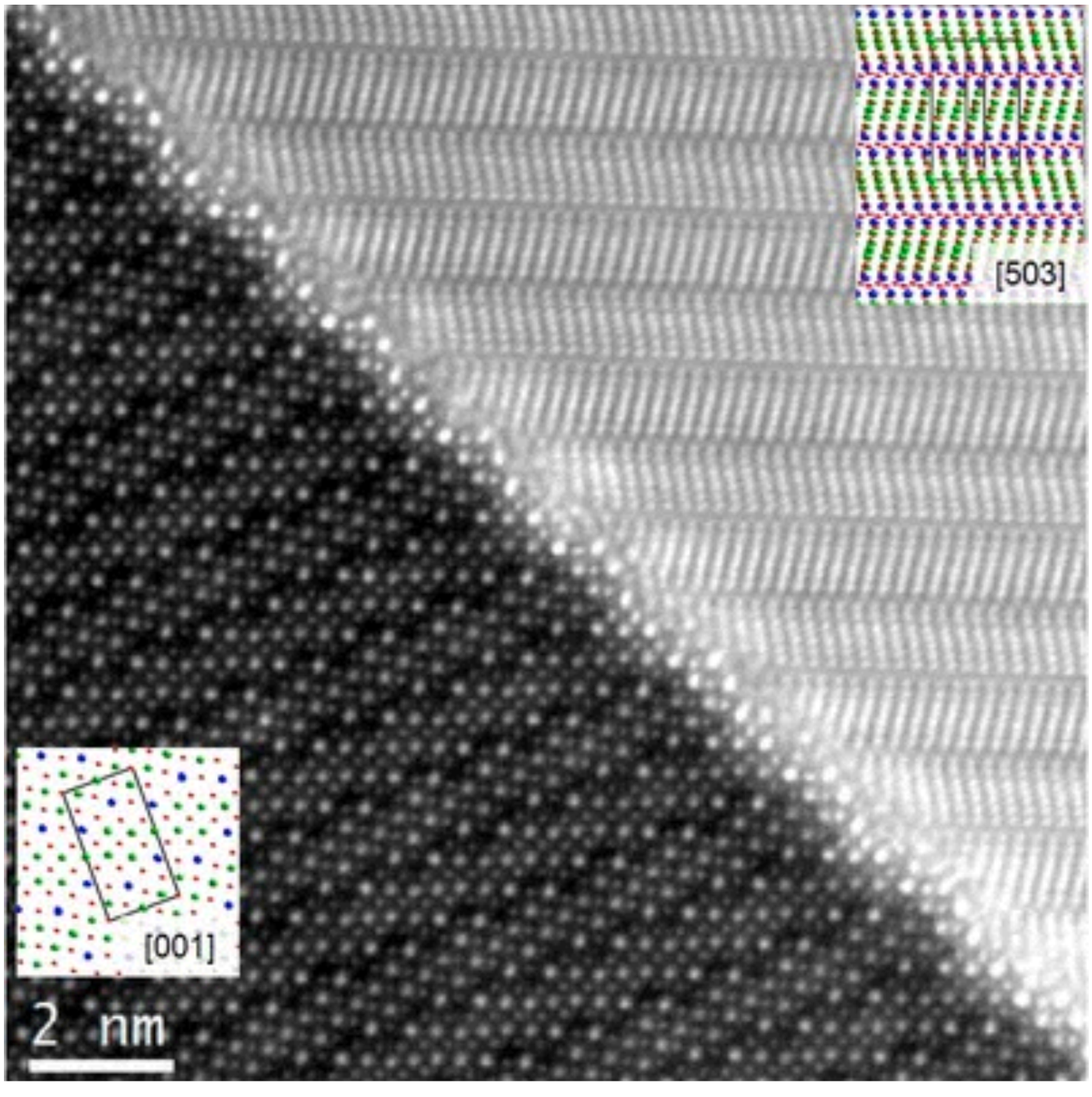
HAADF-STEM image
(chemical sensitivity because of Z-contrast)

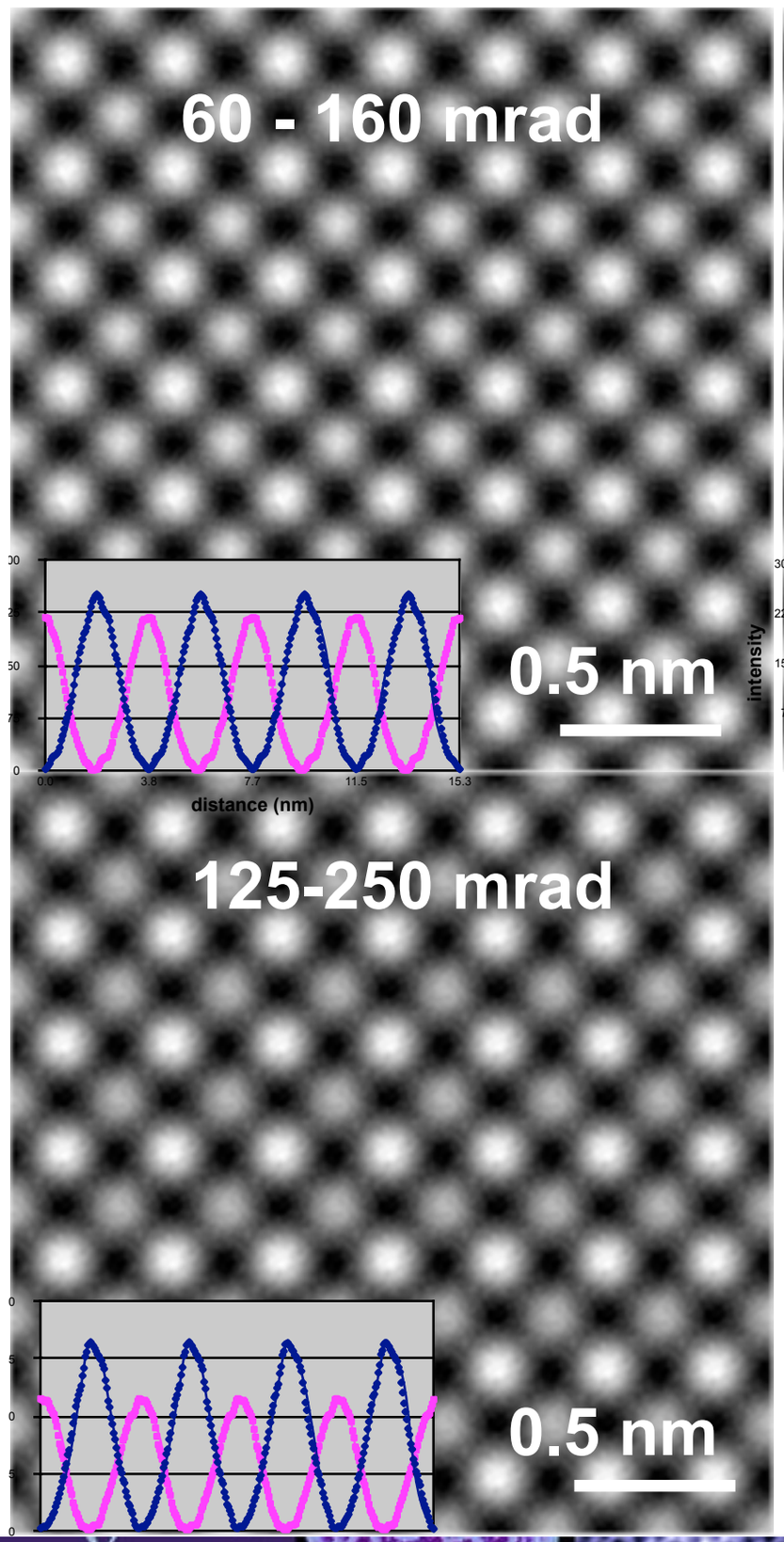
Quantum Well

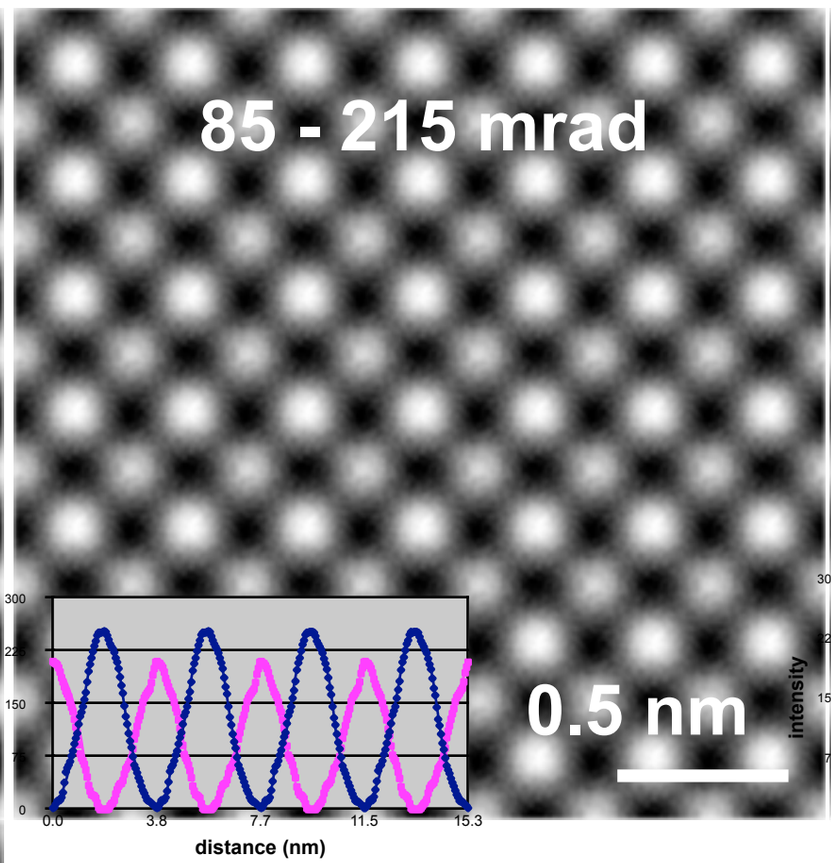


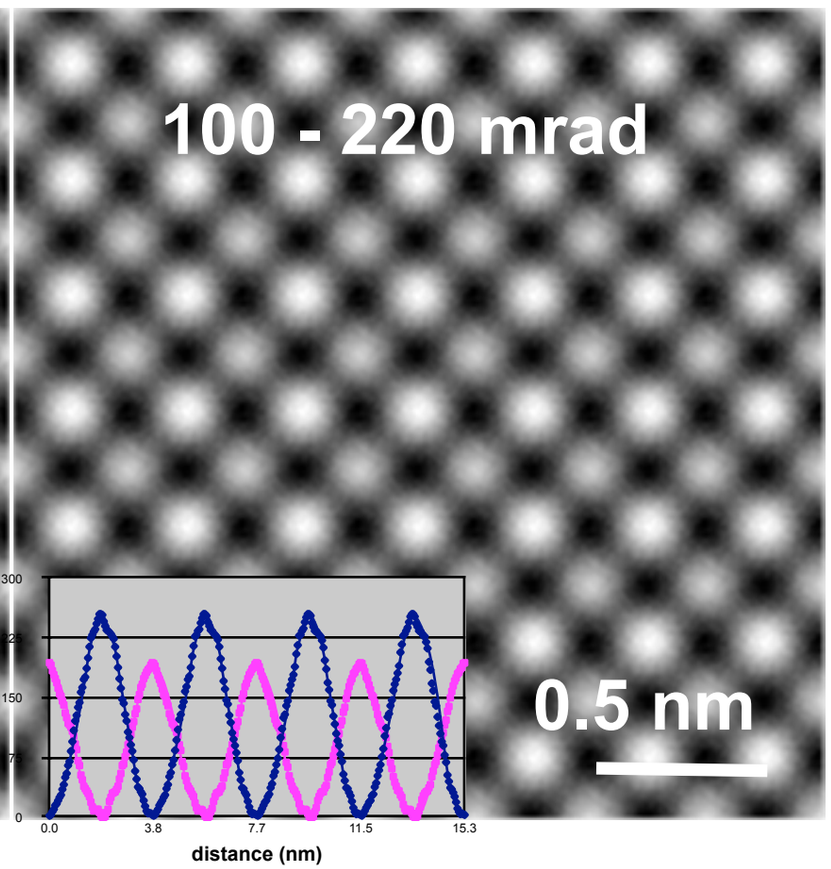
Examples of STEM images

MAADF image of grain boundary in $Ba_{6-3x}Nd_{8+2x}Ti_{18}O_{54}$ (BNT)





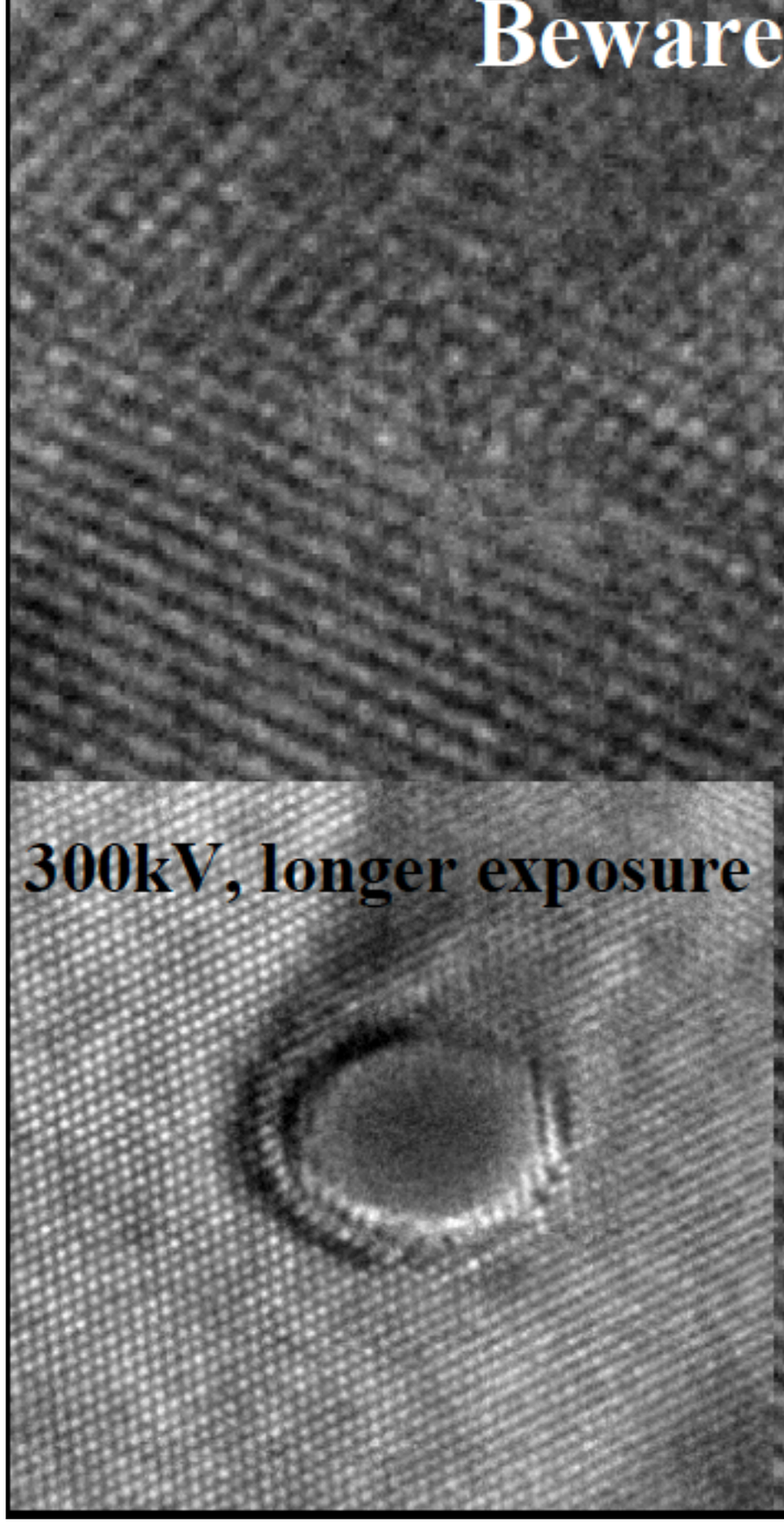




Collection angles

HR STEM HAADF images of $CaTiO_3$ in the [001] zone axis taken at different detector inner and outer collection angles. The intensity ratio between Ti-O/Ca atomic columns is increasing with detector collection range.

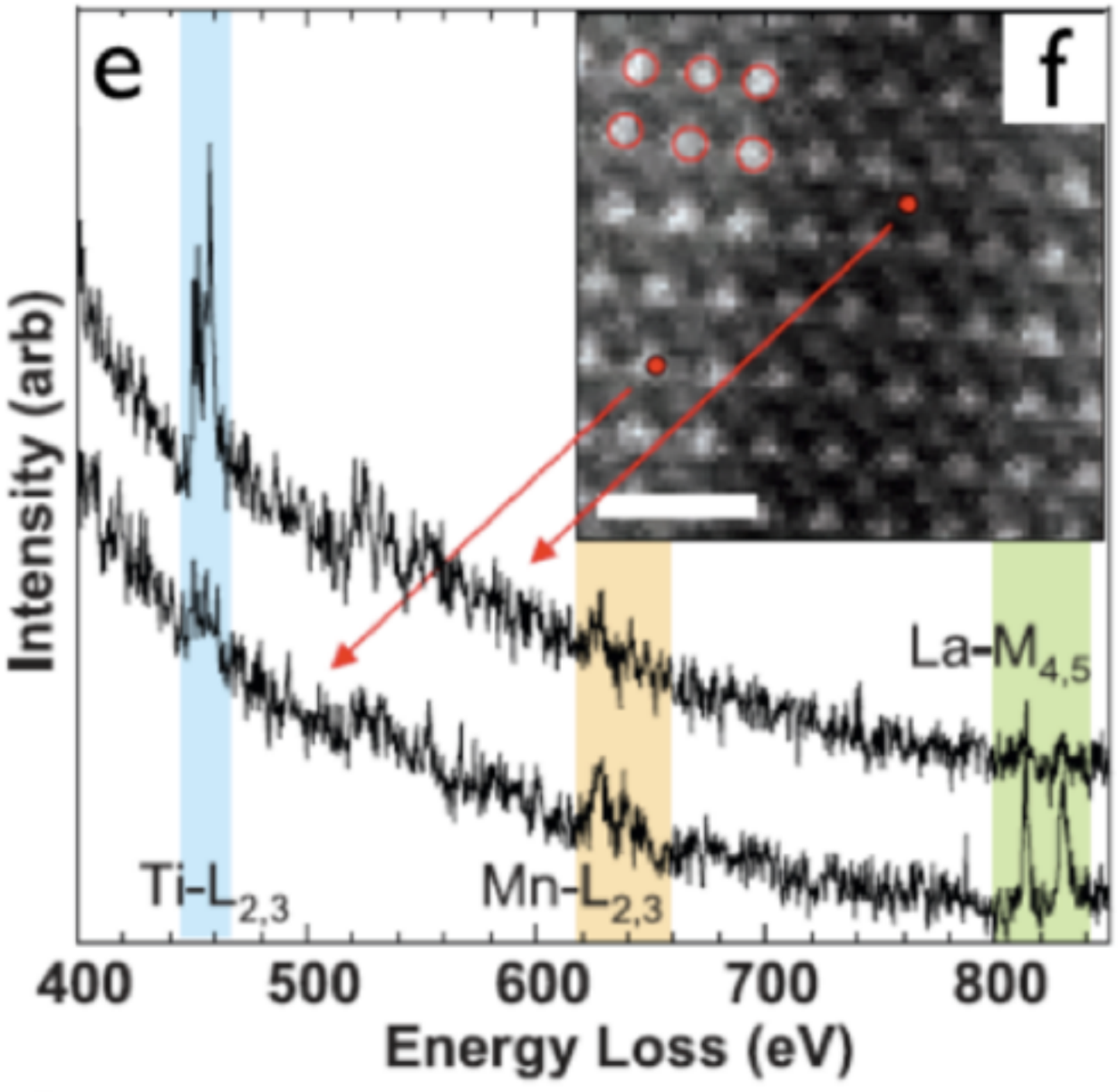
Beware of Beam Damage!



300kV, 1-2sec exposure

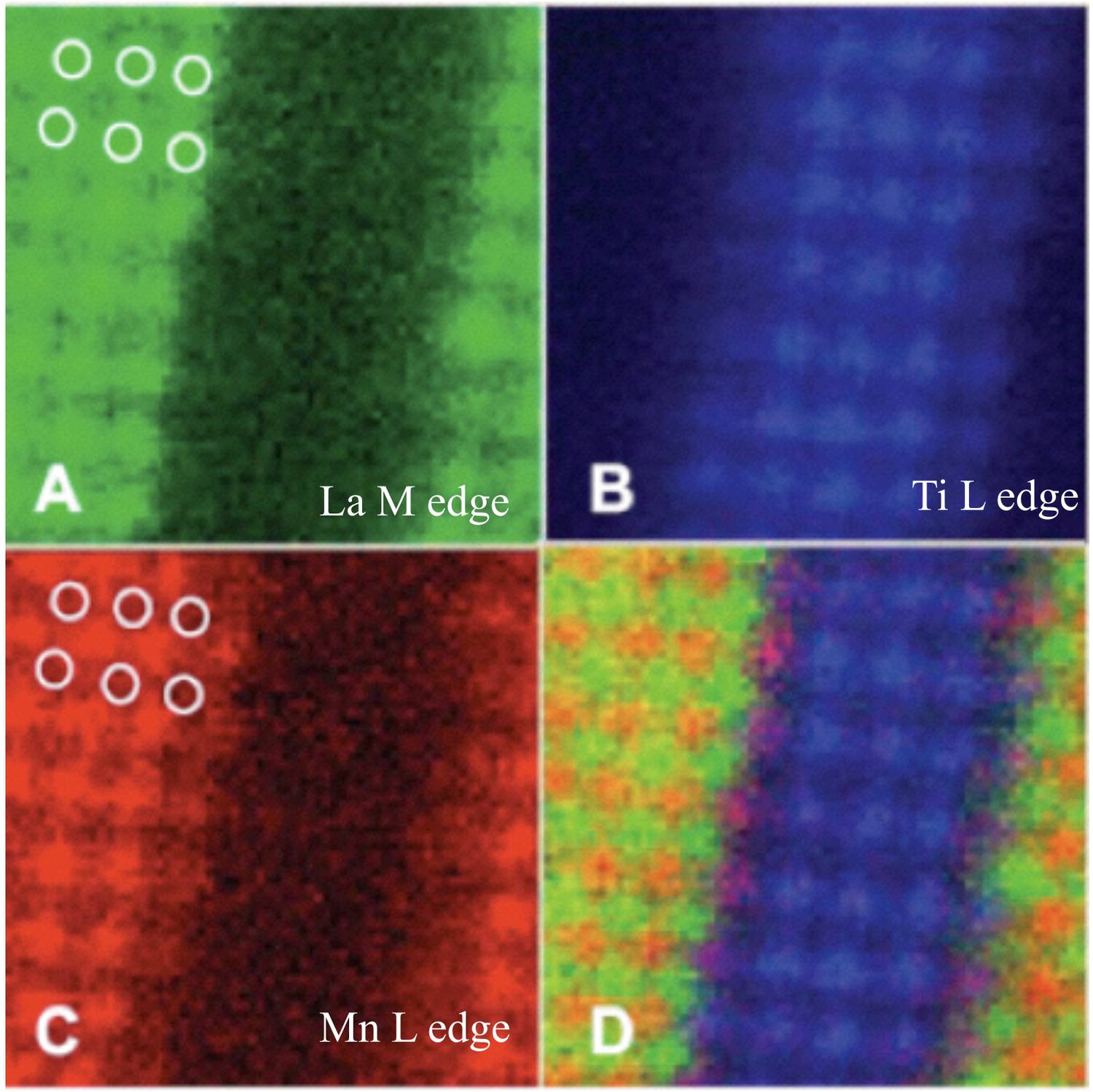
C. Kisielowski, NCEM Berkeley

Atomic resolution of EELS of $La_{0.7}Sr_{0.3}MnO_3/SrTiO_3$ multilayer



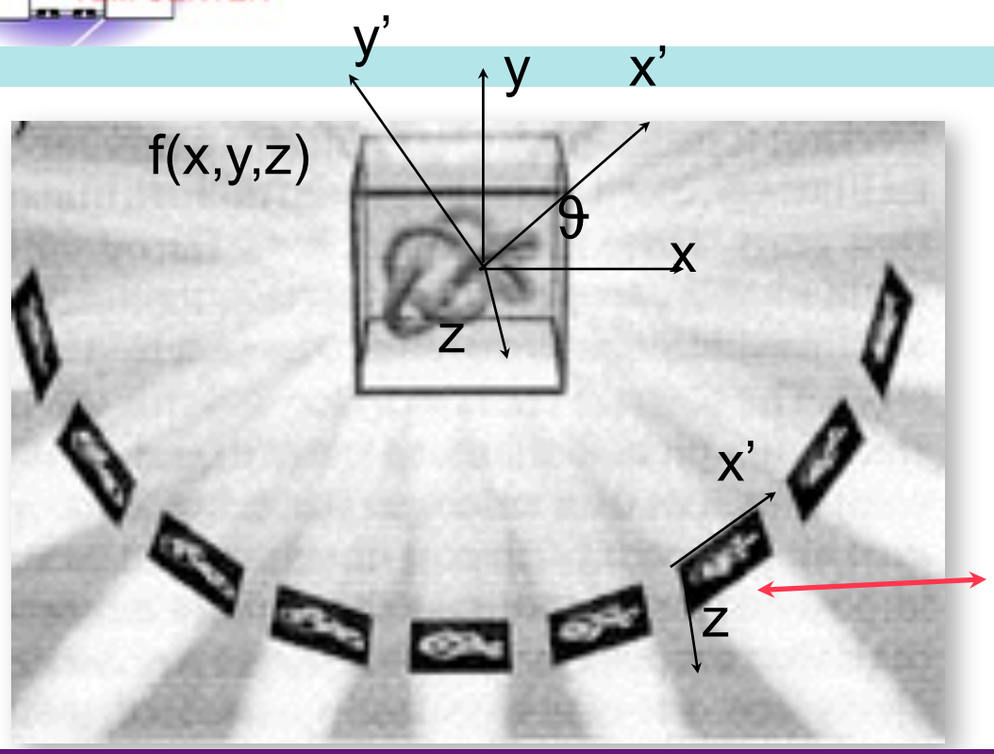
- D. A. Muller, et al, SCIENCE **319** 1073-1075 (2008)

8.7 Atomic Resolution Spectrum Imaging



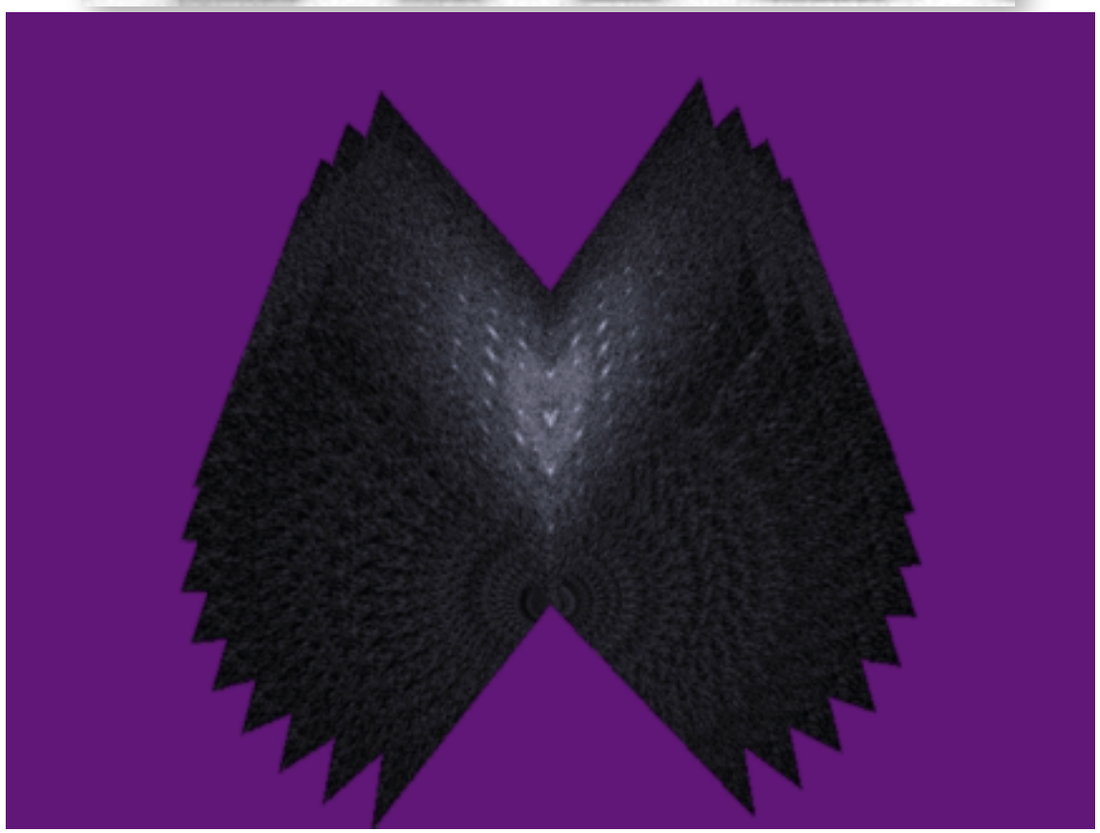
Atomic resolution compositional and bonding maps

8.8 STEM Tomography

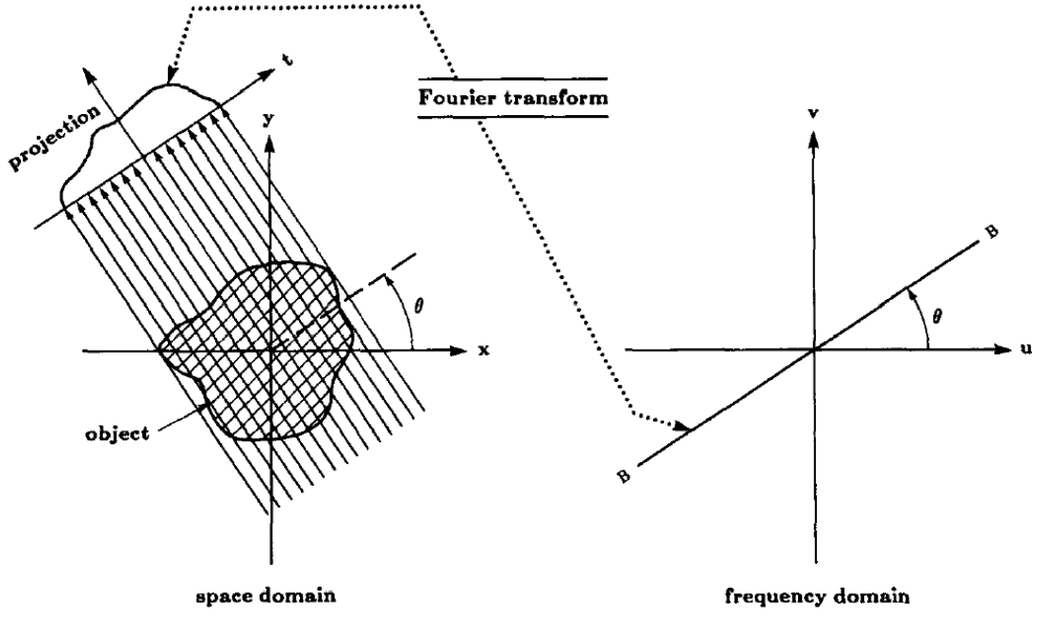


1. Kinematic diffraction
2. Lens aberration can be ignored

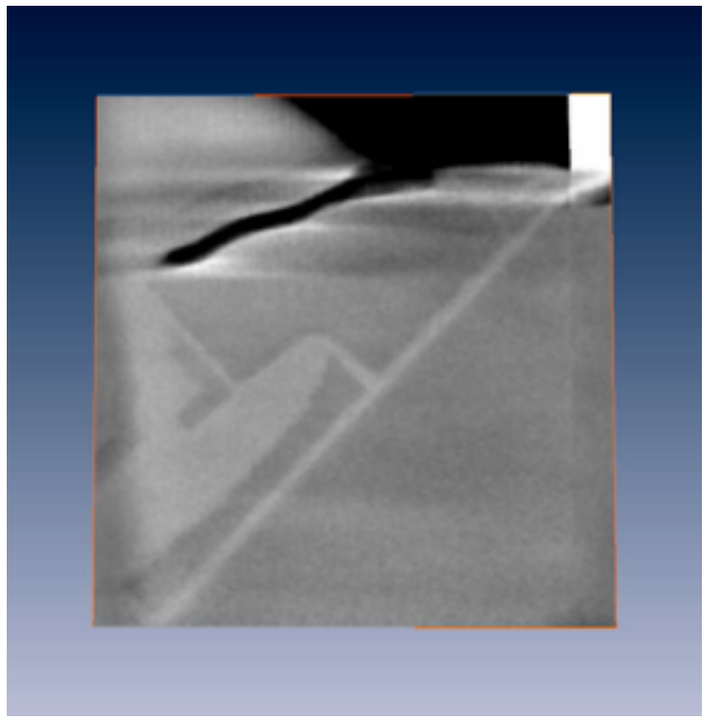
Inverse Randon Transformation
Tomographic Reconstruction

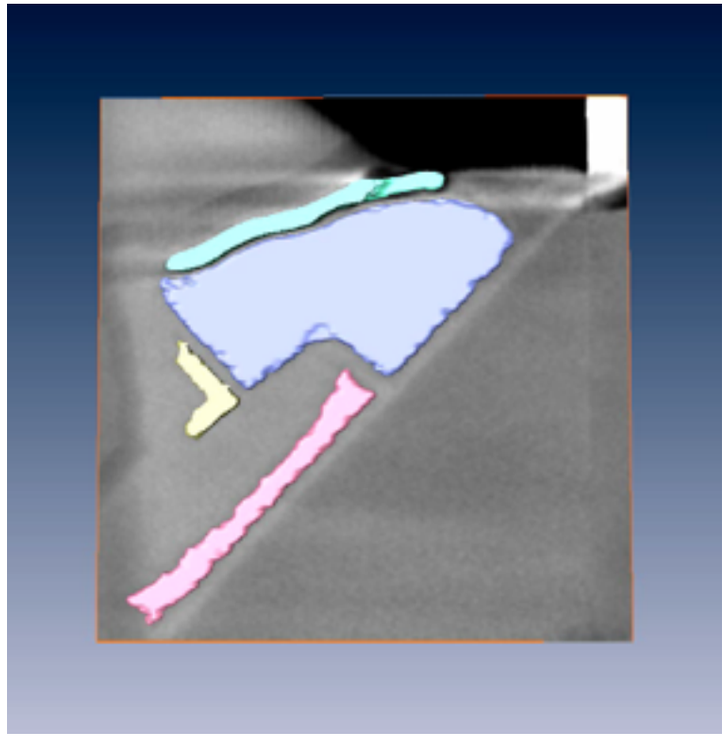


Fourier Projection-Slice Theorem

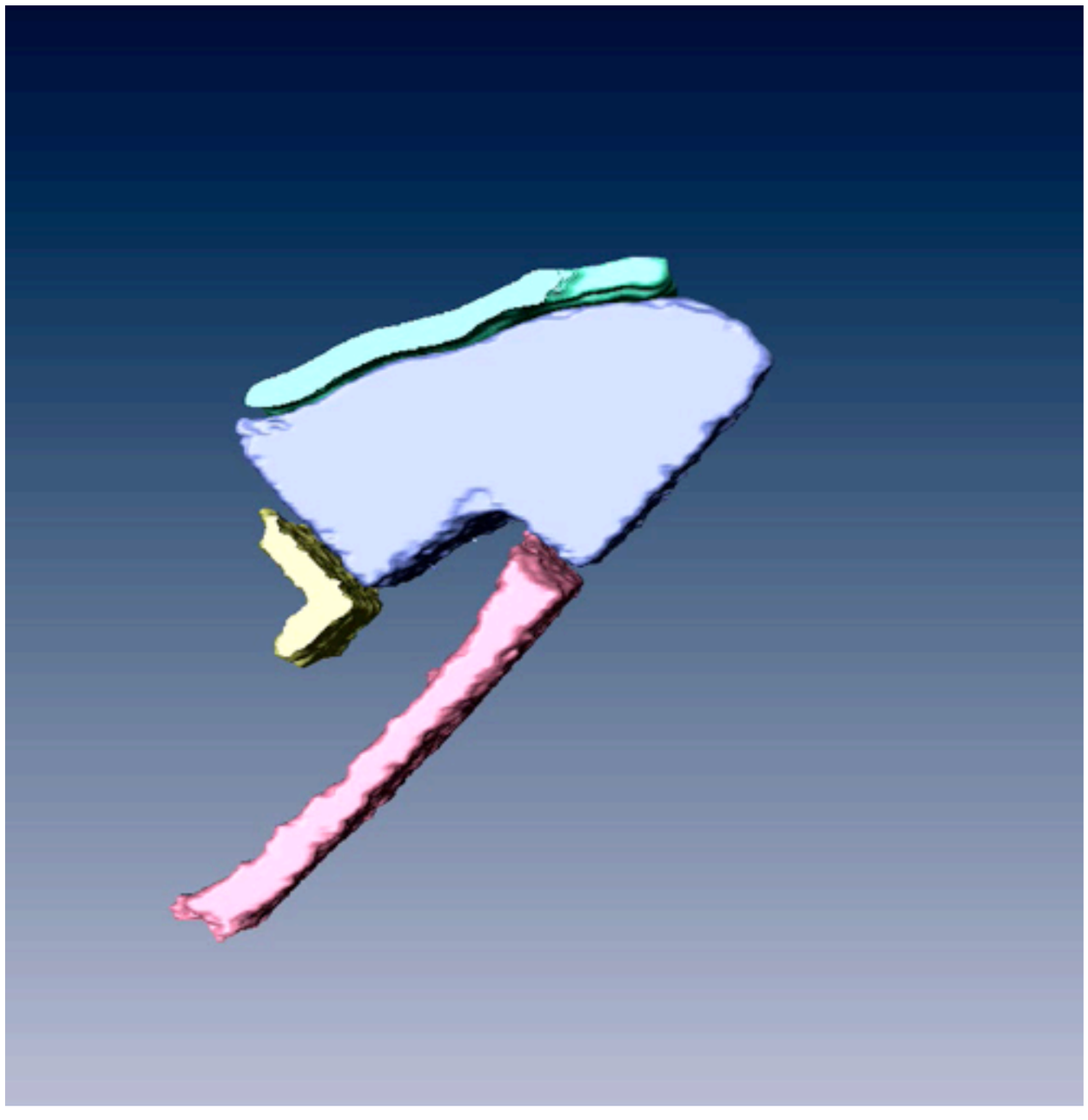


Micro-electronics

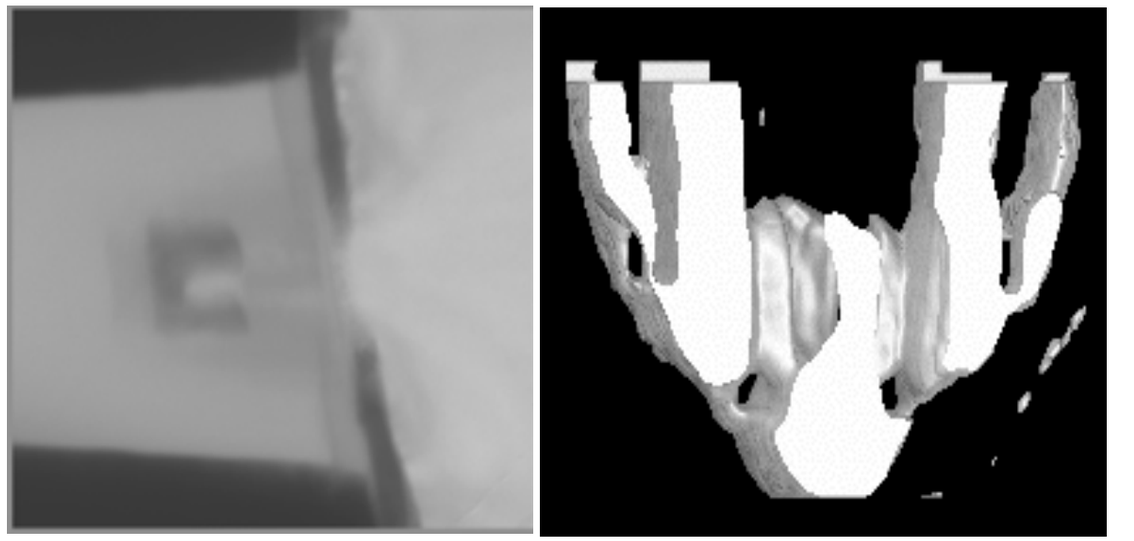


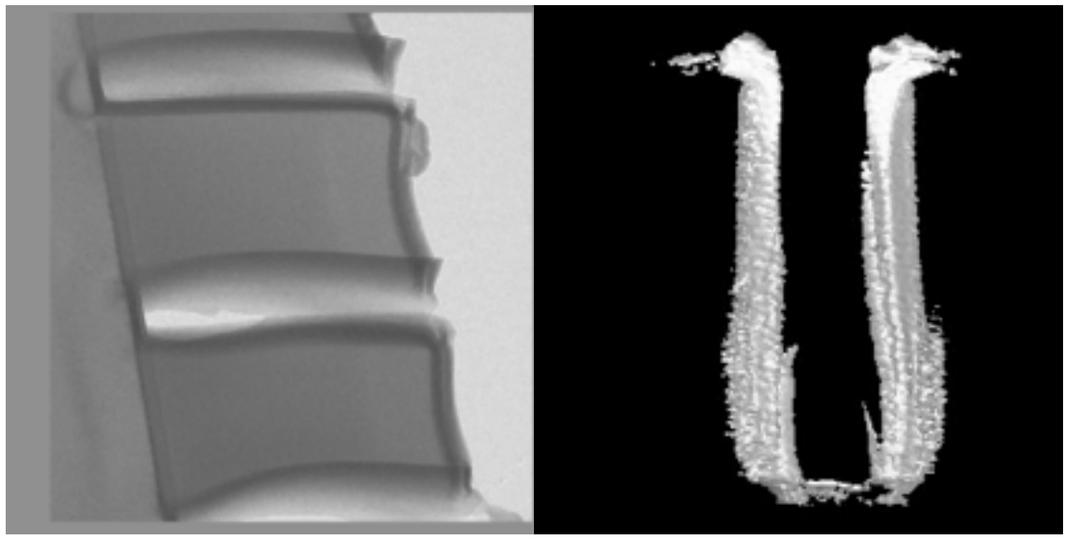


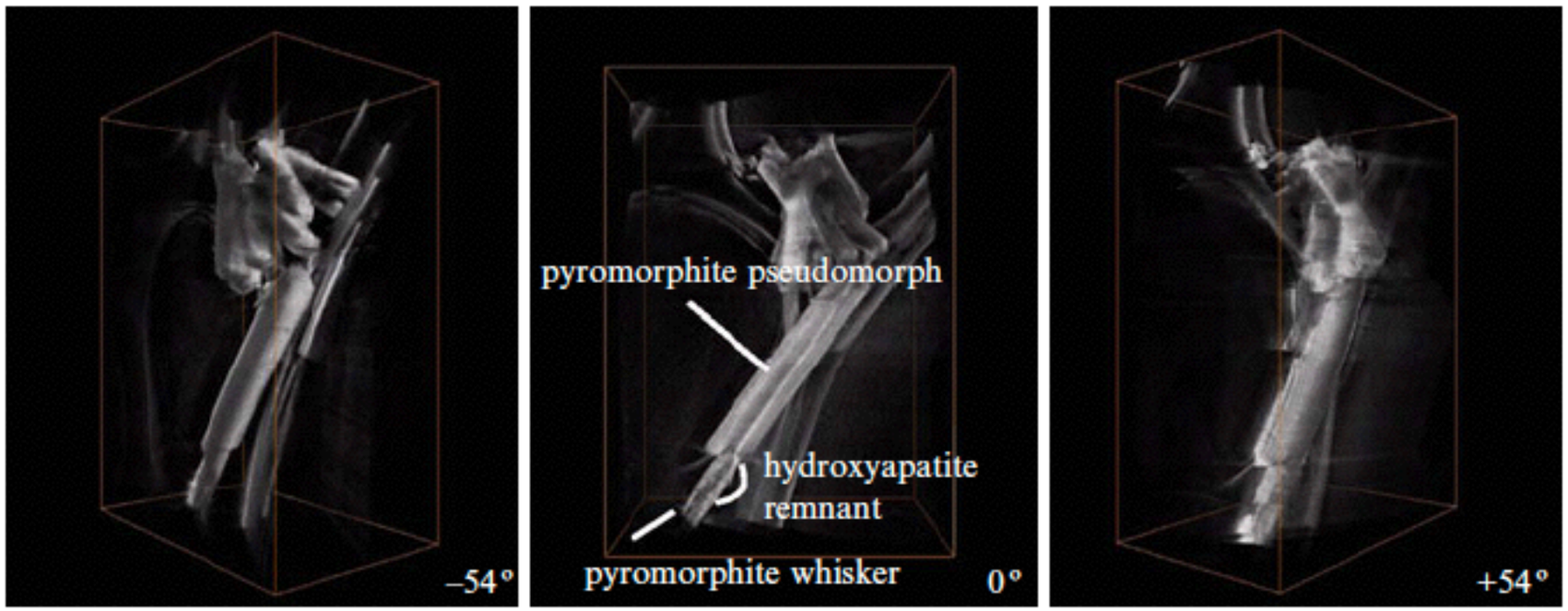
Flash device



8.8 STEM Tomography







Hydroxypyromorphite nanocrystallite whisker and pseudomorph formed on hydroxyapatite