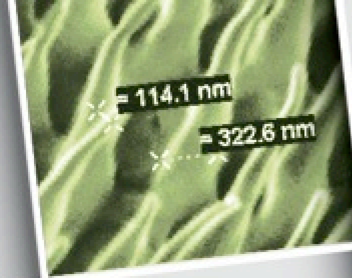
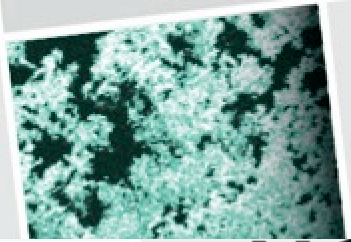




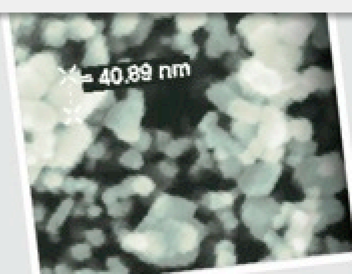
NACK NETWORK
Building College-University Partnerships for Nanotechnology Workforce Development

NCI Southwest

Advances in Transmission Electron Microscopy

Friday, April 21, 2017

Click here to watch the webinar recording

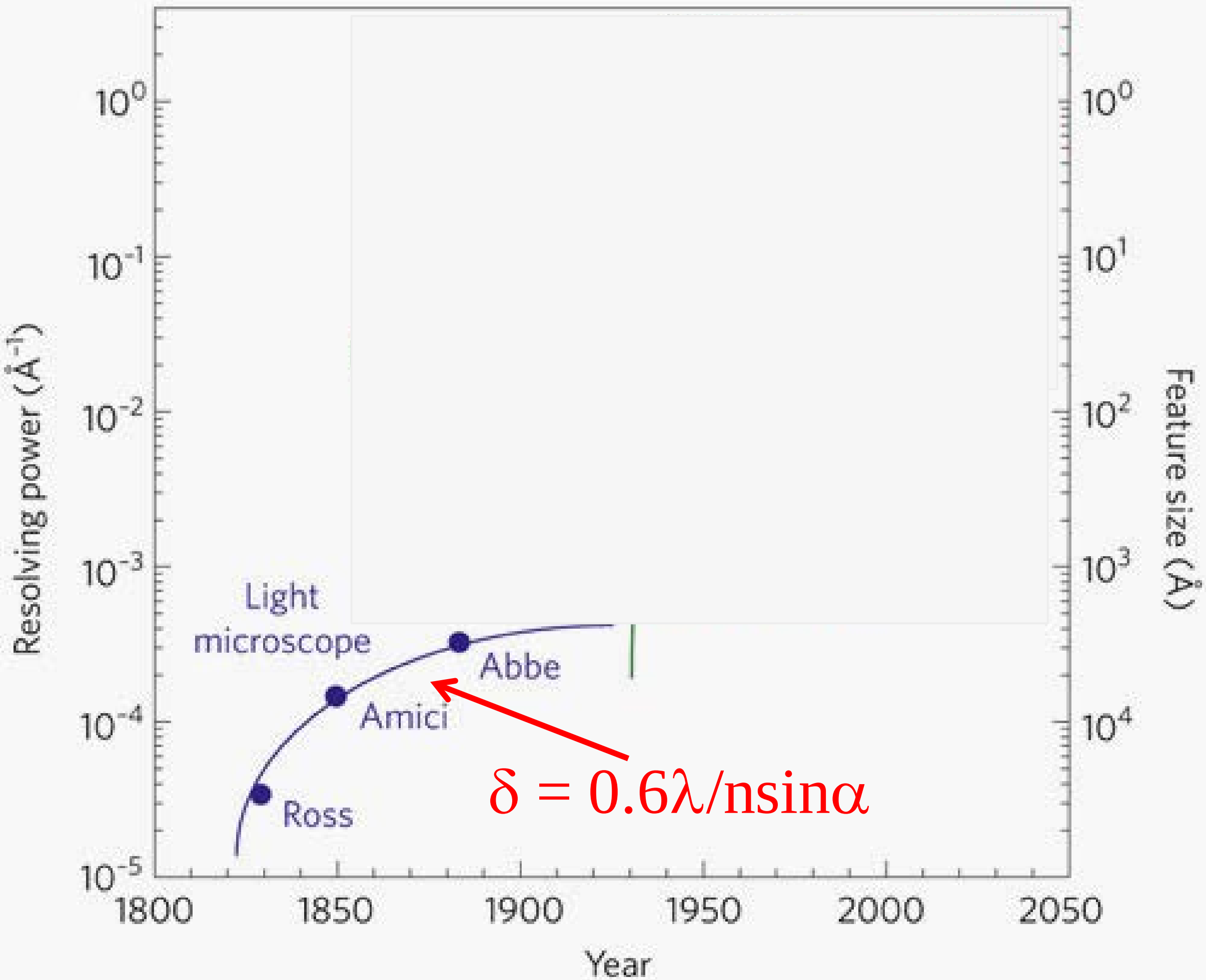


Advances in transmission electron microscopy

Tom Sharp
LeRoy-Eyring Center for Solid State Science
Arizona State University

Improving microscope resolution

DA Muller (2009)
Nature Materials



Resolution of an optical microscope is limited by the diffraction limit:

$\delta = 0.6\lambda/n\sin\alpha$

Resolution could be improved by reducing the wavelength (λ)

The first electron microscope

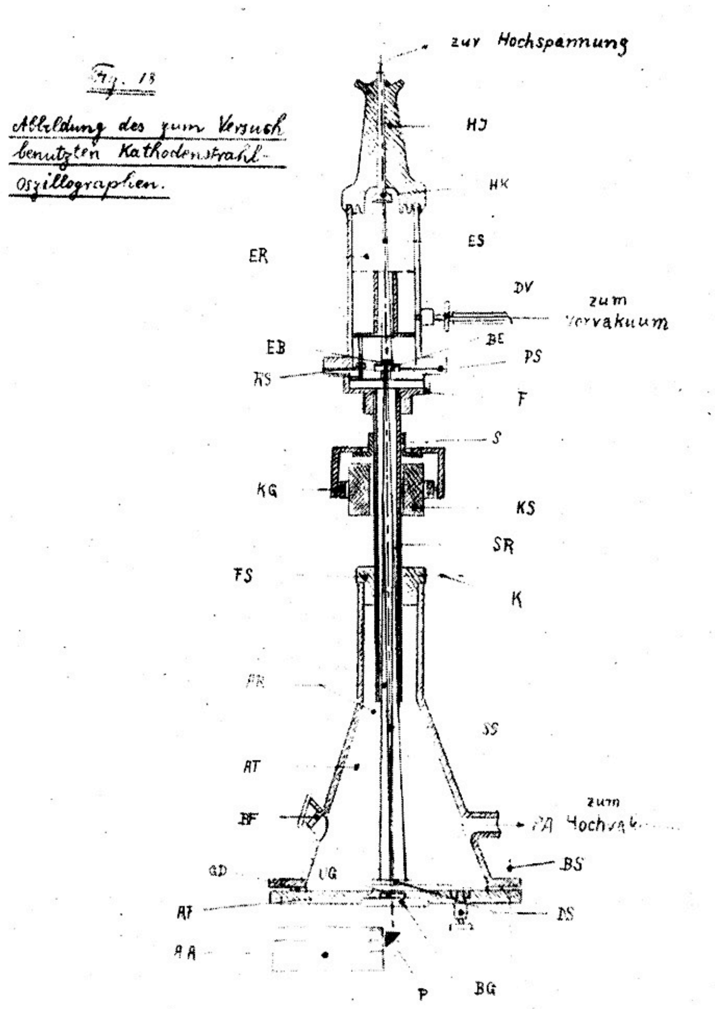
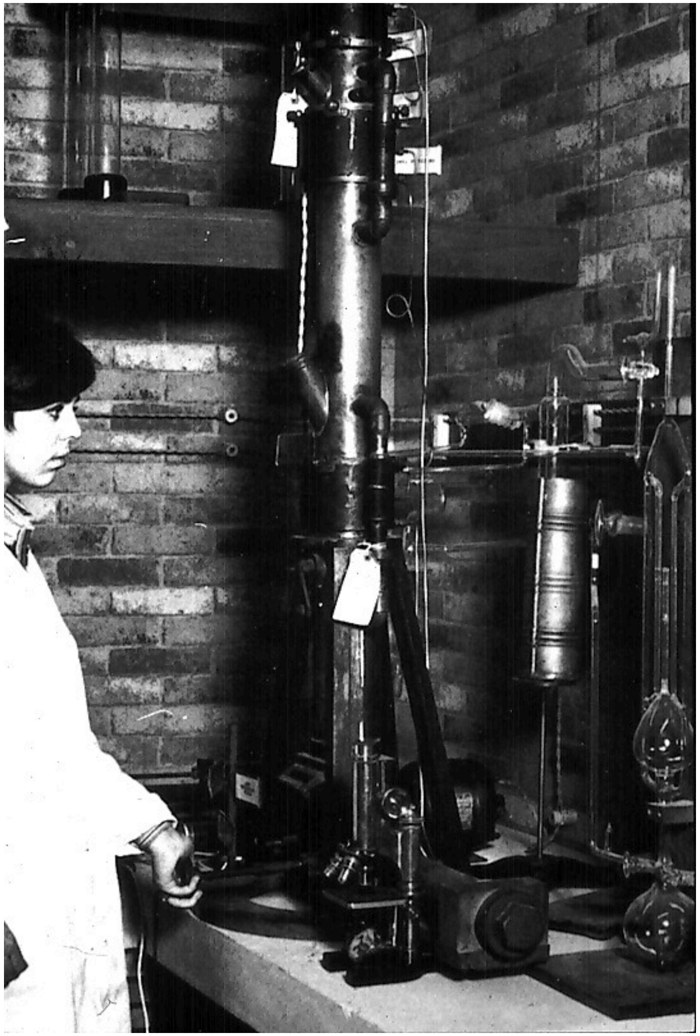
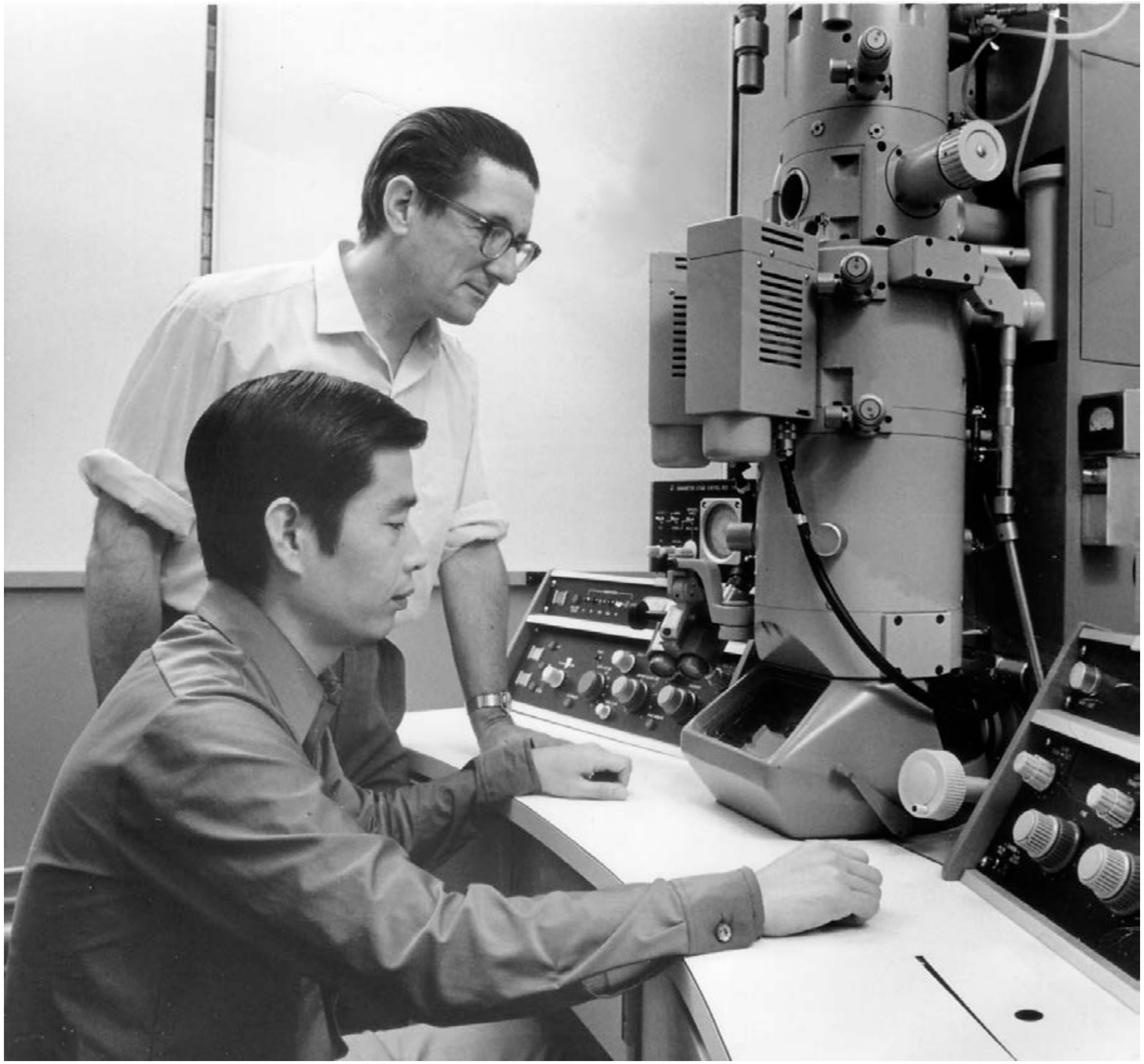


FIG. 1. Sketch by the author of the cathode-ray tube for testing the imaging properties of the nonuniform magnetic field of a short coil (Ruska, 1929; Ruska and Knoll, 1931) (footnote 1).



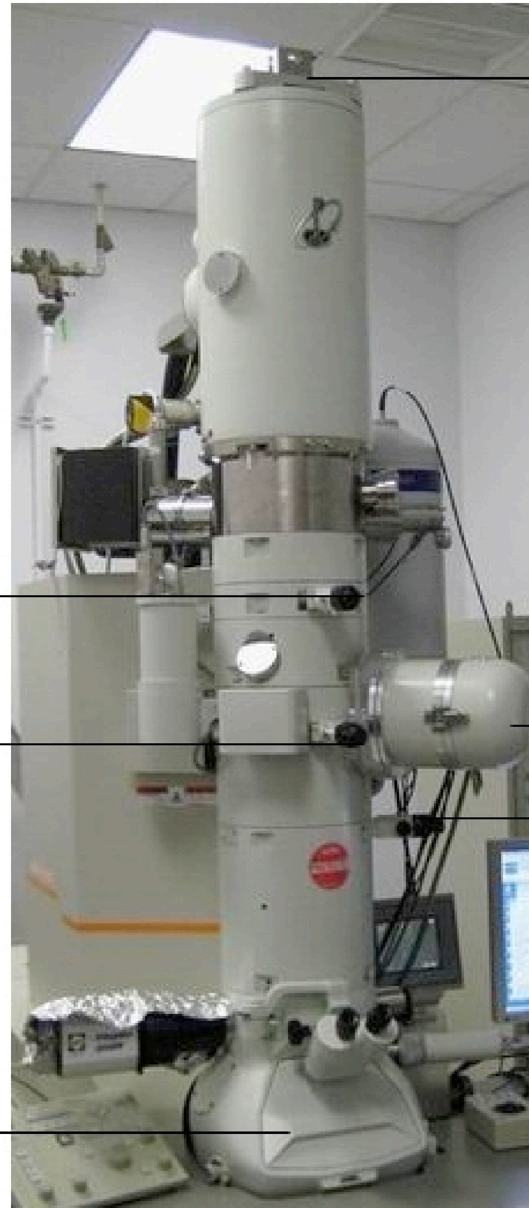
Ruska and Knoll in the 1931.

John Cowley with Sumio Iijima



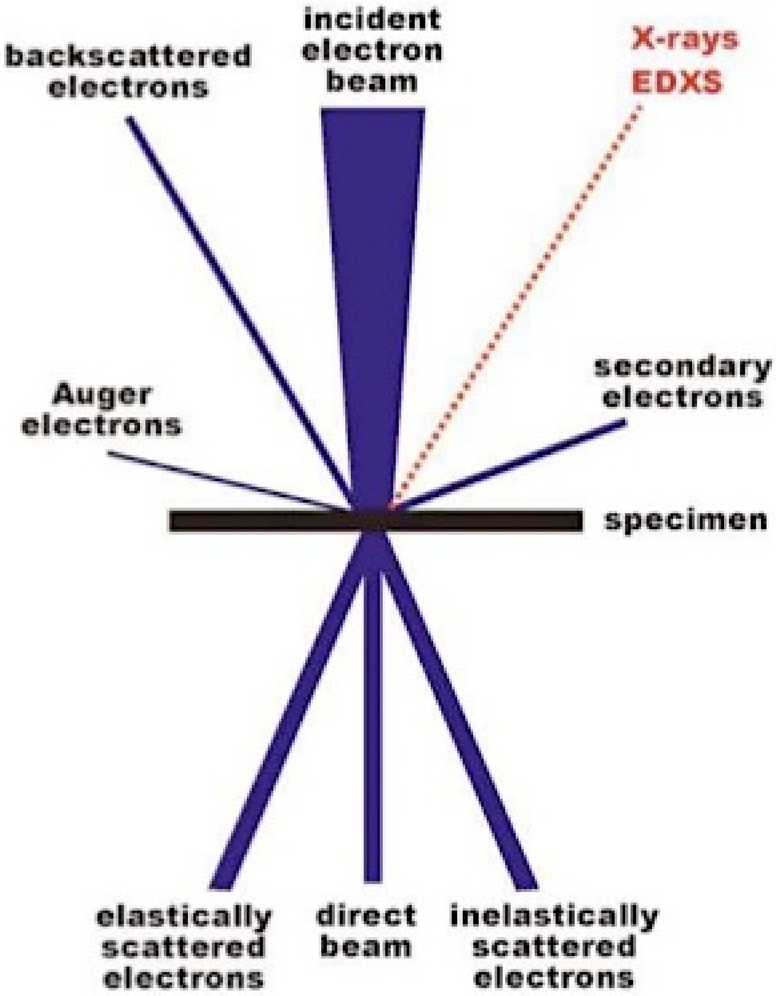
John Cowley with Sumio Iijima at the controls of the JEM-100B (Tempe, 1974)

Layout of a Transmission Electron Microscope

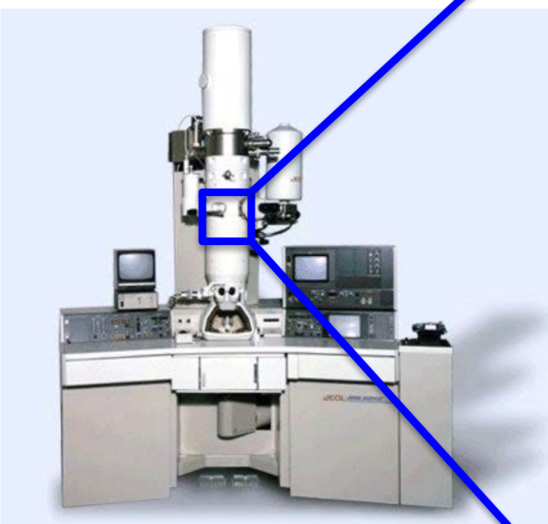


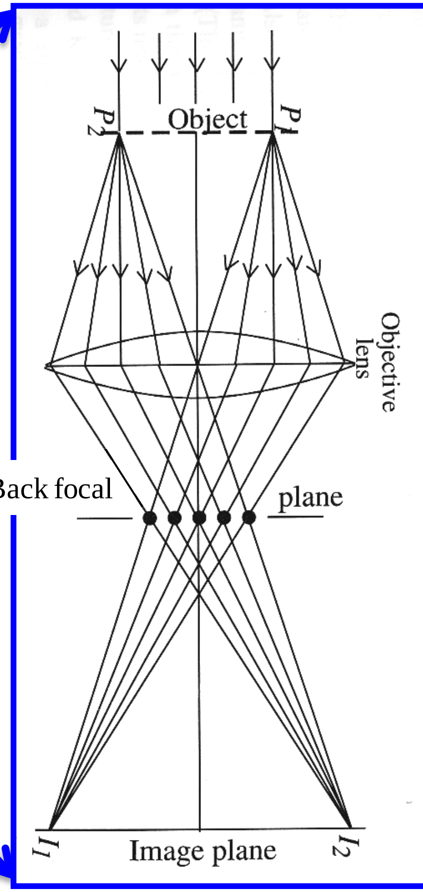
Electron Matter Interactions

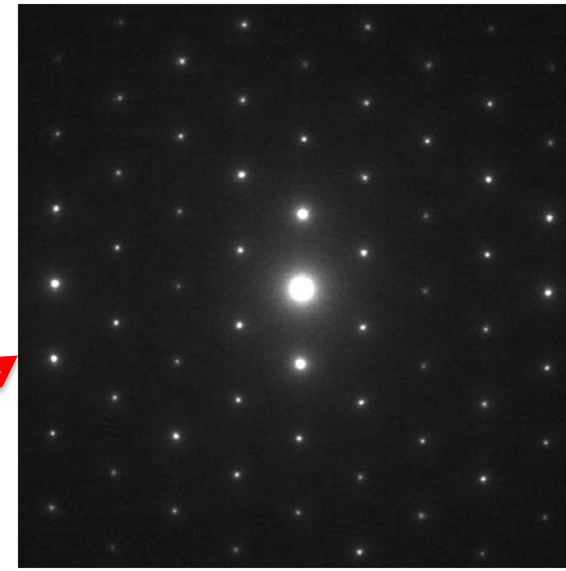
- Electrons are easily scattered.
- Elastic scattering
  - no change in energy.
  - diffraction
- Inelastic scattering
  - transferring energy to the sample.
  - Used for microanalysis:

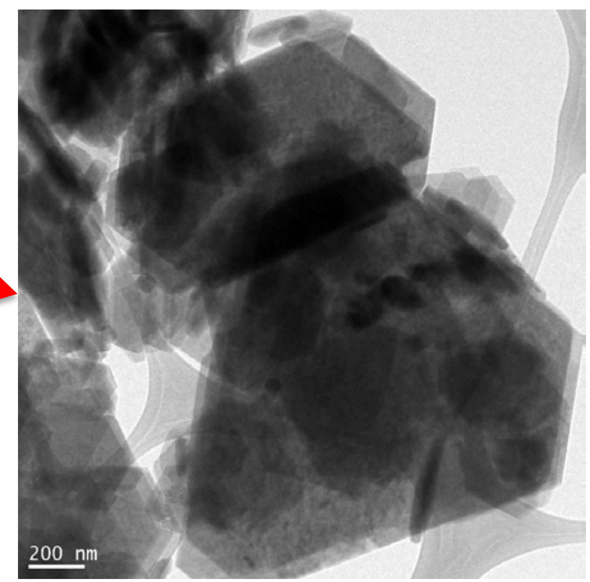


Electron imaging and diffraction







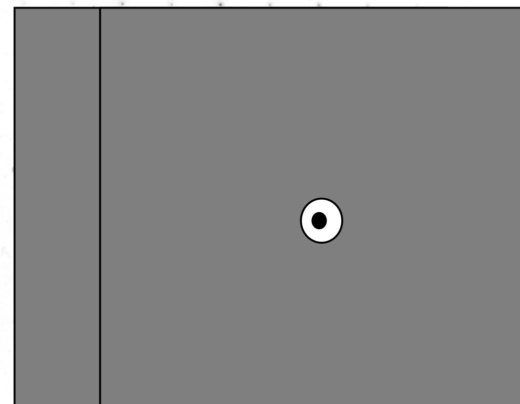


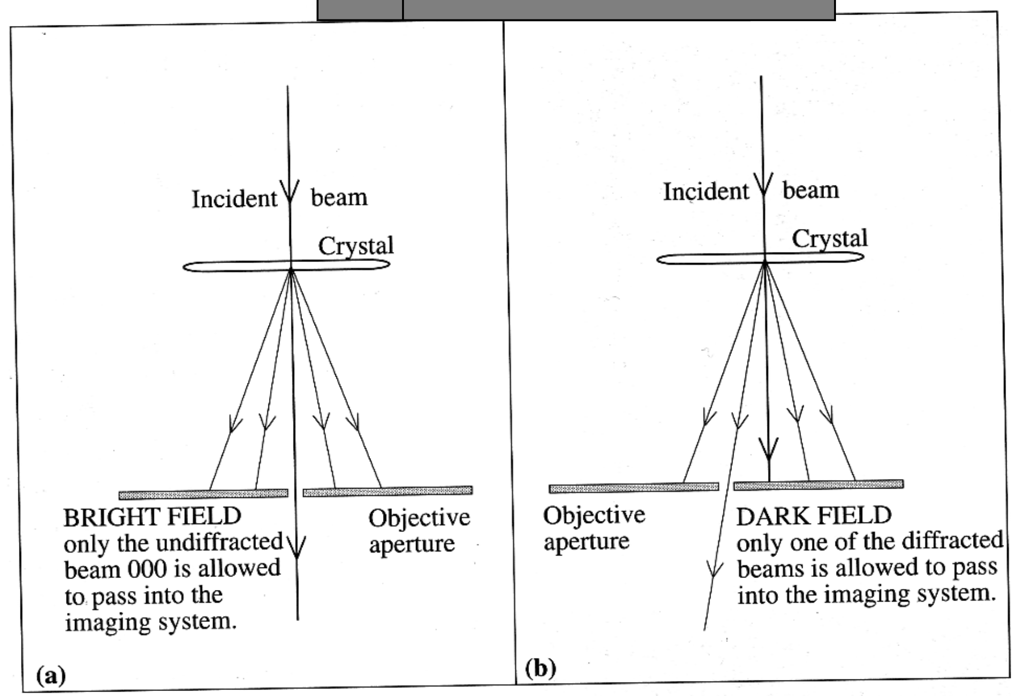
Back focal plane – diffraction pattern
Image plane - image

Kaolinite along [001]

Diffraction Contrast Imaging

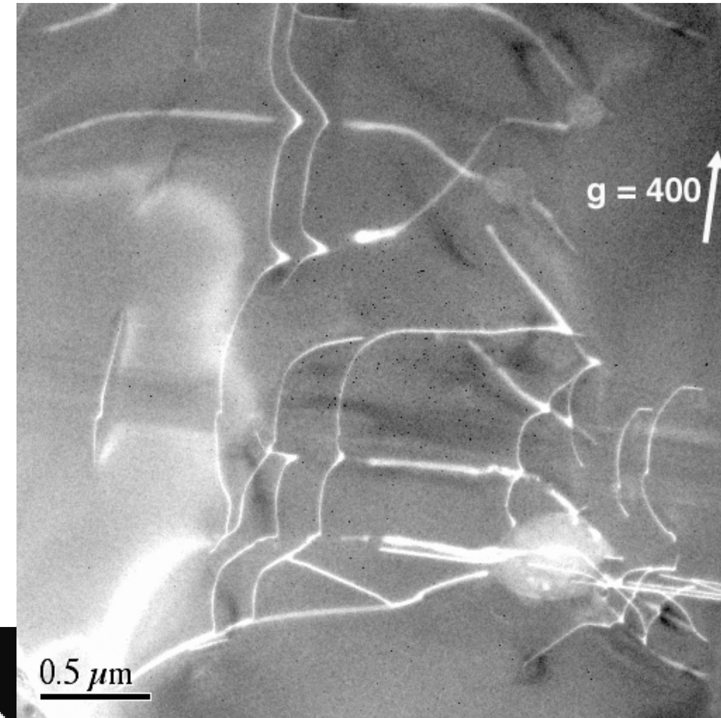
- Each diffracted spot corresponds to a set of lattice planes.
  - Each spot contains information from the entire image (selected)
- Bright-Field Imaging
  - Use only the central spot.
- Dark-Field Imaging
  - Use one of the diffracted spots
  - Tilt the crystal to or near a Bragg condition

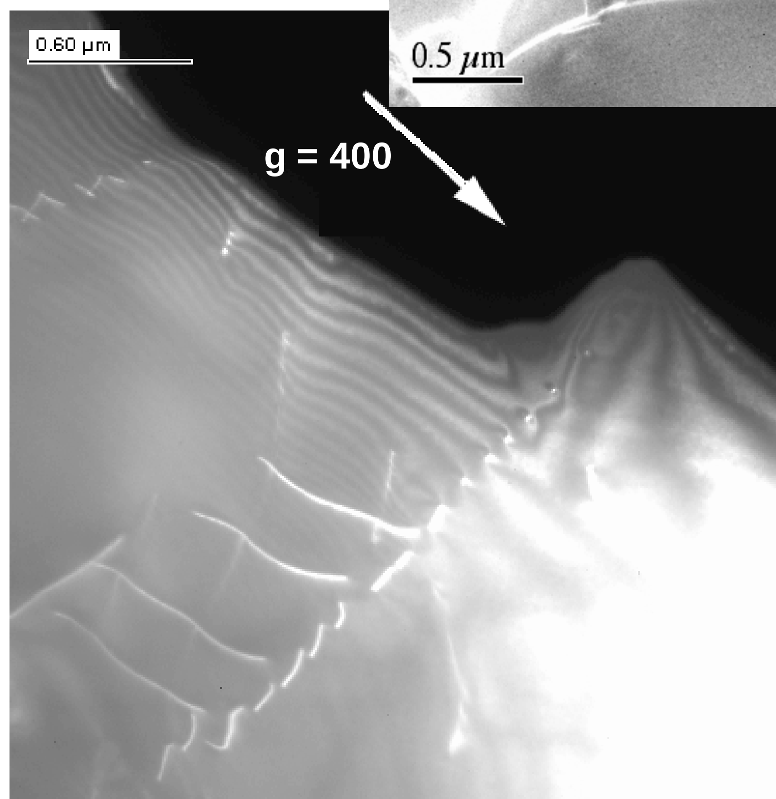




DF Image of Olivine dislocations, b=[100]

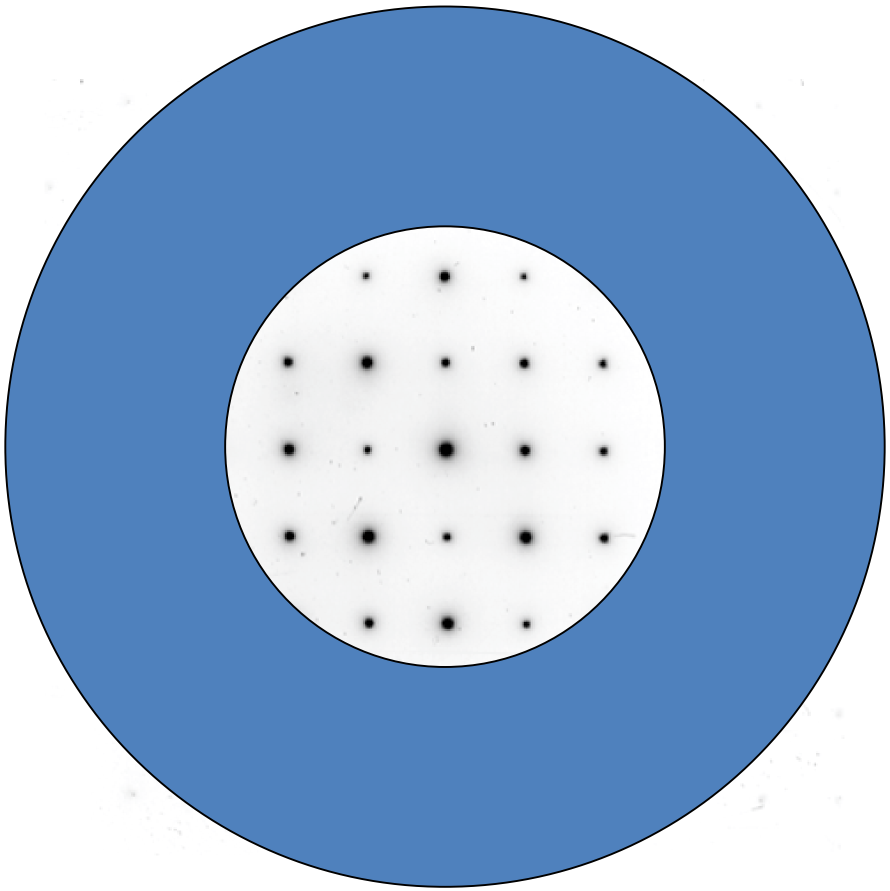
- Complex mixed character
  - Edge and screw dislocations in a (010) foil
  - Slip system [100](010)
- Tilt boundaries along (100)
  - Edge dislocations with **u** ~ [010]
  - Slip system [100](001)





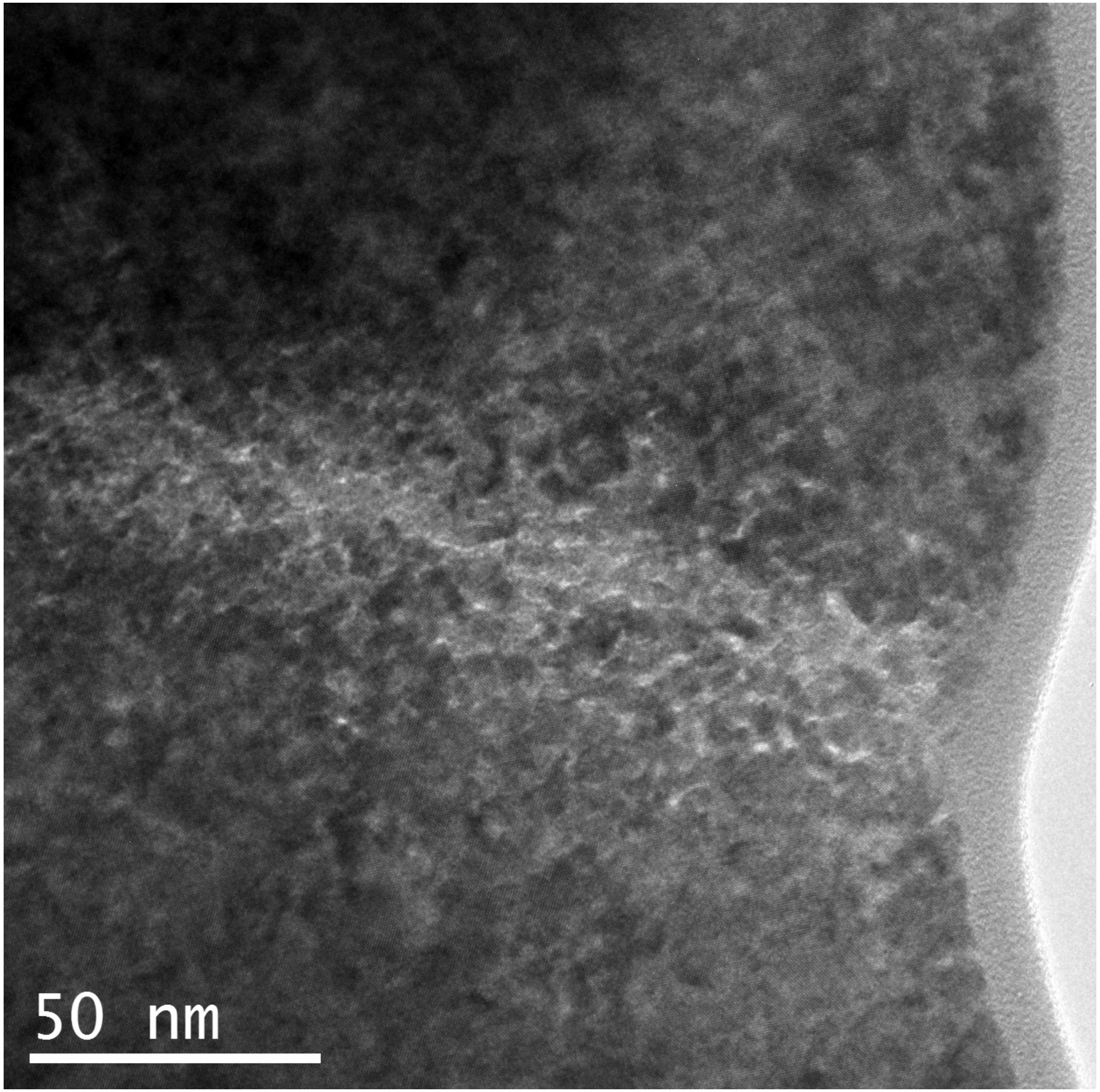
HRTEM (Phase Contrast)

- Imaging down a zone axis many diffracted beams.
  - Use a large objective aperture.
  - Image of the crystal lattice with nearly atomic resolution
- Phase contrast
  - Contrast from phase differences between the diffracted beams.
- Dynamic diffraction
  - Complex image contrast variations with sample thickness and defocus.



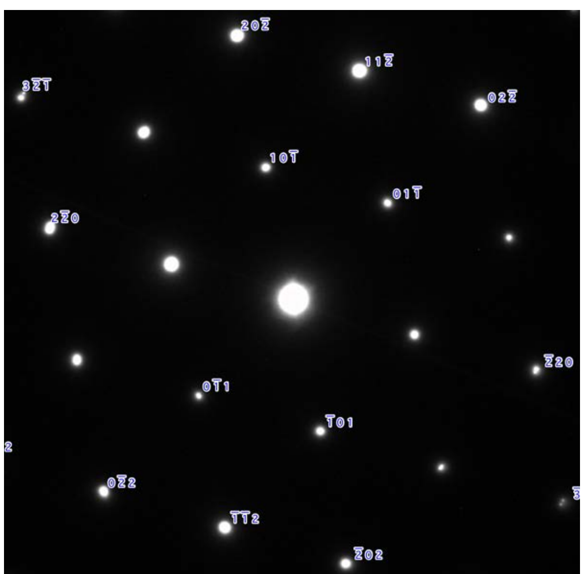
Zircon twin boundary

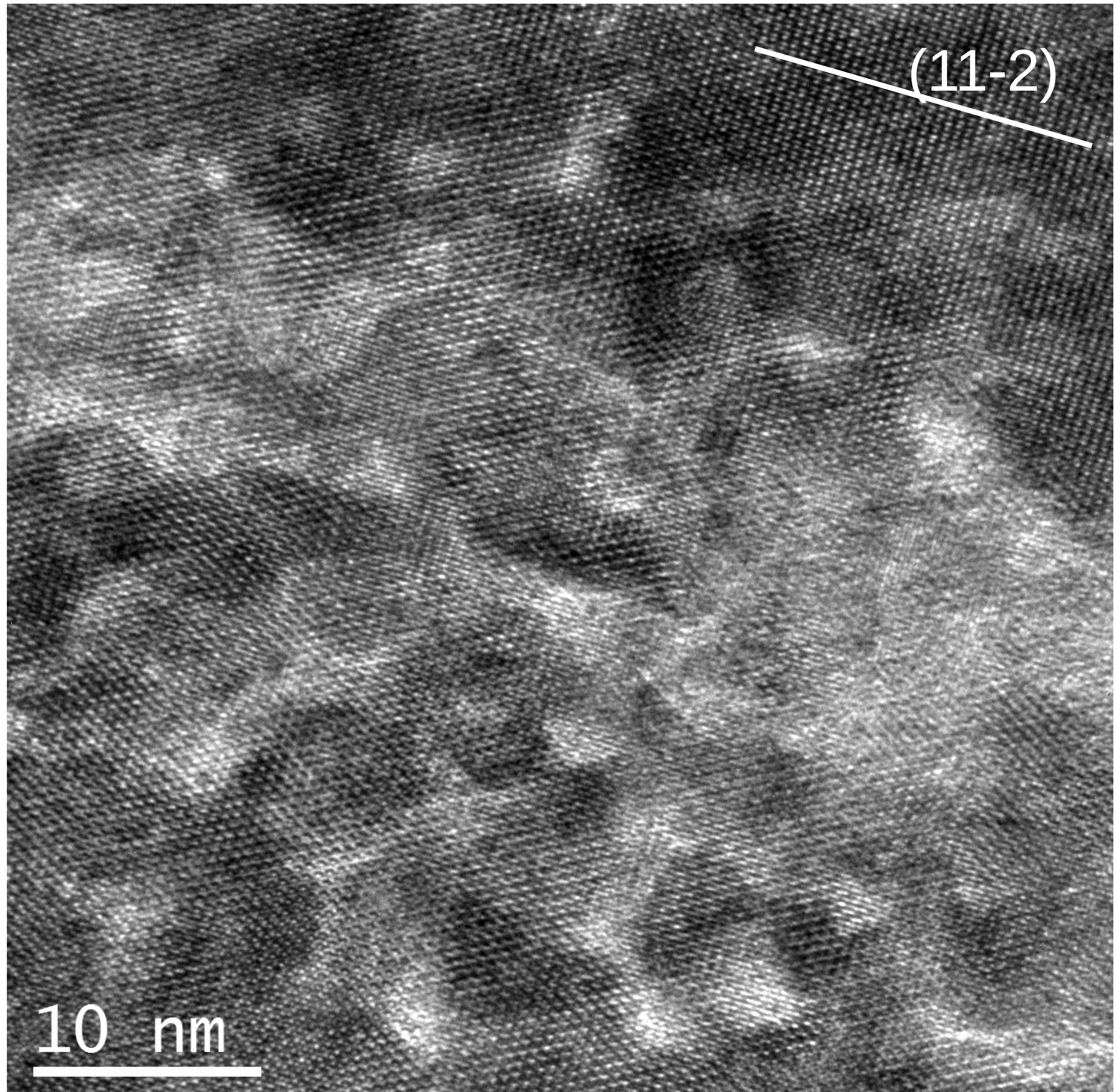
Phase contrast image alon [111] showing damaged regions that appear lighter.



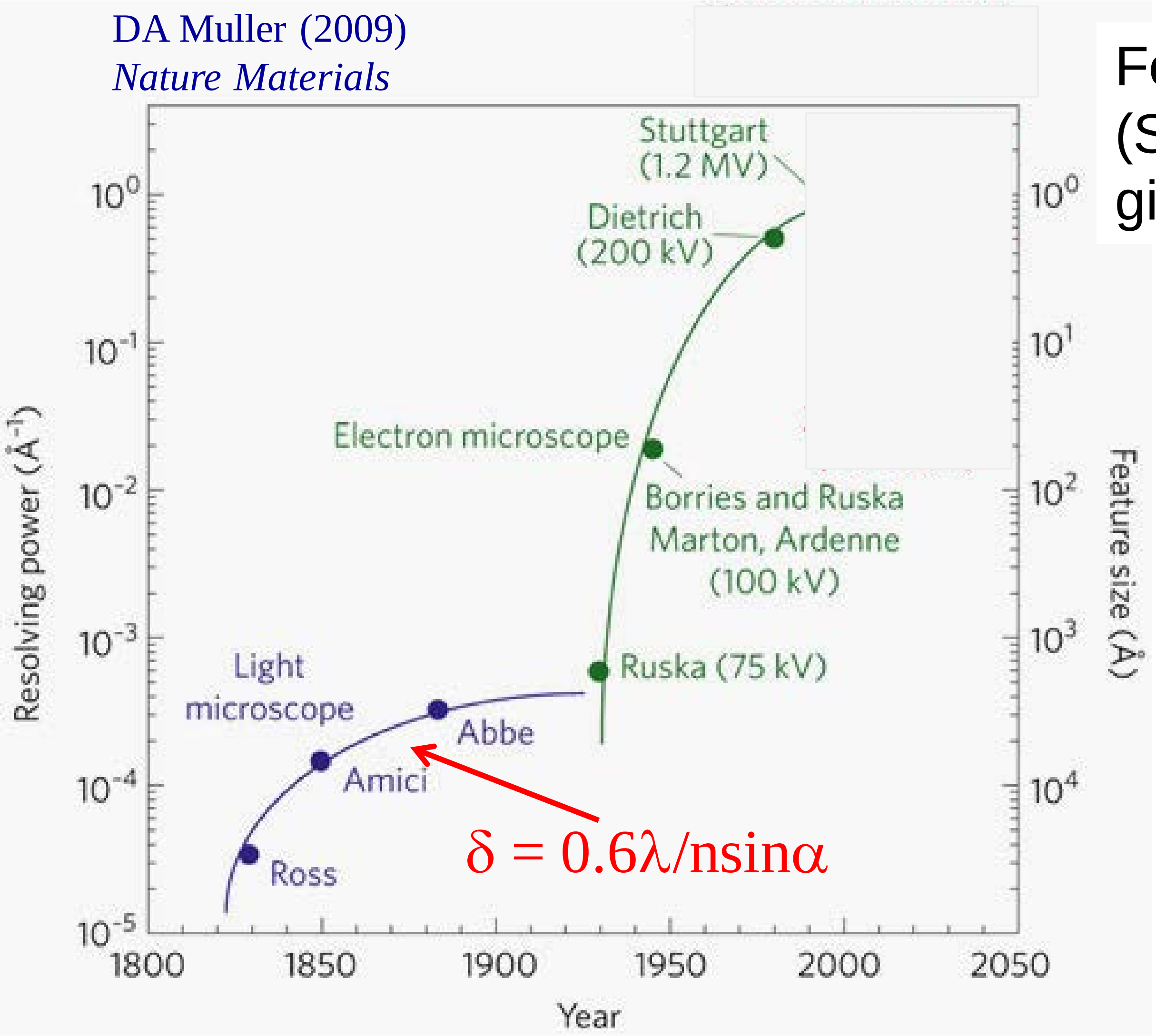
Zircon twin boundary

Higher magnification image of disordered domains.





Improving TEM resolution



For electron microscope the (Scherzer) resolution is given by

$$\delta = 0.66\lambda^{3/4}C_s^{1/4}$$

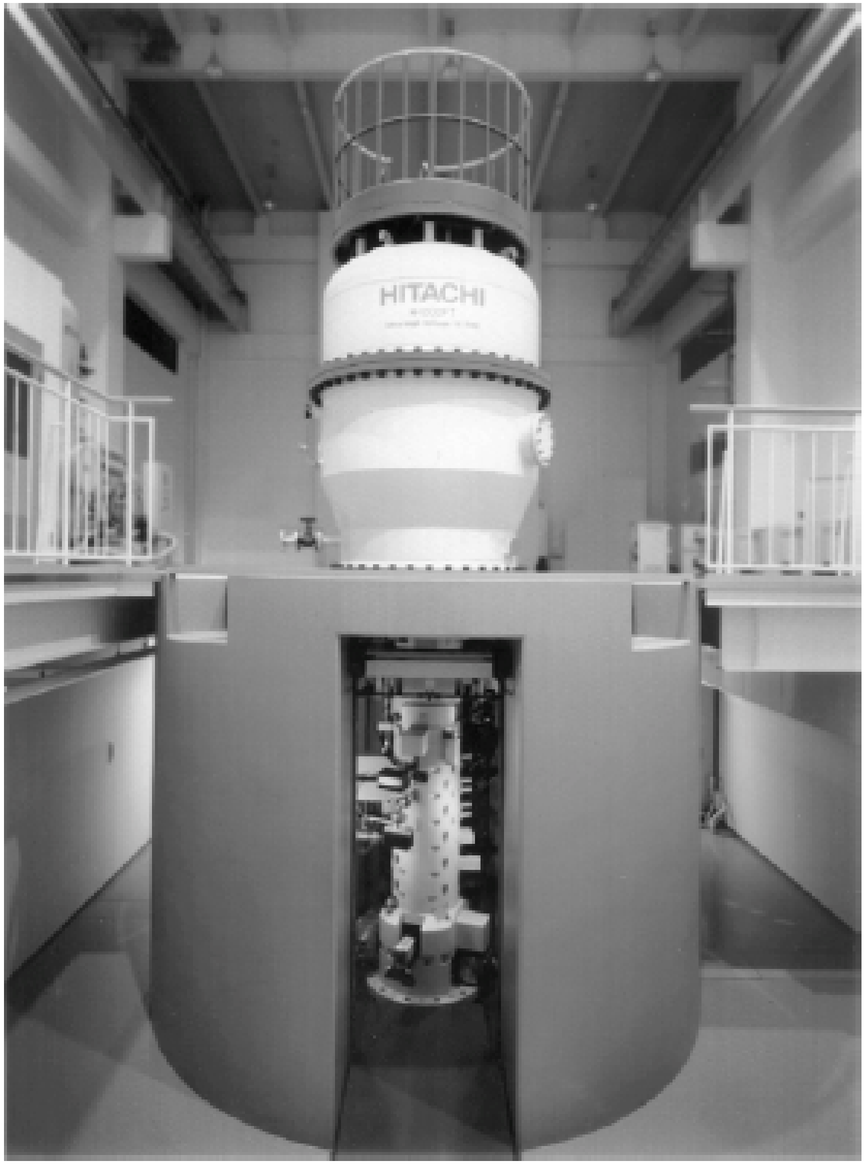
Increase the accelerating voltage to reduce the wavelength.

Historical approach to resolution improvement

Use higher-voltages for shorter wavelengths

Problems:

- Difficult to house a giant TEM
- Electron beam damage

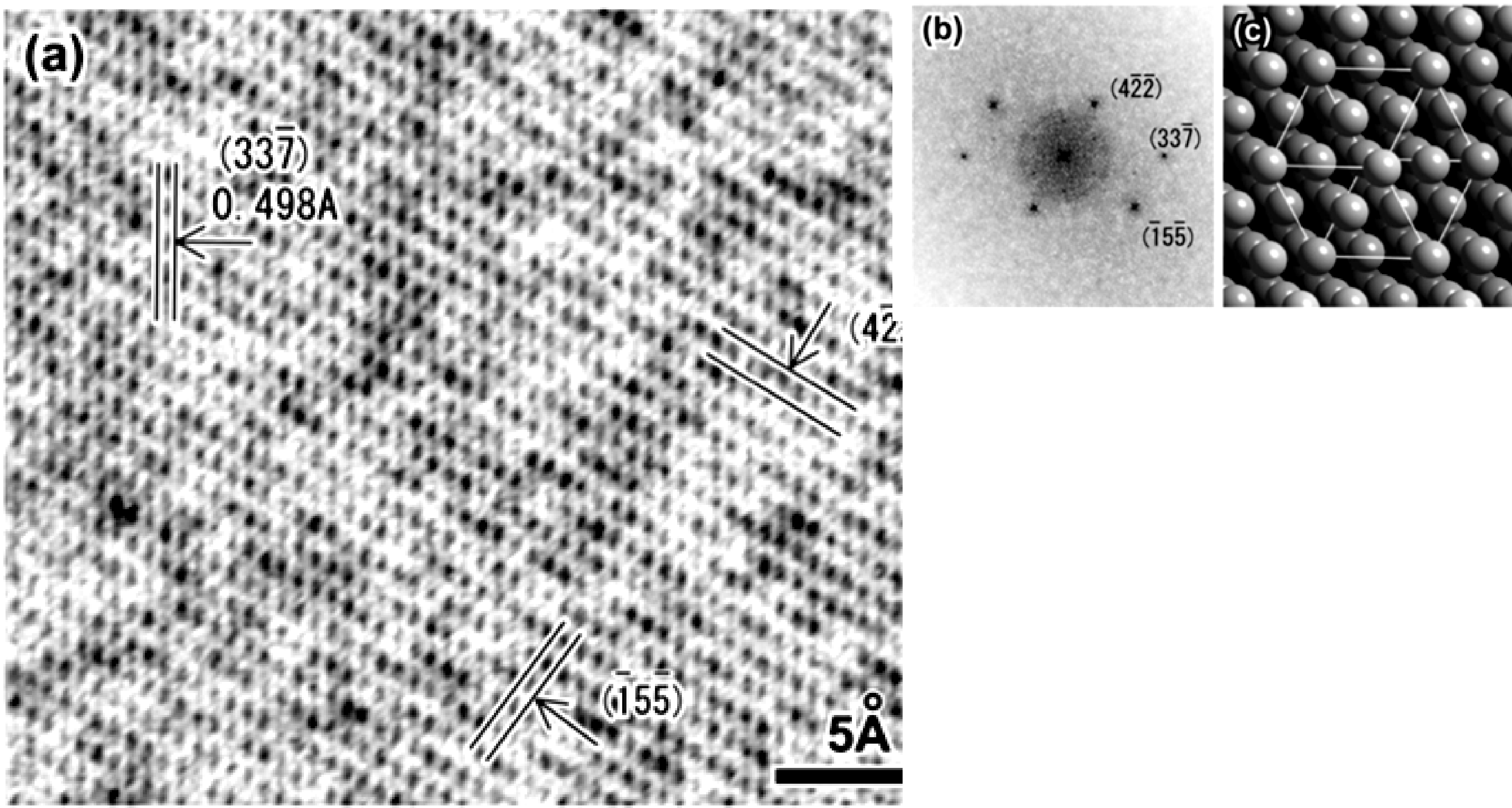


Hitachi 1-MeV FE-HRTEM

Tonomura, J. Electron Micr. **52**, 11 (2003)

“World record” lattice fringe spacing

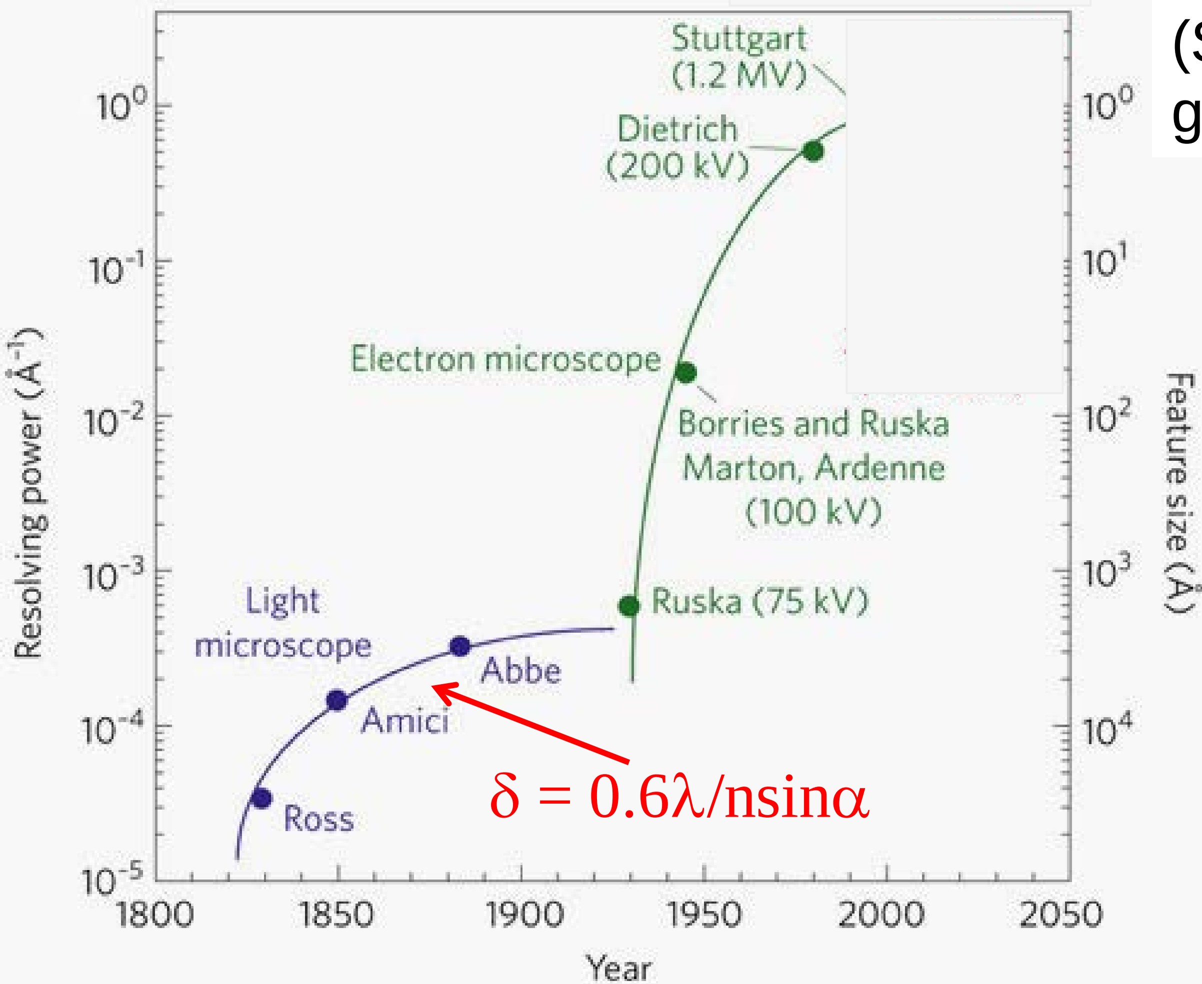
Lattice fringes at 1.0 MeV - beyond 0.5Å



from Kawasaki *et al.,* Appl. Phys. Lett. **76**, 1342 (2000)

Improving TEM resolution

DA Muller (2009)
Nature Materials



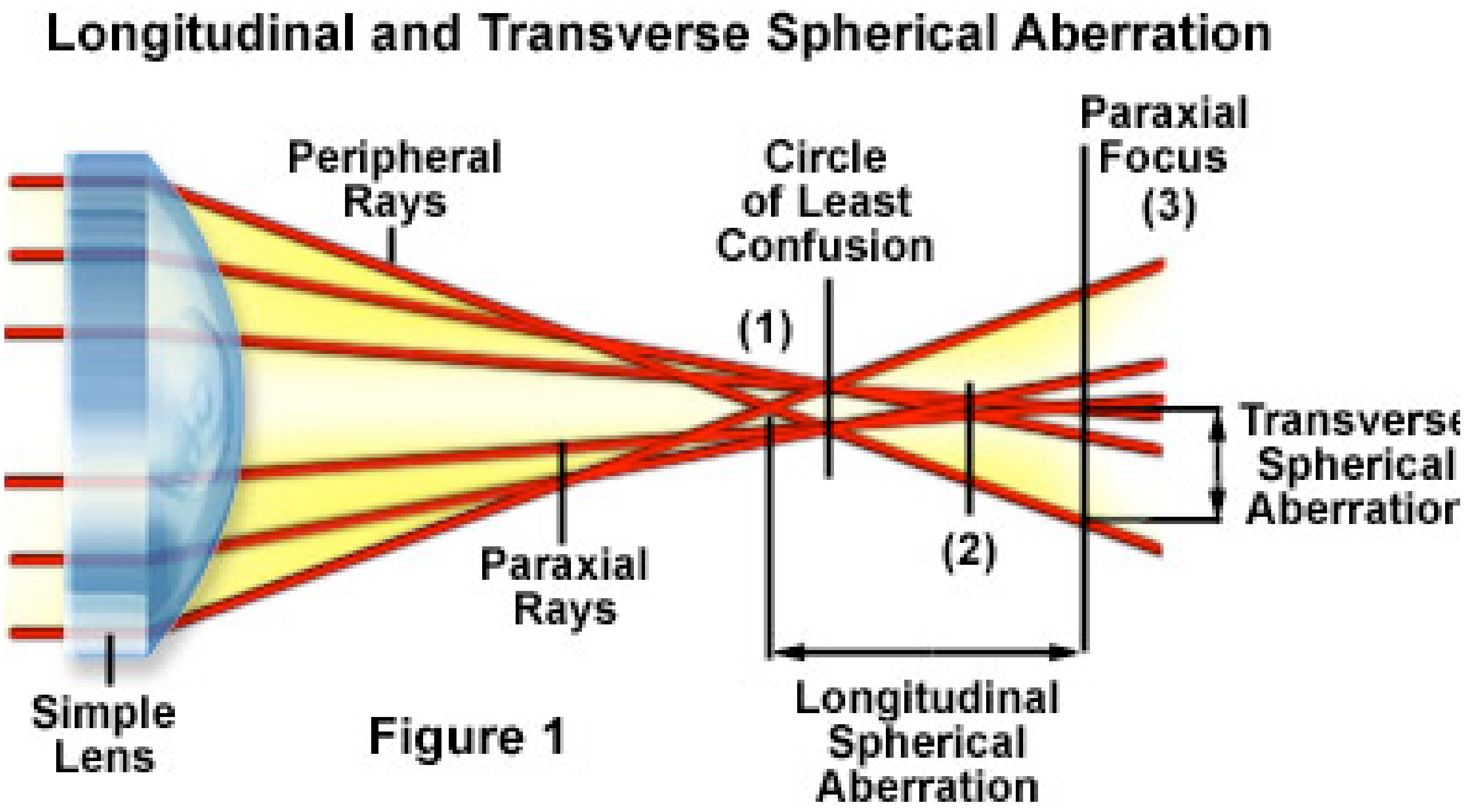
For electron microscope the (Scherzer) resolution is given by

$$\delta = 0.66\lambda^{3/4}C_s^{1/4}$$

Modern microscopes correct the C_s

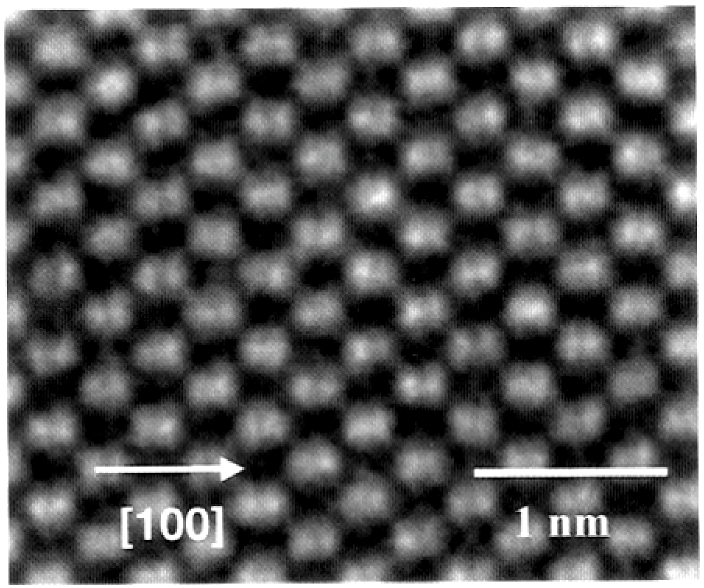
Aberration corrected TEM and STEM

Spherical Aberration

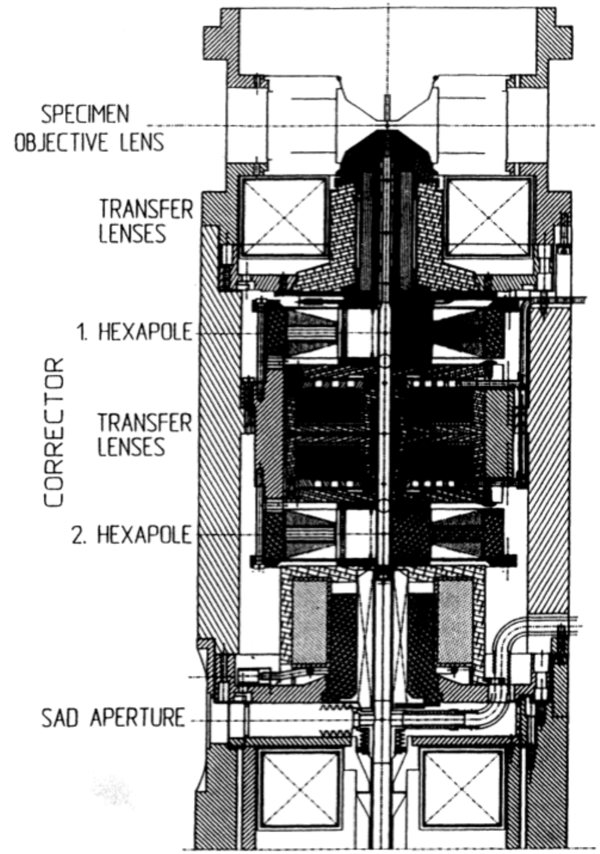


Aberration-correction in TEM

Design of the first successful aberration-corrected 200-keV FEG-TEM



GaAs showing individual atomic columns after application of C_s correction. Atoms separated by 1.4 Å



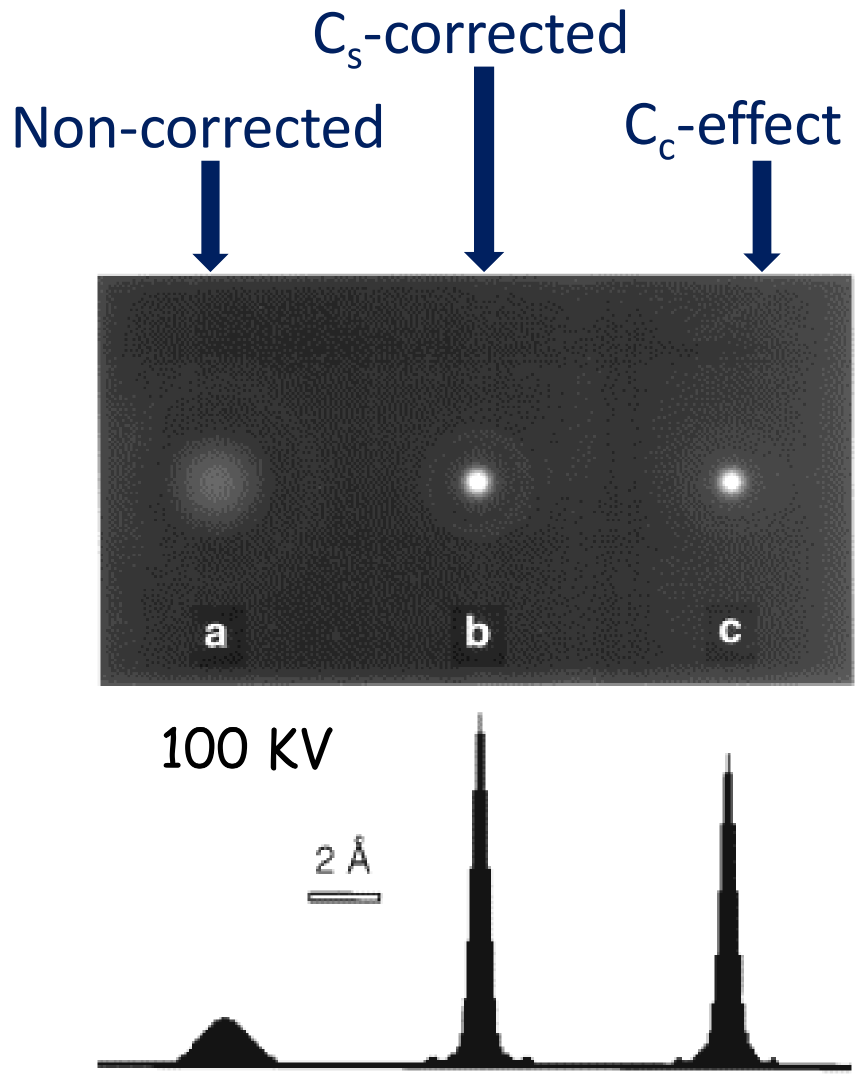
Schematic drawing of aberration correction device placed between objective and diffraction lenses.

from Haider, *et al.*, Ultramicroscopy, **75**, 53 (1998)

Aberration-correction in STEM

Allowing use of large objective aperture

- Smaller probe size
- Higher probe current



(Krivanek)

Southwest Center for Aberration Corrected Electron Microscopy

Building designed to meet environmental needs of aberration-corrected STEM/TEM

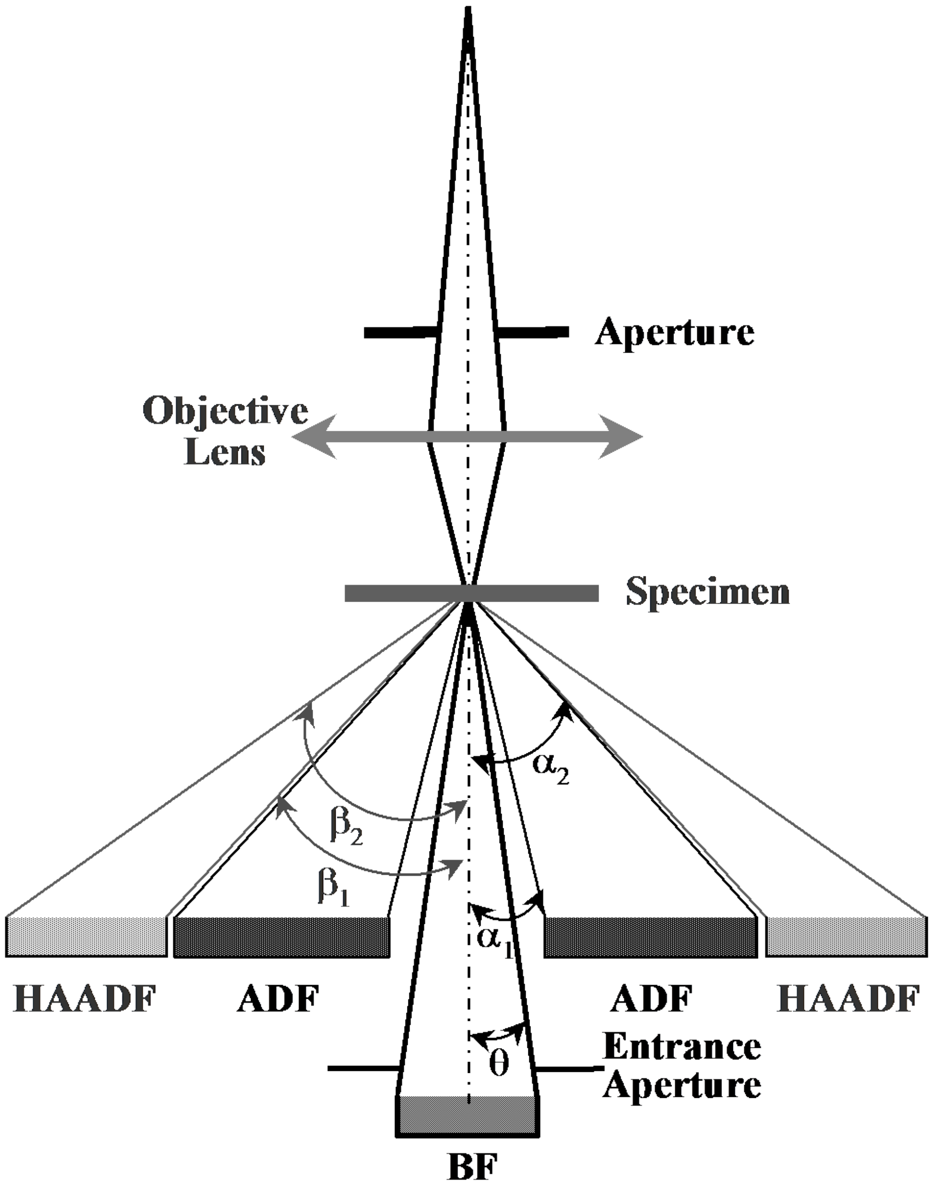
4-foot thick Isolated foundation for vibration isolation
Isolated power with no ground loops in floor or walls
Tight temperature control with minimal airflow

Space for four advanced microscopes:

Jeol ARM200F

- Aberration-Corrected STEM for Imaging and Spectrum Mapping
  - Operates at 80, 120, and 200 kV.
  - Field-emission electron gun
  - Corrector: CEOS CESCOR
  - STEM resolution @ 200 kV ~ 0.8Å
    @ 80 kV ~ 1.2 Å
  - JEOL EDX Detector (0.24 ster)
  - Gatan Enfinium EELS spectrometer

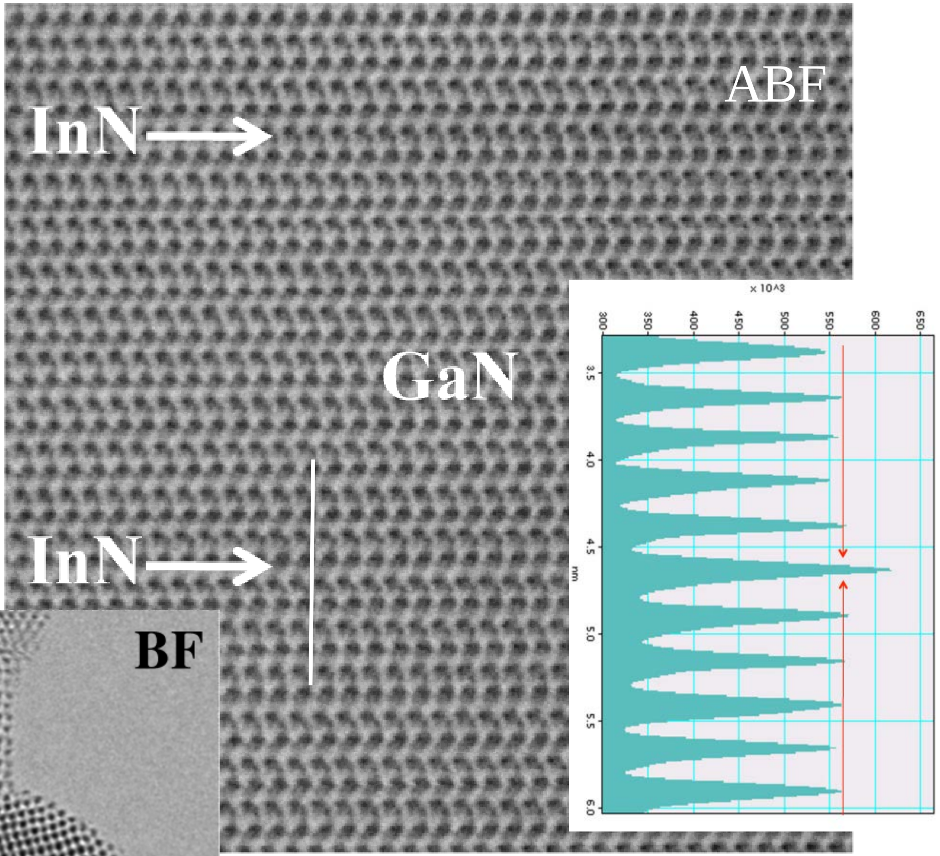
Formation of STEM images



- Bright-field STEM
- Annular bright-field (ABF)
- Large-angle BF (LABF)
- Low-angle ADF (LAADF)
- Medium-angle ADF (MAADF)
- High-angle ADF (HAADF)

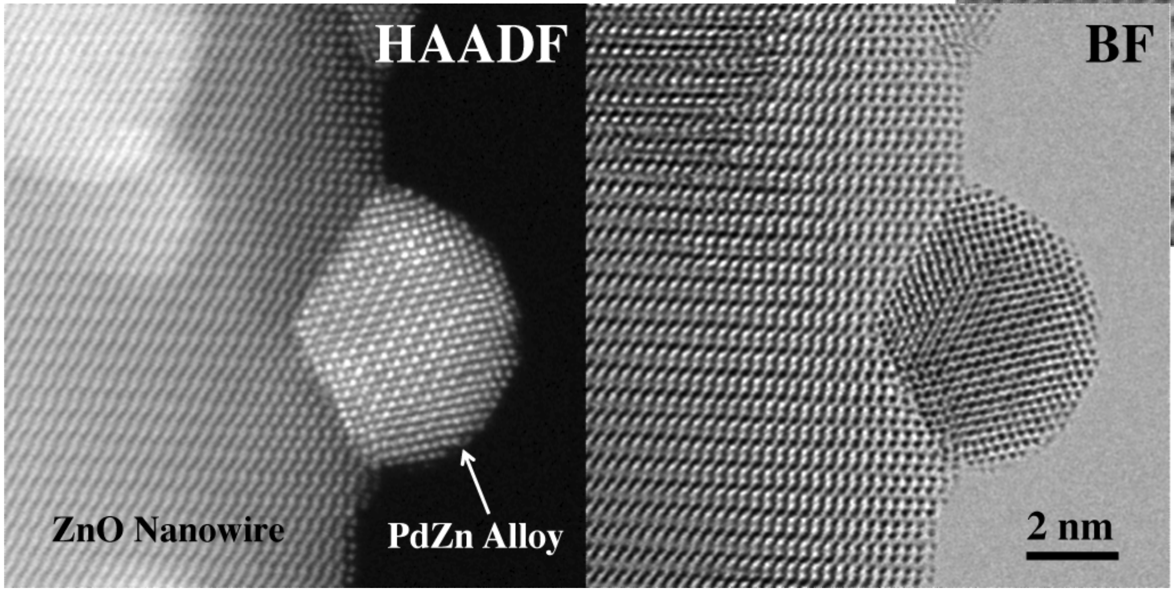
STEM Imaging

- BF: Bright-field; DF: Dark-field
- ABF: annular-bright-field
- MAADF: medium-angle annular-DF
- HAADF: high-angle annular-DF



Courtesy of Lin Zhou

Annular-bright-field image and line scan showing one-monolayer-thick InN quantum wells in GaN matrix.

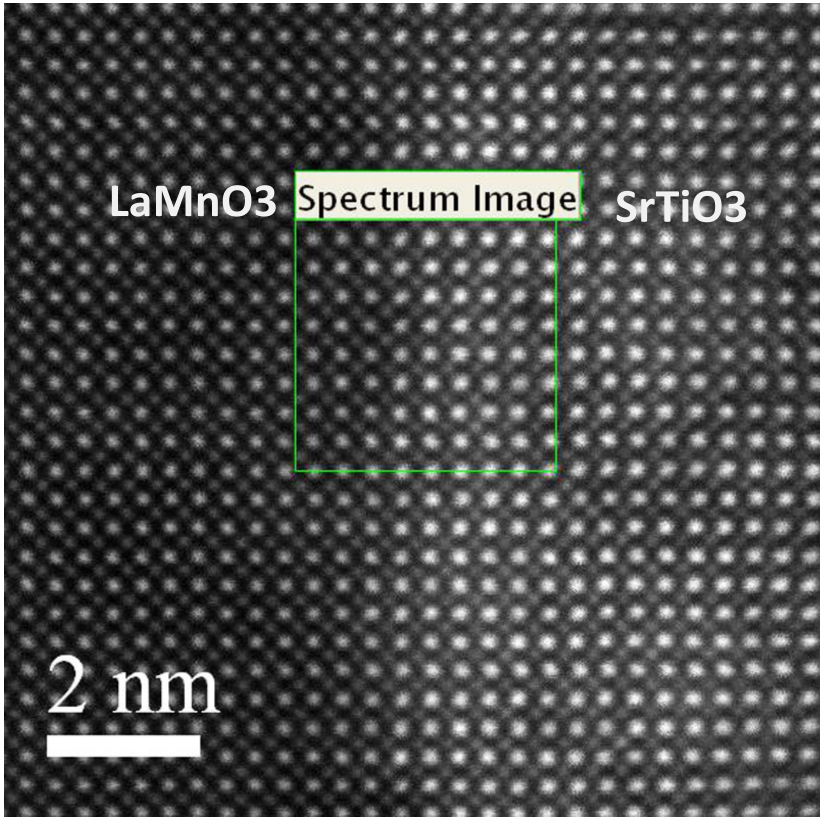


Simultaneous HAADF and BF images of endotaxially anchored PdZn alloy nanoparticle on ZnO nanowire.

Courtesy of Jingyue Liu

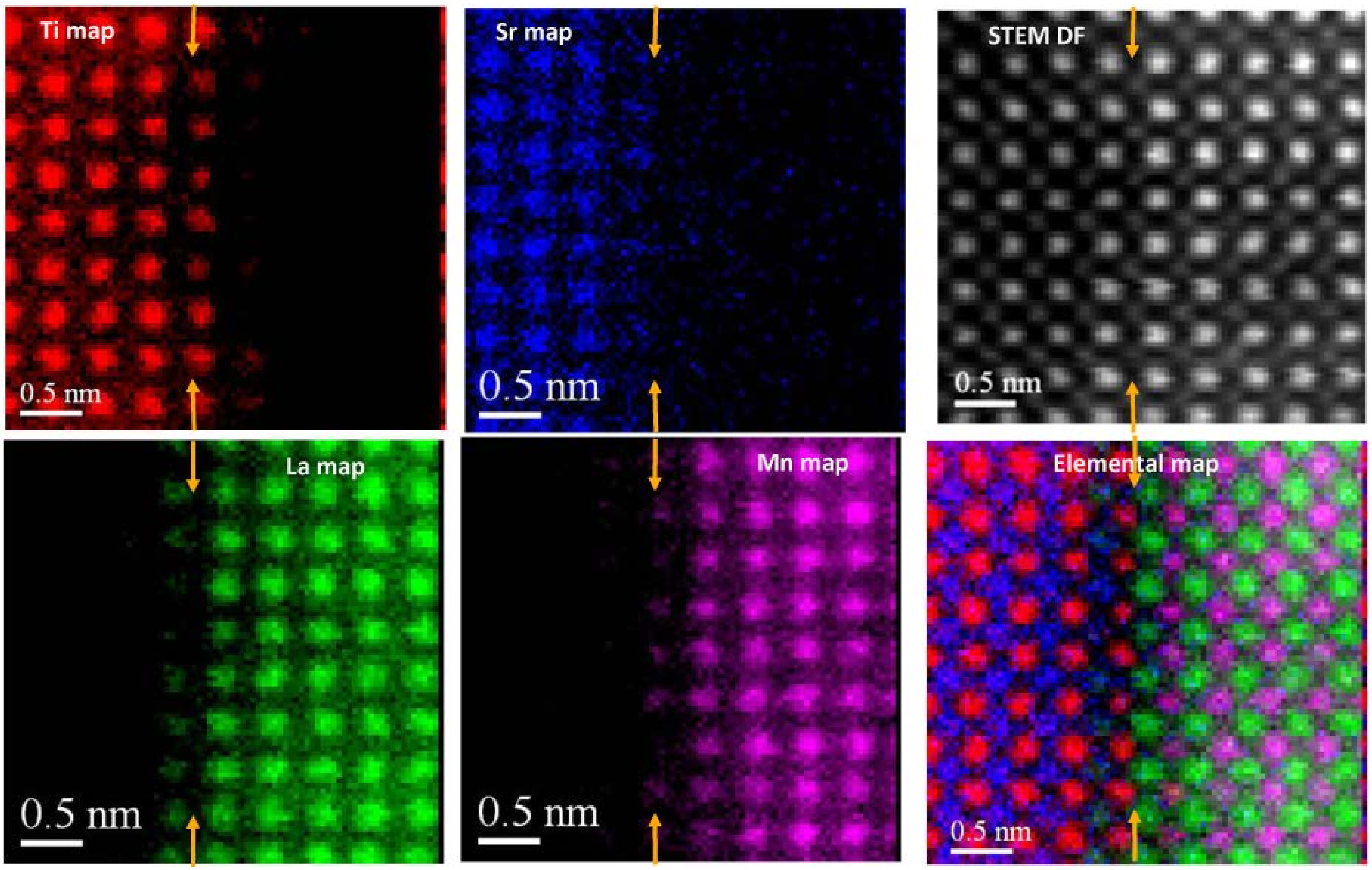
Imaging of a $LaMnO_3/SrTiO_3$ interface

- Fast collection of EELS spectra combined with STEM imaging
- Atomic-resolution chemical mapping



STEM HAADF image of SrTiO3/LaMnO3 interface, used as survey image for EELS Spectrum imaging

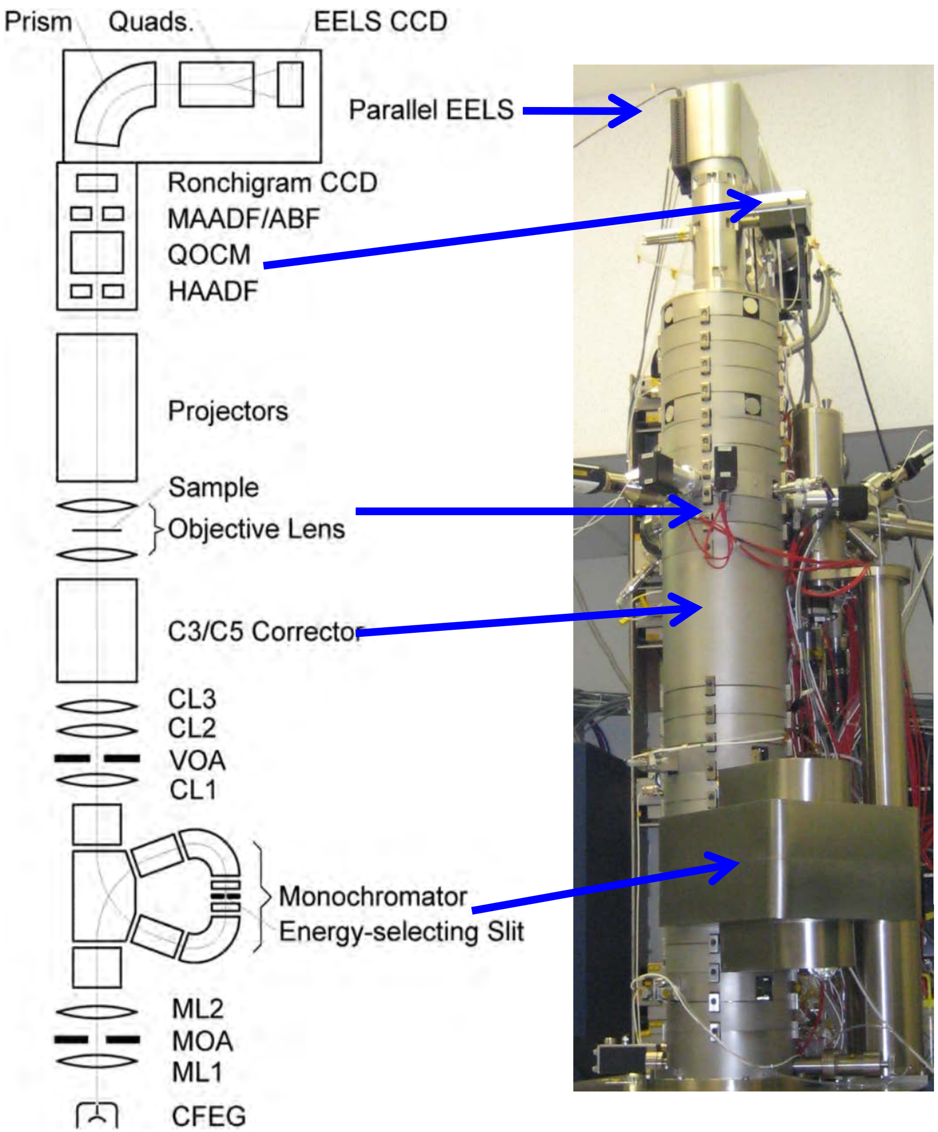
EELS Spectrum Imaging



Chemical mapping across $LaMnO_3/SrTiO_3$ interface.

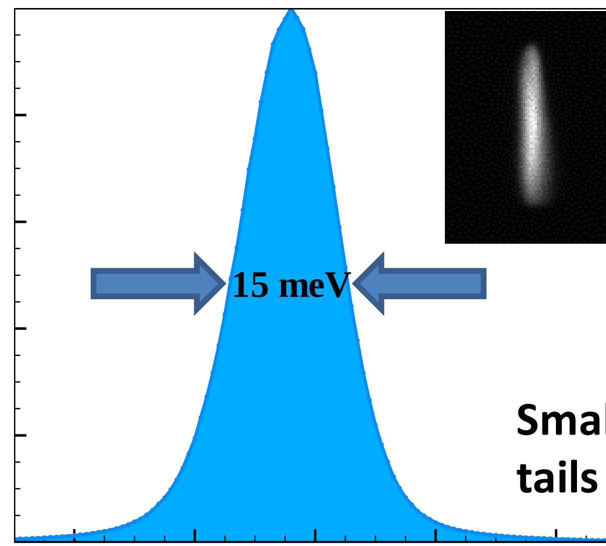
Courtesy of Paolo Longo

NION UltraSTEM Monochromated STEM/EELS at 40/60/100kV



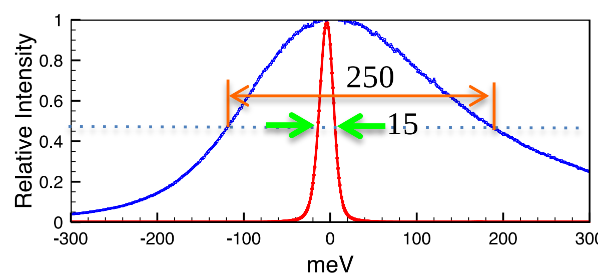
O.L. Krivanek et al. Microcopy **62**(1) 3-21 (2013).

Nion high-energy resolution mono-chromated EELS systems (HERMES)

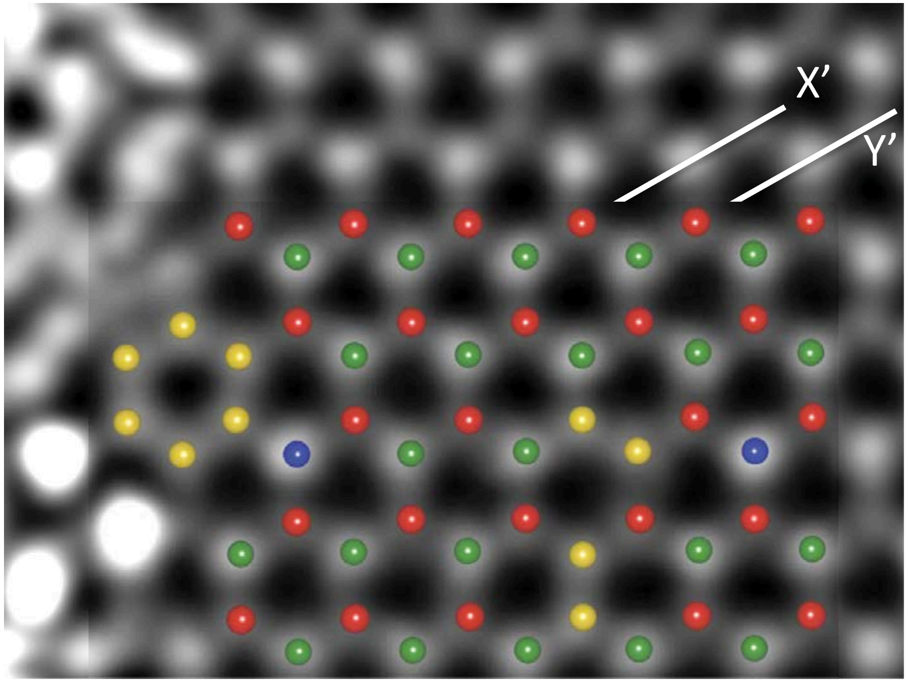


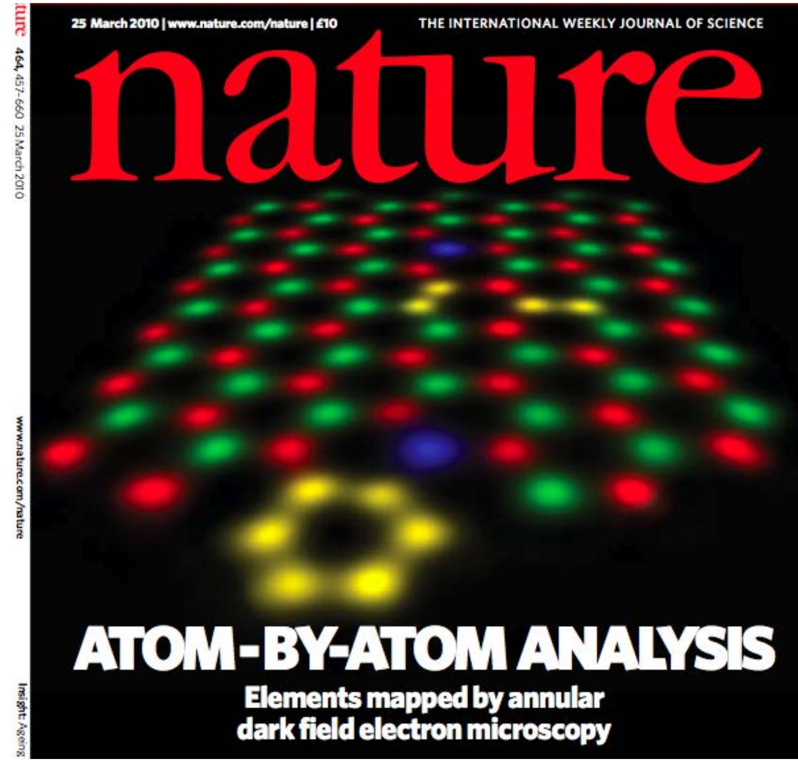
Typical on ASU 15 - 30 meV

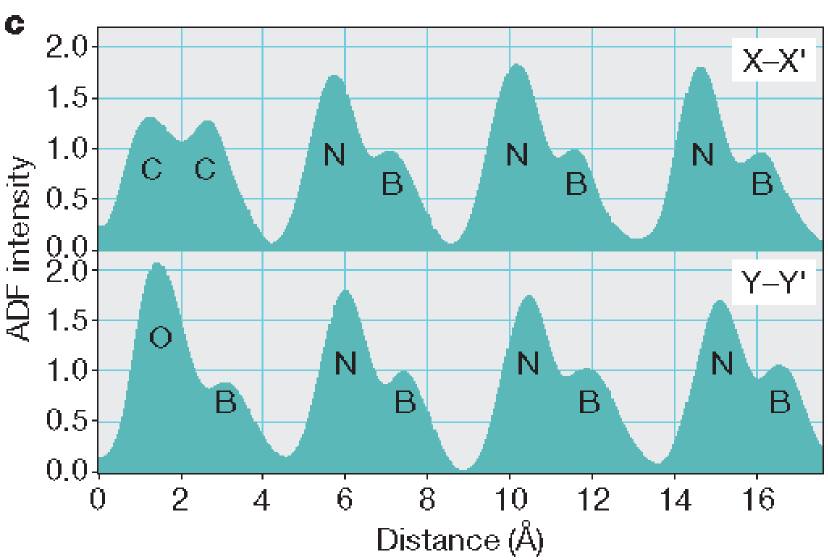
Small zero-loss tails

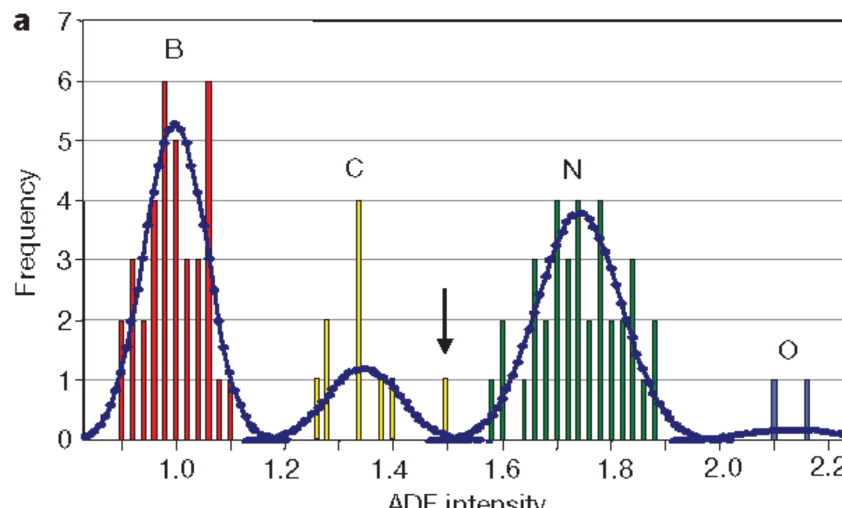


ASU record 12 meV



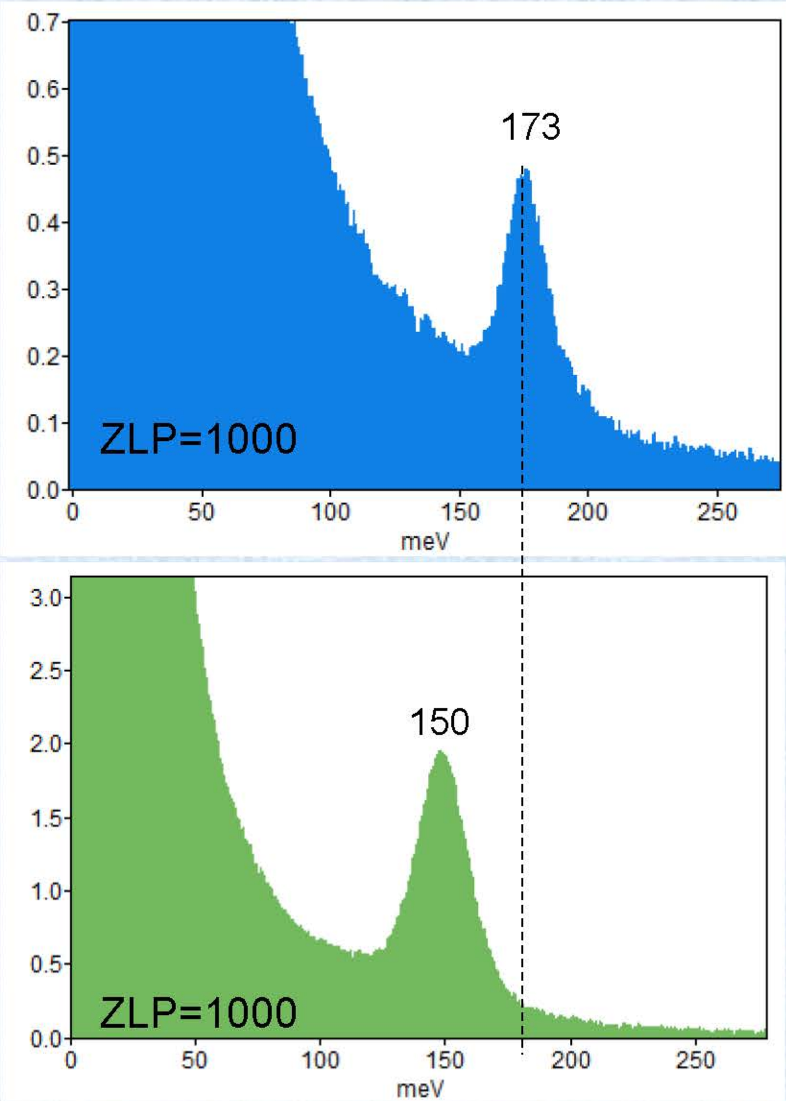




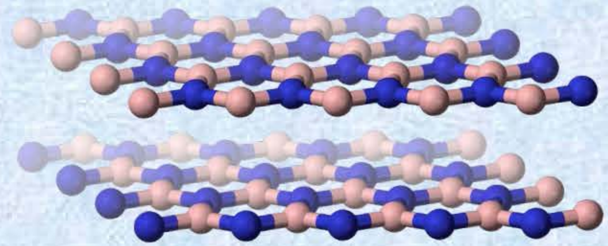


From Krivanek, *et al.* Nature , 29 March, 2010

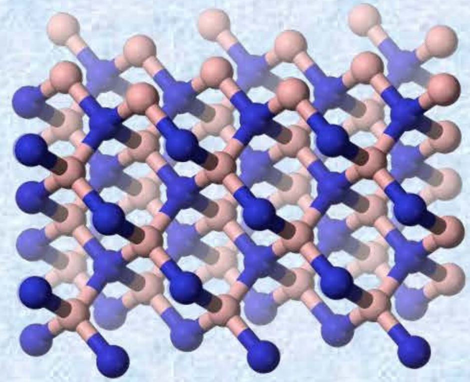
Boron Nitride phonons: hexagonal vs. cubic



Hexagonal BN

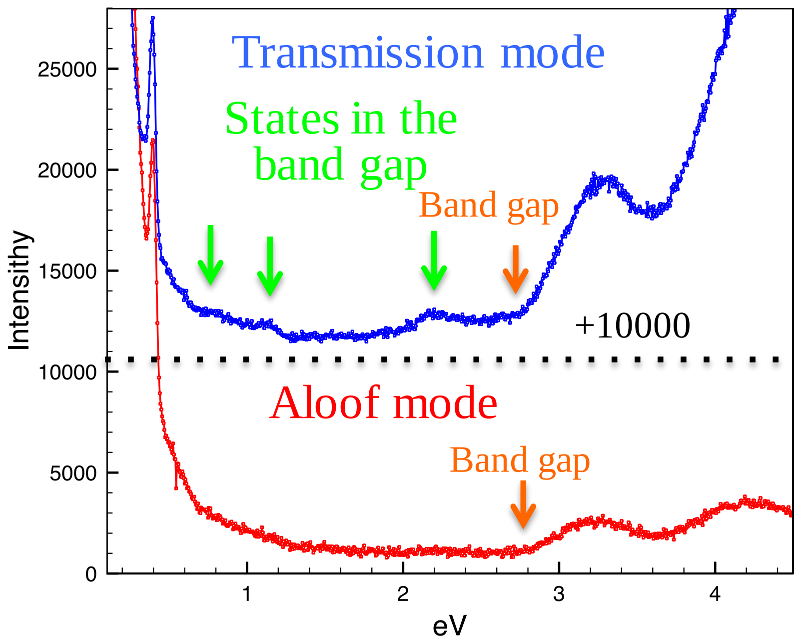


Cubic (zinc blende) BN



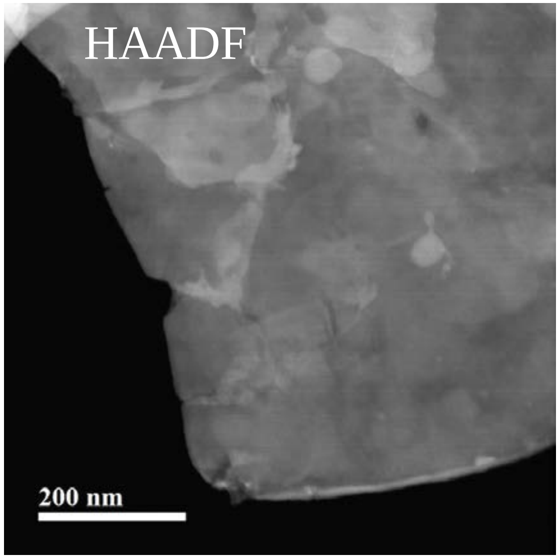
Quentin Ramasse, Fredrik Hage (Daresbury SuperSTEM), Rebecca Nicholls (Oxford) and Keith Refson (Rutherford Appleton Labs)

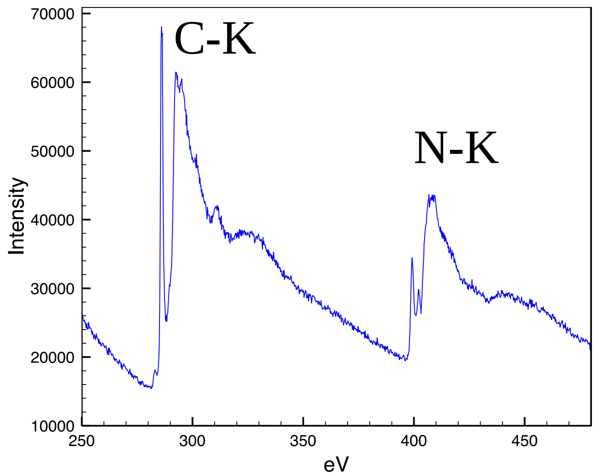
Aloof beam spectroscopy of radiation sensitive materials



VEELS of C_3N_4 in transmission and aloof modes.

Diane Haiber et al M&M 2016





HAADF images of C_3N_4 and EELS spectrum showing C-K and N-K edges

FEI Titan ETEM

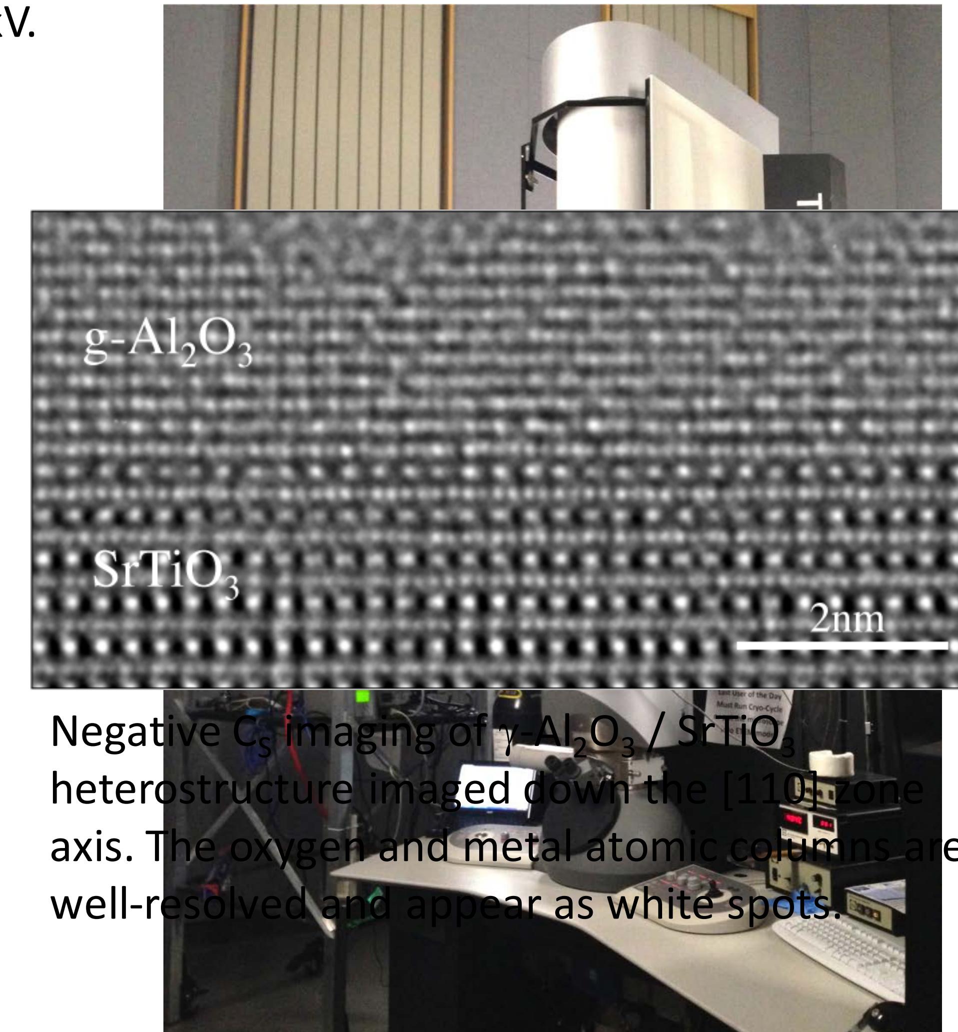
(S)TEM that operates at 80, 200, & 300 kV.

X-FEG:
Ultrahigh-brightness electron gun

Monochromator:
Energy resolution ~ 0.15 eV

Imaging Corrector: CEOS CETCOR
Information limit @ 300 kV < 0.9 Å
@ 80 kV ~ 1.9 Å (mono off)
@ 80 kV ~ 1 Å (mono on)

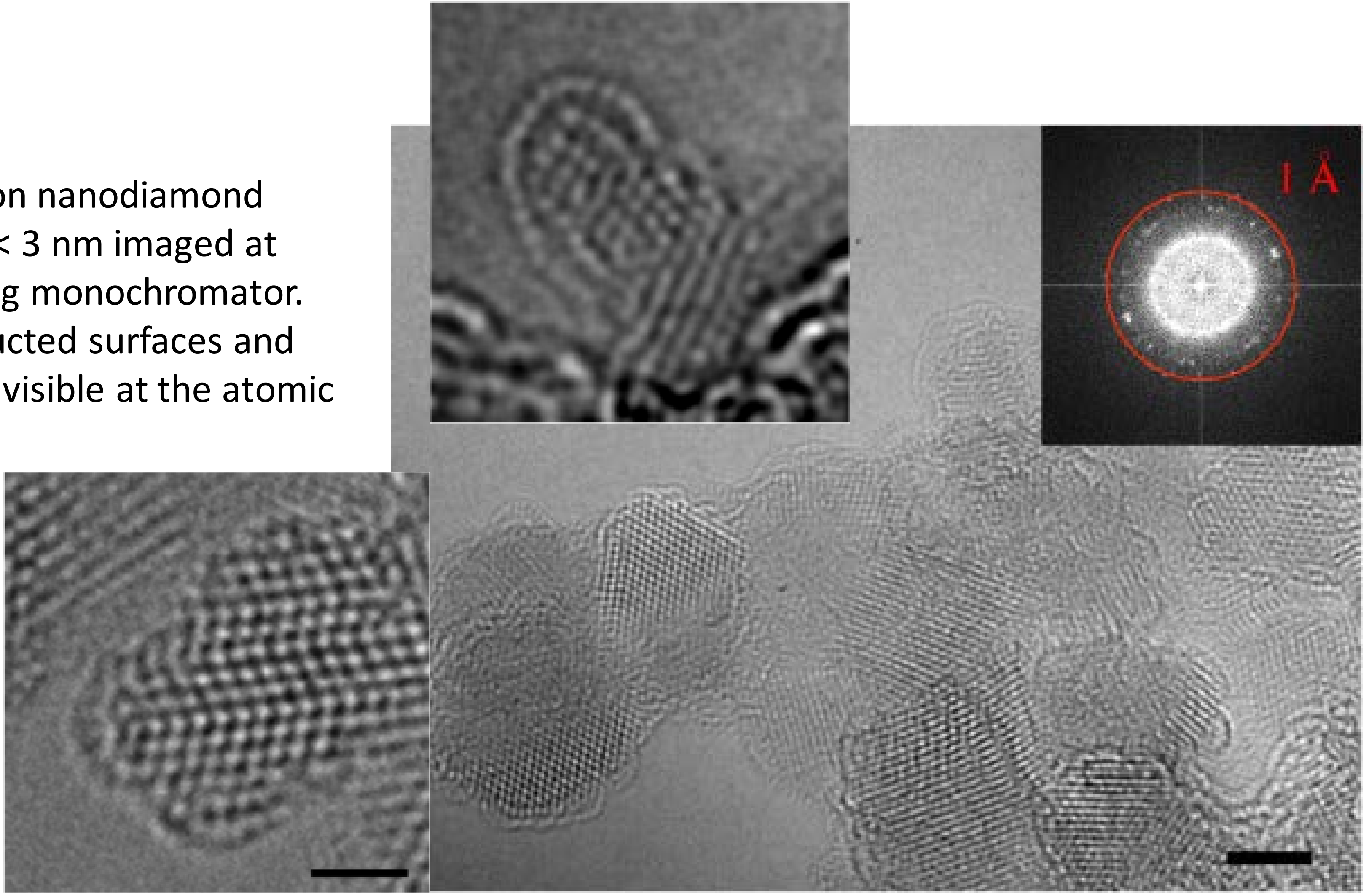
Analytical (S)TEM:
EDAX EDX Detector (0.13sr)
Gatan Imaging Filter/EELS spectrometer



Negative C_S imaging of γ-Al_2O_3 / $SrTiO_3$ heterostructure imaged down the [110] zone axis. The oxygen and metal atomic columns are well-resolved and appear as white spots.

Low Voltage Imaging

Detonation nanodiamond particles < 3 nm imaged at 80kV using monochromator. Reconstructed surfaces and twins are visible at the atomic level.



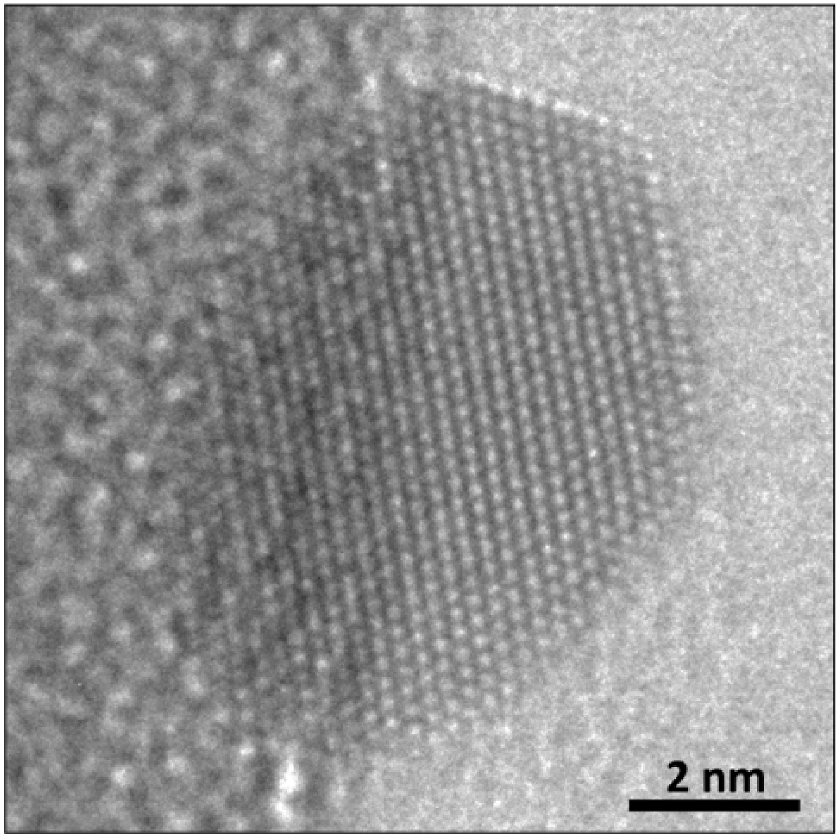
Environmental TEM

Environmental TEM:
Samples to be exposed to gaseous environment.

TEM allows rapid imaging and movies with atomic resolution.

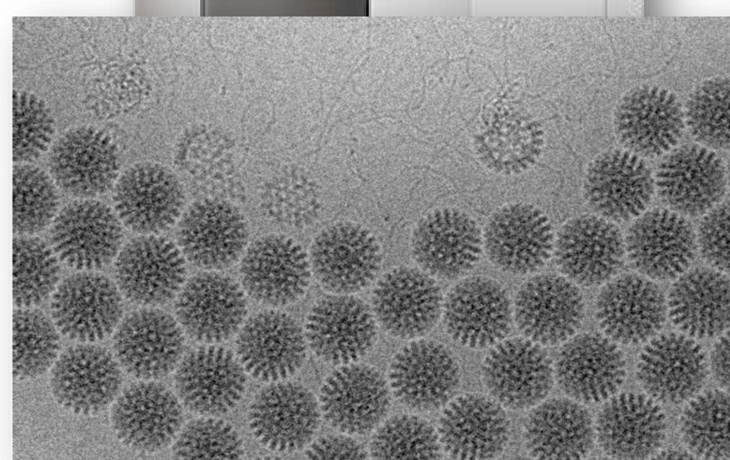
In-house gas system allows precise control and accurate mixing.

Heating and Cooling Holders: Sample observation at temperatures up to 1100∘C or down to -170 ∘C.

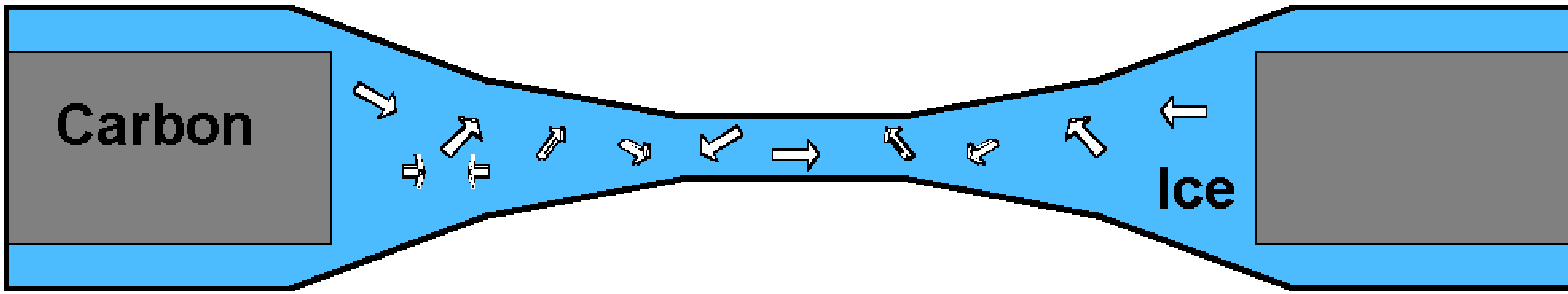


FEI Titan Krios

- FEI Titan Krios with a Gatan K2 Summit single-electron detector
- 2-3Å resolution in biological macromolecules
- Single particle analysis of proteins
- Cryo-electron tomography of cell structures



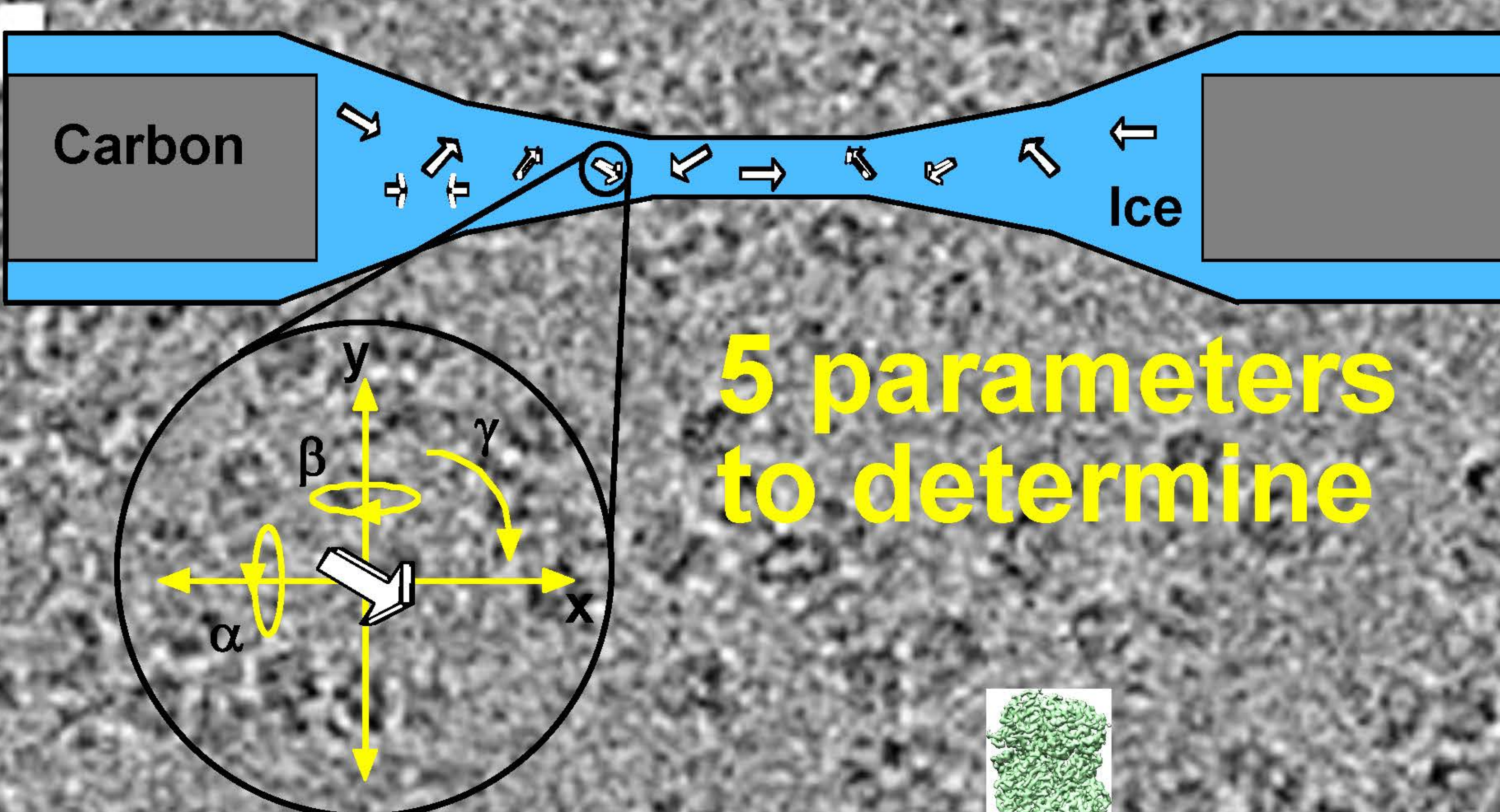
Why Cryo?



- Sample is frozen in aqueous solution
  → native conditions
- Frozen state prevents dehydration in microscope vacuum
- Low temperature delays effects of radiation damage

Nikolaus Grigorieff, ASU Cryo-EM Winter School

Aligning Particles



5 parameters to determine

100Å

Webinar Recordings & Slides

To access this recording and slides

NACK NETWORK — Building College-University Partnerships for Nanotechnology Workforce Development

http://nano4me.org/webinars.php

Or

NCI Southwest

http://ncisouthwest.org/index.php/webinars/

Thank You!

Thank you for attending the
NACK Network & NCI-SW webinar

Please take a moment to complete our survey