STEM Imaging

Lecture 17

Much of the material for this class is courtesy
Nigel Browning of UC Davis & LLNL
and
Dave Muller of Cornell

Outline

How does it work?

Inelastic scattering (some review, some new)

Instrumental and alignment concerns

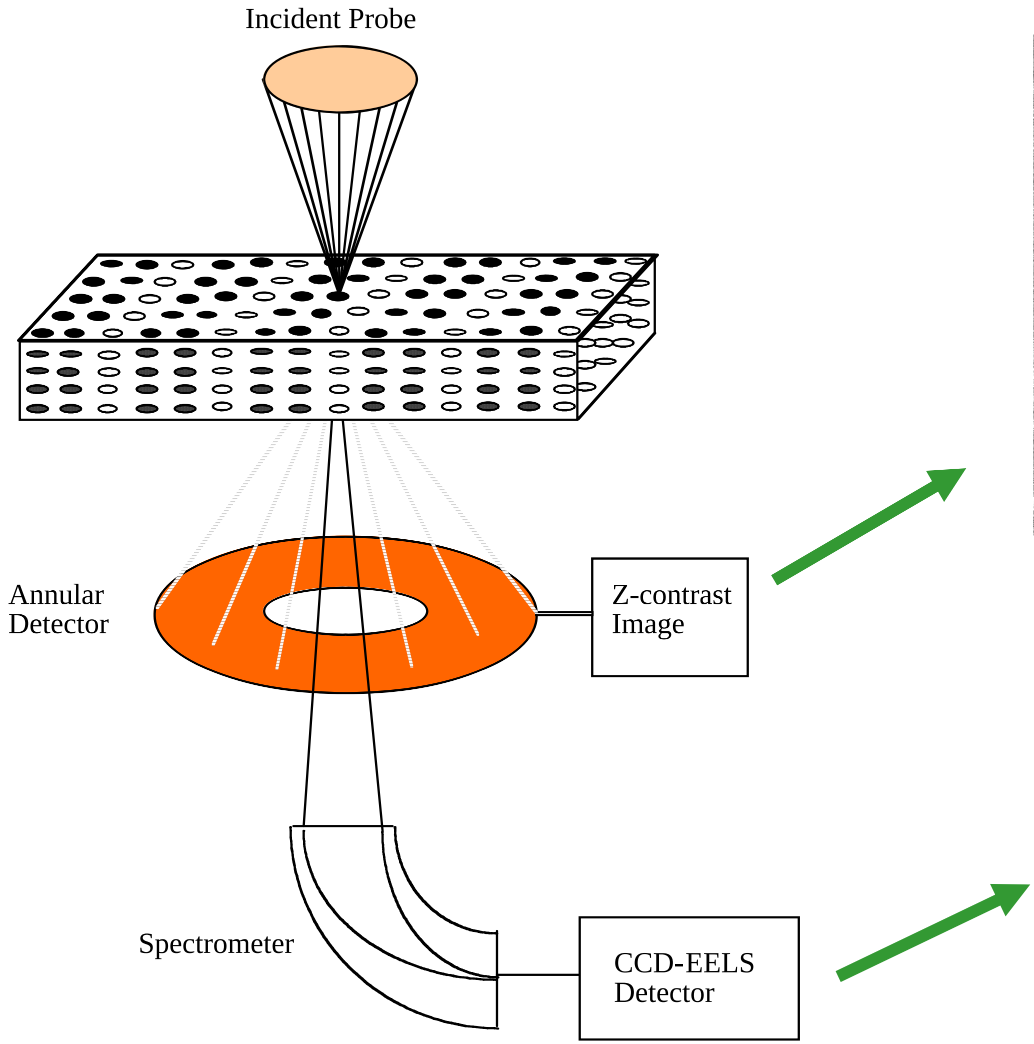
Image artifacts

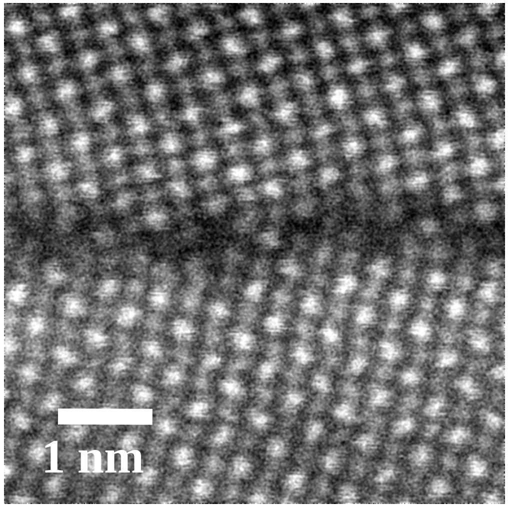
Examples spread throughout

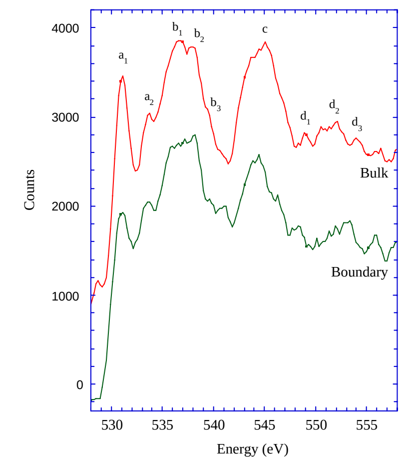
Operational principle

Resolution

Image Properties

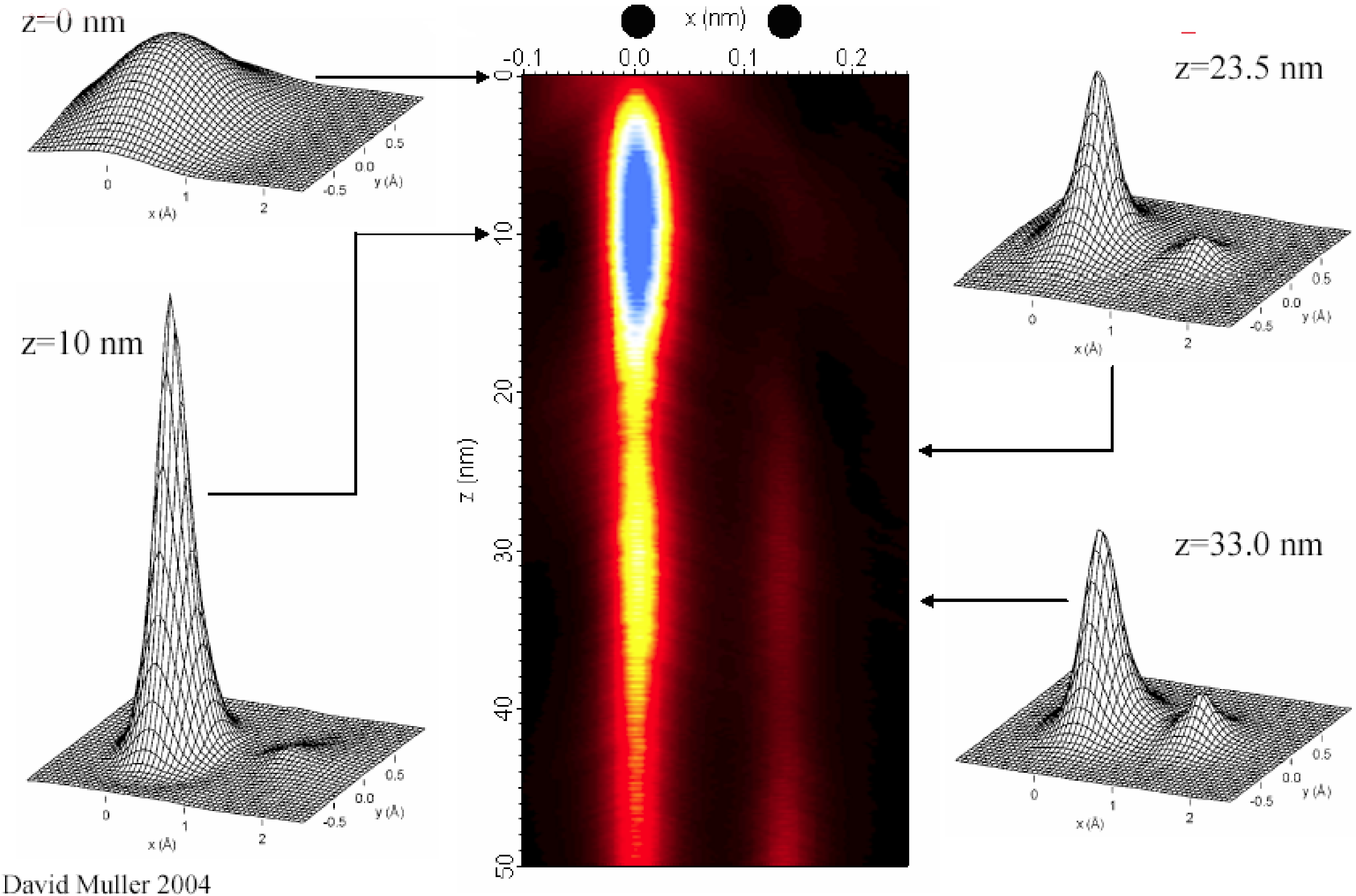






Courtesy Nigel Browning

Probe channeling



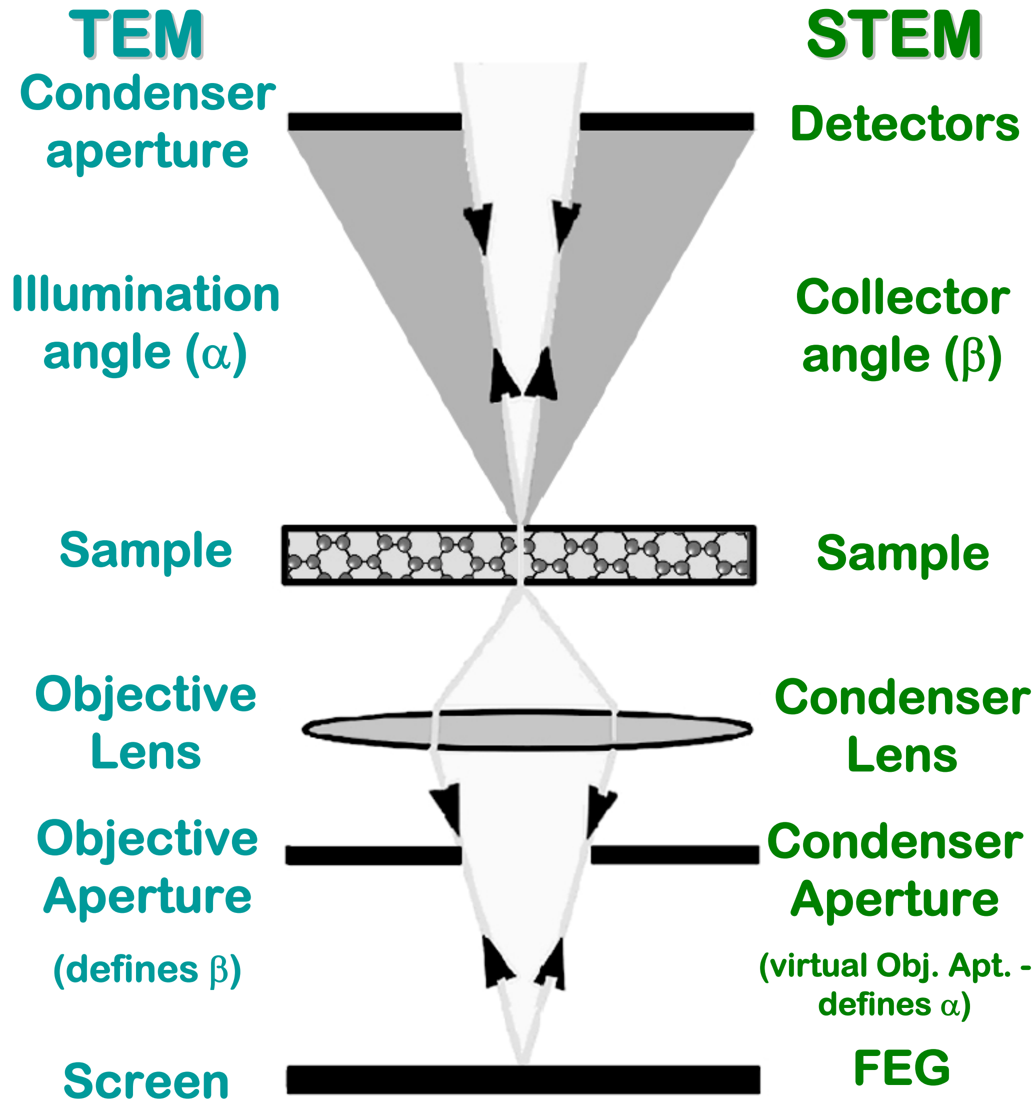
David Muller 2004

Reciprocity

In certain instances, TEM & STEM images are strictly equivalent

"Theorem of Reciprocity"

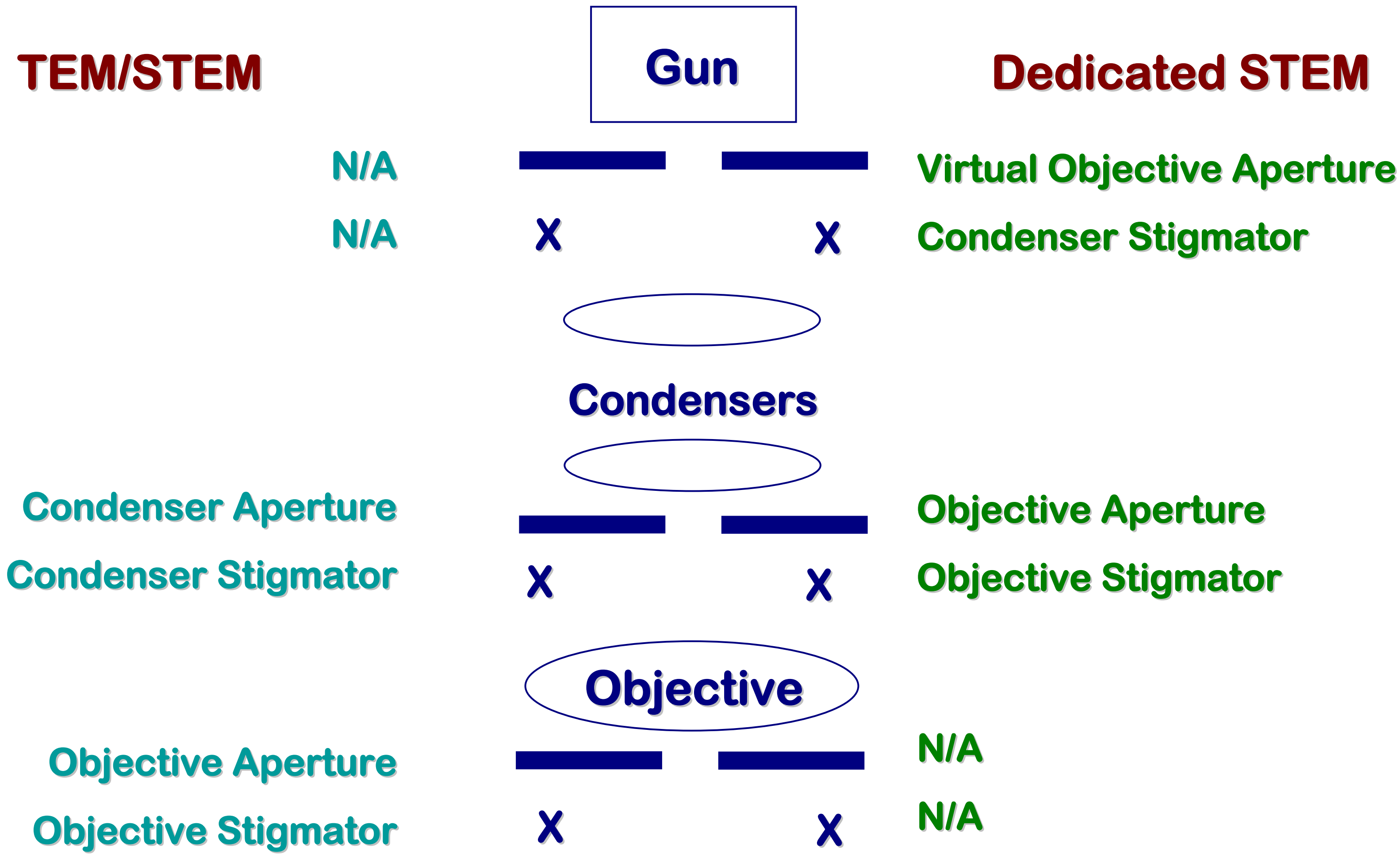
	TEM	STEM
Coherent illumination	Small Condenser Aperture	Small Collector Aperture
Incoherent illumination	Hollow cone illumination	HAADF



Courtesy Nigel Browning & Dave Muller

Nomenclature

depends on machine type



Courtesy Nigel Browning

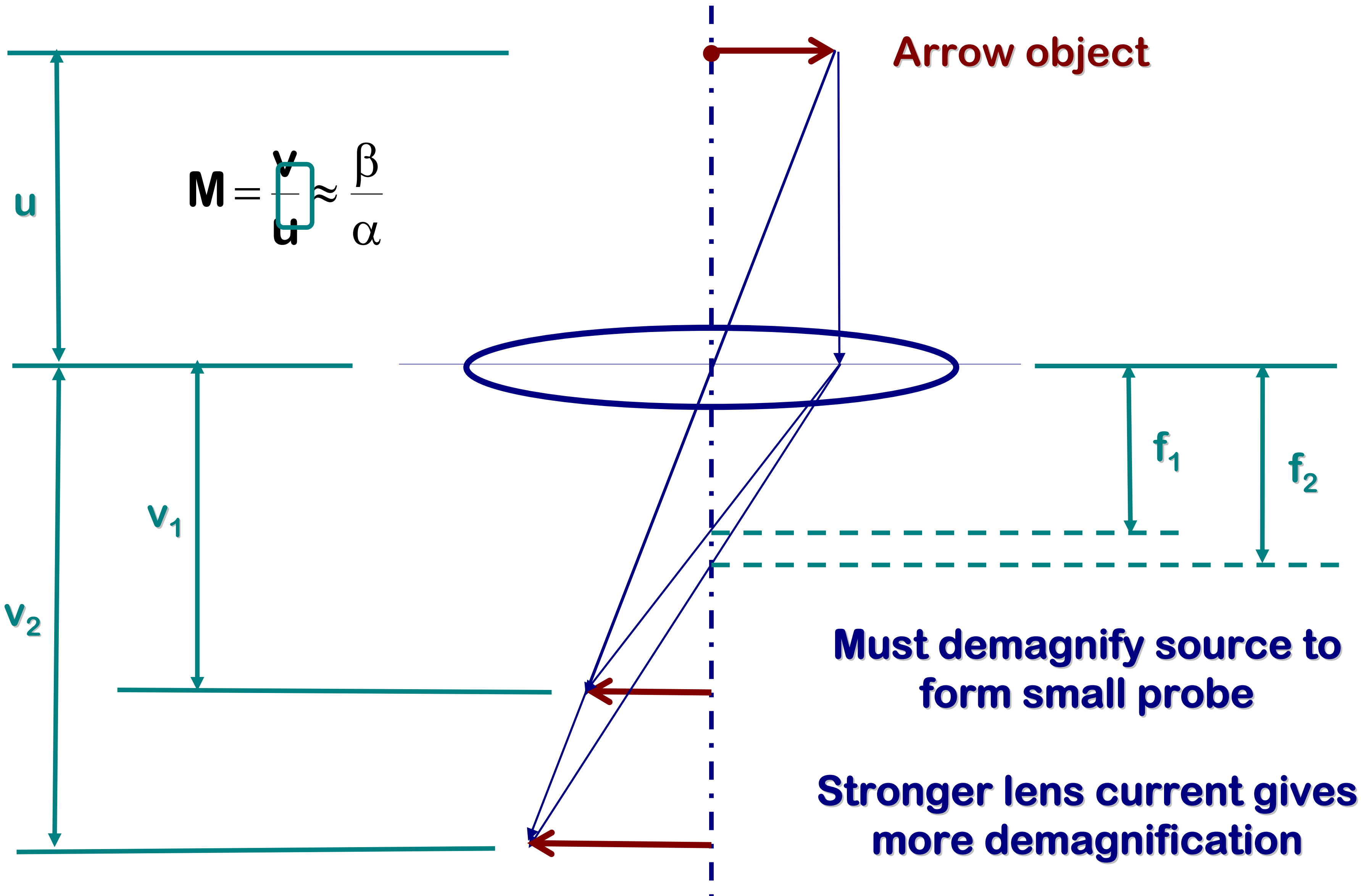
Electron sources

A small bright source is necessary for STEM

		Thermal emission		Field emission			
		W	LaB_6	Shottky ZrO/W	Thermal FE W (100)	Cold FE W (310)	
Brightness ($A/cm^2/sr$) at 200kV		~$5x10^5$	~$5x10^6$	~$5x10^8$	~$5x10^8$	~$5x10^8$	
Electron Source Size		50μm	10μm	0.1-1μm	10-100nm	10-100nm	
Energy Width (ev)		2.3	1.5	0.6-0.8	0.6-0.8	0.3-0.5	
Operating Conditions	Vacuum (Pa)	10^{-3}	10^{-5}	10^{-7}	10^{-7}	10^{-8}	10^{-9}
	Temperature (K)	2800	1800	1800	1600	300	
Emission	Current (μA)	~100	~20	~100	20-100	5-20	
	Short term stability	1%	1%	1%	7%	5%	2%
	Long term stability	1%/hr	3%/hr	1%/hr	6%/hr	20%	10%
Maintenance		Not necessary	Not necessary	Start-up takes time	Build up necessary after change	Flash every few hours	
Price & Operation		Low & simple	Low & simple	High & easy	High & easy	High & complicated ?	
Lifetime		3 months	1 year	>4 years	?	?	1 year

Courtesy Nigel Browning

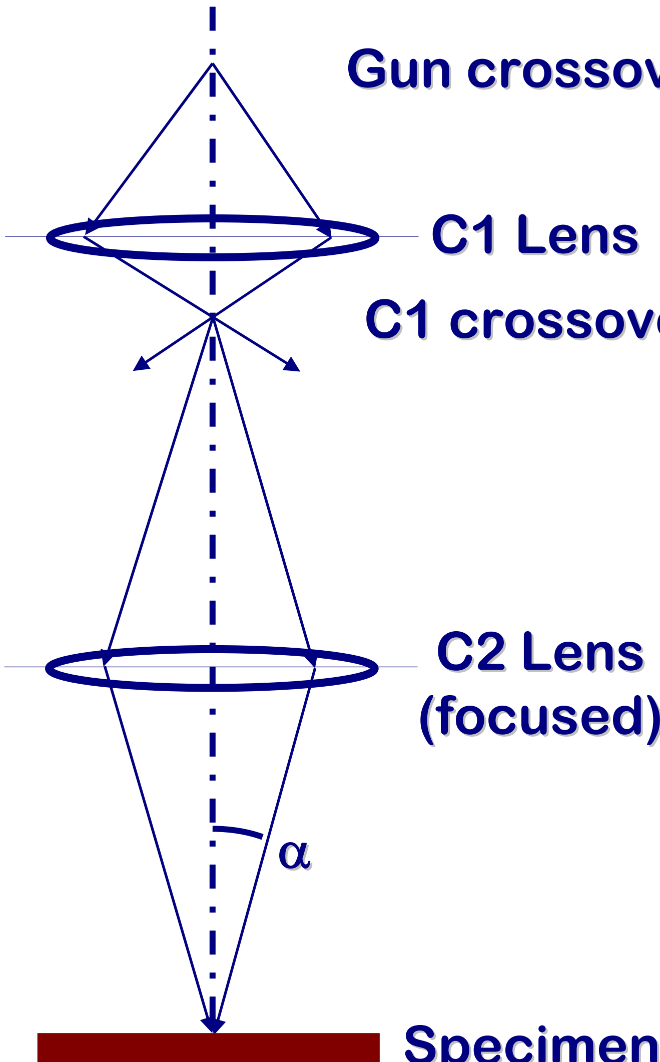
Demagnification

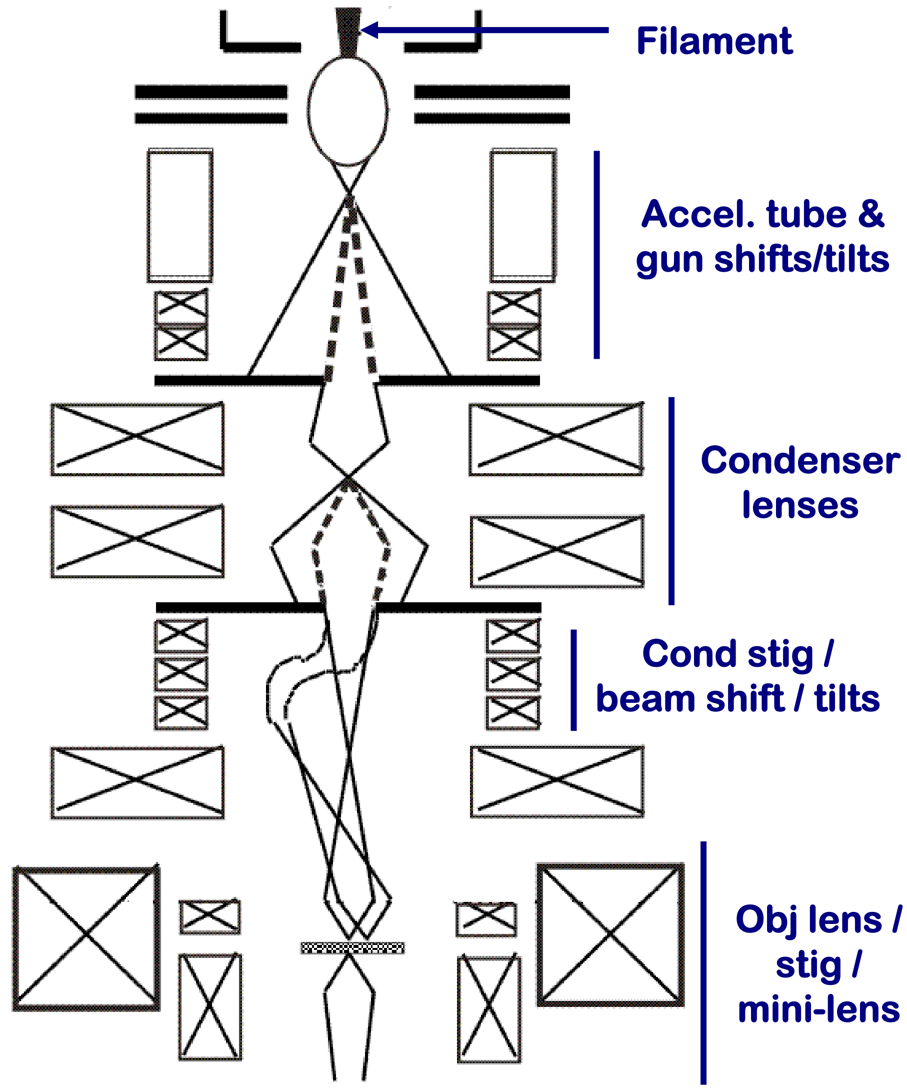


Must demagnify source to form small probe

Stronger lens current gives more demagnification

Probe forming optics





James and Browning, *Ultramicroscopy* 78, 125 (1999)

Scattering

For perfect crystals, four sources that give high-angle scatter

- Rutherford scattering
  - Elastic
  - Proportional to square of atomic weight ($\propto Z^2$)
- Higher Order Laue Zone reflections
  - Elastic
  - Important in thin samples
- Thermal diffuse scattering (TDS)
  - Scatter from phonons (lattice vibrations)
  - Not strictly proportional to Z^2
    - Depends on b (low angles screened by other planes in lattice)
    - Measured between $Z^{1.5}$ and $Z^{1.7}$
- Electron Compton scattering
  - Inelastic scatter off of the electrons

See Z.L. Wang, "Elastic and inelastic scattering in electron diffraction and imaging", Plenum, 1995
and C.B. Boothroyd, et al. Proc. MRS, Fall 1996.

Types of STEM images

Bright-field

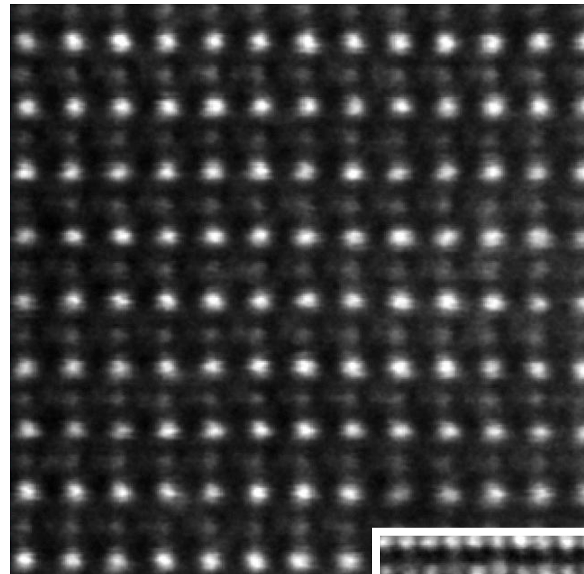
- Collect central beam with a small collection angle
- Contains elastic (Rutherford), phonon, plasmon and Compton

Low-angle annular dark field

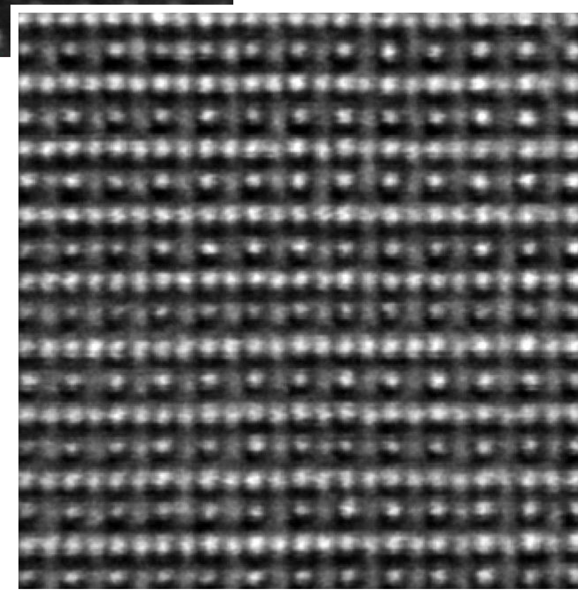
- Collection angle of 25 - 50 milliradians (mrad)
- Mostly phonon scatter

High-angle annular dark field

- Collection angle of 50 - 250 mrad
- Largely phonon scatter (TDS)



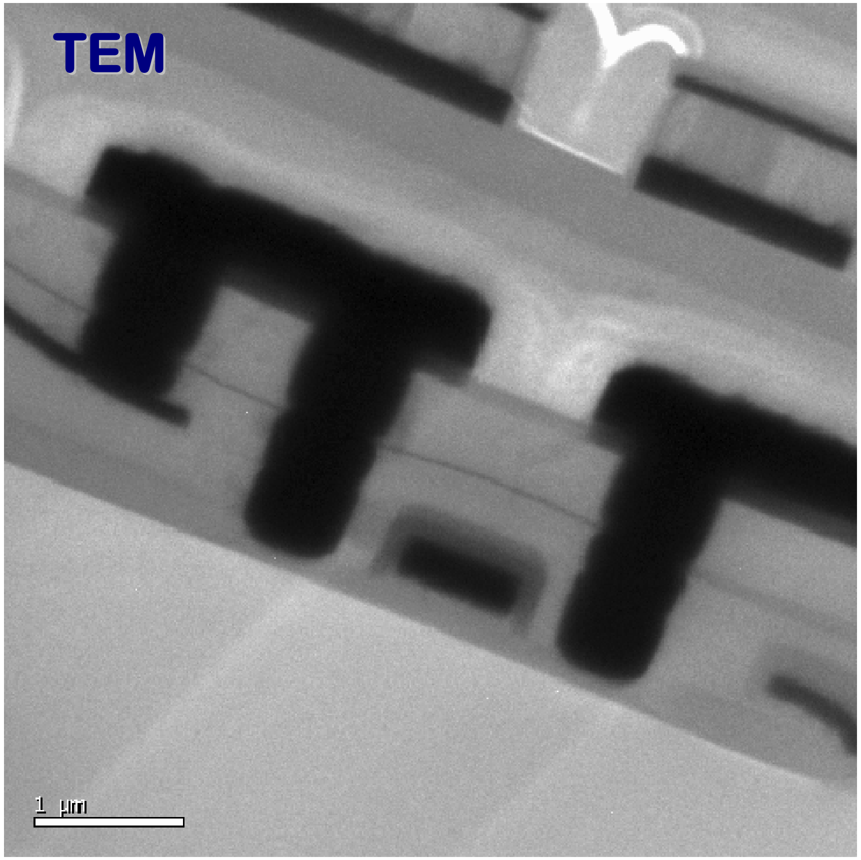
Coherent BF-STEM image of $SrTiO_3$ <110>

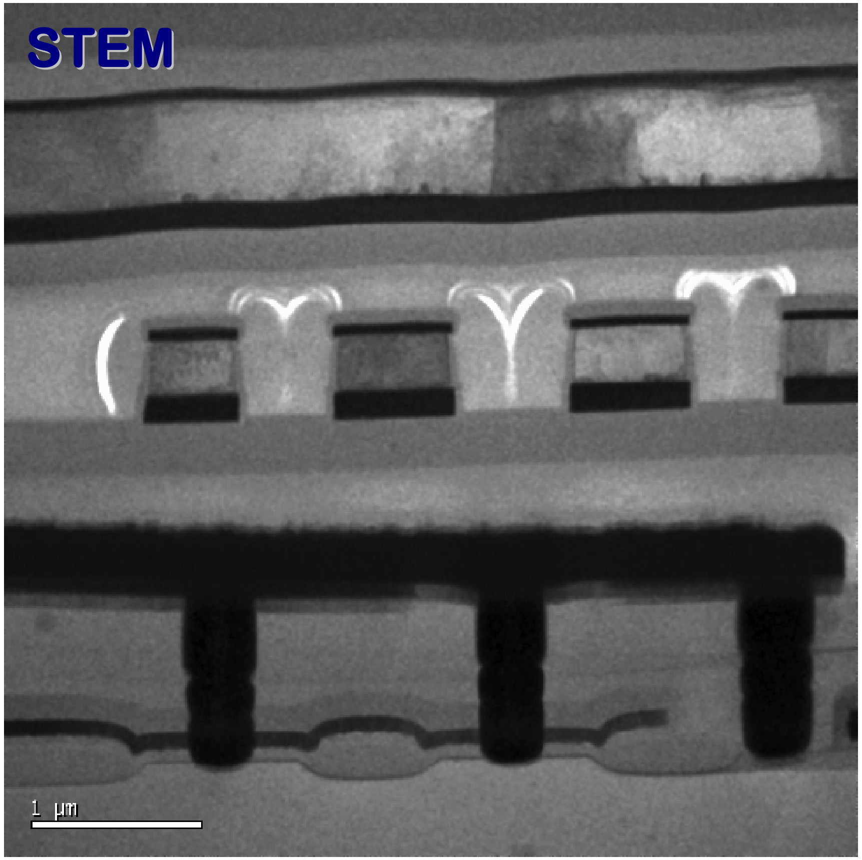


HAADF-STEM

Images from S.J. Pennycook

BF-STEM



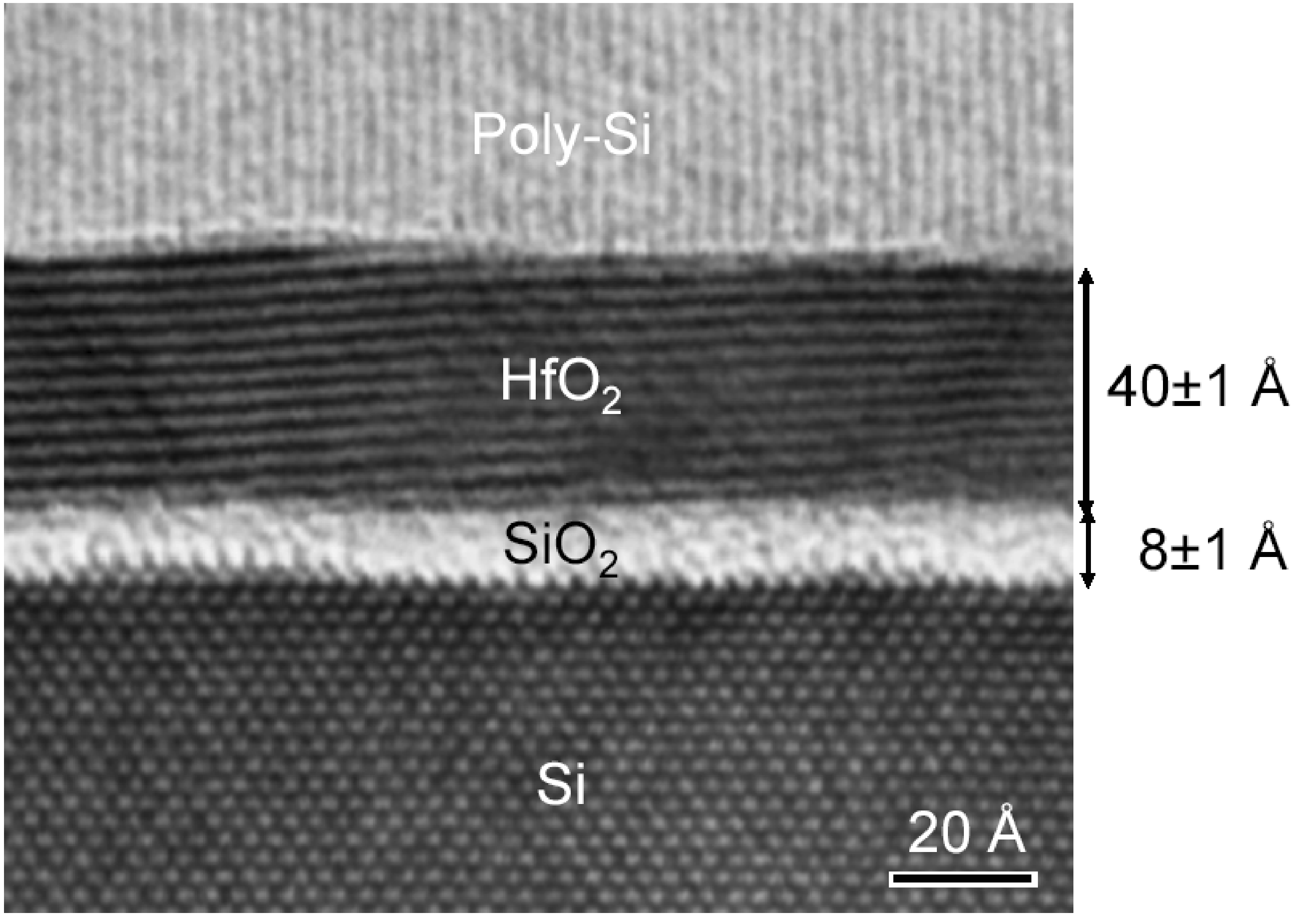


Images courtesy Dave Muller

In STEM, energy losses in sample to do not contribute to chromatic aberration (averaged over collector …)

Finding increasing use in semiconductor quality control

BF-STEM

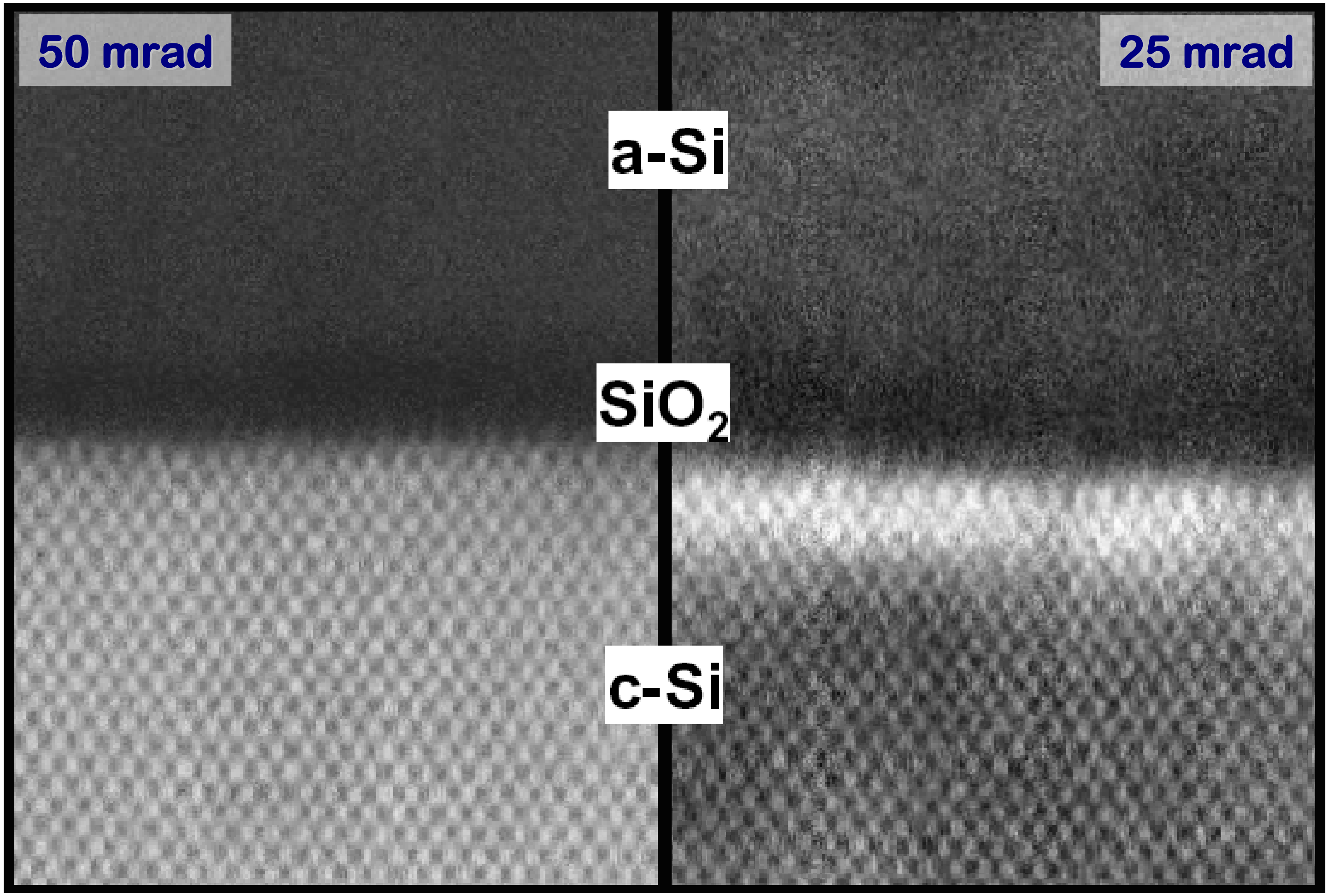


"Reciprocity"

But - HRTEM is certainly quicker, no 'scan noise'

Images courtesy Dave Muller

Low-angle annular dark field (LAADF)



Strain fields cause de-channeling and scattering to small angles

Images courtesy Dave Muller

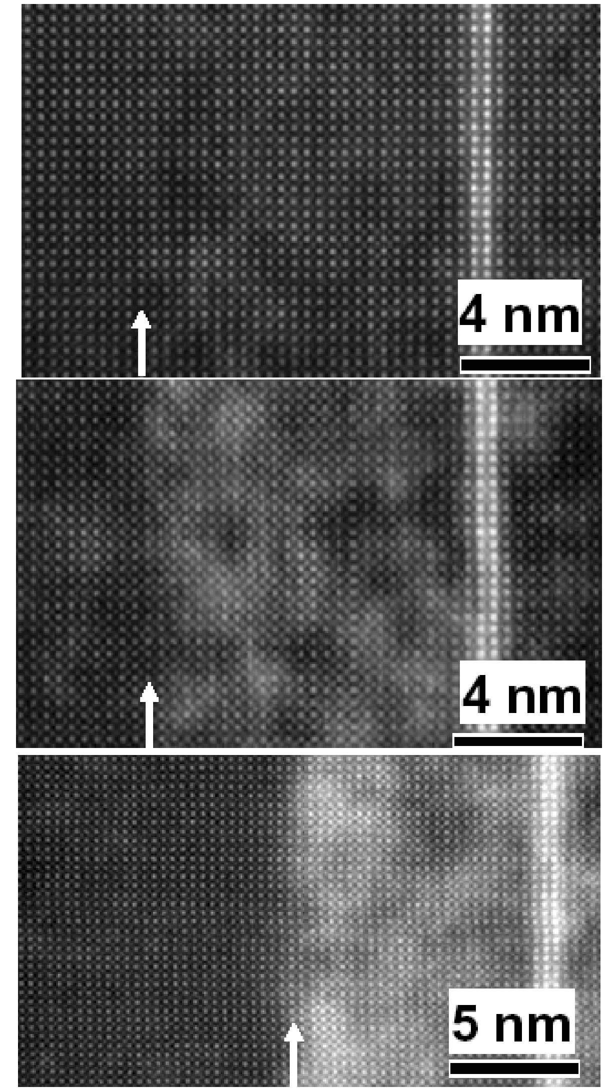
Low-angle annular dark field (LAADF)

Here contrast is correlated with oxygen vacancies

HAADF
"Z" map

LAADF
"Strain" map
Thin x/s

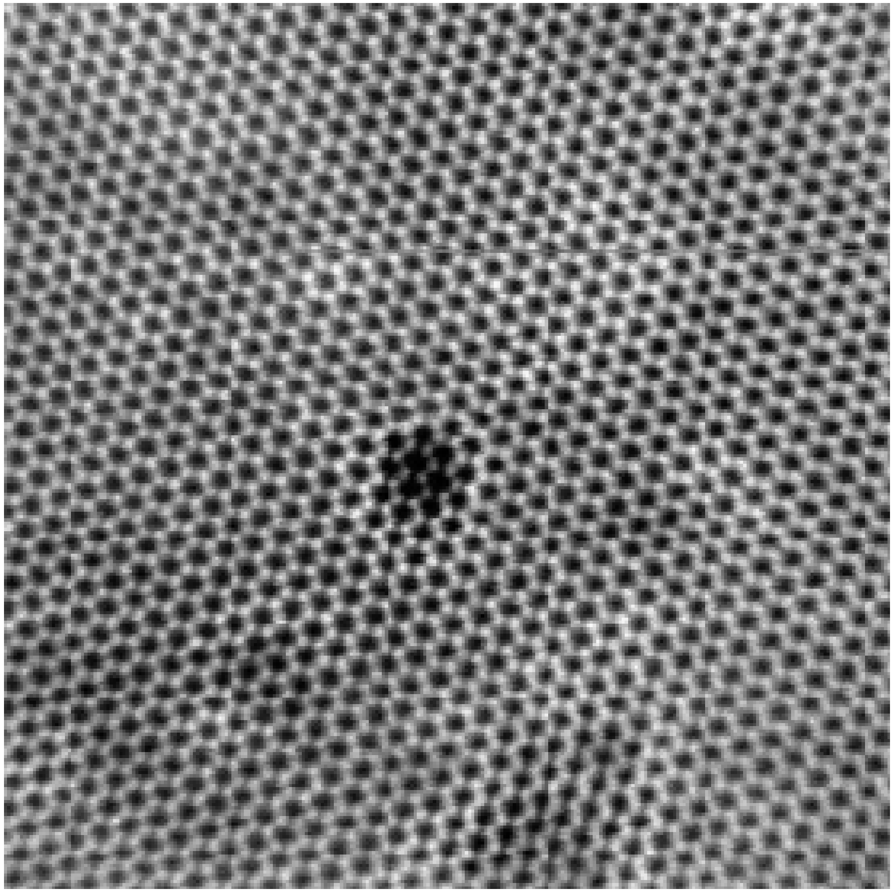
LAADF
"Strain" map
Thick x/s



Images courtesy Dave Muller

High angle annular dark field (HAADF)

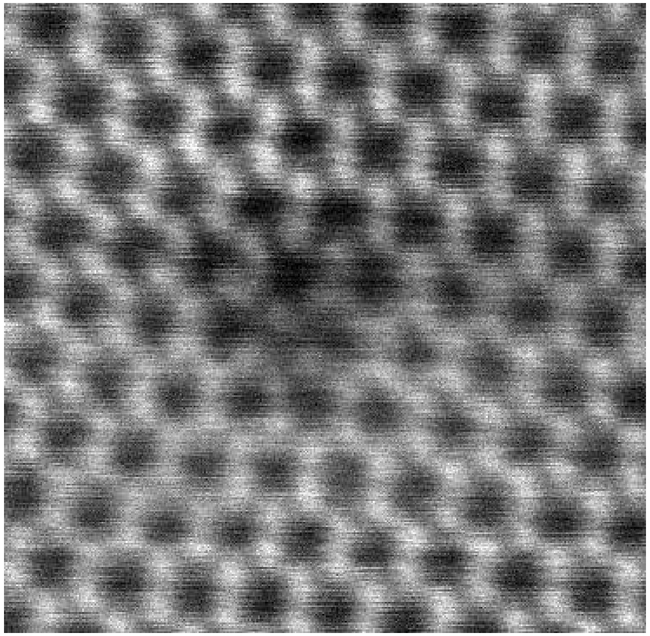
- No contrast reversals with thickness
- Directly 'interpretable' images
  - If you see a white blob, there's an atom column there
  - Caveat: the person taking (& processing) the image knew what they were doing …

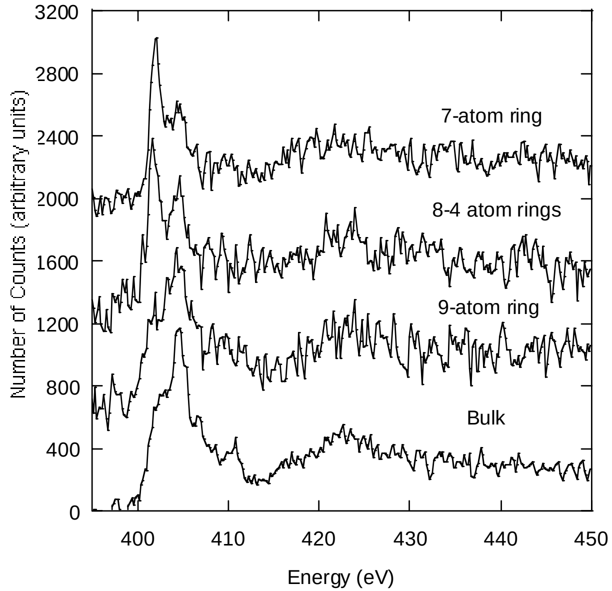


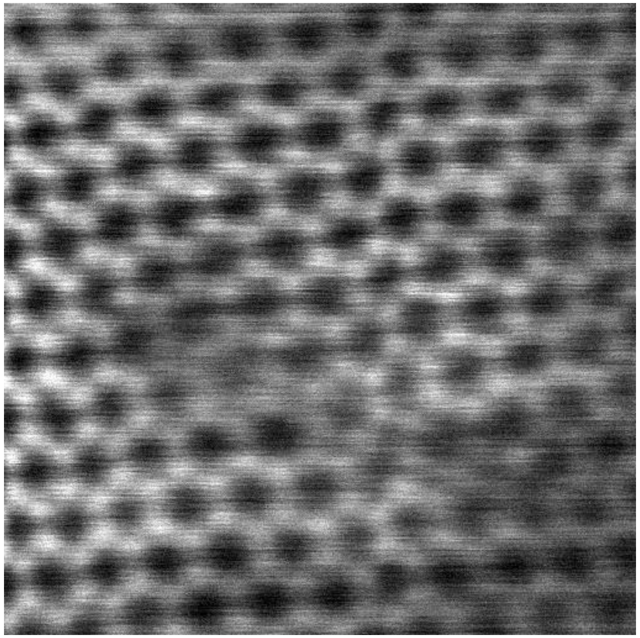
Screw dislocation core in GaN

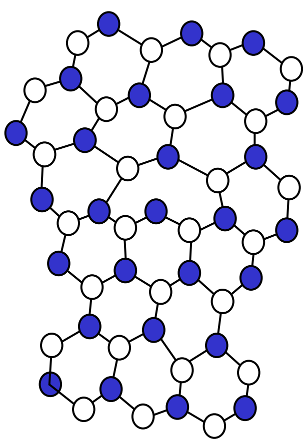
Image courtesy Ilke Arlsan, Nigel Browning

HAADF of dislocation cores





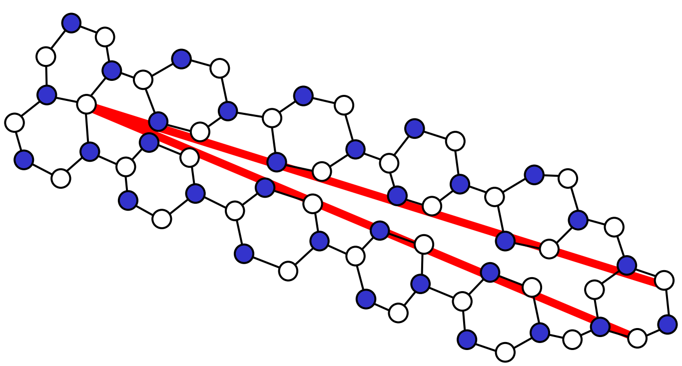




$$\frac{1}{3}[113] \rightarrow \frac{1}{3}[110] + [001]$$

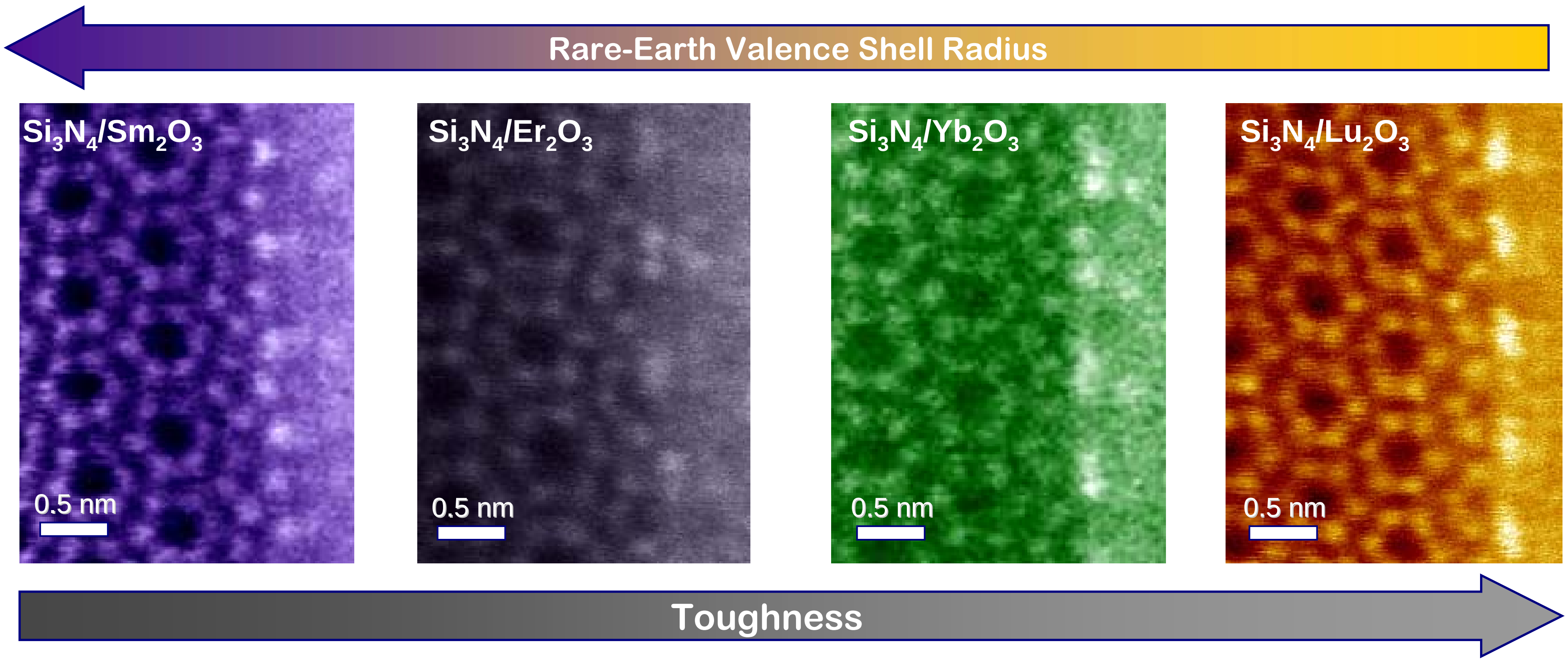
or

$$\frac{1}{3}\left[11\bar{2}3\right] \rightarrow \frac{1}{3}\left[11\bar{2}0\right] + [0001]$$



Arslan, Bleloch, Stach and Browning, PRL 2005

HAADF images



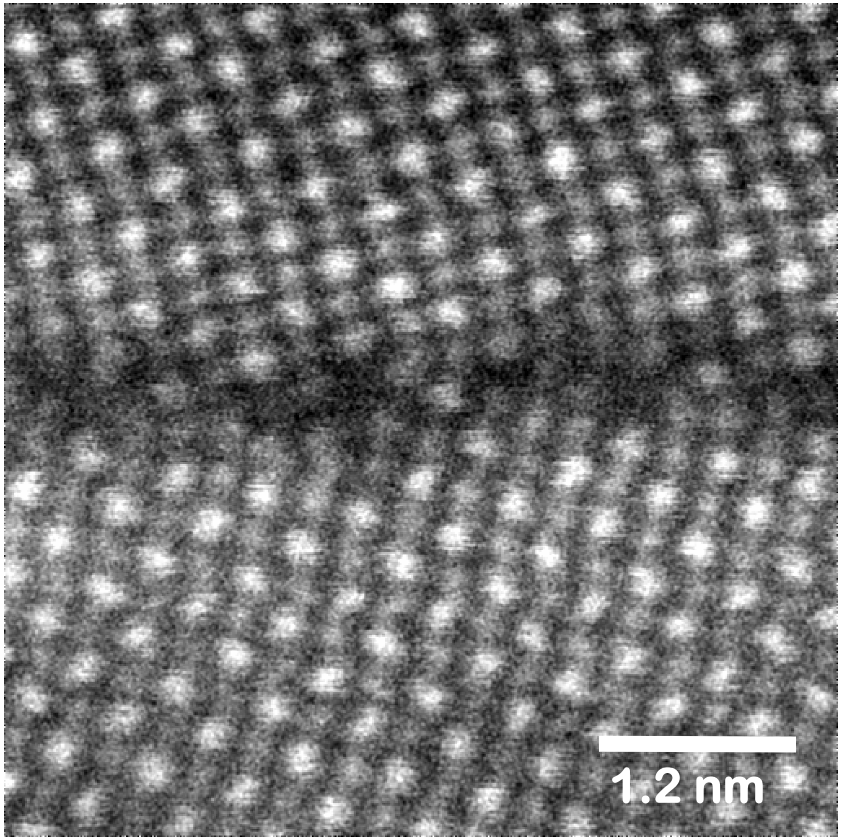
This is in image of a Si3N4 sample at a grain boundary lined with amorphous glass.

The effect of different dopant types can be seen

From Zeigler, et al., Science 2004

HAADF of grain boundaries

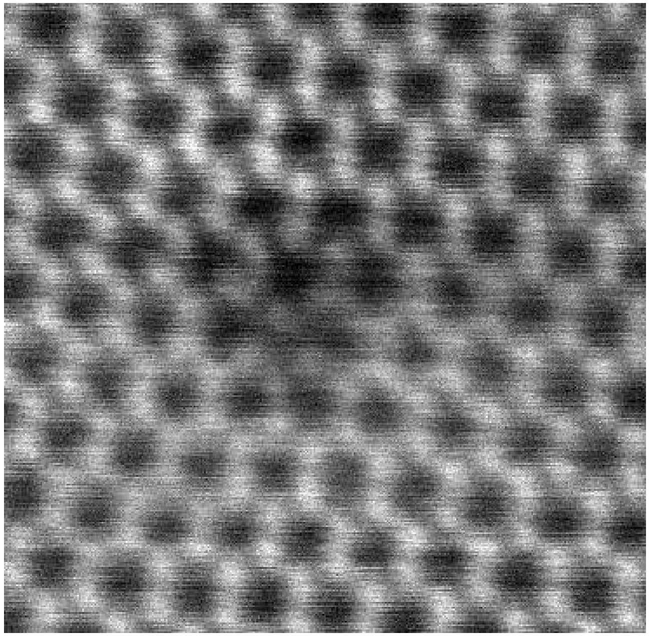
- HAADF often used to study grain boundaries and interfaces
- Direct atom positions combined with EELS spectroscopy
- Difference in intensity due to locally different phonon scattering
  - Local lattice relaxation allows different phonon modes
  - "Huang Scatter"

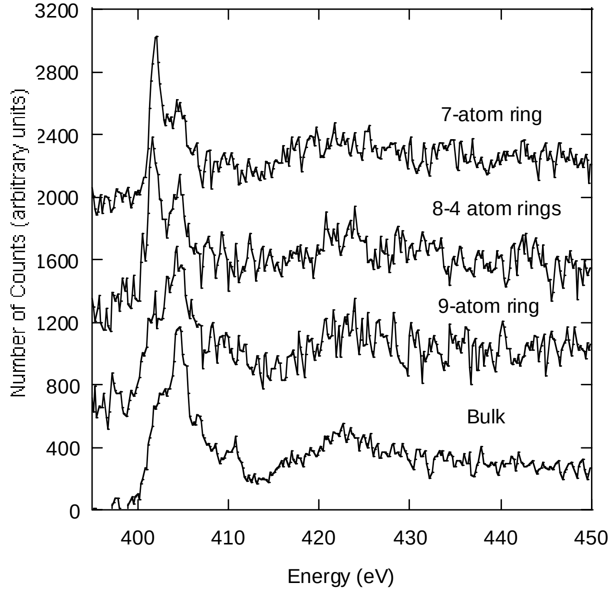


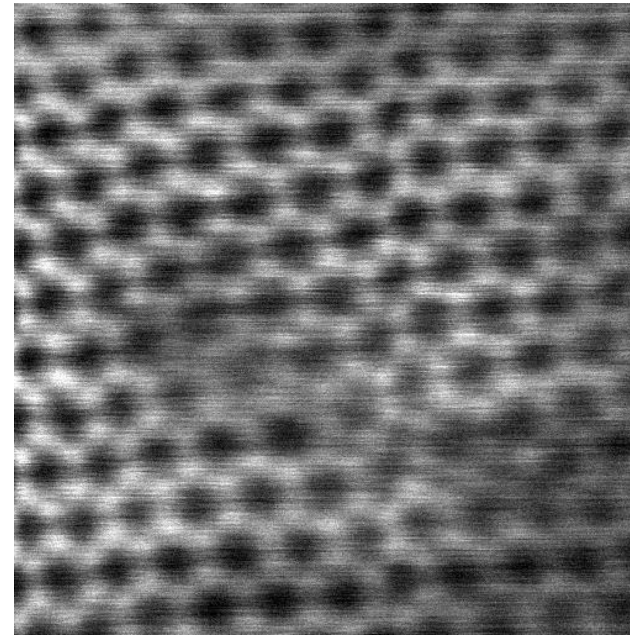
Grain boundary in $SrTiO_3$

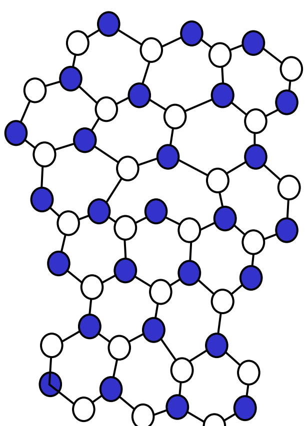
Image courtesy Nigel Browning

HAADF of dislocation cores









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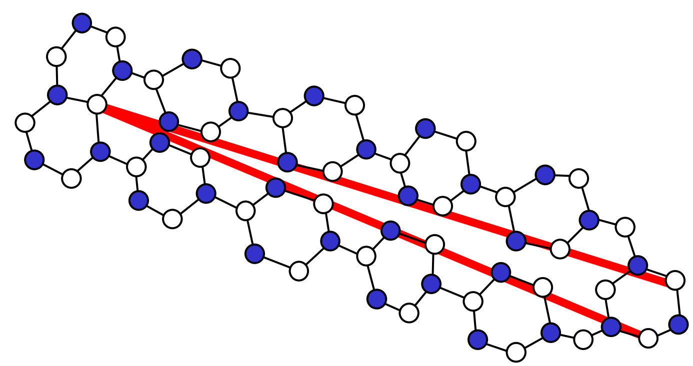
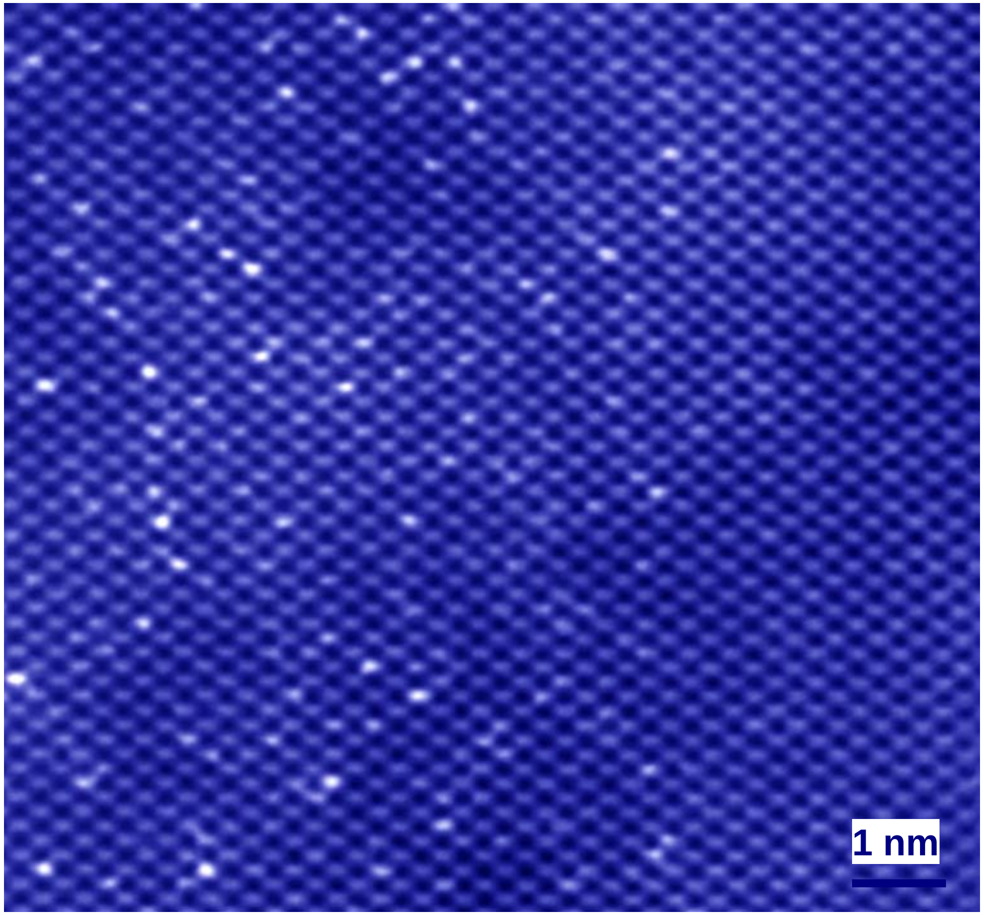


Arslan, Bleloch, Stach and Browning, PRL 2005

Imaging individual dopant atoms (Sb in Si)



Null test:

No Sb in substrate

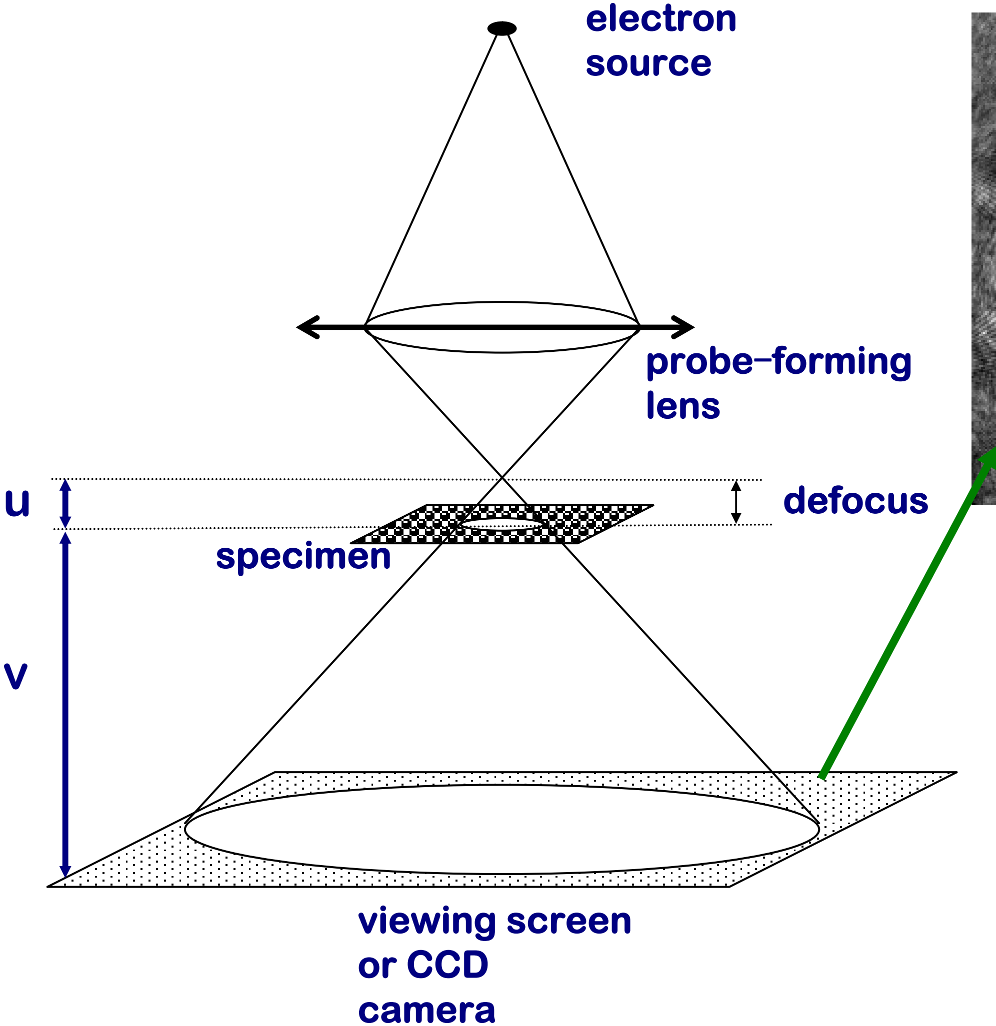
Sb source turned on here

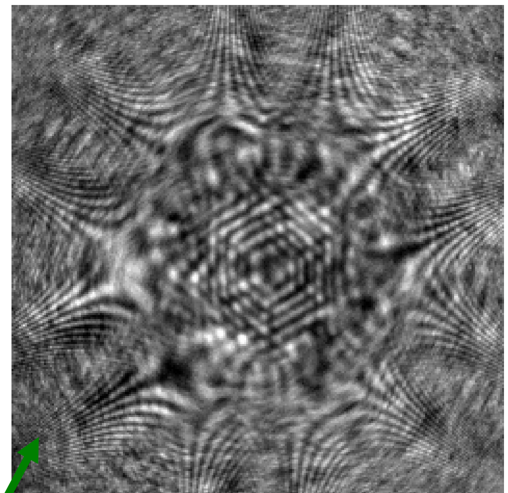
No Sb in substrate

P. Voyles, D. Muller, J. Grazul, P. Citrin, H. Gossmann, *Nature* **416** 826 (2002)

Alignment

Electron "Ronchigram"





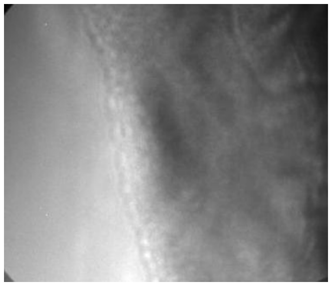
$$M = \frac{v}{u}$$

- Easiest method of alignment
- Easiest way to find optic axis
- Easiest way to correct astigmatism
- Easiest way to find focus
- Works best on amorphous material
- Start with largest aperture and work your way down

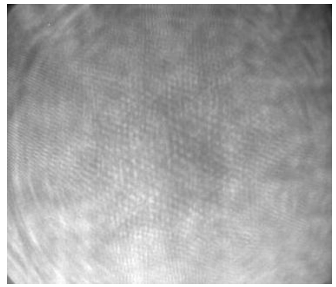
Cowley, *J. Elec Microsc Tech* 3, 25 (1986); E.N. James & N.D. Browning, *Ultramicroscopy*, 78, 1999

Ronchigrams from Si <110>

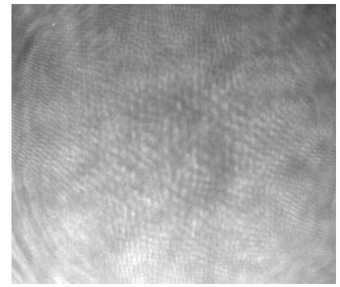
overfocused



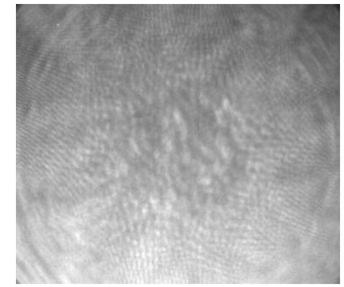
Δf=150 nm



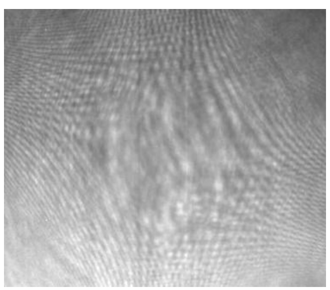
Δf=100 nm



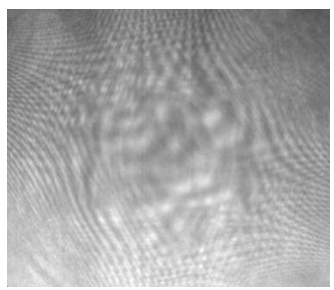
Δf=50 nm



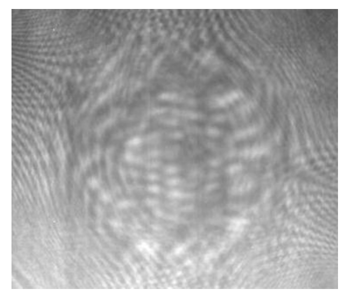
Δf=0 nm



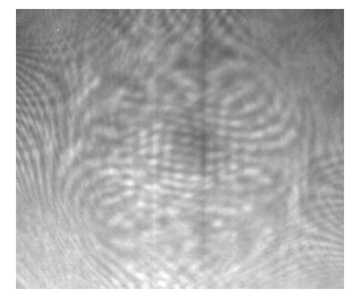
Scherzer focus



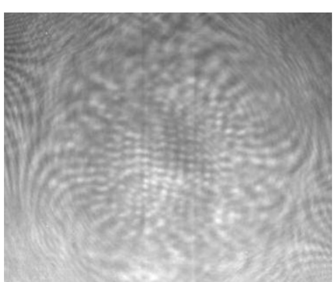
Δf=-100 nm



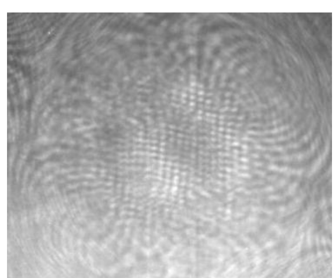
Δf=-150 nm



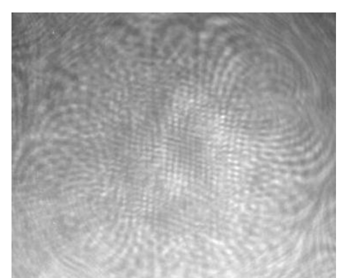
Δf=-200 nm



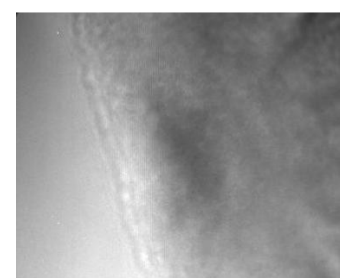
Δf=-300 nm



Δf=-350 nm



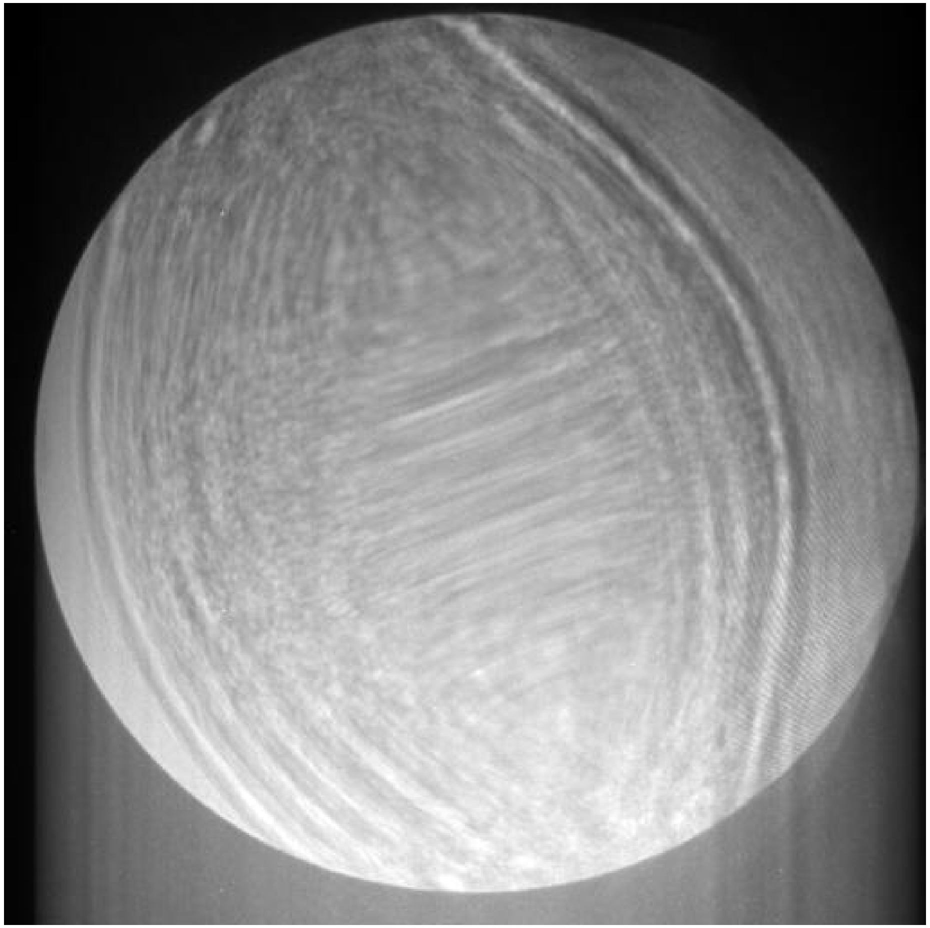
underfocused



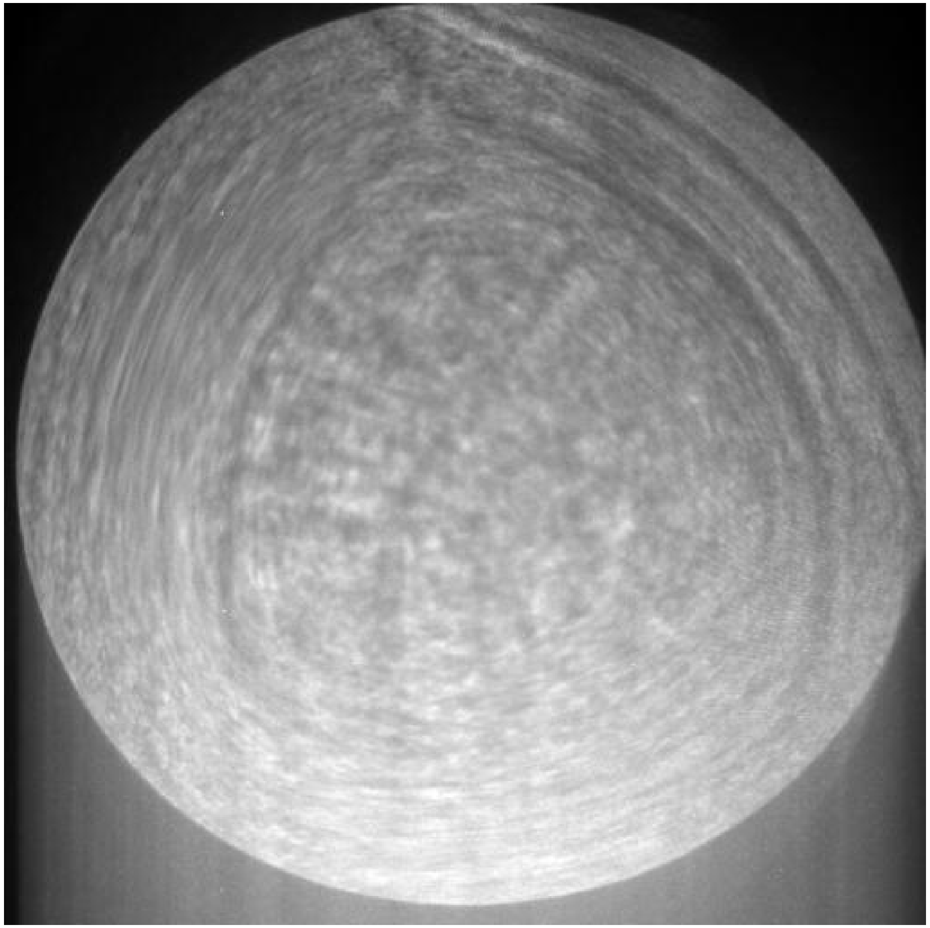
Courtesy Nigel Browning

Correcting for astigmatism

Two fold astigmatism

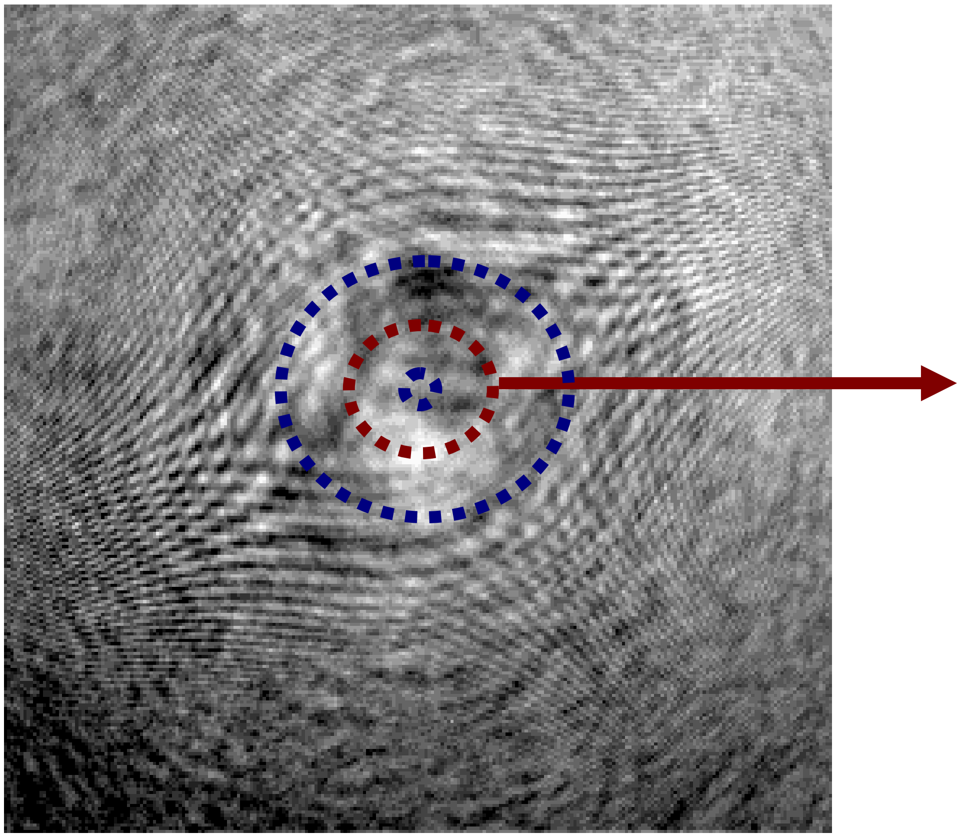


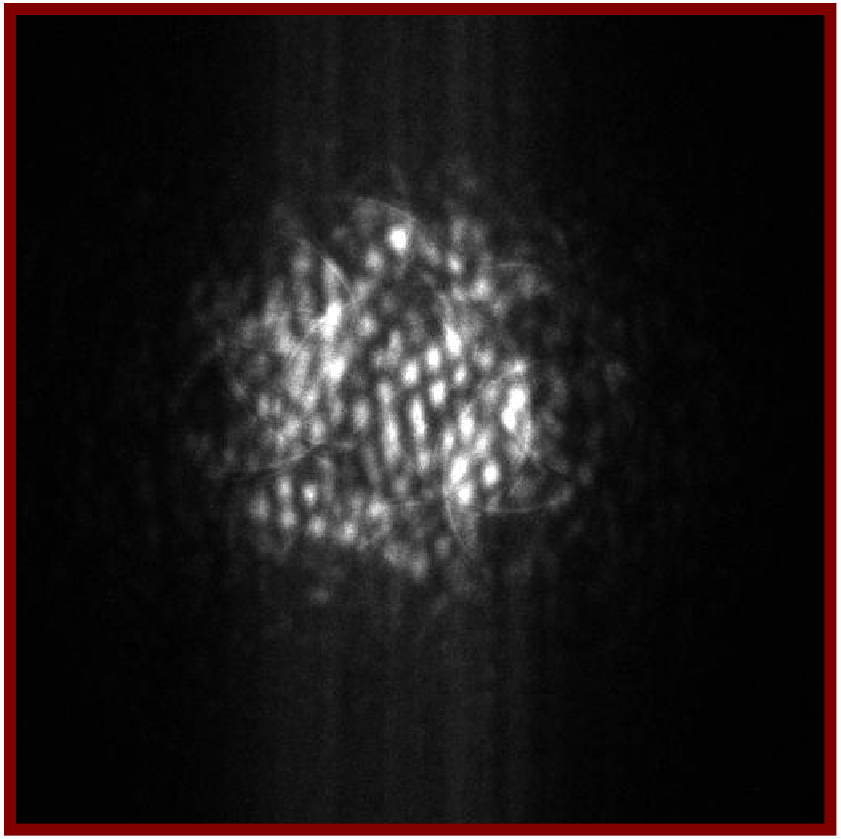
Three fold astigmatism



Courtesy Nigel Browning

Forming the Smallest Probe

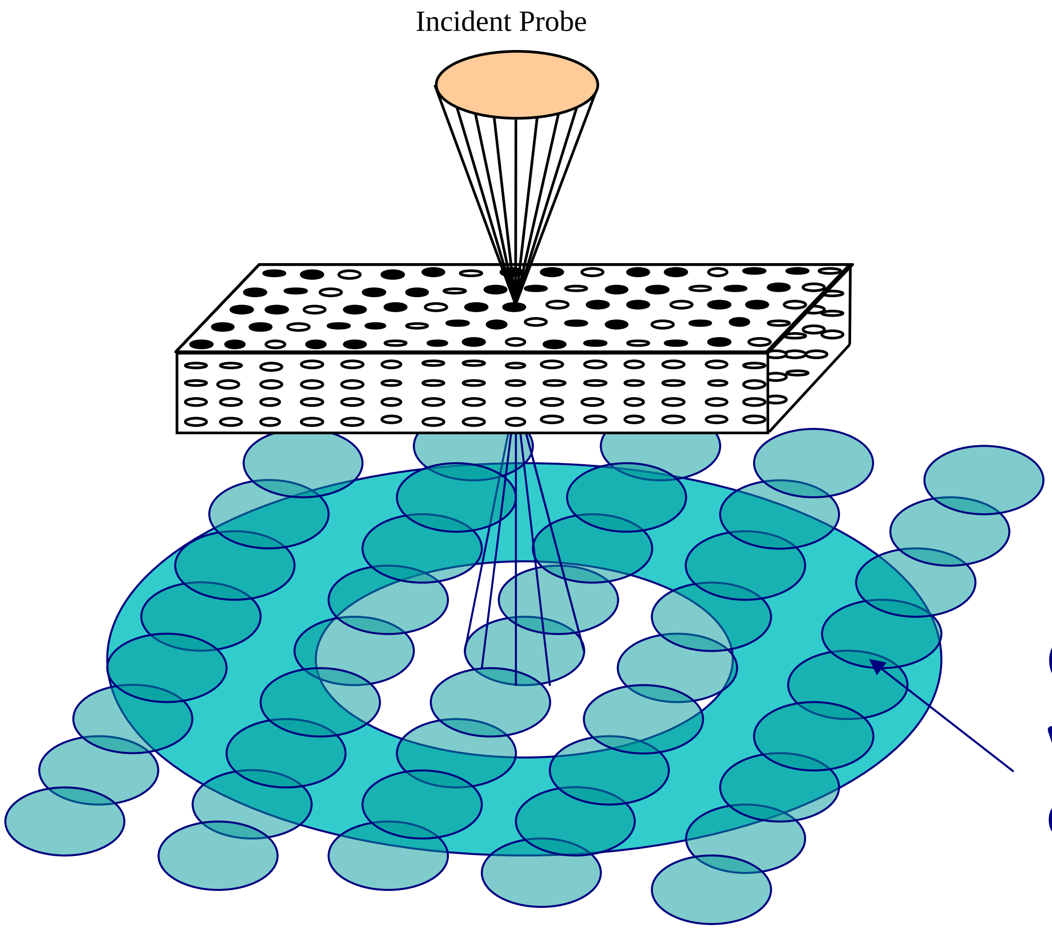




Put aperture over area of constant phase in Ronchigram to give CBED pattern

Courtesy Nigel Browning

Forming the dark field image



Overlap of disks bright when probe on a column, dark between columns

High-angle detector integrates many spots in the CBED pattern

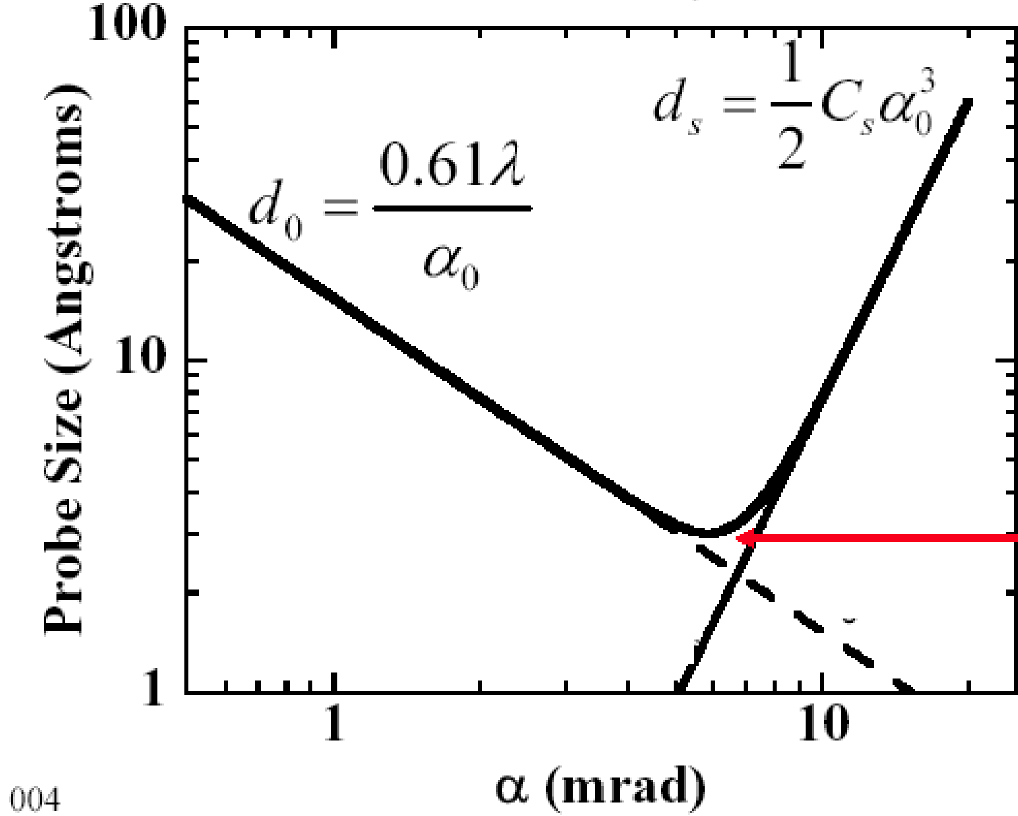
Courtesy Nigel Browning

Optimum resolution

balance C_s and aperture angle

Remember aperture introduced diffraction effect on resolution

$$d_{tot}^2 \approx d_0^2 + d_s^2$$



Optimal aperture
And minimum
Spot size

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

Courtesy Dave Muller

Optimum resolution

Using wave optics, find:

Minimum spot size: $d_{\min} = 0.43 C_s^{1/4} \lambda^{1/4}$

Optimum aperture size: $\alpha_{\text{opt}} = \left(\frac{4\lambda}{C_s} \right)^{1/4}$

At 200kV, λ = 0.0257Å

C_s = 1.0 mm, $d_{\min}$ = 1.55Å and α_{opt} = 10 mrad

C_s = 1.2 mm, $d_{\min}$ = 1.59Å and α_{opt} = 9.6 mrad

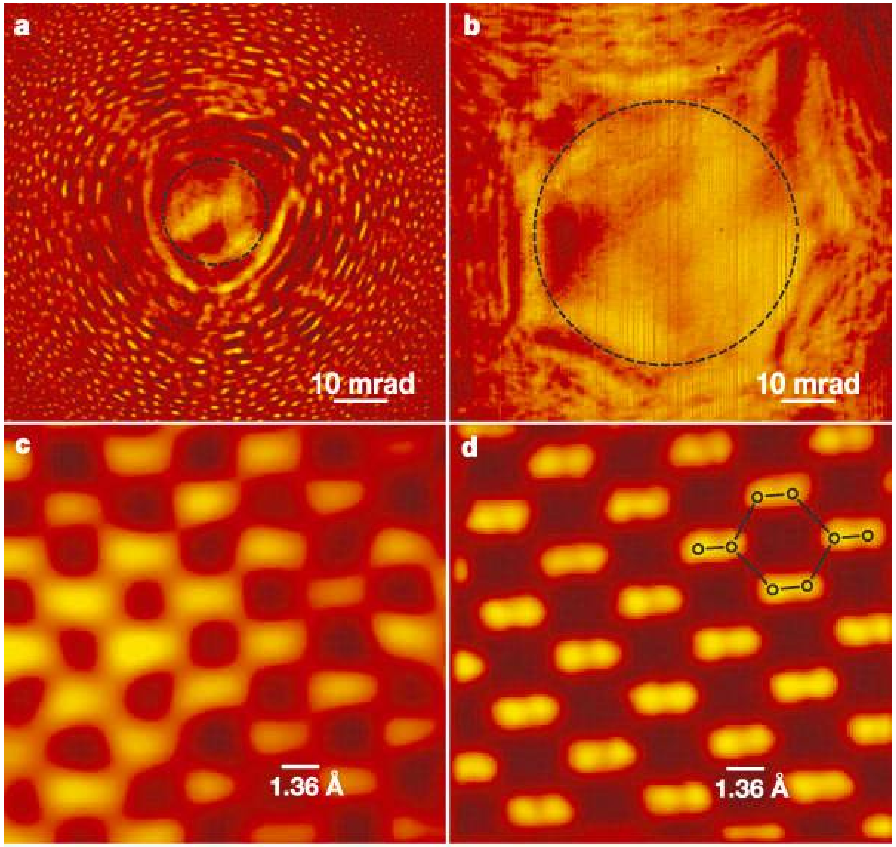
C_s = 0.5 mm, $d_{\min}$ = 1.28Å and α_{opt} = 12 mrad

C_s = 0.6 mm, $d_{\min}$ = 1.34Å and α_{opt} = 11 mrad

Courtesy Dave Muller

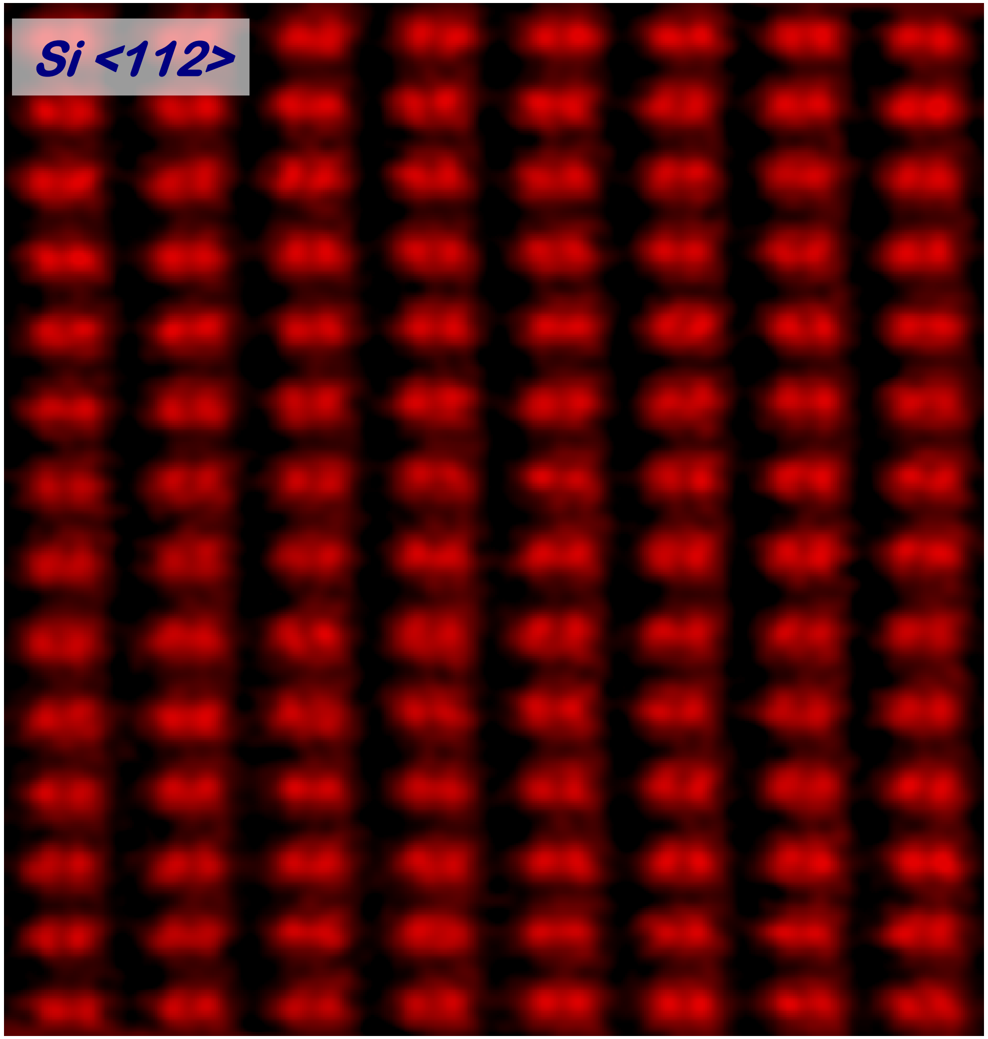
Effect of C_s Correction

- Cs correction opens up the aperture angle
- Allows a much smaller probe



Batson, Dellby and Krivanek, Nature **418**, 617 (2002)

Effect of C_s correction



Direct resolution at 0.78 Å

Information transfer to 0.607 Å

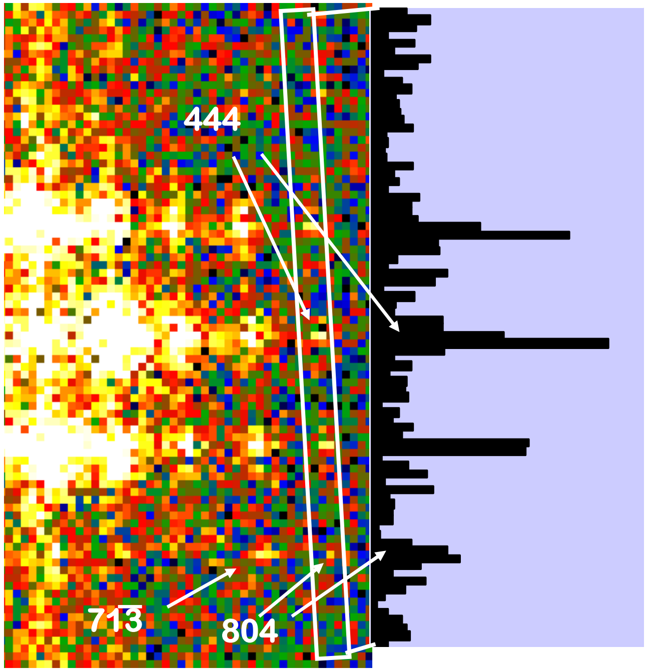


Image by Matt Chisholm,

Processing by Albina Borisevich and Andy Lupini

Aberration correction by Pete Nellist et al., Nion Co.

Nellist et al., Science 305, 2004.

Stability issues for STEM

(and for TEM!)

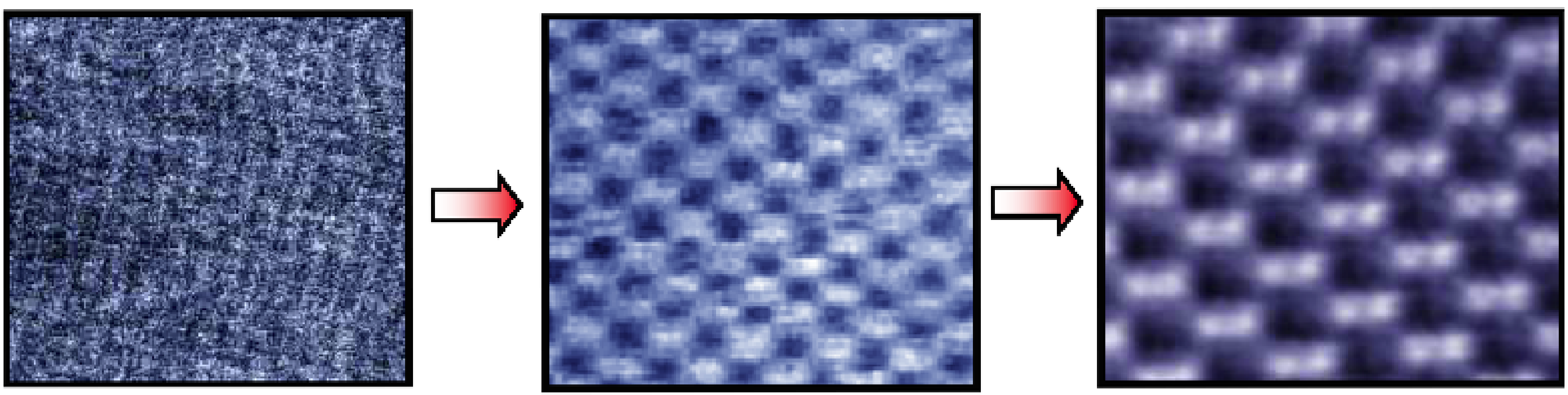
Stray Fields/Ground loops

Air Flow/Temperature Control

Pressure Variations

Mechanical Vibrations

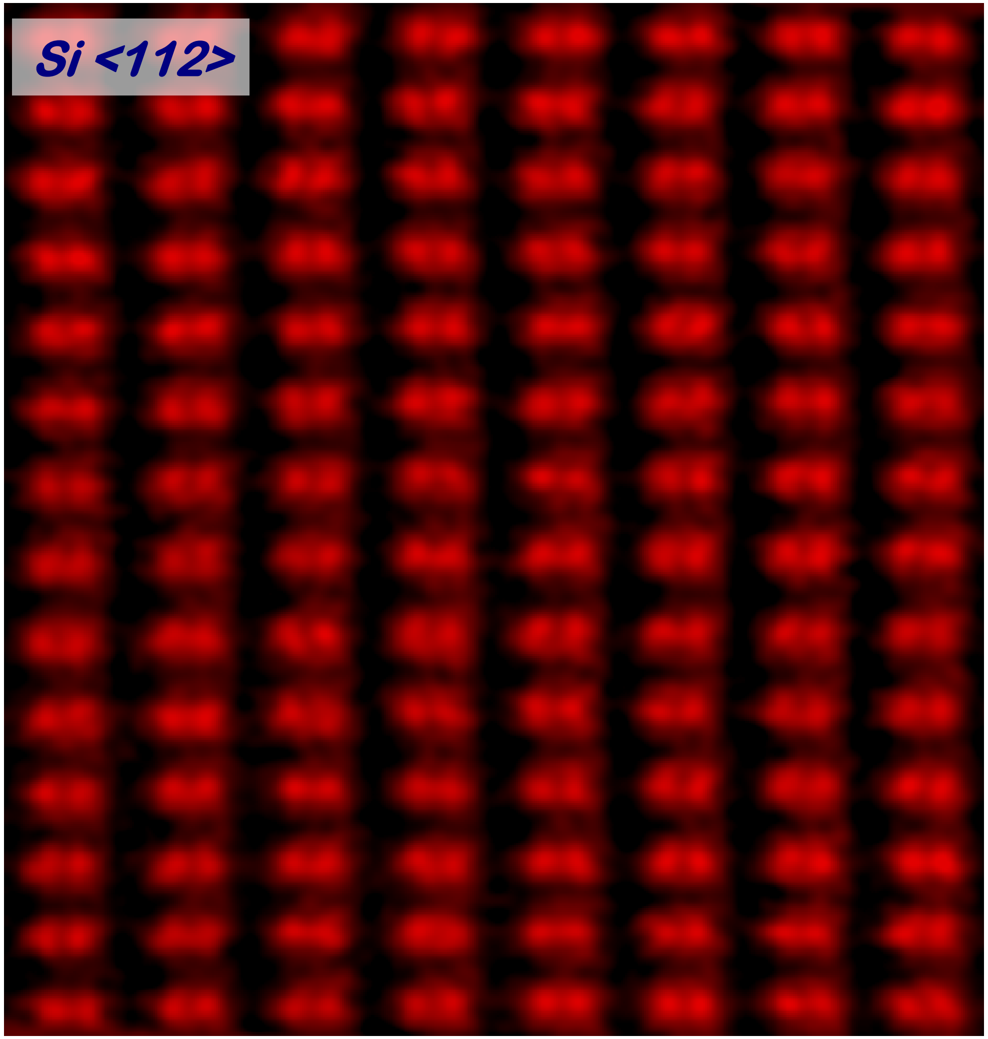
Specimen Preparation



3 weeks | 6 months | 1 year

D. A. Muller, J. Grazul, *Journal of Electron Microscopy*, **50** (2001) 219.

Effect of C_s correction



Direct resolution at 0.78 Å

Information transfer to 0.607 Å

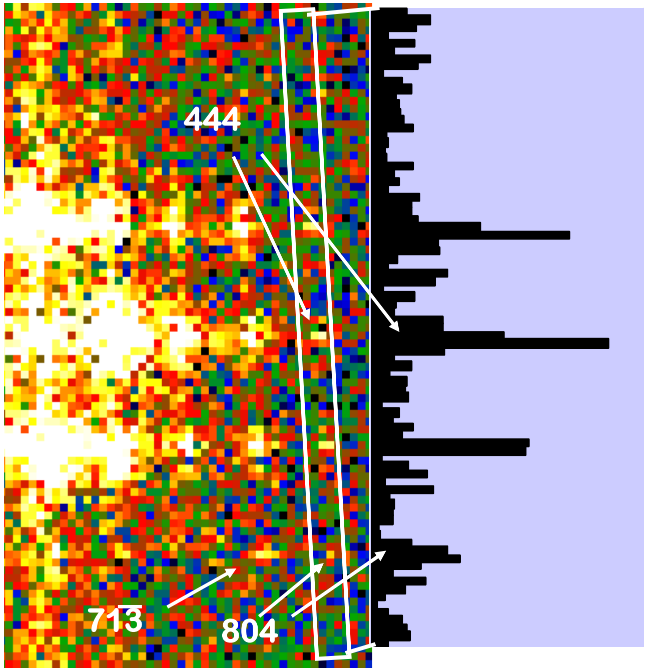


Image by Matt Chisholm,

Processing by Albina Borisevich and Andy Lupini

Aberration correction by Pete Nellist et al., Nion Co.

Nellist et al., Science 305, 2004.

Image ‘artifacts’

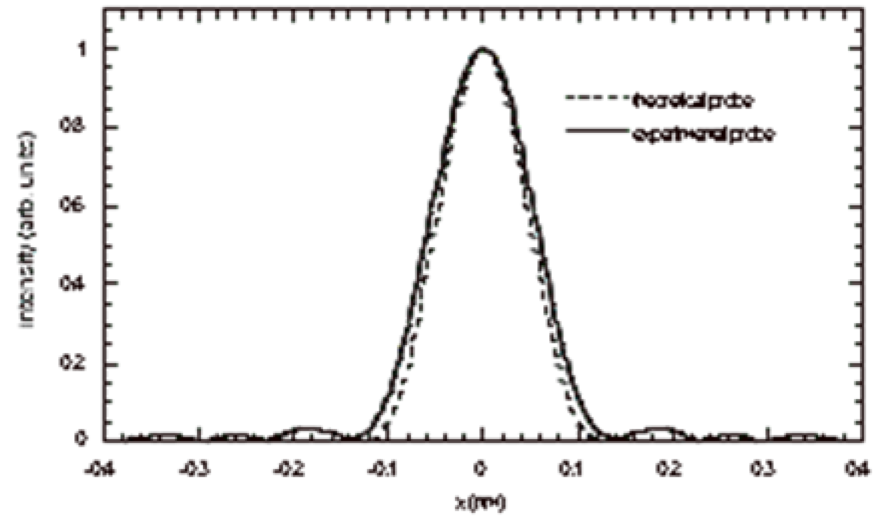
“probe tails”

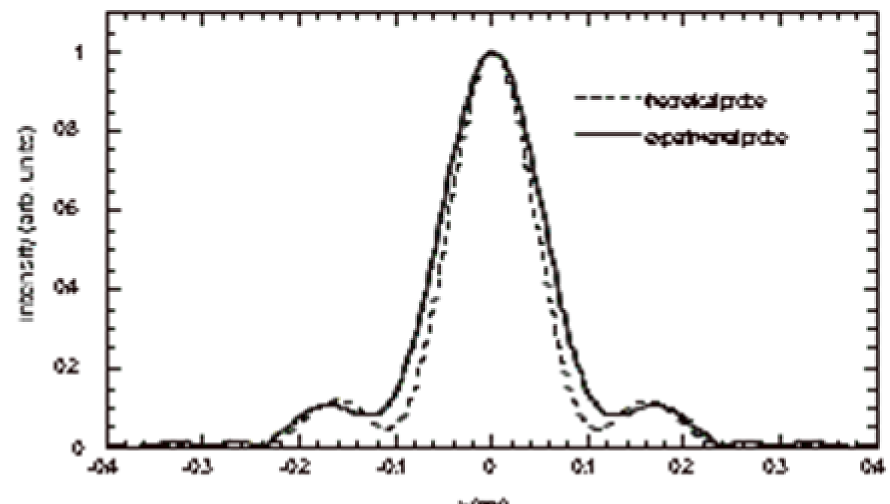
Can tweak probe shape to get a narrower probe

But, gives ‘tails’ in the distribution

These give ‘extra spots’ in the diffraction pattern

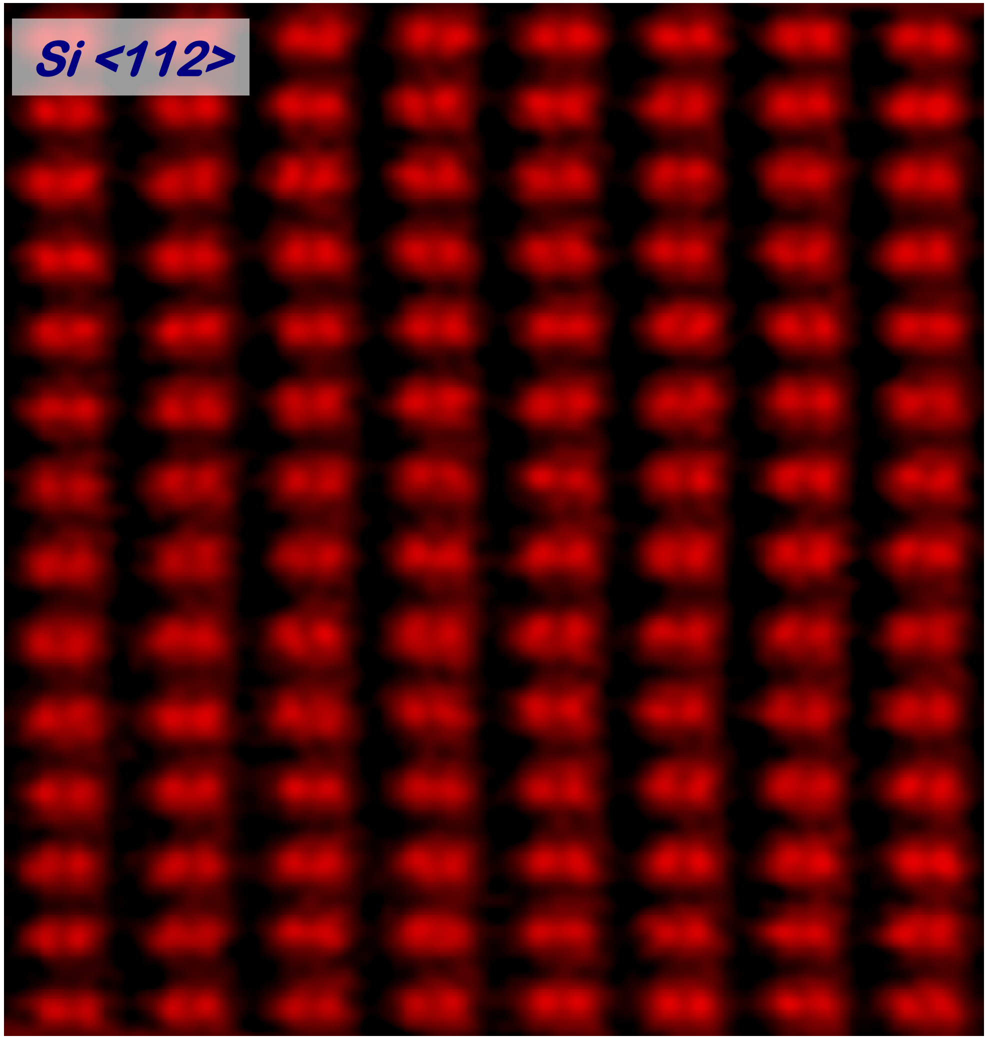
- The presence of extra spots can be confuse resolution determination
- Now people used images along known projections





James and Browning, *Ultramicroscopy* 78, 125 (1999)

Effect of C_s correction



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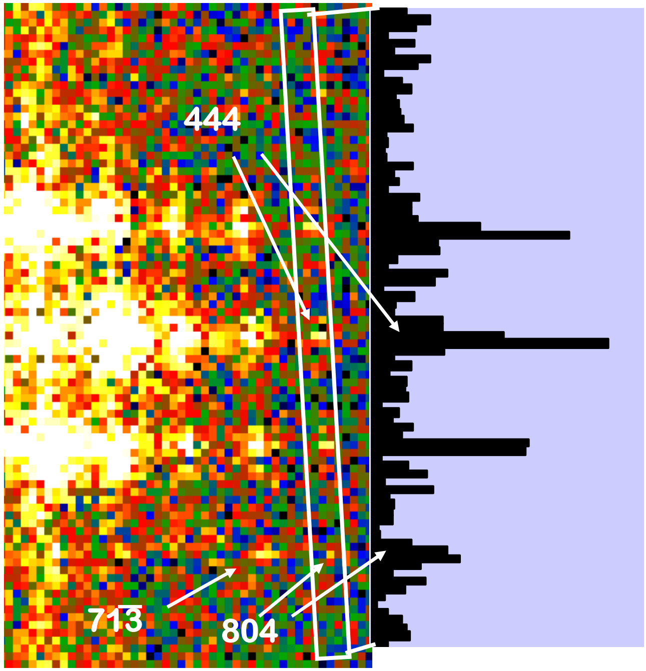

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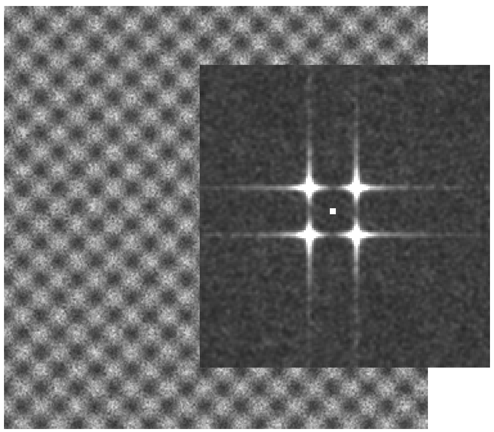
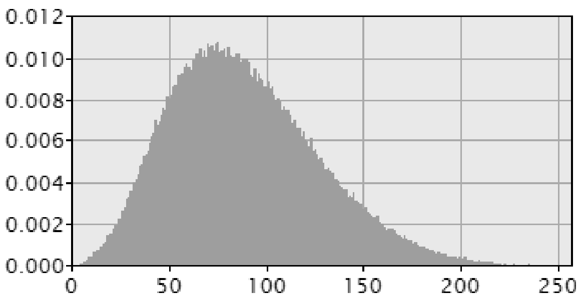
Image artifacts

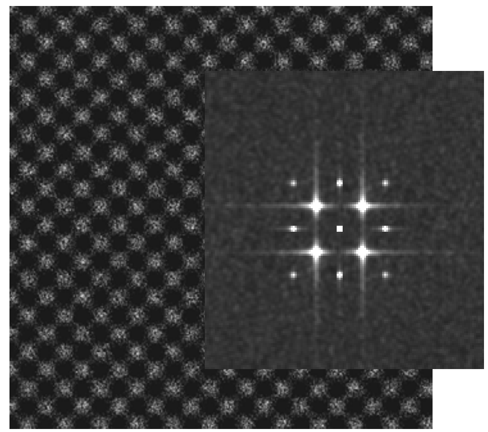
'clipping'

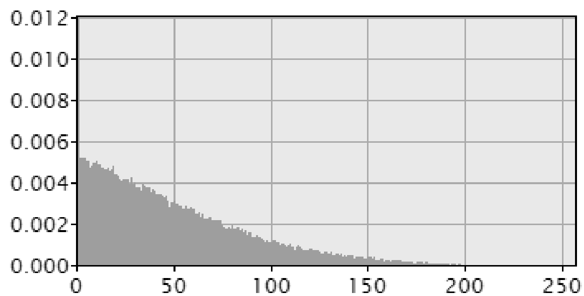
Unclipped



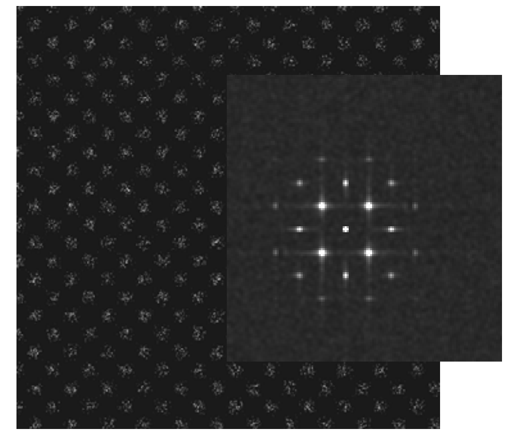


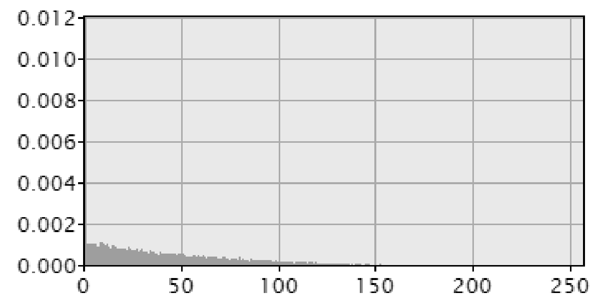
40% clipped





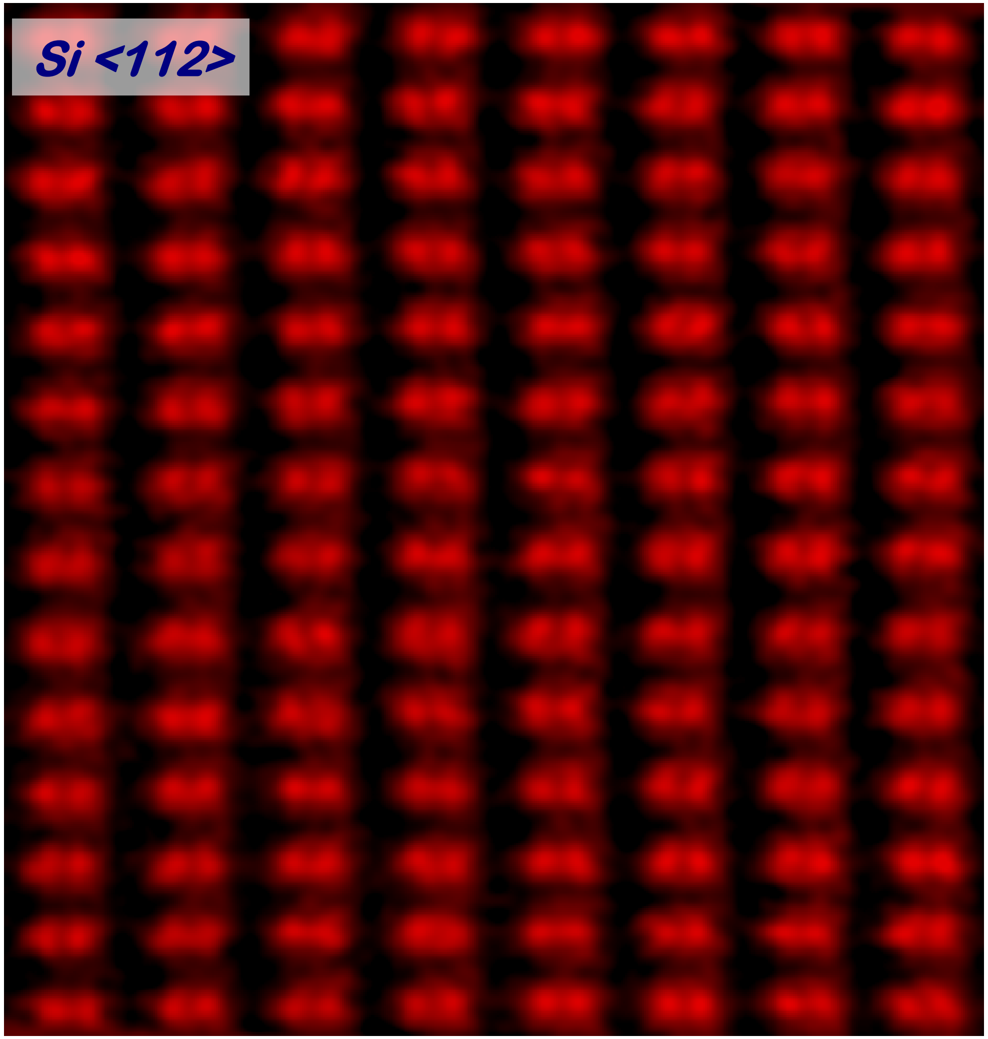
60% clipped





From S.J. Pennycook

Effect of C_s correction



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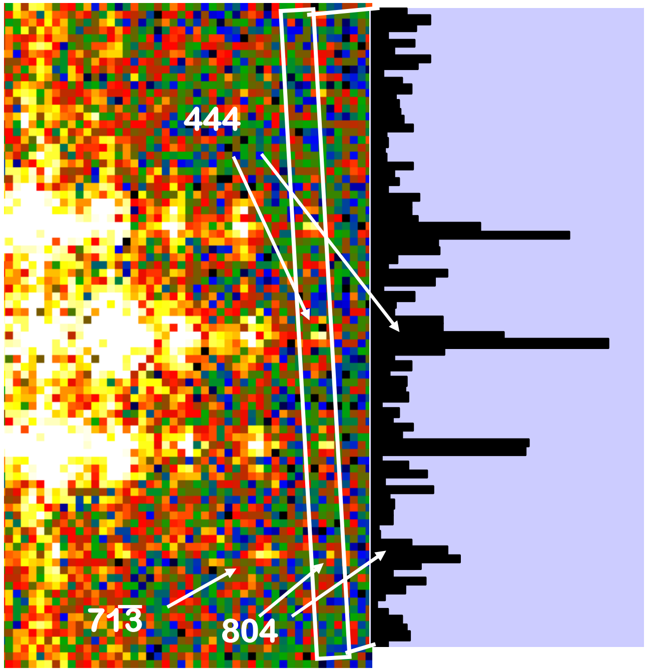

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