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- Thickness dependence of images
- Common mistakes
- Alignment Tricks

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**Scanning Transmission Electron Microscopy**

200 kV Incident Electron Beam ($\Delta E=1$ eV)

1 atom wide (0.2 nm) beam is scanned across the sample to form a 2-D image

Annular Dark Field (ADF) detector

Electron Energy Loss Spectrometer

Elastic Scattering ~ "Z contrast"

Increasing energy loss


Reciprocity (or STEM vs. CTEM)

CTEM
- Gun
- Illumination angle
- Specimen
- Objective Aperture
- Viewing Screen

STEM
- Collectors
- Aperture
- Specimen
- Objective Aperture
- Gun

Reciprocity (for zero-loss images):
*A hollow-cone image in CTEM ↔ an annular-dark field image in STEM.*

However: In STEM, energy losses in the sample do not contribute to chromatic aberrations (Strong advantage for STEM in thick specimens)
Imaging Thick Samples at 200 kV

- Less blurring, more contrast in thick samples with STEM
- No Signal in W plugs, diffraction in poly
  unsuitable for tomography
Phase Contrast for Different Illumination Angles

(10.5 mr Objective, $E_0=200kV$, $Cs=1$ mm, Scherzer Defocus)

- For distances larger than 1 nm, there is little phase contrast to start with.
- When the illumination angles exceeds the objective aperture, all phase contrast is suppressed!
Amplitude Contrast for Different Illumination Angles

- For distances larger than 1 nm, there is little phase contrast to start with.
- When the illumination angles exceeds the objective aperture, all contrast reversals are removed and the resolution is increased!

(10.5 mr Objective, $E_0=200$kV, $Cs=1$ mm, Scherzer Defocus)
Why Increased Resolution? (coherent vs. incoherent imaging)

- Coherent imaging PSF is the probe wavefunction,
- Incoherent imaging PSF is the square of wavefunction

Eg: \( \phi(r) \approx \exp\left(-\frac{r^2}{\sigma^2}\right) \)

\[ |\phi(r)|^2 \approx \exp\left(-2\frac{r^2}{\sigma^2}\right) \Rightarrow \sigma' = \sigma/\sqrt{2} \]
Increasing the Collector Angle ($\theta_c$)

$\theta_c \ll \theta_{obj}$
- Phase contrast
- Diffraction contrast

$\theta_c \gg \theta_{obj}$
- No Phase contrast
- No Diffraction contrast

The incoherent BF image is the complement of the ADF image.
WiSi40 - 40Å HfO₂ on O₃ Underlayer + 850C/Spike/NO (6/12/2)

Bright-field STEM with small collector- like conventional TEM
Imaging Thick Samples at 200kV

ADF-STEM (θ_c>45 mr)

ADF-STEM (θ_c>75 mr)

- No more diffraction contrast
- Signal in W plug not monotonic, could be mistaken for voids
- Effect reduced by increasing the collector angle
Annular Dark Field STEM at 200 kV

- Interpretable (monotonic, single valued) signal in Silicon to ~1 um depth
- There is a geometric limit to the detector angles (~200-400 mr)

Electrons fall outside the detector

I/I₀ vs Thickness (um)

30-120 mr
50-200 mr
100-400 mr
Effect of Camera Length

Channeling enhances on-column Sb intensity.
Strain Contrast at Si/SiO$_2$ Interfaces
(JEOL 2010F, 200 kV, $C_s$=1mm)

ADF Inner angle:
50 mrad  25 mrad

Strain Fields cause dechanneling (and scattering to small angles)

Strain Contrast vs. Sample Thickness

Contrast at a c-Si/-aSi is strongly depends on sample thickness

100 kV, 45 mrad ADF inner angle

Strain Contrast effects at the interface:
for 130 Å thick sample, ~0%; for 340 Å thick sample, 15%.

Contrast from Random Strain Fields
(treated as a static Debye-Waller Factor)

No channeling  ➔ No thickness dependence

Contrast from Random Strain Fields
(using frozen phonons in multislice)

LAADF

HAADF

Channeling Thickness dependence

Contrast from Random Strain Fields

(using frozen phonons in multislice)

Channeling Thickness dependence
Contrast from Random Strain Fields
(treated as a static Debye-Waller Factor)

Imaging Light Atoms

Dechanneling contrast from the Strain Field around impurities

Polysilicon gate

B segregated to the interface?

4 nm Gate Oxide

Si substrate

Implanted B

Single atom contrast is expected at 77K (Hillyard and Silcox)
**Imaging Vacancies?**

(grow 25 layers of SrTiO$_3$-$\delta$ on SrTiO$_3$)


Detection sensitivity: 1-4 Oxygen vacancies
Ronchigrams

- Most accurate manual method of alignment
- Easy to find the optic axis
- Easy to correct serious astigmatism
- Easy to bring the sample into focus
- Works best on an amorphous layer
- Start with the largest aperture

Ronchigrams – no \( C_s \)

(beam is at cross-over before sample)

Sample is magnified, erect
**Ronchigrams – no $C_s$**

(beam is at cross-over after sample)

Sample is magnified, inverted
Ronchigrams – no \( C_s \)

(beam is at almost cross-over on the sample)

Almost infinite magnification
Ronchigrams with Spherical Aberration, $C_s$

For $C_s > 0$, rays far from the axis are bent too strongly and come to a crossover before the gaussian image plane.

Almost infinite magnification, Only at small angles
Spherical Aberration

Apparent deflection at the object is proportional to the cube of the distance off-axis within the imaging lens. Deflection towards axis is always too strong.

Phil Batson, IBM
Shadow Map: “Ronchigram”

Shadow Map: “Ronchigram”


Phil Batson, IBM
Shadow Map: “Ronchigram”

Source

Objective

Specimen

Screen

20 nm


Phil Batson, IBM
Shadow Map: “Ronchigram”


Phil Batson, IBM
Shadow Map: “Ronchigram”

Source
Objective Specimen Screen


Phil Batson, IBM
The Electron Ronchigram

\[ M = \frac{v}{u} \]


Nigel Browning, UC Davis
Effect of $C_s$ on Ronchigram

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

Nigel Browning, UC Davis
Ronchigrams from Si <110>

Nigel Browning, UC Davis
Correcting for Astigmatism

Two fold astigmatism

Three fold astigmatism

Nigel Browning, UC Davis
Forming the Smallest Probe

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

Nigel Browning, UC Davis
Ronchigram focus series on a-C

This aperture is too big
Correcting Severe Astigmatism in Ronchigrams

(don’t image probe)
Ronchigrams on Crystals

Tilted

On Axis (not in focus)
Ronchigrams on Crystals

under

In focus

over

More over

way over
Measuring the Aperture Size
(Using [110] Silicon as a reference)

Scan the beam over a small area to remove ronchigram structure

Distance AB
= 4 x Bragg Angle
Figure 10. Measurement of STEM convergence angles in an FEI F20 SuperTWIN. The diffraction pattern is “calibrated” on the 200 reflection of Si, oriented onto the 110 axis. The convergence semi-angle (α) is proportional to the ratio of the disc width to the disc spacing (a/b). As b is independent of the chosen aperture the other three apertures can be calibrated by just recording the width of the zero order disk (α). The dotted line inside the 50μm aperture represents the relative scale of the 50 μm aperture.
**Reality Check**

(Can I see a lattice spacing)

Disks must overlap to form a lattice fringe

(more overlap, more contrast)*

No lattice fringes this direction
Balancing Spherical Aberration against the Diffraction Limit

(Less diffraction with a large aperture – must be balanced against $C_s$)

A more accurate wave-optical treatment, allowing less than $\lambda/4$ of phase shift across the lens gives

Minimum Spot size:  
$$d_{\text{min}} = 0.43 C_s^{1/4} \lambda^{3/4}$$

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

At 200 kV, $\lambda=0.0257$ Å,

- $C_s = 1.0$ mm, $d_{\text{min}} = 1.55\text{Å}$ and $\alpha_{\text{opt}} = 10$ mrad
- $C_s = 1.2$ mm, $d_{\text{min}} = 1.59\text{Å}$ and $\alpha_{\text{opt}} = 9.6$ mrad
- $C_s = 0.5$ mm, $d_{\text{min}} = 1.28\text{Å}$ and $\alpha_{\text{opt}} = 12$ mrad
- $C_s = 0.6$ mm, $d_{\text{min}} = 1.34\text{Å}$ and $\alpha_{\text{opt}} = 11$ mrad
Multislice simulated Annular-Dark-Field Images of Silicon [110] in a 200 kV STEM

(Cs=0.5 mm, Probe forming aperture=11.9 mr, ADF inner angle=30 mr)

(a) The projected potential along [110]
(b) The ADF image for a 2.7 Å thick crystal
(c) The ADF image for a 81 Å thick crystal
(d) The ADF image for a 2.7 Å thick crystal (1.6 Å information limit)

Note: (i) The dumbbells are visible even when the (400) spot is excluded
(ii) Except the dumbbell spacing is not 1.36Å, but closer to 1.6Å
Figure 3.17: Simulated strength of 2.7 Å fringe (normalized with respect to overall linescan intensity) in 190 Å of Si (110) versus nominal probe size. For large probe sizes the fringe is beyond the resolution and is not visible, for small probes the is no 'crosstalk' between the atomic columns located 1.36 Å apart and the 'forbidden' fringe is again not visible.
Beam Spreading

Electron Range (in $\mu$m):

$$R \approx \frac{0.064}{\rho} E_0^{1.5}$$

(density $\rho$ in g/cm$^3$, $E_0$ in keV)

$R \approx 100 \mu$m at 200 keV
Beam Spreading

At 300 kV

Beam Spreading $\propto \frac{(Zt)^{1.5}}{E_0}$

At 100 kV
0.16 nm for 10 nm thick C
1.8 nm for 50 nm thick C

How does an amorphous layer on the entrance surface degrade resolution?

- Increased Apparent Source size
- Loss of resolution, loss of apparent brightness

- No FIB’ed samples!
- Ignore for probes larger than 0.4 Å
**Phase vs. ADF Contrast**

ADF Signal is much weaker than HR-TEM

Imaging a Single Antimony Atom in 4.5 nm of Silicon
(the atom is 2.1 nm from the top surface)

Multislice simulations assume a 200 kV electron beam

Sb contrast:  HRTEM 0%, EWR: 10% (5% at 1.2Å) , ADF-STEM:65%

Why do we get more signal in ADF? Channeling!
**ADF in Thicker Samples**

- *Simple specimen transmission function model:*
  \[ I_{ADF} \sim |h|^2 \otimes |t|^2 \]

- Suggests that wave amplitude is important, not phase as in conventional HRTEM
- Interaction of the fast electrons with the periodic lattice including phonons is difficult
- Numerical simulations

David Muller 2006
Probe Channeling

z=0 nm

z=10 nm

z=23.5 nm

z=33.0 nm
ADF Signal tracks Probe Amplitude

Si [110] at 300K
(200 kV, C_s=1 mm)

\[ I_{ADF} \sim \int_0^{z_0} |h(\vec{r}, z)|^2 \otimes r |t(\vec{r}, z)|^2 \, dz \]
Channeling down

\[ \frac{dl}{dz} \ (1 / \text{Å}) \]

- [110] Si multislice
- fit to Bloch wave
- fit uncertainty band
- amorphous Si

\[ 1.6 \times 10^{-4} \]

\[ z \ (\text{Å}) \]

0 200 400 600 800
Signal vs Collection Angle

[011]Si, 300 kV, 9 mr probe angle

\[ \frac{\theta_c}{\theta_0} = 4.5 \]

\[ \frac{\theta_c}{\theta_0} = 2.4 \]

\[ \frac{\theta_c}{\theta_0} = 1.3 \]

\[ \frac{dI}{dt} \text{ (% of incident beam)} \]

\[ \times 0.14 \]

\[ \times 0.25 \]

Isolated Atom signal
Imaging Thick Cross-Sections

- ADF Images decay gracefully with increasing thickness
- Apparent Oxide Thickness is unchanged with thickness
- Apparent Interface Roughness increases from 1.6 to 2.7 Å rms
- “white band” develops (depends on thickness and ADF angles)

Gate Oxide Thickness: 20 Å
Scattering from one Sb atom $\propto$ Si scattering at the same depth.
Dopants as probes of Beam Spreading

(Multislice for 75.8 nm Si in 100 kV $C_s$-corrected STEM)

Channeling Down Si [110]

- Probe doesn’t stay between atom columns - oscillates
- Almost entirely on atom columns at 100, 400 Å
- When on-column, scattering is large
  Will reduce dumbbell contrast
Si Dumbell Contrast vs. Probe Angle

Contrast vs. Depth (Å) for different probe angles:
- 10 mr
- 20 mr
- 50 mr
- 100 mr
On-column vs Off

- On-column Sb
- Off-column Sb

Sb depth (Å)

$\frac{I_{Sb}}{I_{Si}}$
Effect of Camera Length

Clipping an Image is Bad
(and easy to do)

Black level set too high

Equivalent to multiplying by square wave
Fourier Transform of a Square Wave

\[ f(t) = \frac{A\tau}{T} \sum_{n=1,3,5} \frac{2A\tau \sin(n\pi\tau/T)}{n\pi\tau/T} \cos(2\pi nf_0 t) \]

\( \text{sinc}(\pi n\tau/T) \)

\( f = 1/n\tau \)
Fourier Transform of a Square Wave

\[ FT \]

\[ \tau \]

\[ d \]

\[ x \]

\[ \frac{1}{d} \quad \frac{3}{d} \quad \frac{5}{d} \]

\[ k \]

\[ f = \frac{1}{n \tau} \]

sinc(\(\pi n \tau / T\))

multiplication

Clipping adds extra spots

To diffractogram
Original image

clipped image

Original histogram

clipped histogram

Power Spectrum
Cut-off at 0.3 nm
Summary

- BF STEM – fake TEM
- LAADF STEM – strain contrast, single vacancy
- HAADF – depth dependent imaging of single dopants

- Check histograms to avoid clipping (extra spots)
- Ronchigrams – easier than imaging probe for align
Comparison of Brightness Measurements For Cold and Thermal Field Emitters

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Current State of the Art:

• Few good measurements of Brightness.

• Need to measure or extract the source size (easy to overestimate)

• No reliable studies of Brightness vs. Field, Temperature or monochromation
Sample Tilt in ADF-STEM

(200kV, 10 mrad)

200Å of [001] SrTiO₃

300Å of [011] Si

Relative Intensity

Tilt angle (degree)

Up to~ 5 mrad of mistilt is OK before fringe contrast is reduced

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