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

Operational principle

Spectrometer

Annular Detector

Z-contrast Image

Incident Probe

CCD-EELS Detector

Counts

Energy (eV)

0 1000 2000 3000 4000

b1 b2 b3 a1 a2 d1 d2 d3 c

Bulk Boundary

1 nm

 Courtesy Nigel Browning
Probe channeling

David Muller 2004
In certain instances, TEM & STEM images are strictly equivalent

“Theorem of Reciprocity”

<table>
<thead>
<tr>
<th>TEM</th>
<th>STEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coherent illumination</td>
<td>Small Condenser Aperture</td>
</tr>
<tr>
<td>Incoherent illumination</td>
<td>Hollow cone illumination</td>
</tr>
</tbody>
</table>

Incoherent illumination

HAADF

HAADF

FEG

Courtesy Nigel Browning & Dave Muller
Nomenclature

*depends on machine type*

<table>
<thead>
<tr>
<th>TEM/STEM</th>
<th>Gun</th>
<th>Dedicated STEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>N/A</td>
<td></td>
<td>Virtual Objective Aperture</td>
</tr>
<tr>
<td>N/A</td>
<td>X</td>
<td>Condenser Stigmator</td>
</tr>
<tr>
<td>Condenser Aperture</td>
<td></td>
<td>Objective Aperture</td>
</tr>
<tr>
<td>Condenser Stigmator</td>
<td>X</td>
<td>Objective Stigmator</td>
</tr>
<tr>
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<td></td>
<td>N/A</td>
</tr>
<tr>
<td>Objective Stigmator</td>
<td>X</td>
<td>N/A</td>
</tr>
</tbody>
</table>

Courtesy Nigel Browning
# Electron sources

## A small bright source is necessary for STEM

<table>
<thead>
<tr>
<th></th>
<th>Thermal emission</th>
<th>Field emission</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Electron sources</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thermal FE W (100)</td>
<td>~5x10⁸</td>
<td>~5x10⁸</td>
</tr>
<tr>
<td>Cold FE W (310)</td>
<td>~5x10⁸</td>
<td>~5x10⁸</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Brightness (A/cm²/sr) at 200kV</strong></th>
<th>Thermal FE W (100)</th>
<th>Cold FE W (310)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>~5x10⁵</td>
<td>~5x10⁸</td>
</tr>
<tr>
<td>LaB₆</td>
<td>~5x10⁶</td>
<td>~5x10⁸</td>
</tr>
<tr>
<td>Shotky ZrO/W</td>
<td>~5x10⁸</td>
<td>~5x10⁸</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Electron Source Size</strong></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50µm</td>
<td>10-100nm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Energy Width (ev)</strong></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>2.3</td>
<td>0.6-0.8</td>
</tr>
<tr>
<td>LaB₆</td>
<td>1.5</td>
<td>0.6-0.8</td>
</tr>
<tr>
<td>Shotky ZrO/W</td>
<td>0.6-0.8</td>
<td>0.3-0.5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Operating Conditions</strong></th>
<th></th>
<th></th>
</tr>
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<tbody>
<tr>
<td>Vacuum (Pa)</td>
<td>10⁻³</td>
<td>10⁻⁵</td>
</tr>
<tr>
<td></td>
<td>10⁻⁷</td>
<td>10⁻⁷</td>
</tr>
<tr>
<td></td>
<td>10⁻⁸</td>
<td>10⁻⁹</td>
</tr>
<tr>
<td>Temperature (K)</td>
<td>2800</td>
<td>1800</td>
</tr>
<tr>
<td></td>
<td>1800</td>
<td>1600</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Emission</strong></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Current (µA)</td>
<td>~100</td>
<td>20-100</td>
</tr>
<tr>
<td>Short term stability</td>
<td>1%</td>
<td>7%</td>
</tr>
<tr>
<td>Long term stability</td>
<td>1%/hr</td>
<td>6%/hr</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Maintenance</strong></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Not necessary</td>
<td>Start-up takes time</td>
<td>Build up necessary after change</td>
</tr>
<tr>
<td>Build up</td>
<td></td>
<td>Flash every few hours</td>
</tr>
<tr>
<td>replaces</td>
<td></td>
<td></td>
</tr>
<tr>
<td>only</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Price &amp; Operation</strong></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Low &amp; simple</td>
<td>High &amp; easy</td>
<td>High &amp; complicated?</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Lifetime</strong></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>3 months</td>
<td>1 year</td>
<td>&gt;4 years</td>
</tr>
<tr>
<td></td>
<td></td>
<td>?</td>
</tr>
</tbody>
</table>

Courtesy Nigel Browning
Demagnification

\[ M = \frac{v}{u} \approx \frac{\beta}{\alpha} \]

Must demagnify source to form small probe

Stronger lens current gives more demagnification
Probe forming optics

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

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)

Images from S.J. Pennycook
In STEM, energy losses in sample do not contribute to chromatic aberration (averaged over collector ...)
Finding increasing use in semiconductor quality control
“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

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

\[ \frac{1}{3} [1\bar{1}3] \rightarrow \frac{1}{3} [1\bar{1}20] + [0001] \]

Arslan, Bleloch, Stach and Browning, PRL 2005
This is an 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

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

or

\[ \frac{1}{3}[1\bar{1}23] \rightarrow \frac{1}{3}[1\bar{1}20] + [0001] \]

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

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

Ronchigrams from Si <110>

overfocused

Δf=0 nm

Δf=150 nm

Δf=100 nm

Δf=50 nm

Scherzer focus

Δf=-100 nm

Δf=-150 nm

Δf=-200 nm

Δf=-300 nm

Δf=-350 nm

Δf=-200 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

Incident Probe

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

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

$$d_s = \frac{1}{2} C_s \alpha_0^3$$

$$d_0 = \frac{0.61 \lambda}{\alpha_0}$$

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

Courtesy Dave Muller
Optimum resolution

Using wave optics, find:

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

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

At 200kV, \( \lambda = 0.0257\text{Å} \)

\[ \text{Cs} = 1.0 \text{ mm, } d_{\text{min}} = 1.55\text{Å and } \alpha_{\text{opt}} = 10 \text{ mrad} \]
\[ \text{Cs} = 1.2 \text{ mm, } d_{\text{min}} = 1.59\text{Å and } \alpha_{\text{opt}} = 9.6 \text{ mrad} \]
\[ \text{Cs} = 0.5 \text{ mm, } d_{\text{min}} = 1.28\text{Å and } \alpha_{\text{opt}} = 12 \text{ mrad} \]
\[ \text{Cs} = 0.6 \text{ mm, } d_{\text{min}} = 1.34\text{Å and } \alpha_{\text{opt}} = 11 \text{ mrad} \]
Effect of $C_s$ Correction

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

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.
Stability issues for STEM
(and for TEM!)

- Stray Fields/Ground loops
- Air Flow/Temperature Control
- Pressure Variations
- Mechanical Vibrations
- Specimen Preparation

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.
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

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.
Image artifacts
‘clipping’

Unclipped

40% clipped

60% clipped

From S.J. Pennycook
Effect of $C_s$ correction

Direct resolution at 0.78 Å

Information transfer to 0.607 Å

Si $<112>$

Image by Matt Chisholm,
Processing by Albina Borisevich and Andy Lupini
Aberration correction by Pete Nellist et al., Nion Co.