Optimizing Automated Particle Analysis for Forensic Applications

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The Big Picture

- X-ray Microanalysis
  - Electron Probe Microanalysis (EPMA)
  - Energy Dispersive X-ray Microanalysis (XEDS)
  - Microanalysis of challenging samples
    - Particles, fibers, films, inclusions, ...
  - Microanalysis of particle data sets

- Customers
  - Material science, forensics, manufacturing,
Our Tools

- **Instruments**
  - 2 electron microprobes, 2 FIBS, 2 FEG SEM, 1 W-filament SEM

- **Software**
  - NIST DTSA-II – Simulation & quantification
  - NIST Graf – Quantification, review & data mining of particle data sets

- **Other techniques**
  - STEM, XRF, EBSD, Atom Probe, Confocal, XRD, Auger
High Speed Automated Analysis of Particles using SEM/EDS

• High Speed – 10,000+ particle data sets
  – Moderate quality analyses of many particles
  – Search for a needle-in-a-haystack

• Automated – Configure, start then no operator intervention
  – Minimize operator bias
  – Reduce tedium

• Analysis -
  – Images and quantitative elemental analysis
Conventional Si(Li) Detector – 6.4 µs process time
Major Time Sinks

- Stage motion – Tiling, stage speed
- Searching – Search pixel size, pixel dwell
- Measuring – Accuracy & pixel dwell
- Compositional Analysis – Limits-of-detection
- Mapping – Pixel dwell, area
- Overhead
  - QC
Strategies for Optimizing Stage Movement

• Speed up the stage
  – Particularly backlash removal jogs, post-move vibration

• Minimize stage movement
  – Move in serpentine
  – Subsets
    • Fixed size - Order frames to produce shortest path
    • Unknown size – Can’t optimize path
  – Electronic fields – Move beam not stage
Optimizing the Backscatter Detector

- Consider a probe current of 1 nA and a dwell of 1 µs
  \( (1 \text{ nA})(1 \text{ µs}) = (6.241 \times 10^{18} \text{ e}^-/\text{s})(10^{-9})(10^{-6} \text{ s}) = 6,200 \text{ e}^- \)

- Typical backscatter coefficients range from 5% to 50%

- If we could collect every electron from
  \( \Delta I/I = (3,100)^{1/2}/(3,100) = 1.8\% \)

- We actually collect about 14%
  \( \Delta I/I = (430)^{1/2}/(430) = 4.8\% \)
  \( \Delta I/I = (43)^{1/2}/(43) = 15\% \)
Area of a spherical cap

\[ A = 2\pi Rh \]

\[ \Omega = \frac{2\pi Rh}{R^2} \]

Sample

\[ \Omega_{BSED} = 1.46 \text{ sr} \]
Optimizing EDS

- Maximize solid angle
  - Large area
  - In close
- Many angles better than one
  - Multiple detectors
- Many pulse processors better than one
  - Multiple pulse processors
Secondary detector

X-ray Detectors

Backscatter detector
Take-off angle: 35°
Sample-to-detector: 34 mm
Optimal WD: 17 mm
Detector area: $4 \times 30 \, \text{mm}^2$
Collection efficiency: 0.66%
OCR: 200 cps per nA on Cu

TESCAN MIRA3 with 4 PulseTor 30 mm$^2$ SDD
OXYGEN IN IRON OXIDE PARTICLES

Sample: Raw iron oxide particles from Calvert Cliffs, MD
An SEM is not a Camera – Part 1

An SEM:
- Collects images pixel-by-pixel, row-by-row
- Can stop the raster anywhere
- Can change directions

- We can size a particle quickly regardless of whether it is large or small.
- The “coord-raster” can be used to keep the beam on the particle while collecting EDS.
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An SEM can:
- Dynamically change pixel spacing
- Search on a large pixel spacing
- Measure on a fine pixel spacing

Search
Measure
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An SEM is not a Camera – Part 3

An SEM can:
- Raster the beam outside the nominal field-of-view
- Naturally handle particles that fall on a field edge.
Does it matter?

<table>
<thead>
<tr>
<th>Dimensions</th>
<th>Timing</th>
<th>Precision</th>
<th>Overhead per particle</th>
<th>Time for 100 particle field</th>
</tr>
</thead>
<tbody>
<tr>
<td>Search Pixels</td>
<td>Measure Pixels</td>
<td>Search (seconds)</td>
<td>1 part in 2048</td>
<td>0</td>
</tr>
<tr>
<td>Naive</td>
<td>2048</td>
<td>2048</td>
<td>4.194</td>
<td>0</td>
</tr>
<tr>
<td>Optimized</td>
<td>256</td>
<td>2048</td>
<td>0.066</td>
<td>~0.025</td>
</tr>
</tbody>
</table>

*Optimized*  
Particles are sized and a spectrum collected as soon as discovered

*Naive*  
Particles are sized and spectrum collected at the end-of-frame

Fewer small particles are lost during analysis using the optimized algorithm.
Does it work?

OLD

• 1,000 particle / hour
• Search: 99.4 mm² in 42 minutes at 1 µm pixel spacing
• Size: 10 particles / s
• Quantify: 0.3 particles / s

NEW

• 7,500 particles / hour
• Search: 100 mm² in 13 minutes at 1 µm pixel spacing
• Size: 18 particles / s
• Quantify: 2.5 particles / s
Tying it all together

NIST's contribution

DTSA-II

SEMANTICS

EM Automation
Library in Java

SharkSEM API

PostgreSQL • The world's most advanced open source database.
A Final Word on QC

1) EDS

2) Imaging detectors

3) Magnification

4) Probe current