Improved Quantitative Analysis of Particles with Topography using Multiple EDS Detectors

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Outline

• Geometry – opposing and pointing at the same WD
• Mapping with 2 detectors and recent developments
• Quantification of surfaces with topography*
  • Garnets
  • Glass fractures
  • Spinels

*Primary purpose is for developing an improved strategy for automated particle analysis.
Improved Quant with Multiple Detectors

Why 2 detectors?

1. Reduce or eliminate the effects of topography
2. Reduce shadowing effects
3. Improve count rate efficiency
Improved Quant with Multiple Detectors

Hitachi S3400N with 2 SDDs
Improved Quant with Multiple Detectors

JEOL JSM 7000F with 2 SDDs – interior view from below
Improved Quant with Multiple Detectors

Interior view of 2 SDDs with the sample. Note the white electron beam producing red and green x-ray beams.
Mapping with Multiple Silicon Drift Detectors

• The Problem:

• A topographically complex sample like this fracture surface of an aluminum alloy will have a chemical and a topographic variability. How do we sort this out?

BSE (UL), Al (UR), Cu L (LL), ROI of all energies (LR).

ROI maps as derived images in lieu of BSE?
Mapping with Multiple Silicon Drift Detectors

Approaches to mapping a sample with a complex topography:

• Poor man’s approach – use 1 detector to collect one set of maps, and then “simply” rotate the stage 180 degrees, do a 180 degree scan rotate and collect a second set of maps of the same area which can be added or merged.

• Poor and saner man’s approach – collect one map and follow some of the procedures that will be outlined in the following slides.

• An easier approach – use 2 well-balanced detectors with different azimuth angles (90 – 180 degrees) and collect two sets of maps which can be added or merged.

Why? To reduce the complexity of the maps and make their chemistry easier to interpret.
Mapping with Multiple Silicon Drift Detectors

Sandstone fracture sample shown in SE (UL), Si Map (UR detector at the upper right), Si Map (LL detector at lower left) and the sum of the two maps (LR).

Question: what does the mid-tone area represent in the sum image?
Mapping with Multiple Silicon Drift Detectors

Stereo image of the fracture surface that has been mapped and which is shown in the following slide. The upper pair of images is to be viewed with parallel vision and the lower pair of images should be viewed cross-eyed (scan rotate of -90 degrees).
Mapping with Multiple Silicon Drift Detectors

Maps of a fracture surface. The BSE image (a) is in the upper left (image width is 1.5 mm). A region of interest was created to represent the entire spectrum. Figure d is a sum of both detectors. The summed image has 3 primary gray levels: black, which indicates an area which did not provide an x-ray signal to either detector; gray, which shows an area that provided an x-ray signal to one of the 2 detectors; and white, which represents an area that provided a signal to both detectors.

KEY IDEA: DO NOT SUM THE DETECTORS, TAKE THE MAX!
Mapping with Multiple Silicon Drift Detectors

Taking the Sum ROI image (inverted and binarized) and overlaying it with MAX maps produces the results above. White areas can not be seen with 1 detector.
Mapping with Multiple Silicon Drift Detectors

Taking the Sum ROI image (inverted and binarized) and overlaying it with MAX maps produces the results above. White areas cannot be seen with 2 detectors.
Overlaying 5 MAX maps without taking into account the Sum ROI produces the results above. Black areas are locations that can not be seen with either of the 2 detectors (or might represent a missed element).
Mapping with Multiple Silicon Drift Detectors

Taking the Sum ROI image (inverted and binarized) and overlaying it with 5 MAX maps produces the results above. White areas can not be seen with either detector. Glaciers versus lakes...
Mapping with Multiple Silicon Drift Detectors

Taking the Sum ROI image (inverted and binarized) and overlaying it with 5 MAX maps produces the results above. Cyan areas can not be seen by either detector. Still have topographic effects. To get rid of the topography, normalize the data…
Mapping with Multiple Silicon Drift Detectors

Taking the Sum ROI image (inverted and binarized) and overlaying it with ZAF Quant MAX maps produces the results above. Cyan areas can not be seen by either detector. Artifacts?
• It is possible to increase the area of illumination of a rough sample, or the area that can be seen by an EDS detector by using multiple detectors.

• Overlaying the Sum ROI image (inverted and binarized) helps to illustrate where x rays are not possible.

• Data cubes should not be summed but the max signal selected.

• Data normalization (quantification) can eliminate the pseudo contrast that comes from illumination effects.
Garnet Formula:

(Mg, Ca, Mn, Fe)$_3$Al$_2$Si$_3$O$_{12}$

Atomic %: Divalent 15%, Al 10%, Si 15%, O 60%.

Endmembers:

Pyrope $\text{Mg}_3\text{Al}_2\text{Si}_3\text{O}_{12}$

Almandine $\text{Fe}_3\text{Al}_2\text{Si}_3\text{O}_{12}$

Spessartine $\text{Mn}_3\text{Al}_2\text{Si}_3\text{O}_{12}$

Andradite $\text{Ca}_3\text{Fe}_2\text{Si}_3\text{O}_{12}$

Grossular $\text{Ca}_3\text{Al}_2\text{Si}_3\text{O}_{12}$

Uvarovite $\text{Ca}_3\text{Cr}_2\text{Si}_3\text{O}_{12}$
Improved Quant with Multiple Detectors

Point 1 Upper

Lower

Sum

Max Ch.
Improved Quant with Multiple Detectors

Point 2 Upper

Sum

Max Ch.
Improved Quant with Multiple Detectors

Point 3 Upper

Lower

Sum

Max Ch.
Improved Quant with Multiple Detectors

**Point 4 Upper**

**Point 4 Lower**

**Sum**

**Max Ch.**
Improved Quant with Multiple Detectors

Garnet Spectra --1 Detector

Si Wt% vs Al Wt% graph with marker symbols indicating data points. At% not Wt%
Improved Quant with Multiple Detectors

Summed and Max Channel Spectra

Si Wt% vs Al Wt%

At% not Wt%
Improved Quant with Multiple Detectors

HFW = 2 mm
Improved Quant with Multiple Detectors

Stereo Pairs

Straight-eyed

Cross-eyed
Improved Quant with Multiple Detectors

Glass Spectra Smooth Surface
Improved Quant with Multiple Detectors

Spot Mode Very Bad!

Note the volatility of the Na.

15 kV, 2-3 nA.
Improved Quant with Multiple Detectors

Selected Area
Not So Bad!

Note the lack of volatility of the Na under the same conditions.

--Albite
Improved Quant with Multiple Detectors

All x rays in the region of interest (ROI) between 0 and 10.24 keV.
Improved Quant with Multiple Detectors

Maps of the Si K ROI.
Improved Quant with Multiple Detectors

Maps of the O K ROI.
Improved Quant with Multiple Detectors

Maps of the Ca K ROI.
Improved Quant with Multiple Detectors

Overlay of the C and Si (ROI) maps.
Improved Quant with Multiple Detectors

Good Sum Spectra
1, 5, 6, 7, 9, 10, 11

Also, note the rotation of the image from the maps. The detectors are now above and below.
Improved Quant with Multiple Detectors

G = good
sum spectrum
Improved Quant with Multiple Detectors
# Improved Quant with Multiple Detectors

![Image of a material sample with annotations and a table showing elemental composition](image)

<table>
<thead>
<tr>
<th></th>
<th>NaK</th>
<th>MgK</th>
<th>AlK</th>
<th>SiK</th>
<th>K K</th>
<th>CaK</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flat/Ideal</td>
<td>8.7</td>
<td>2.4</td>
<td>0.9</td>
<td>35.4</td>
<td>0.9</td>
<td>4.1</td>
</tr>
<tr>
<td>Upper</td>
<td>10.6</td>
<td>2.7</td>
<td>1.1</td>
<td>34.2</td>
<td>0.9</td>
<td>3.6</td>
</tr>
<tr>
<td>Lower</td>
<td>5.4</td>
<td>1.7</td>
<td>0.7</td>
<td>35.2</td>
<td>1.6</td>
<td>8.2</td>
</tr>
<tr>
<td>Sum</td>
<td>9.2</td>
<td>2.4</td>
<td>0.9</td>
<td>34.6</td>
<td>1.0</td>
<td>4.8</td>
</tr>
</tbody>
</table>

**Note:**
- The table shows the weight percentage of various elements in different regions of the sample.
- The sample image is labeled SE1.
# Improved Quant with Multiple Detectors

## Weight % of Glass Analyses

<table>
<thead>
<tr>
<th></th>
<th>NaK</th>
<th>MgK</th>
<th>AlK</th>
<th>SiK</th>
<th>K K</th>
<th>CaK</th>
<th>O K*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ideal Flat</td>
<td>8.6</td>
<td>2.4</td>
<td>0.8</td>
<td>35.6</td>
<td>0.9</td>
<td>4.1</td>
<td>47.7</td>
</tr>
<tr>
<td>Single Analysis</td>
<td>8.9</td>
<td>2.4</td>
<td>1.4</td>
<td>34.5</td>
<td>1.0</td>
<td>4.5</td>
<td>47.2</td>
</tr>
<tr>
<td></td>
<td>1.7</td>
<td>0.5</td>
<td>1.7</td>
<td>1.5</td>
<td>0.3</td>
<td>1.2</td>
<td>0.4</td>
</tr>
<tr>
<td>Sum</td>
<td>9.2</td>
<td>2.4</td>
<td>1.1</td>
<td>34.9</td>
<td>0.9</td>
<td>4.2</td>
<td>47.4</td>
</tr>
<tr>
<td></td>
<td>0.7</td>
<td>0.2</td>
<td>0.7</td>
<td>0.6</td>
<td>0.1</td>
<td>0.4</td>
<td>0.2</td>
</tr>
<tr>
<td>Max</td>
<td>9.6</td>
<td>2.5</td>
<td>1.1</td>
<td>34.7</td>
<td>0.9</td>
<td>4.0</td>
<td>47.2</td>
</tr>
<tr>
<td></td>
<td>0.8</td>
<td>0.2</td>
<td>0.8</td>
<td>0.7</td>
<td>0.1</td>
<td>0.4</td>
<td>0.2</td>
</tr>
</tbody>
</table>

*Oxygen determined by stoichiometry

Mean  
Std. Dev.
Improved Quant with Multiple Detectors

Upper and Lower

Ideal analysis shown as a magenta circle
Improved Quant with Multiple Detectors

Ideal analysis shown as a magenta circle.
Improved Quant with Multiple Detectors

Max Channel

Ideal analysis shown as a magenta circle
Spectra collected from, or derived from point 1. The count rates were 10 kcps vs 15 kcps but the spectra are very similar.
Spectra collected from, or derived from point 2. The count rates were 1 kcps vs 14 kcps and the spectra are very dissimilar.
Spectra collected from, or derived from point 3. The count rates were 5 kcps vs 13 kcps and the spectra are very dissimilar.
Spectra collected from, or derived from point 4. The count rates were 4 kcps vs 13 kcps. The spectra are dissimilar but produced a good sum spectrum.
Improved Quant with Multiple Detectors

Spectra collected from or derived from point 7. The count rates were 10 kcps vs 15 kcps but the spectra are very similar.
Where do we stand on the usefulness of multiple detectors for improving our quant?

• This looks very promising.

• Some common sense precautions are helpful like don’t go near the edges of particles and analyze the centers.

• So far, a simple summing of the two spectra appears to be the best strategy.

• Might use a secondary threshold to analyze the brightest part of a grain.

• More data are needed (garnet sand paper, ball mill the glass sample to get smaller grains).