Improved Quantitative Analysis of Particles with Topography using Multiple EDS Detectors

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AMETEK

Improved Quant with Multiple Detectors

Outline

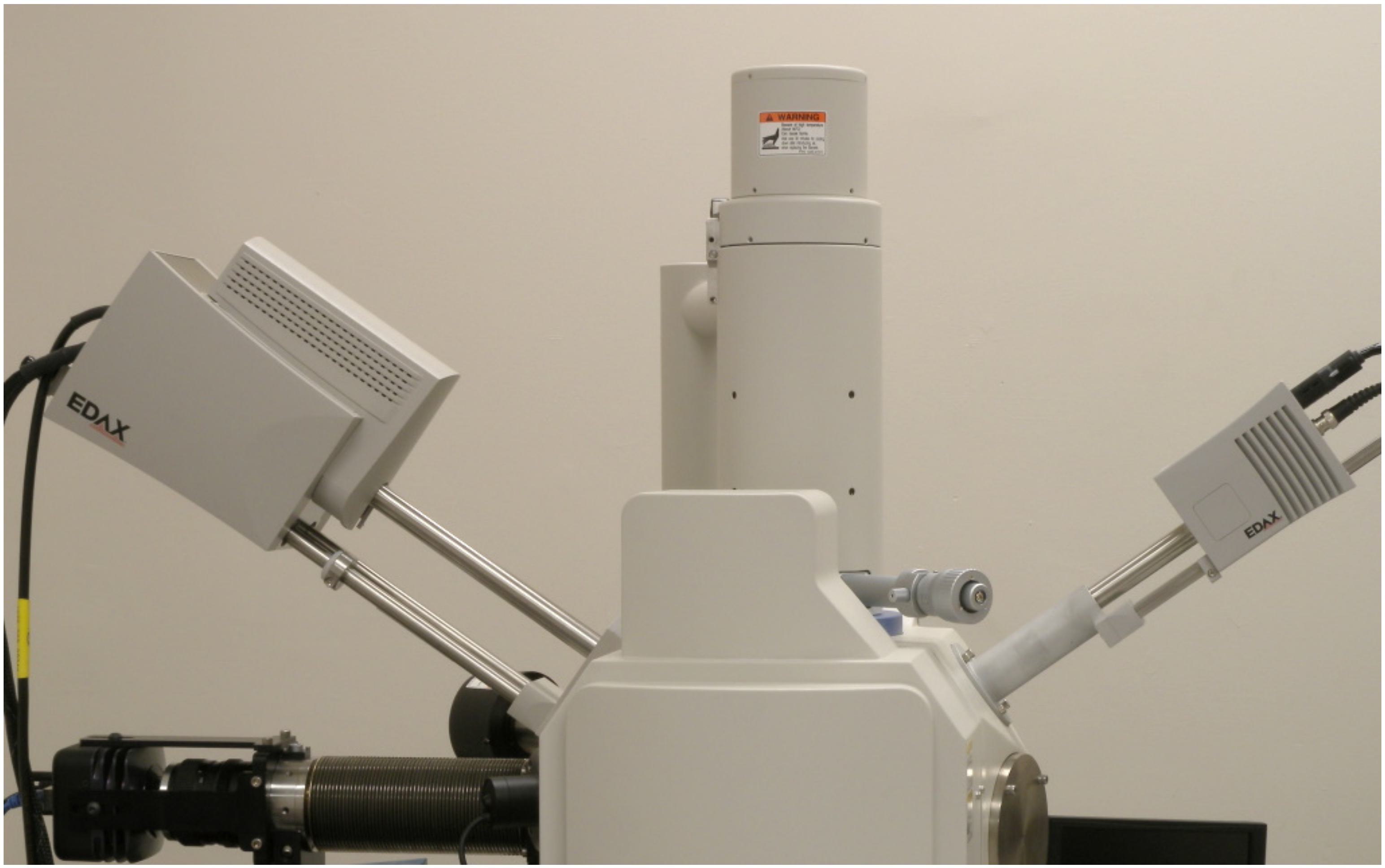
- Geometry –opposing and pointing at the same WD
- Mapping with 2 detectors and recent developments
- Quantification of surfaces with topography*
    - Garnets
    - Glass fractures
    - Spinels

*Primary purpose is for developing an improved strategy for automated particle analysis.

Improved Quant with Multiple Detectors

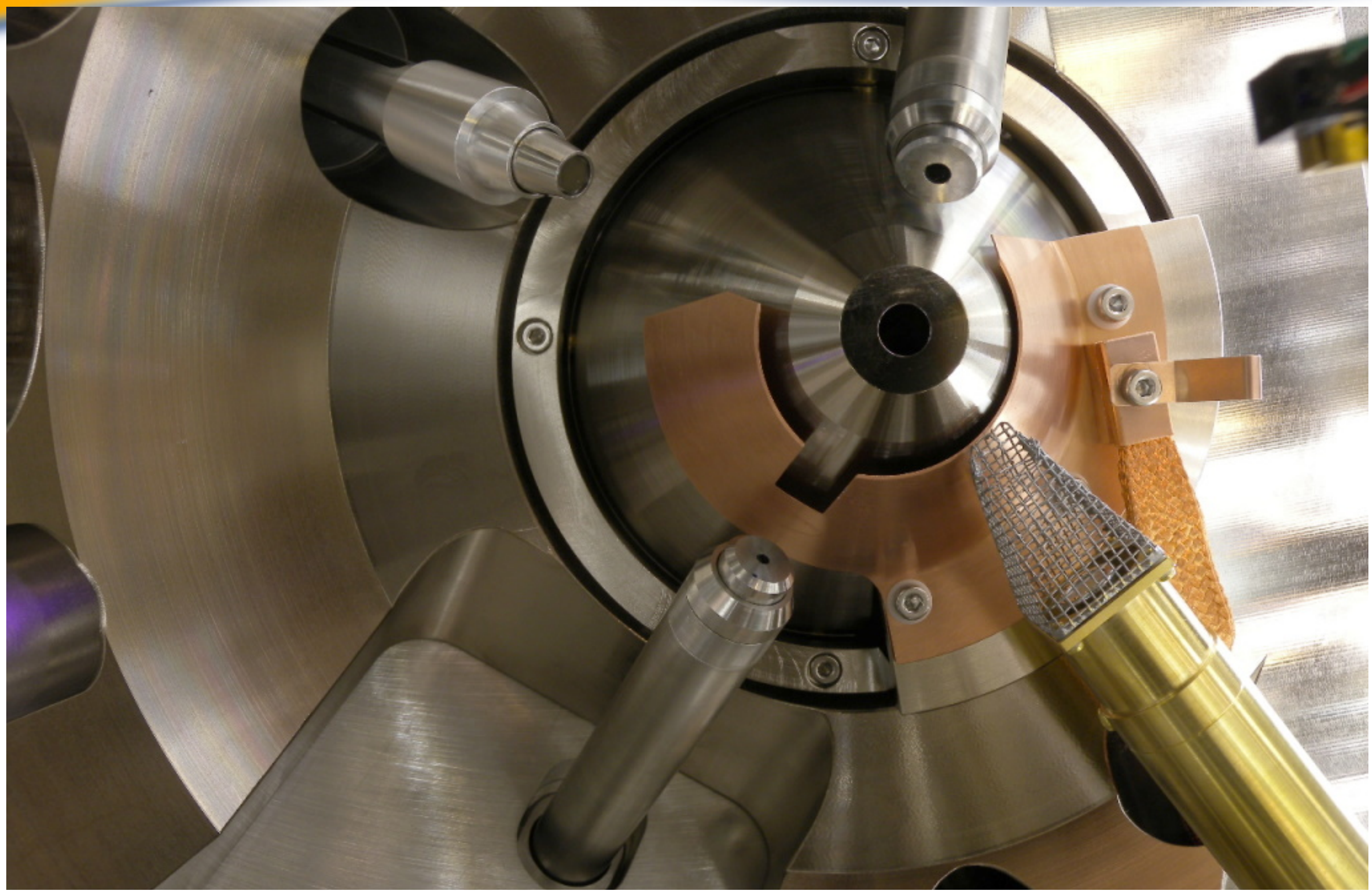
1. Reduce or eliminate the effects of topography
2. Reduce shadowing effects
3. Improve count rate efficiency

Improved Quant with Multiple Detectors



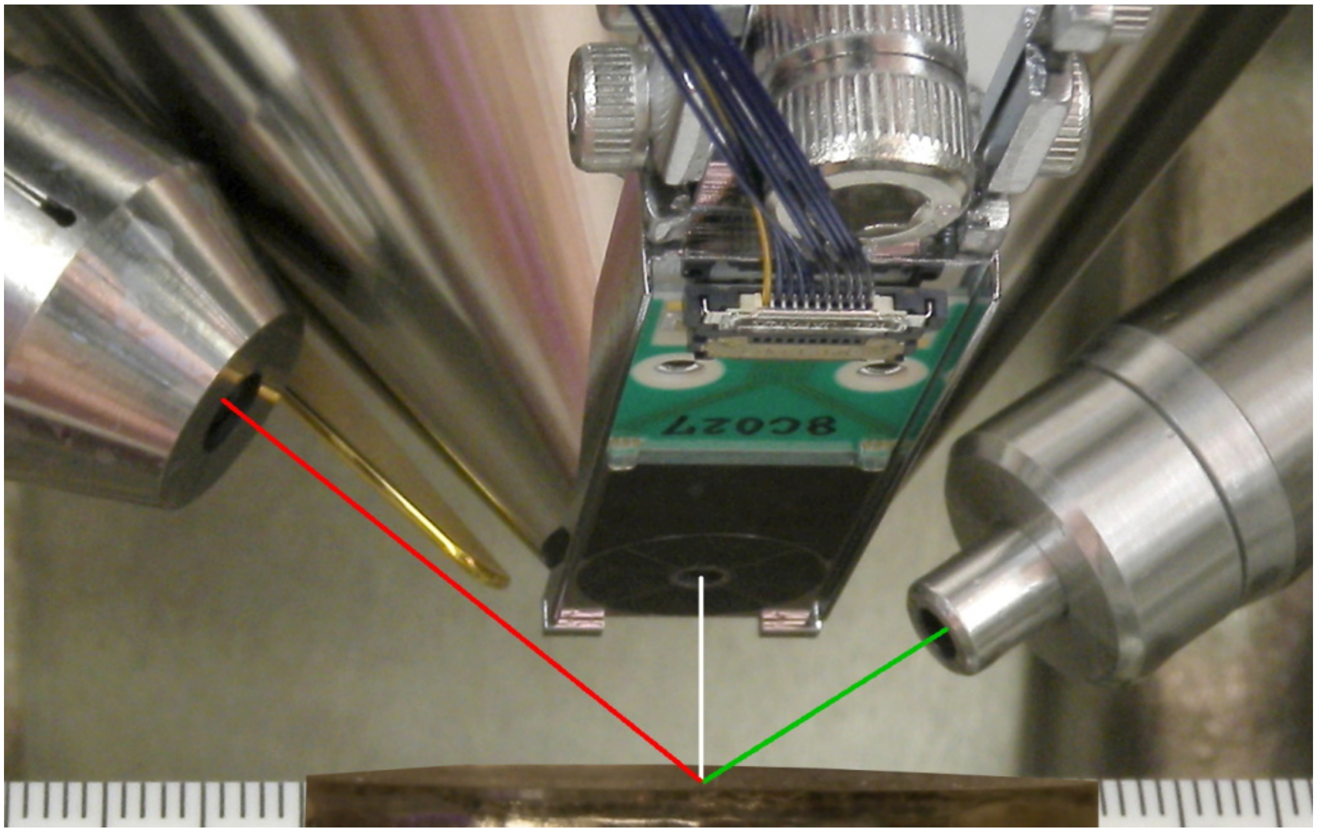
Hitachi S3400N with 2 SDDs

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.

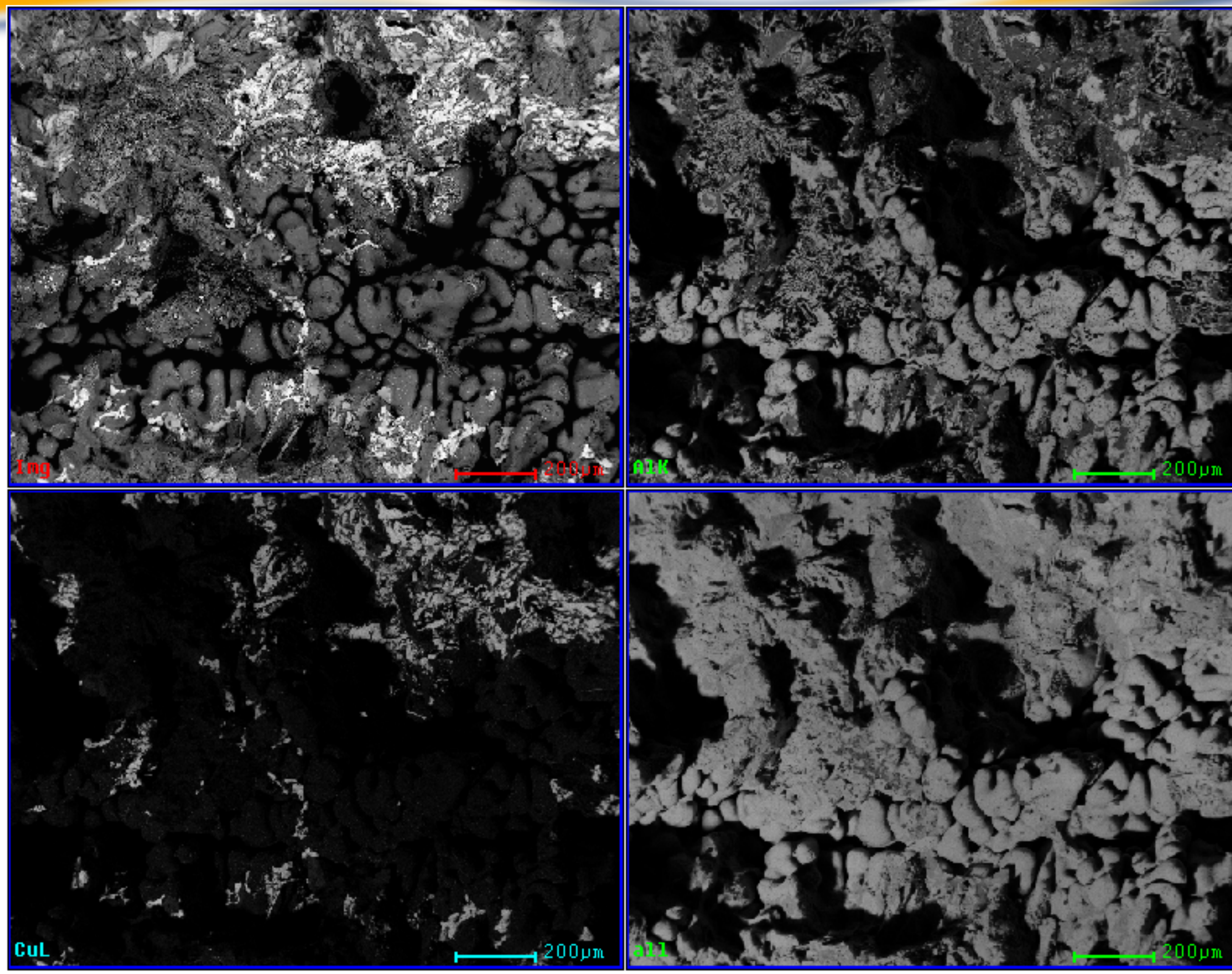
Mapping with Multiple Silicon Drift Detectors

•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:

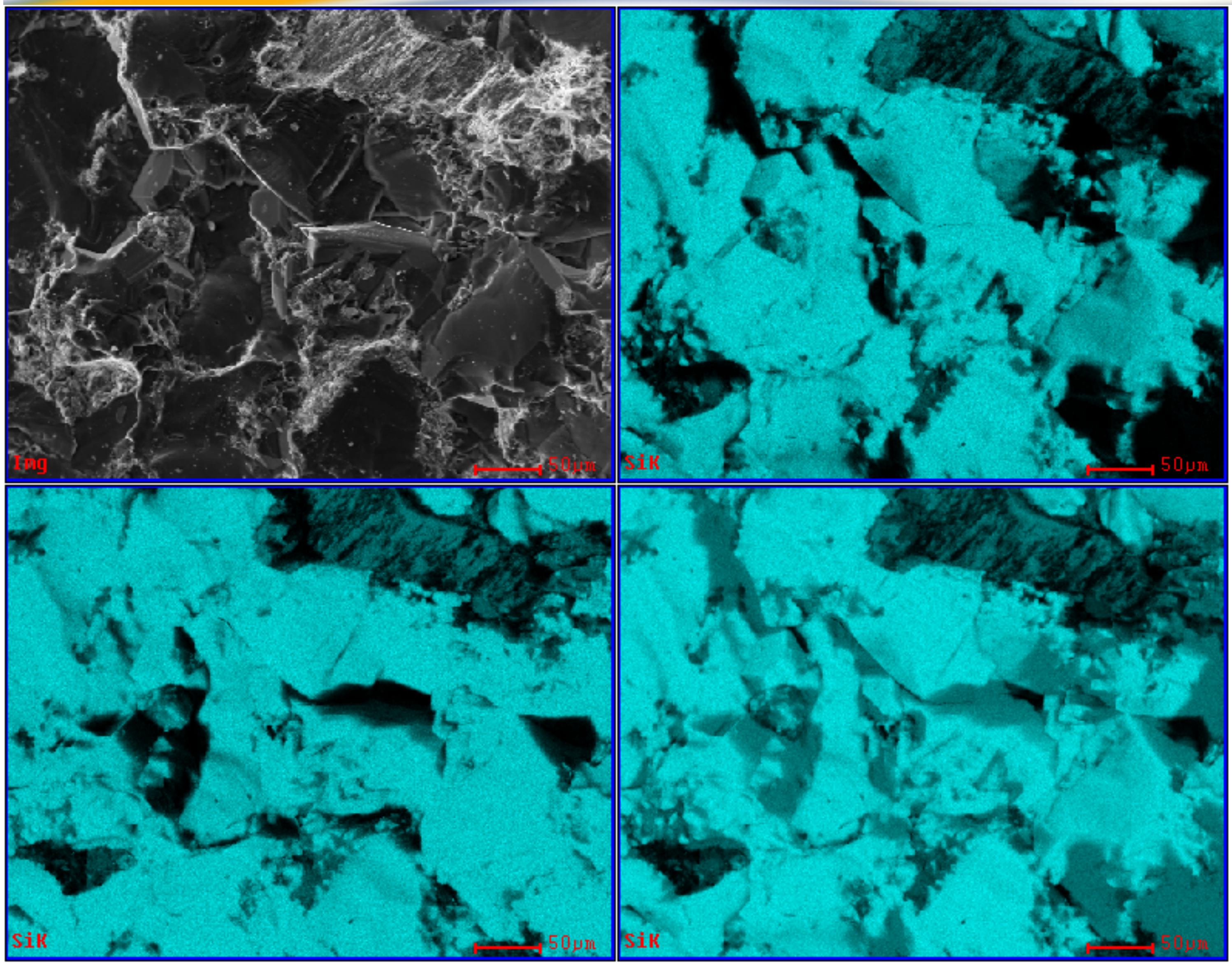
•Poor man's approach –use 1 detector to collect one set of maps, and then "simply" rotate the stage 180 degrees, do a 180 degree scan rotate and collect a second set of maps of the same area which can be added or merged.

•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.

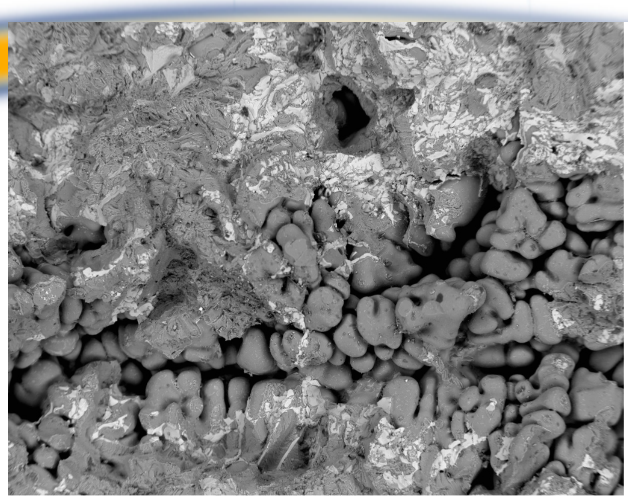
Mapping with Multiple Silicon Drift Detectors

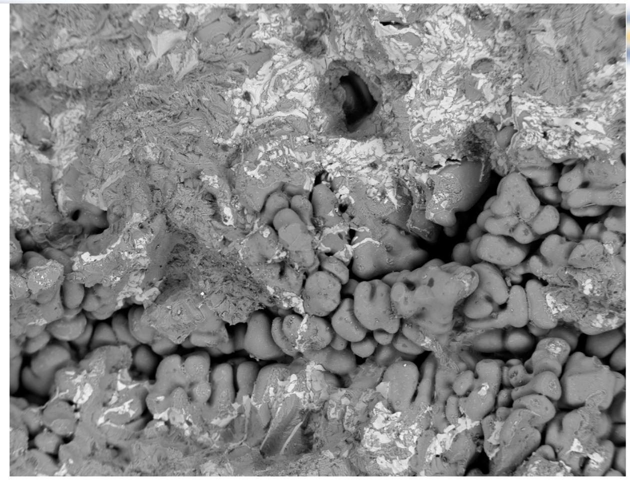


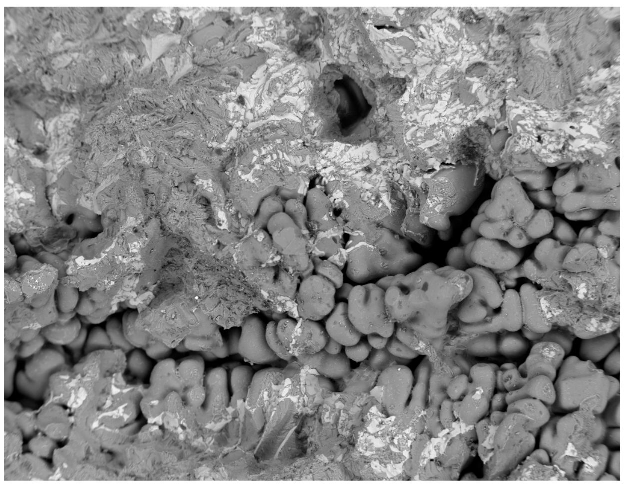
Sandstone fracture sample shown in SE (UL), Si Map (UR detector at the upper right), Si Map (LL detector at lower left) and the sum of the two maps (LR).

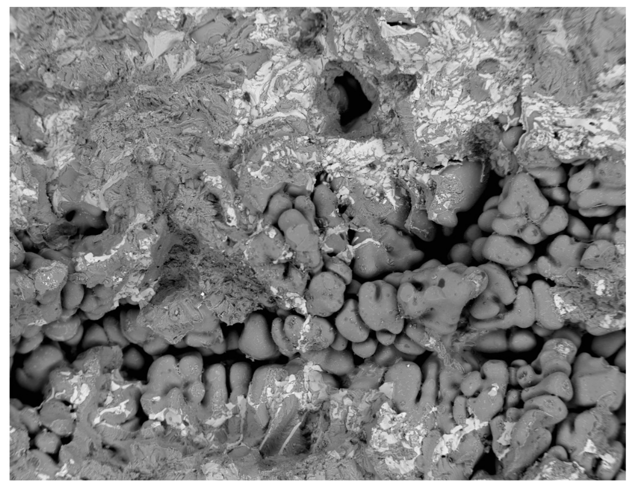
Question: what does the mid-tone area represent in the sum image?

Mapping with Multiple Silicon Drift Detectors





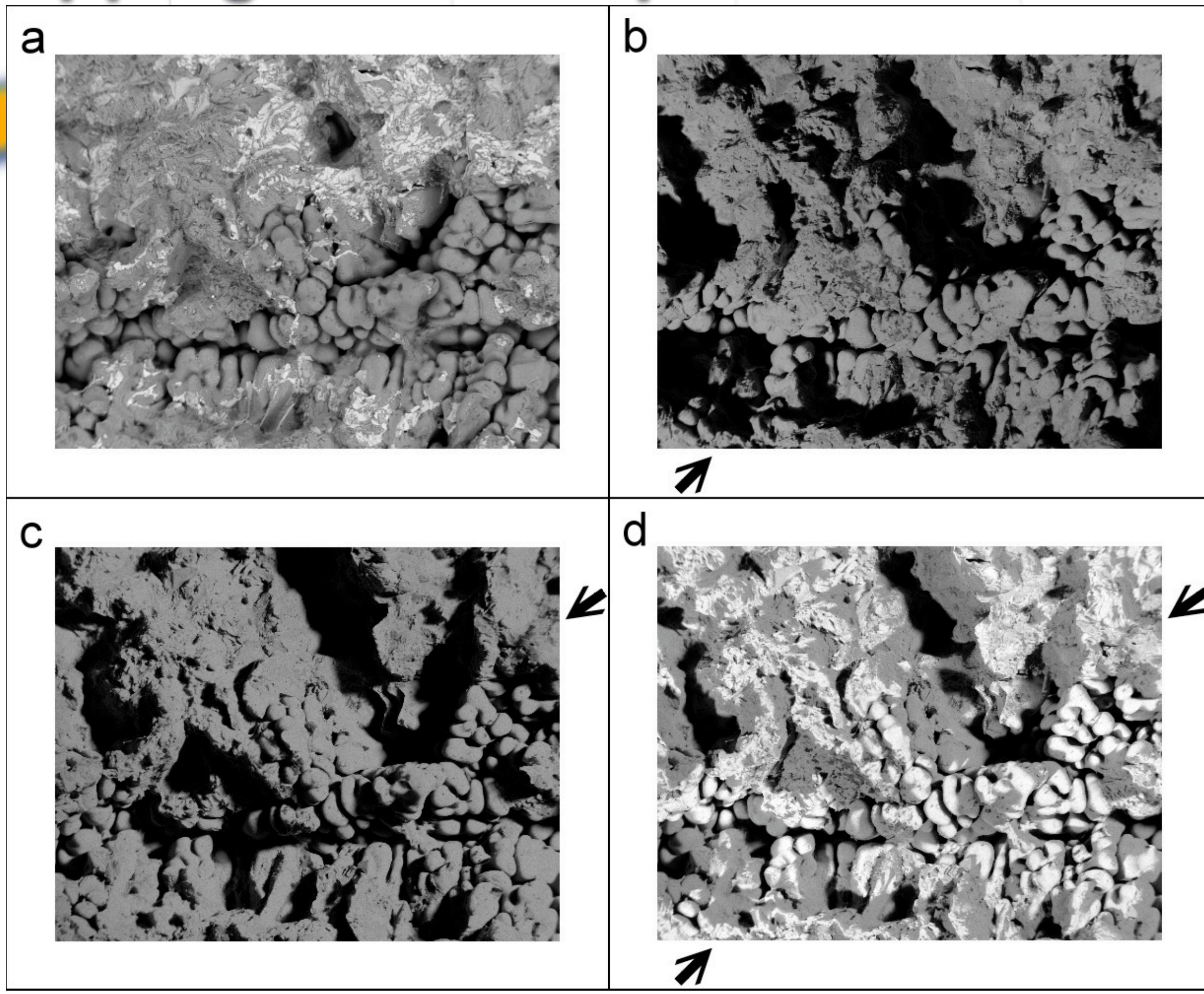




15 kV
512x400
HFW 1.5 mm

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



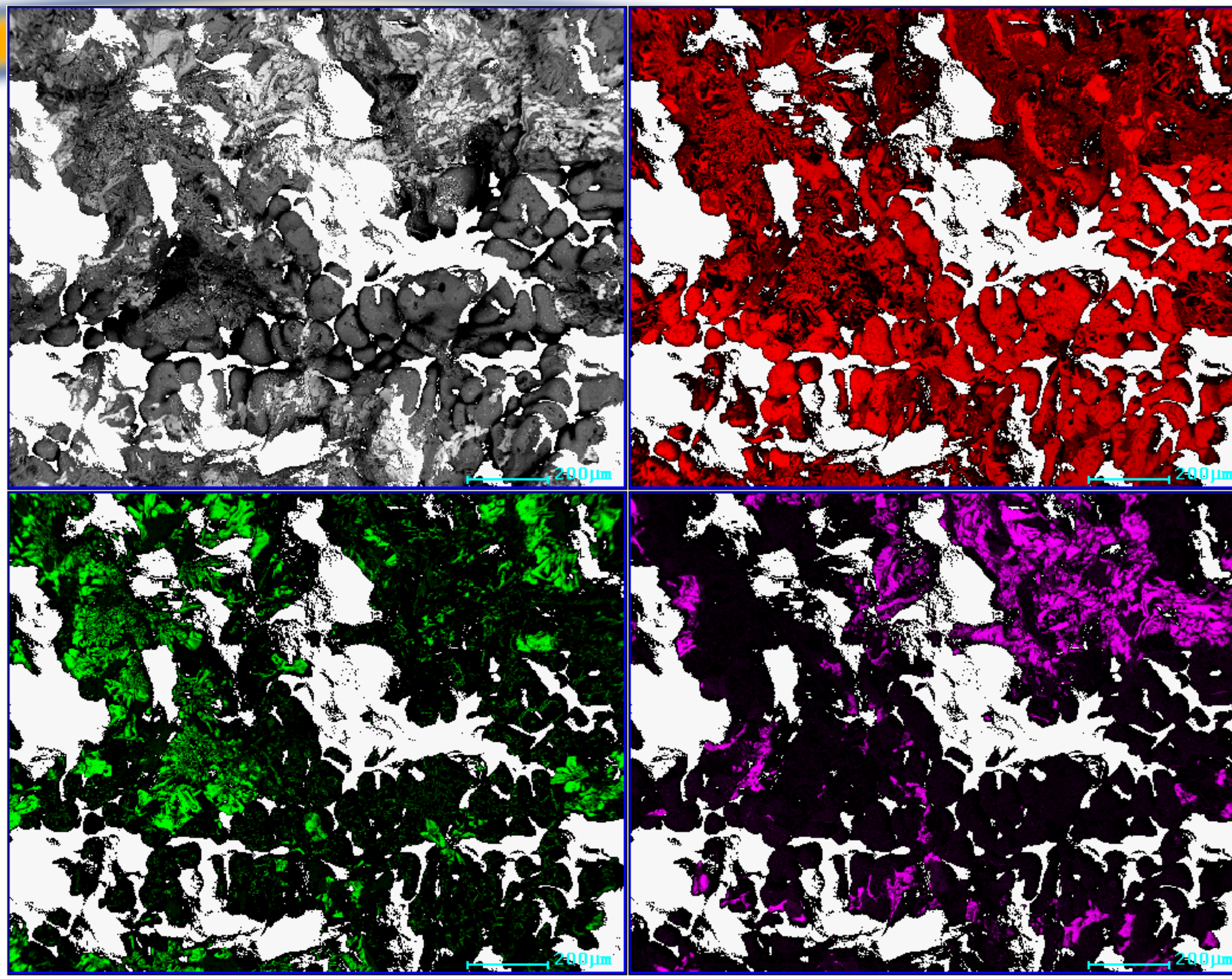
Maps of a fracture surface. The BSE image (a) is in the upper left (image width is 1.5 mm). A region of interest was created to represent the entire spectrum. Figure d is a sum of both detectors. The summed image has 3 primary gray levels: black, which indicates an area which did not provide an x-ray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

BSE

Al

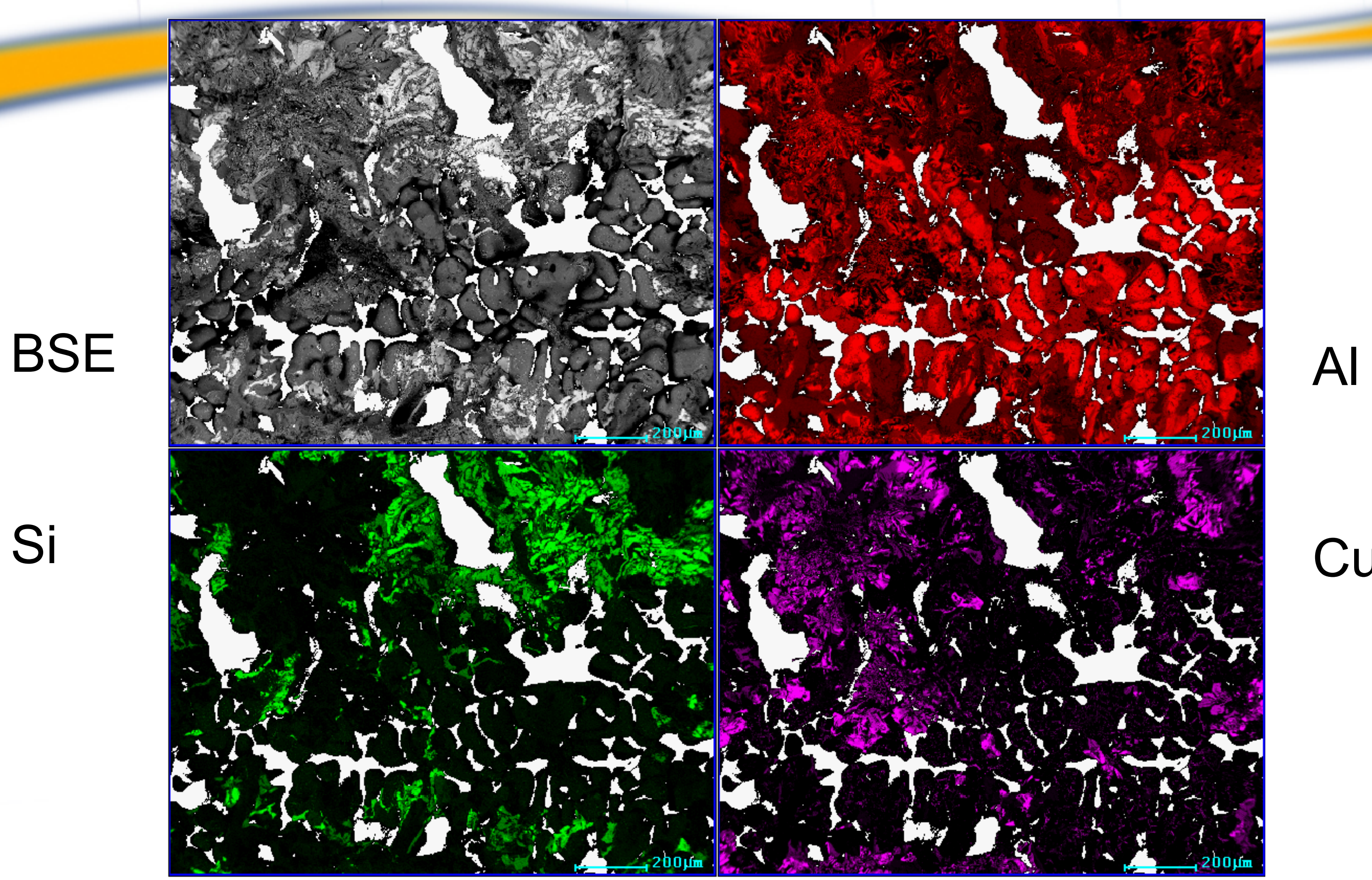


Si

Cu

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

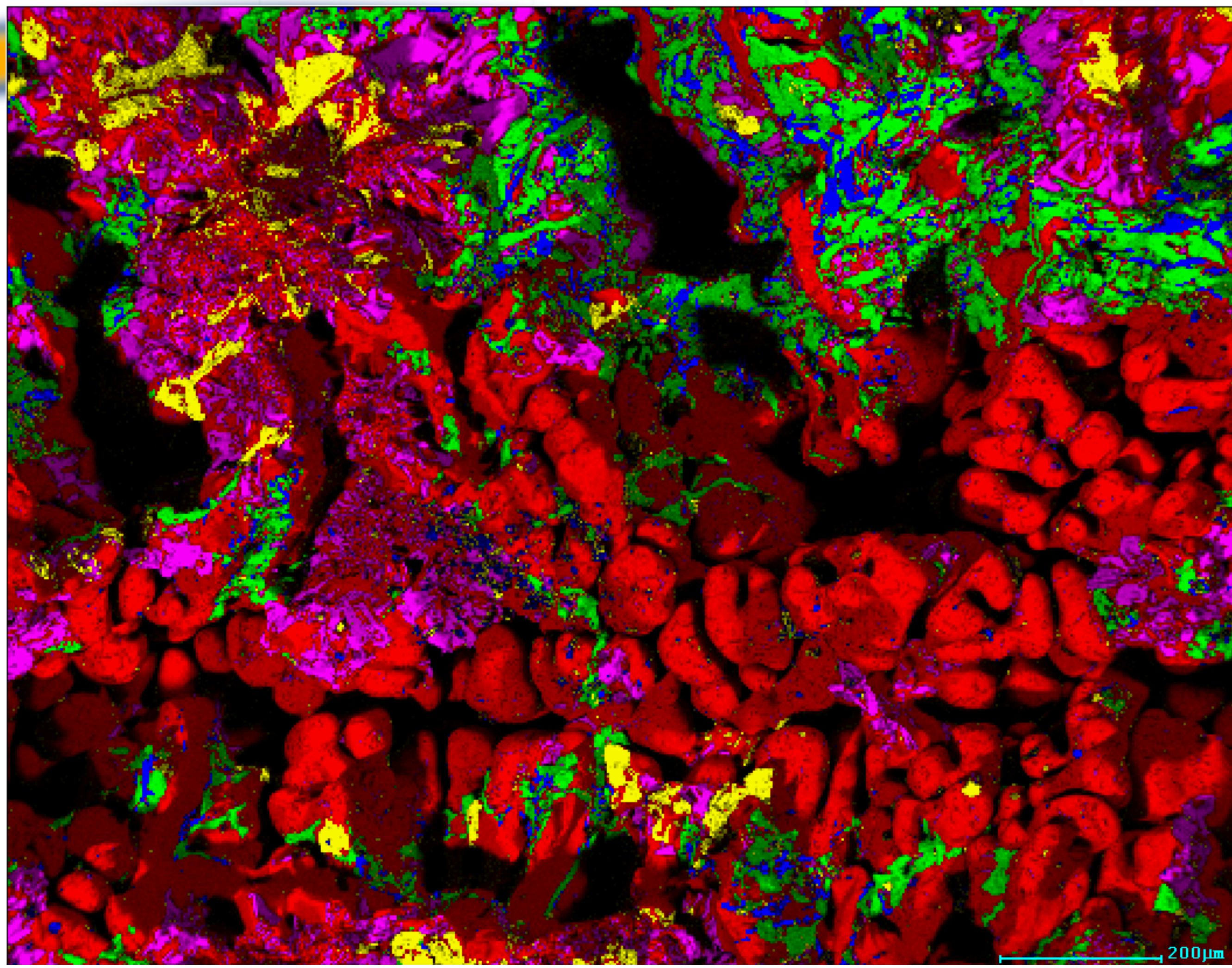


Taking the Sum ROI image (inverted and binarized) and overlaying it with MAX maps produces the results above. White areas can not be seen with 2 detectors.

Mapping with Multiple Silicon Drift Detectors

Al
Cu
Fe
Mg
Si

Null
?

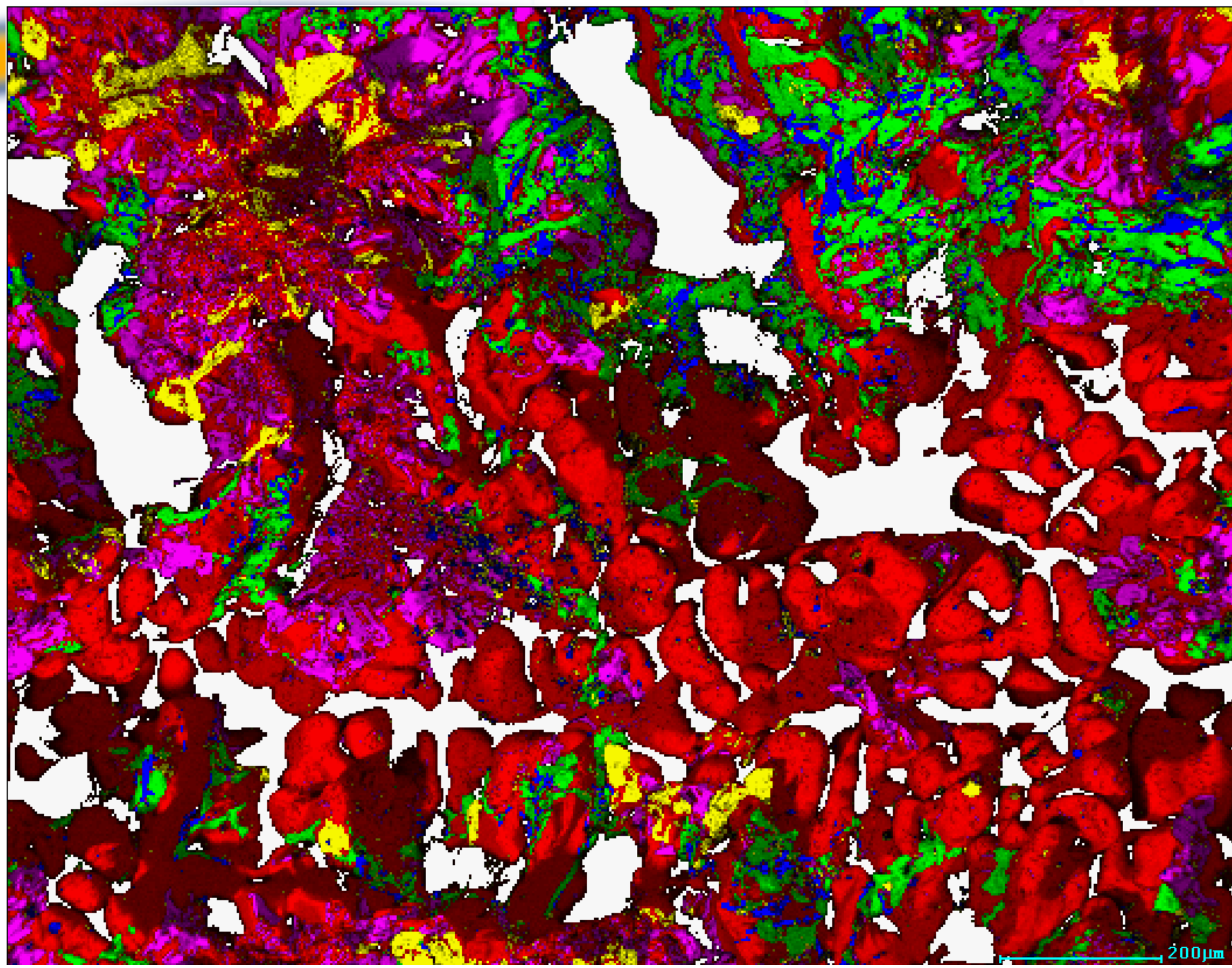


Overlaying 5 MAX maps without taking into account the Sum ROI produces the results above. Black areas are locations that can not be seen with either of the 2 detectors (or might represent a missed element).

Mapping with Multiple Silicon Drift Detectors

Al
Cu
Fe
Mg
Si

Null

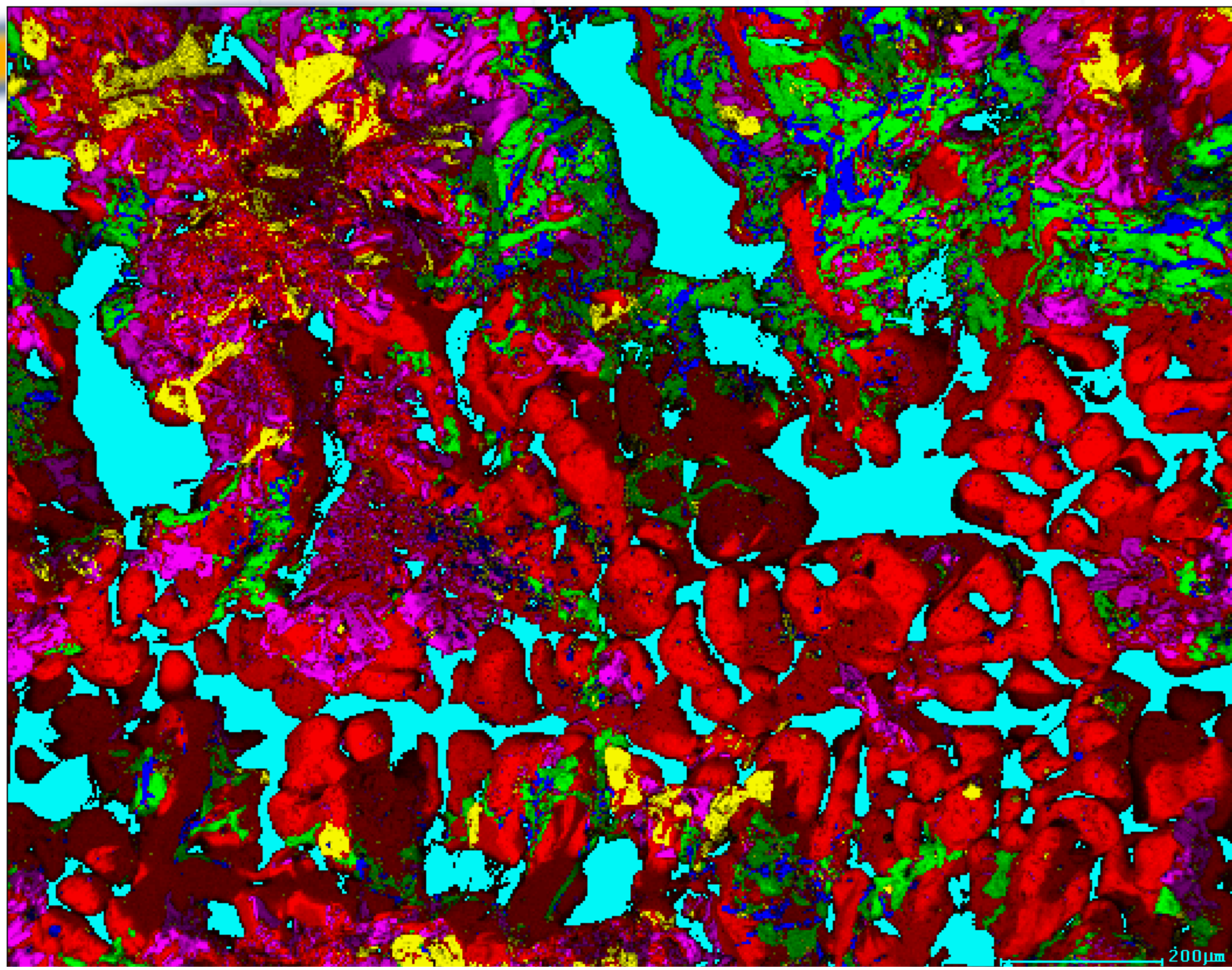


Taking the Sum ROI image (inverted and binarized) and overlaying it with 5 MAX maps produces the results above. White areas can not be seen with either detector . Glaciers versus lakes…

Mapping with Multiple Silicon Drift Detectors

Al
Cu
Fe
Mg
Si

Null

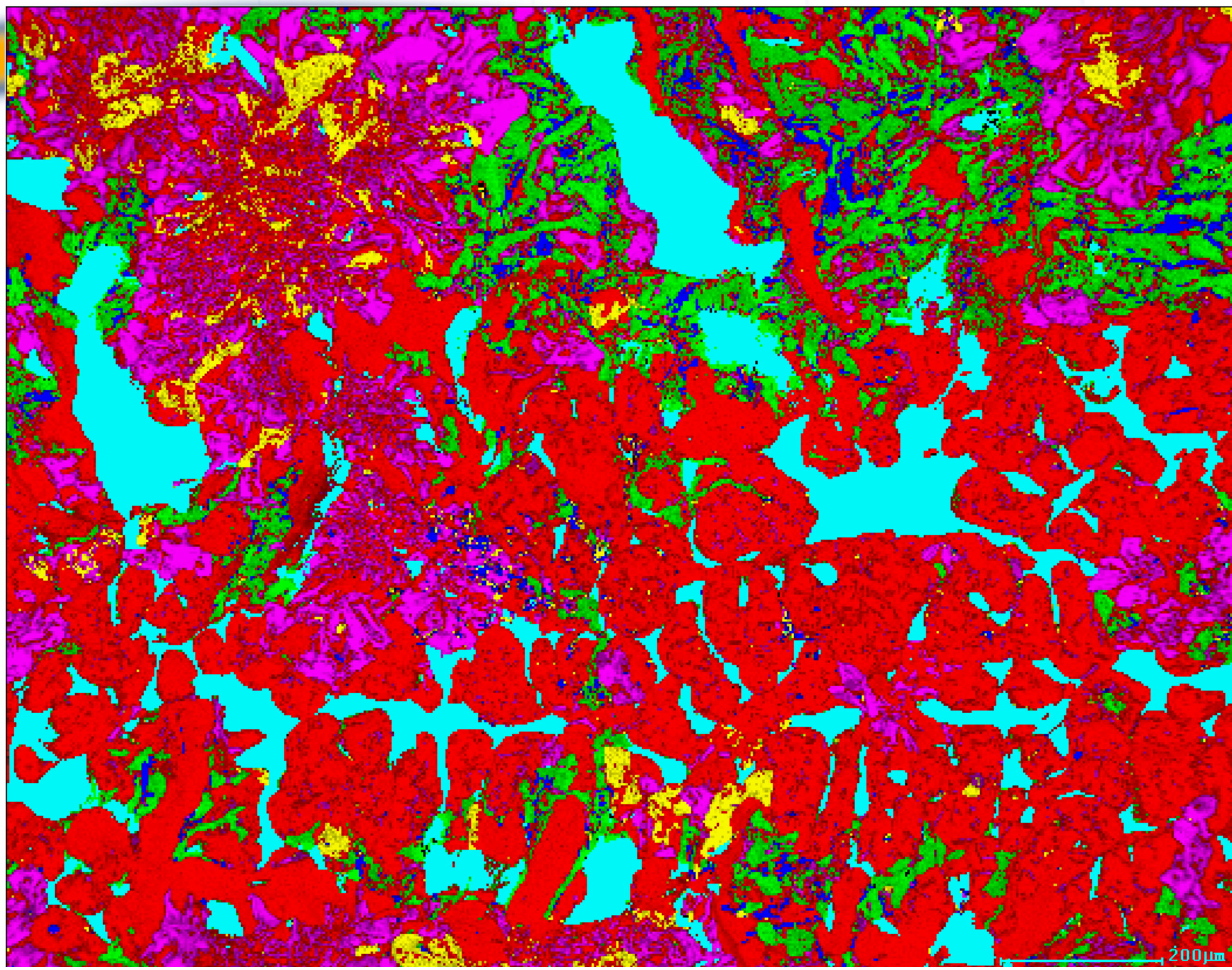


Taking the Sum ROI image (inverted and binarized) and overlaying it with 5 MAX maps produces the results above. Cyan areas can not be seen by either detector. Still have topographic effects. To get rid of the topography, normalize the data…

Mapping with Multiple Silicon Drift Detectors

Al
Cu
Fe
Mg
Si

Null



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?

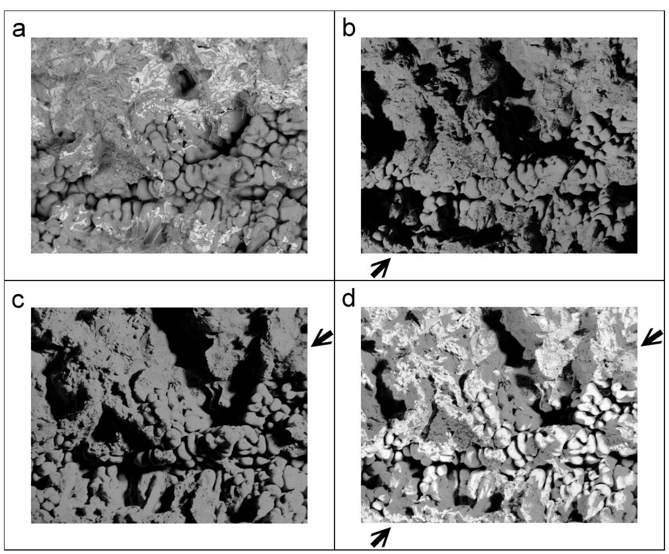
Summary Part 1

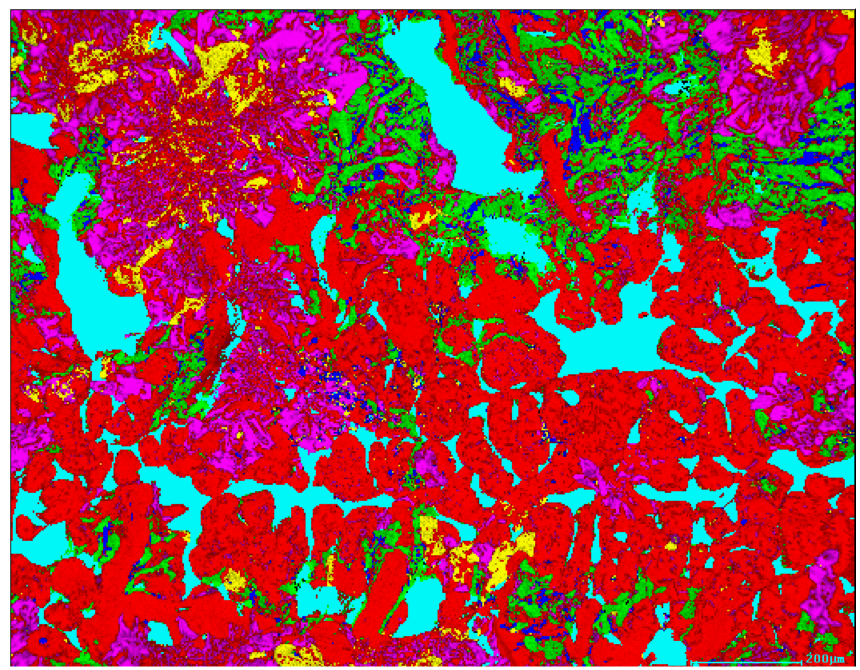
•It is possible to increase the area of illumination of a rough sample, or the area that can be seen by an EDS detector by using multiple detectors.

•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.





Improved Quant with Multiple Detectors

Garnet Formula:

$(Mg, Ca, Mn, Fe)_3Al_2Si_3O_{12}$

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

Endmembers:

Pyrope $Mg_3Al_2Si_3O_{12}$

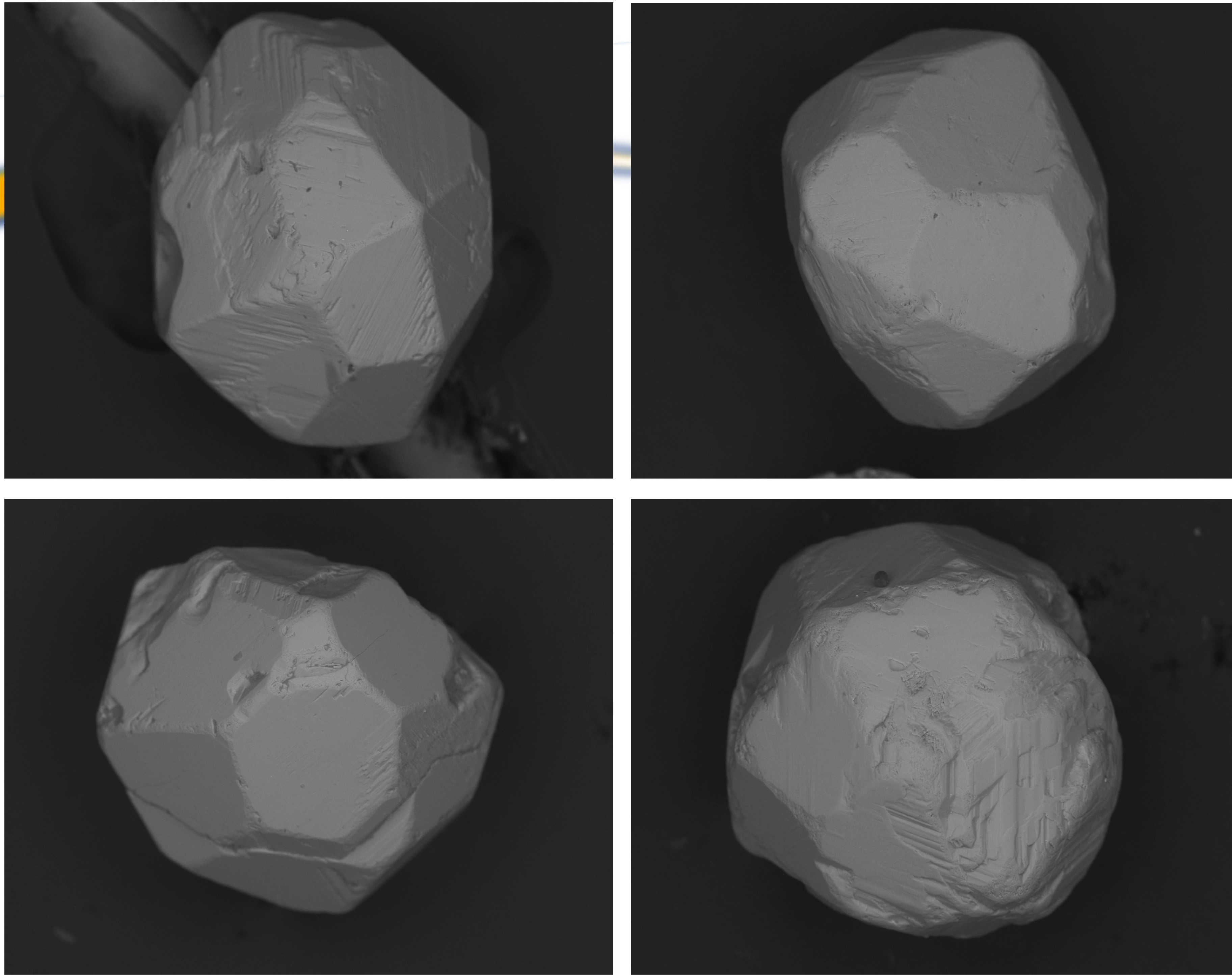
Almandine $Fe_3Al_2Si_3O_{12}$

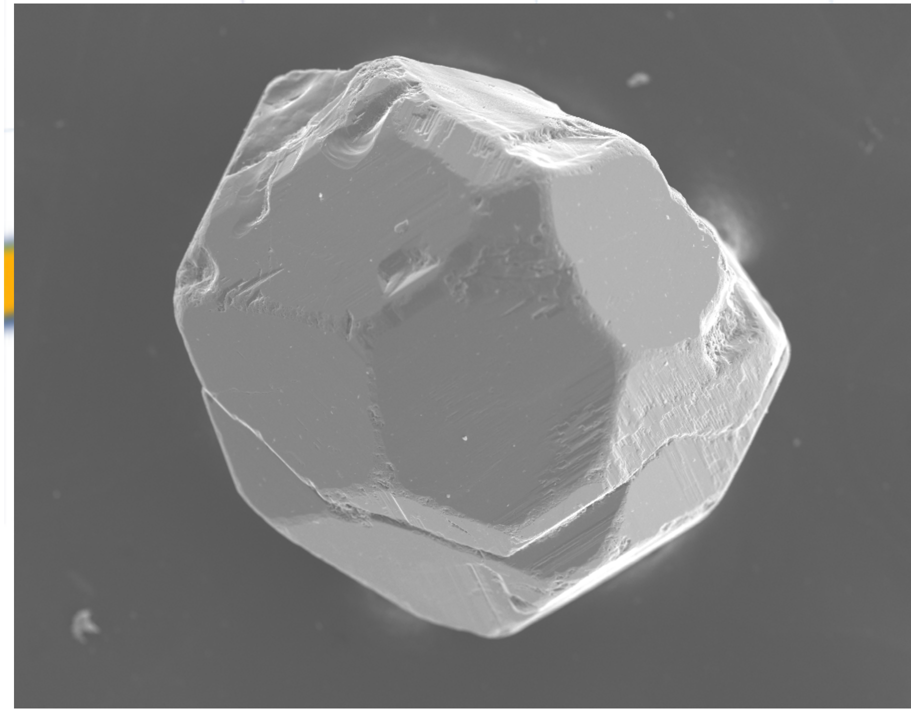
Spessartine $Mn_3Al_2Si_3O_{12}$

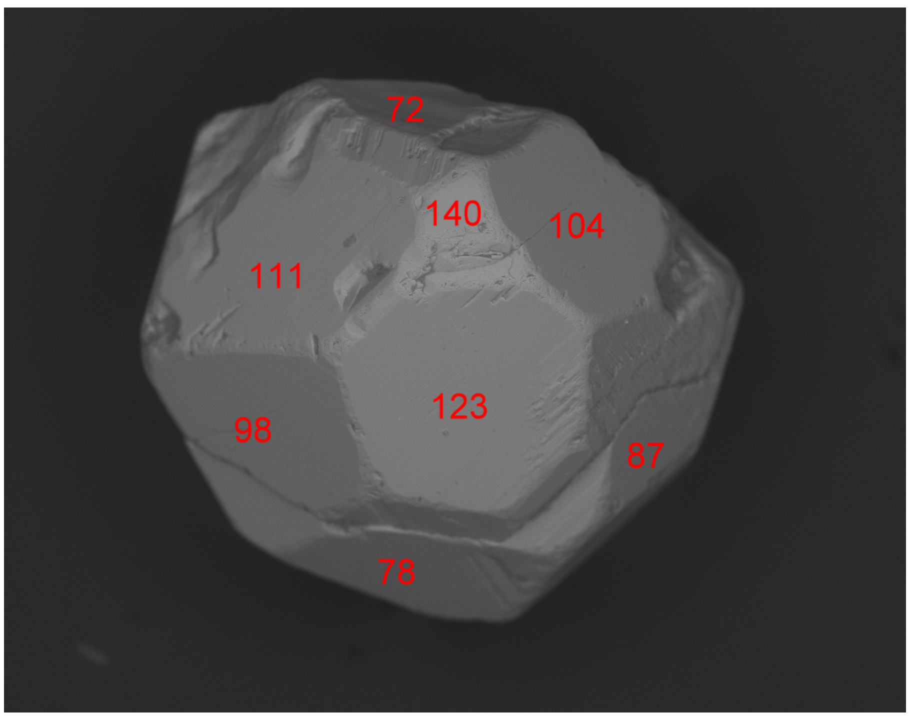
Andradite $Ca_3Fe_2Si_3O_{12}$

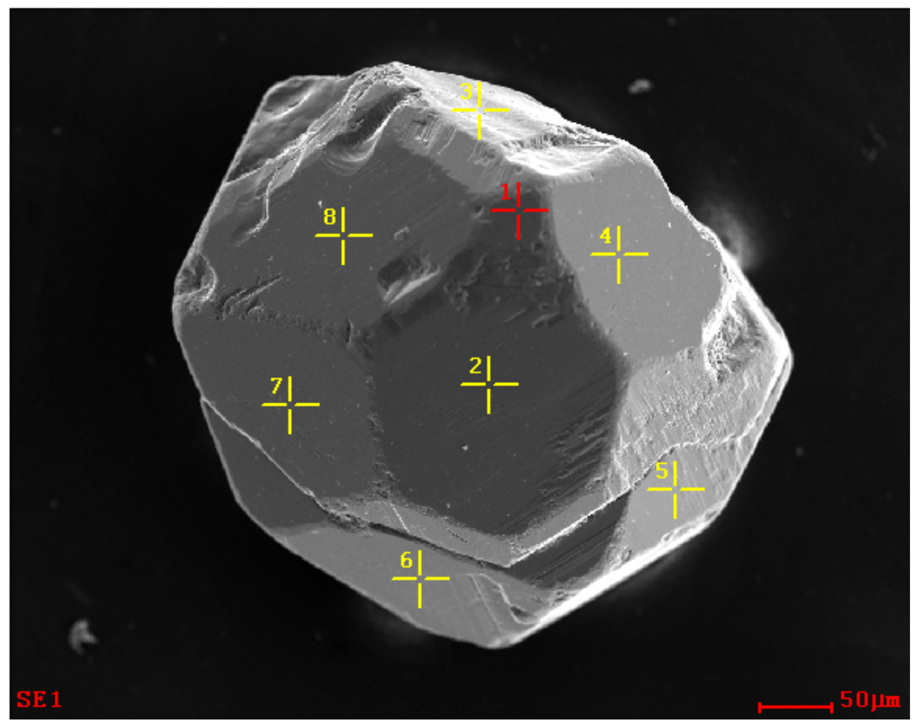
Grossular $Ca_3Al_2Si_3O_{12}$

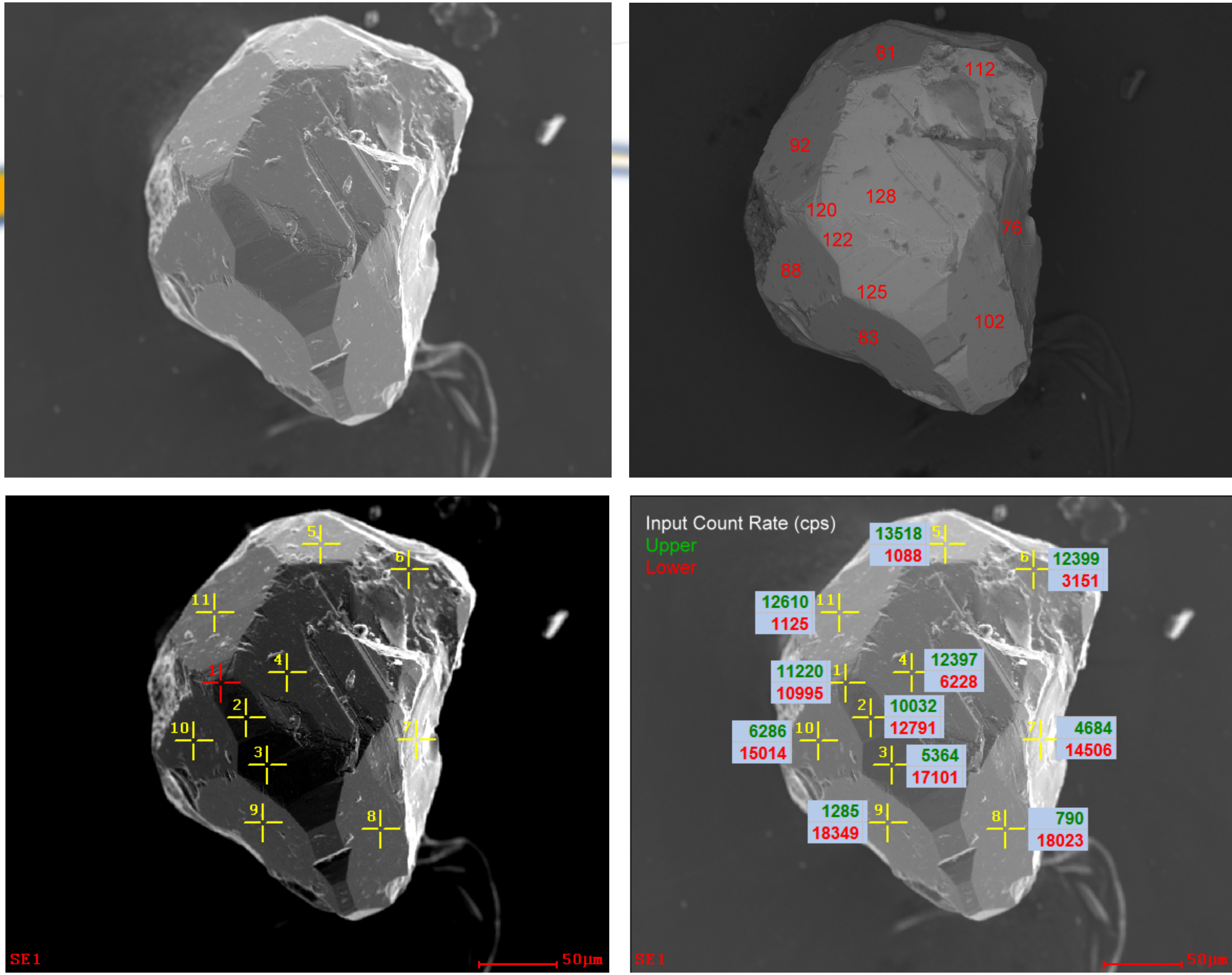
Uvarovite $Ca_3Cr_2Si_3O_{12}$



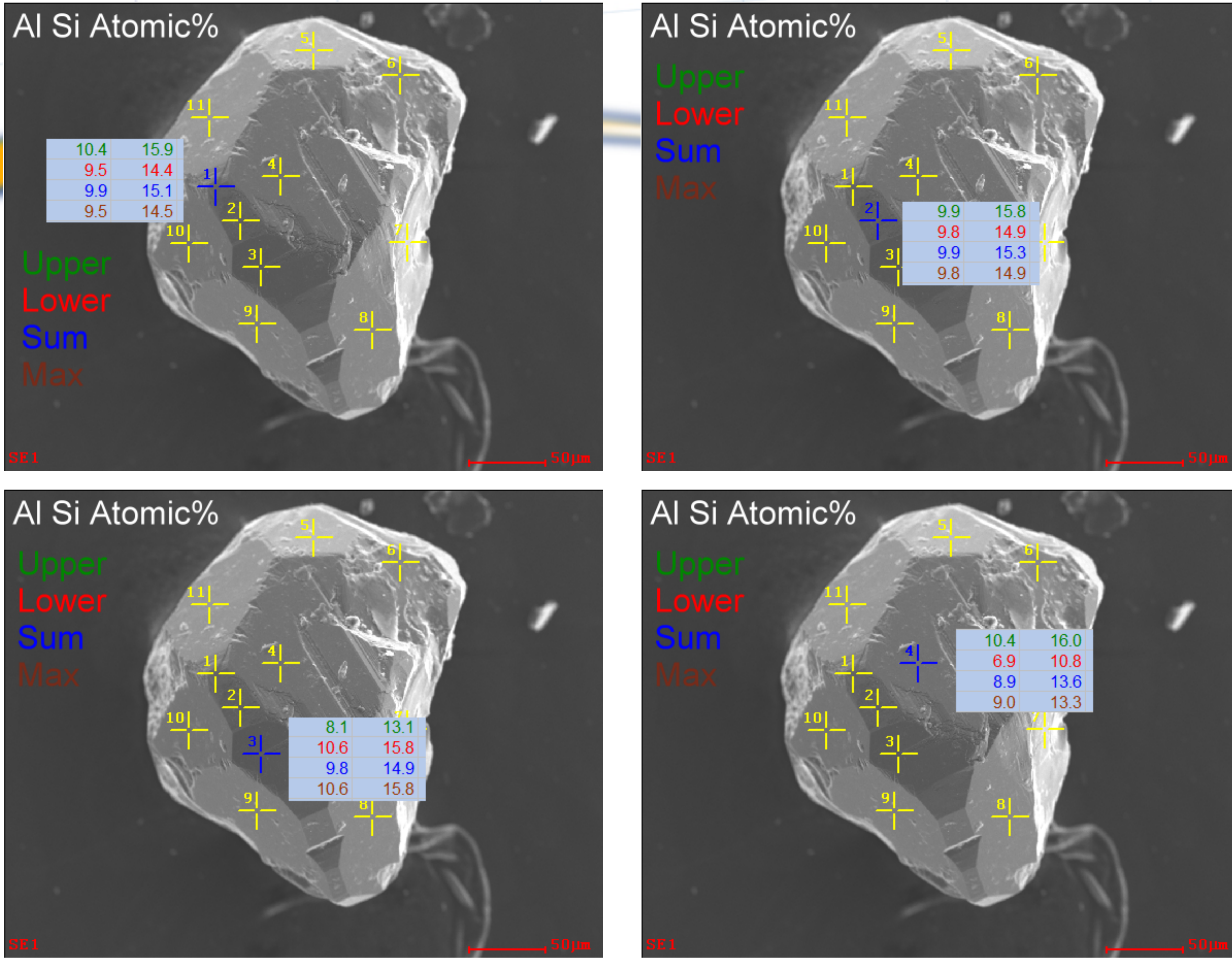



72
140
104
111
123
98
87
78

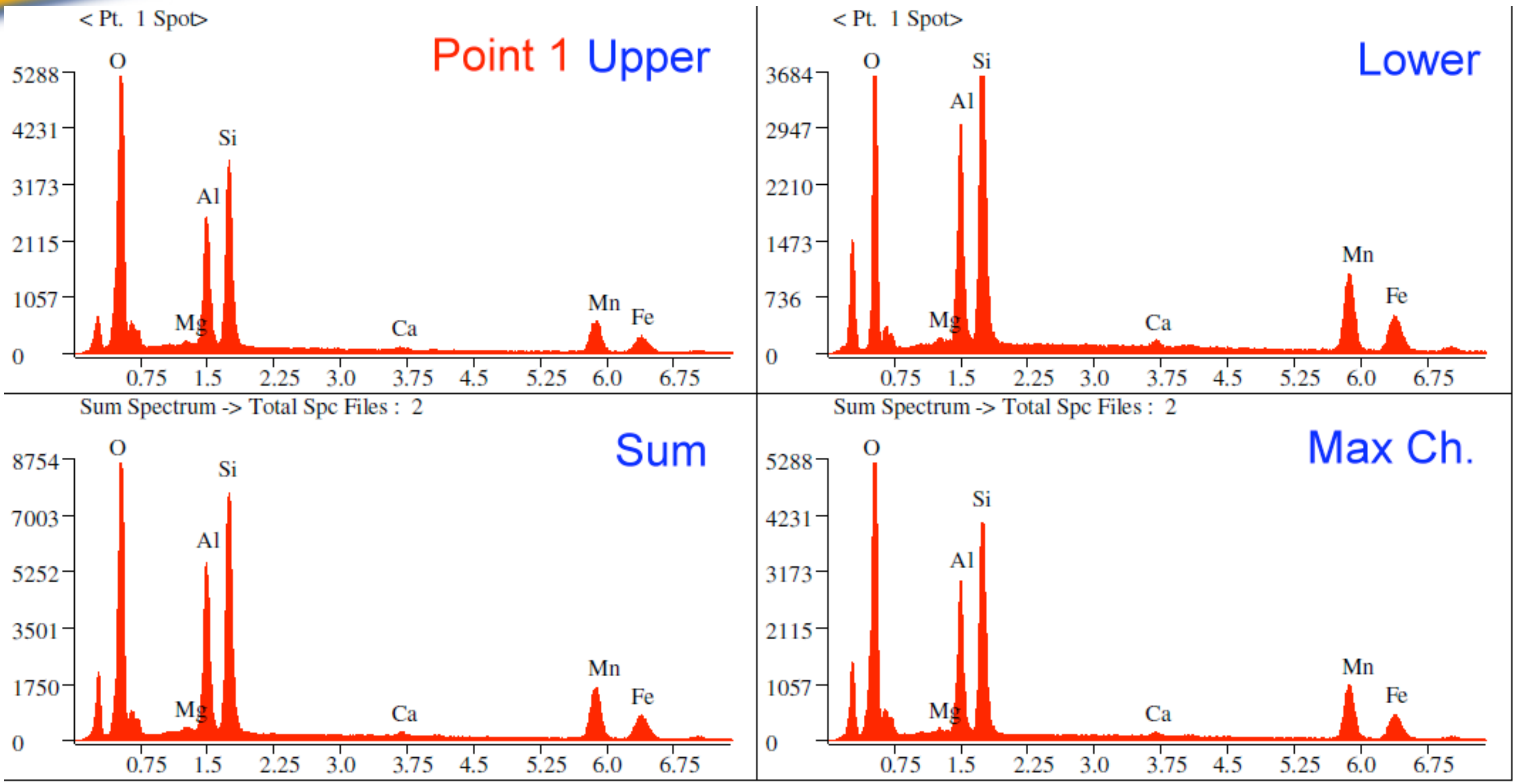

3
1
8
4
2
7
5
6
SE1
50µm



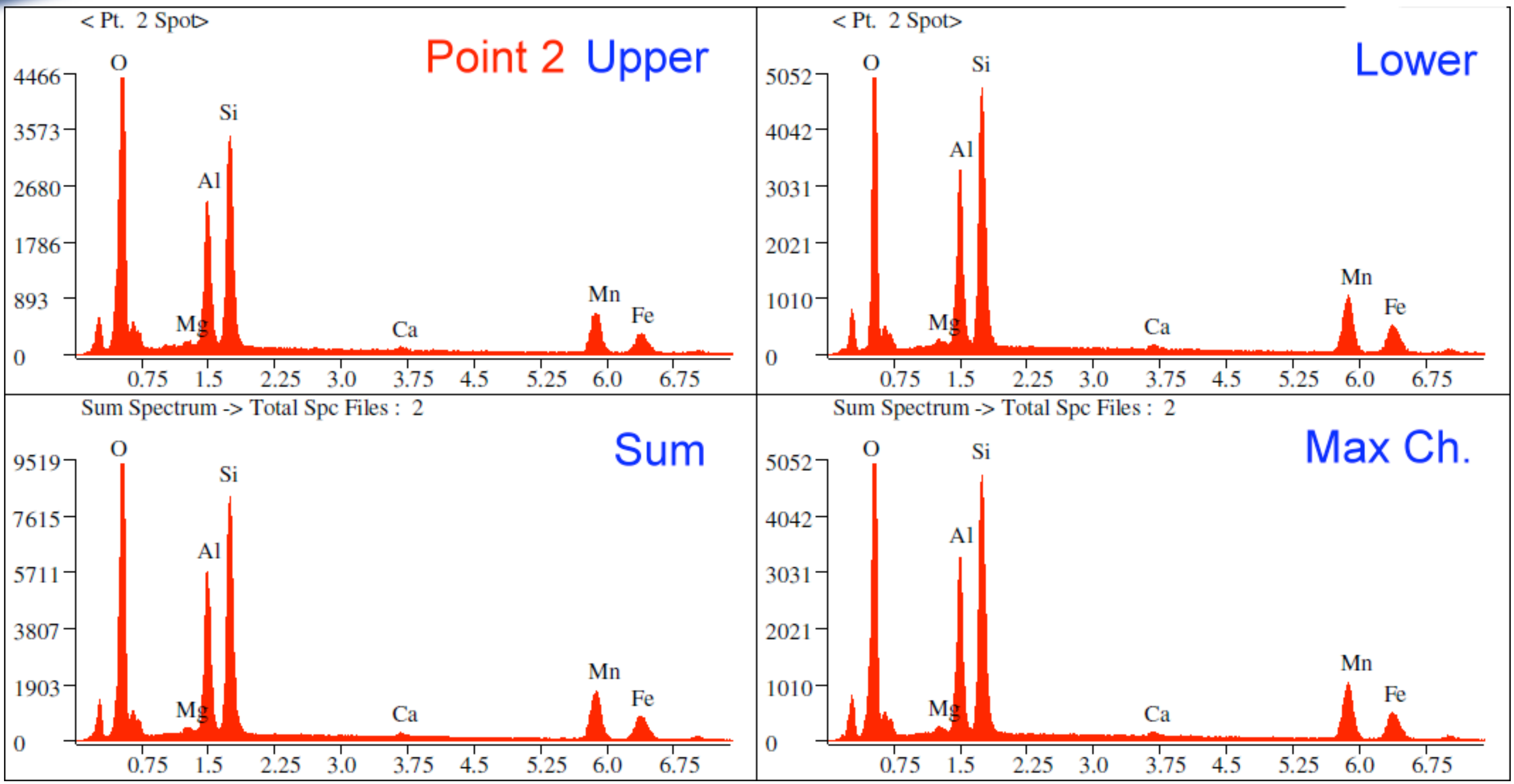
81
112
92
128
120
76
122
88
125
102
83
5
6
11
4
1
2
10
7
3
9
8
SE1
50µm
Input Count Rate (cps)
Upper
Lower
13518
1088
12399
3151
12610
1125
11220
10995
12397
6228
10032
12791
6286
15014
5364
17101
4684
14506
1285
18349
790
18023
SE1
50µm


Al Si Atomic%
10.4 15.9
9.5 14.4
9.9 15.1
9.5 14.5
Upper
Lower
Sum
Max
SE1
50µm
Al Si Atomic%
Upper
Lower
Sum
Max
9.9 15.8
9.8 14.9
9.9 15.3
9.8 14.9
SE1
50µm
Al Si Atomic%
Upper
Lower
Sum
Max
8.1 13.1
10.6 15.8
9.8 14.9
10.6 15.8
SE1
50µm
Al Si Atomic%
Upper
Lower
Sum
Max
10.4 16.0
6.9 10.8
8.9 13.6
9.0 13.3
SE1
50µm

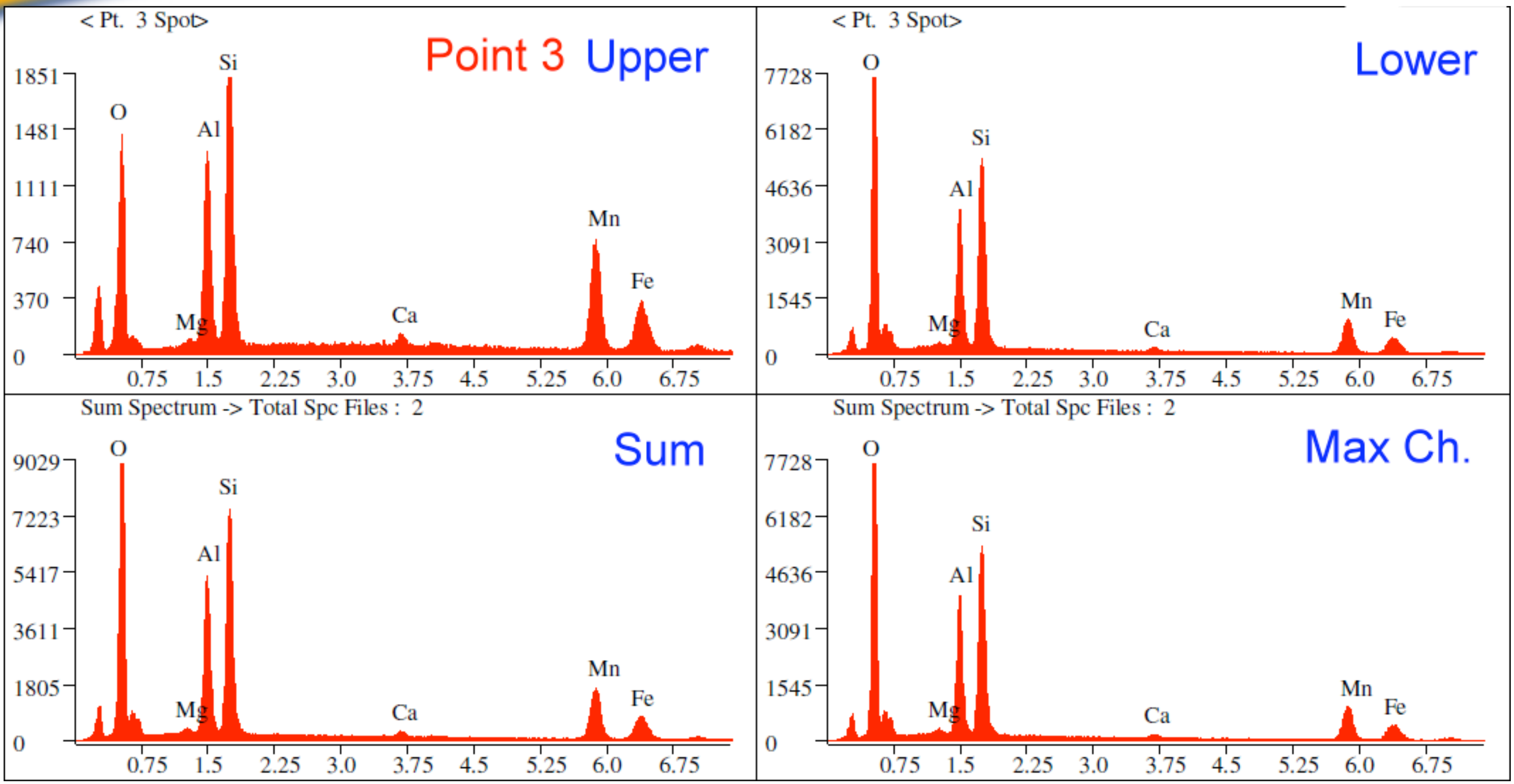
Improved Quant with Multiple Detectors



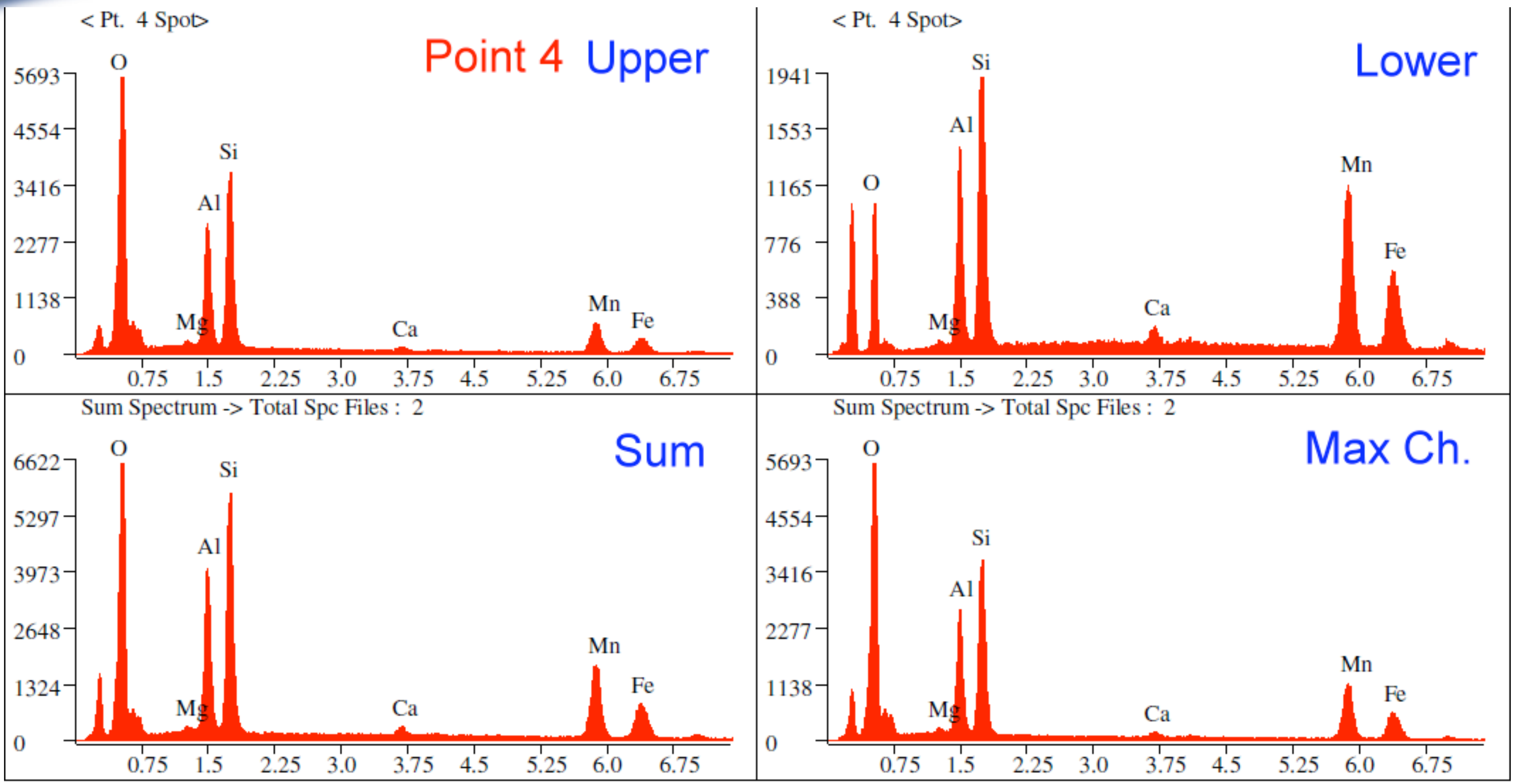
Improved Quant with Multiple Detectors

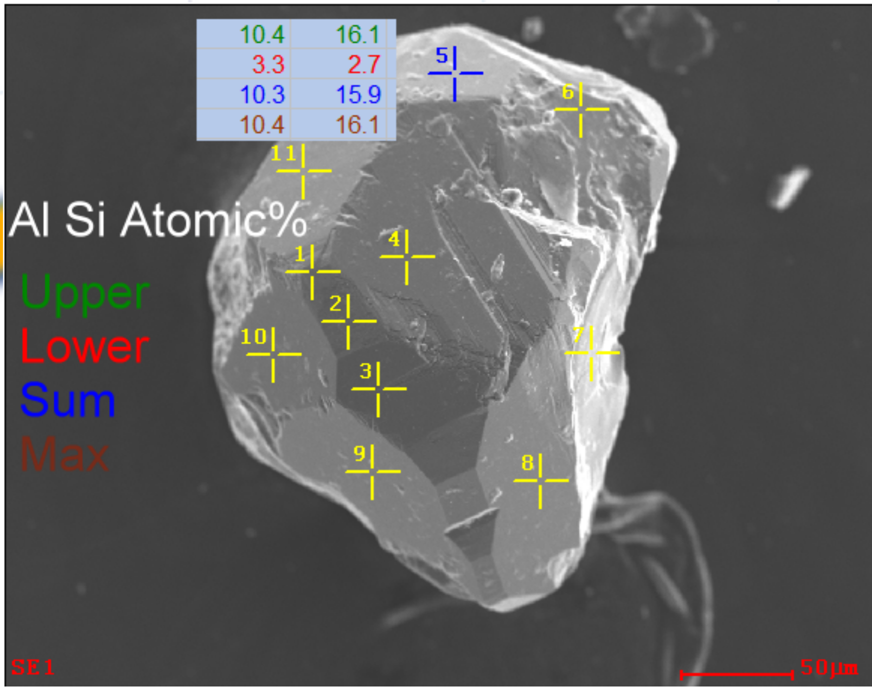


Improved Quant with Multiple Detectors

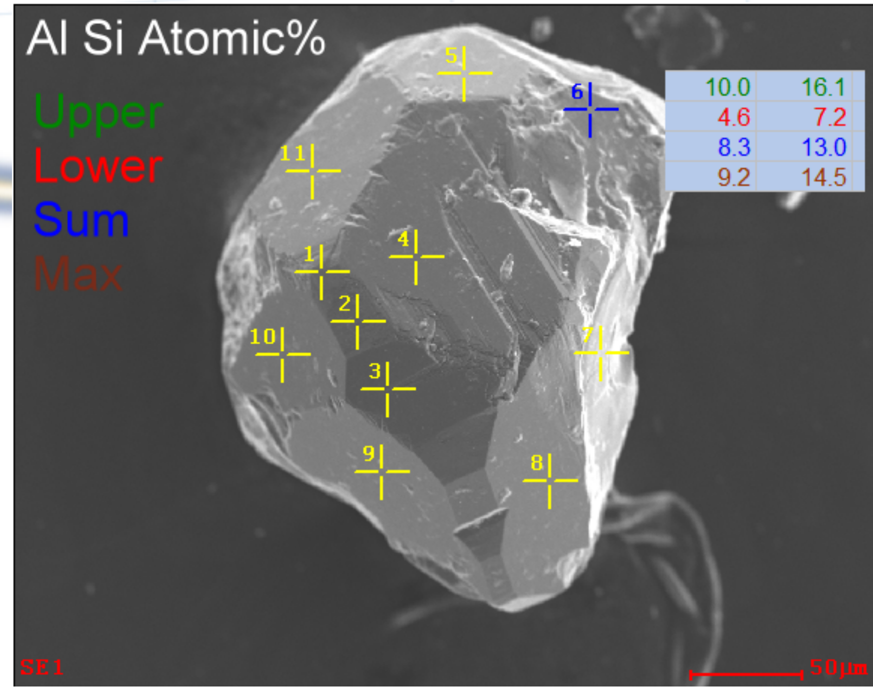


Improved Quant with Multiple Detectors

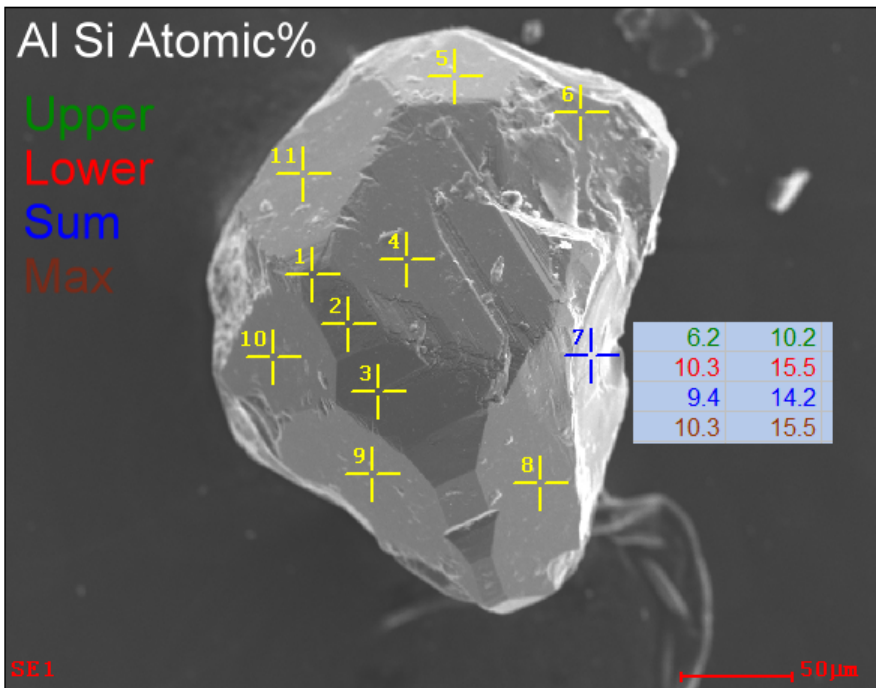




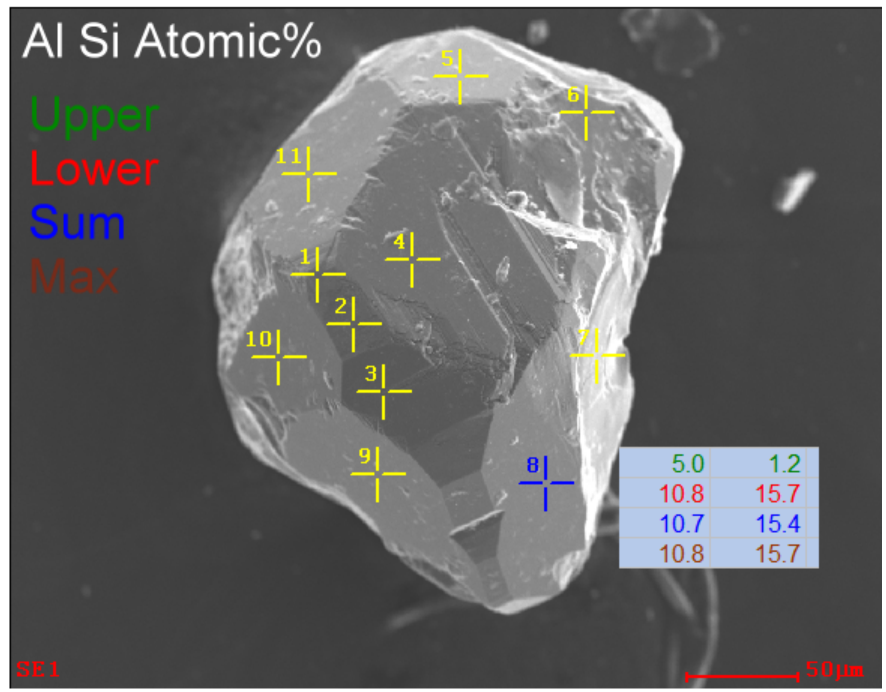
Al Si Atomic%
Upper
Lower
Sum
Max
10.4 16.1
3.3 2.7
10.3 15.9
10.4 16.1
SE1
50µm



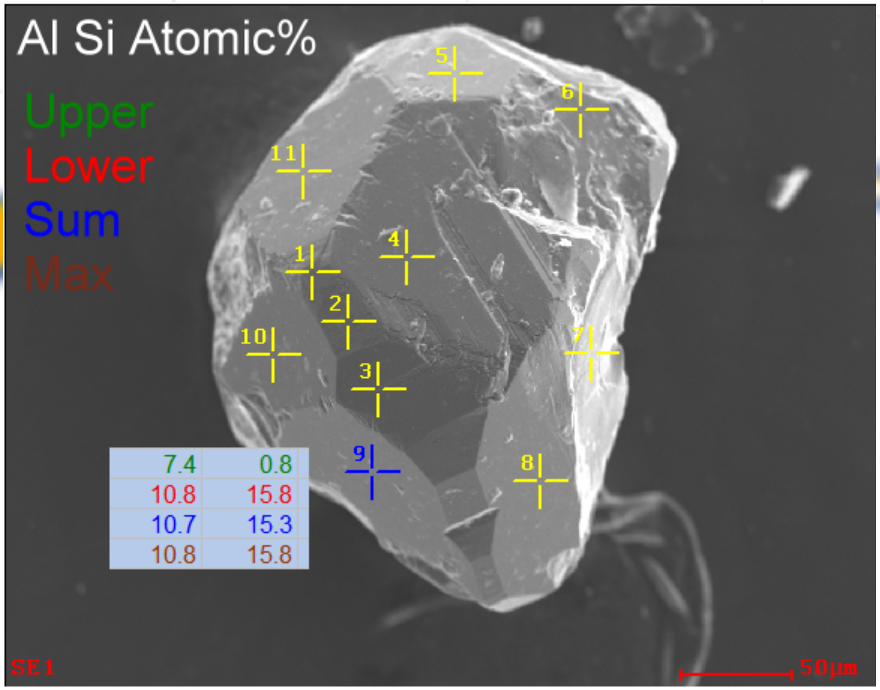
Al Si Atomic%
Upper
Lower
Sum
Max
10.0 16.1
4.6 7.2
8.3 13.0
9.2 14.5
SE1
50µm

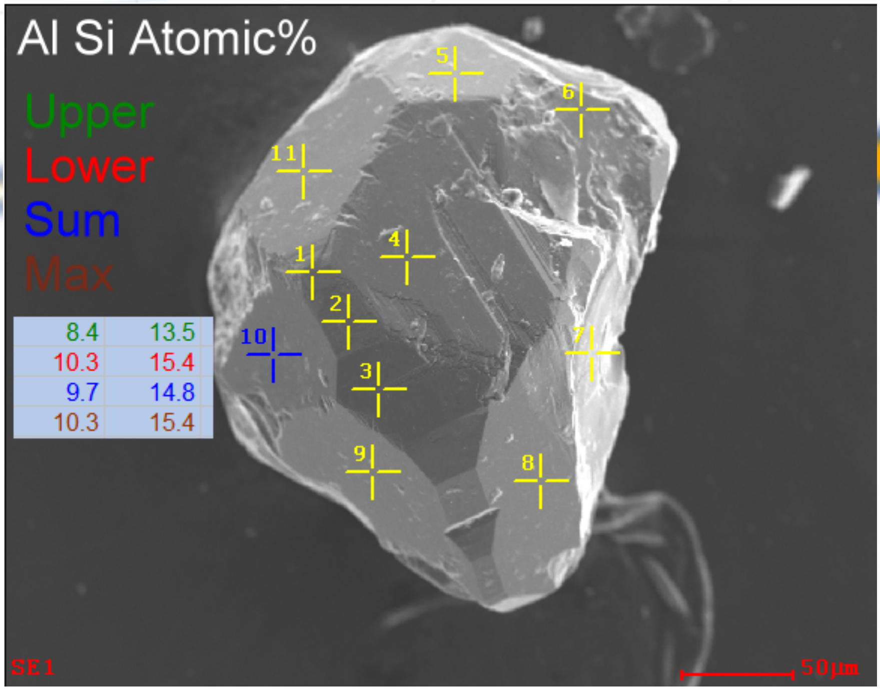


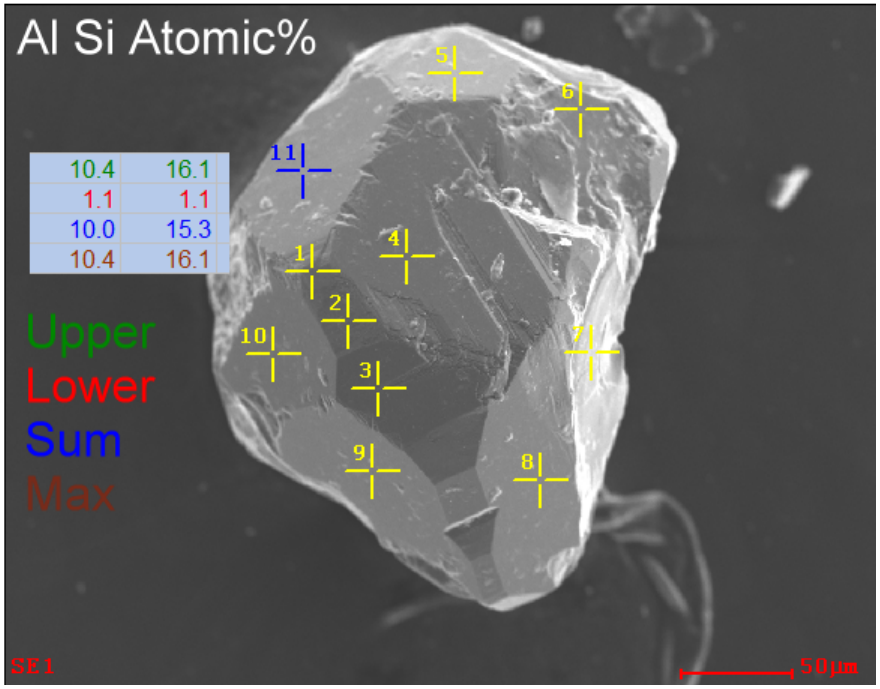
Al Si Atomic%
Upper
Lower
Sum
Max
6.2 10.2
10.3 15.5
9.4 14.2
10.3 15.5
SE1
50µm



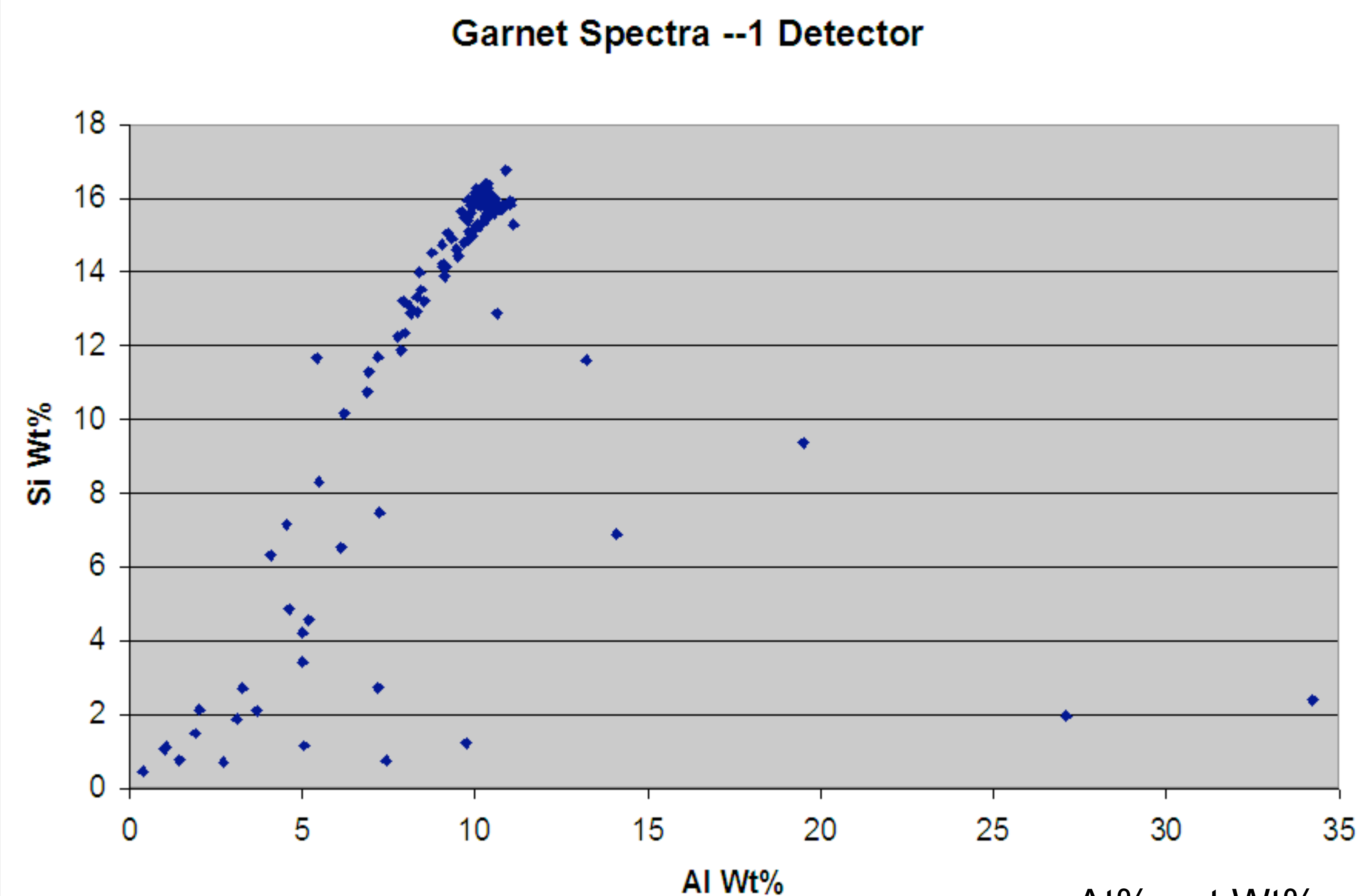
Al Si Atomic%
Upper
Lower
Sum
Max
5.0 1.2
10.8 15.7
10.7 15.4
10.8 15.7
SE1
50µm


Al Si Atomic%
Upper
Lower
Sum
Max
7.4 0.8
10.8 15.8
10.7 15.3
10.8 15.8
1
2
3
4
5
6
7
8
9
10
11
SE1
50µm


Al Si Atomic%
Upper
Lower
Sum
Max
8.4 13.5
10.3 15.4
9.7 14.8
10.3 15.4
1
2
3
4
5
6
7
8
9
10
11
SE1
50µm

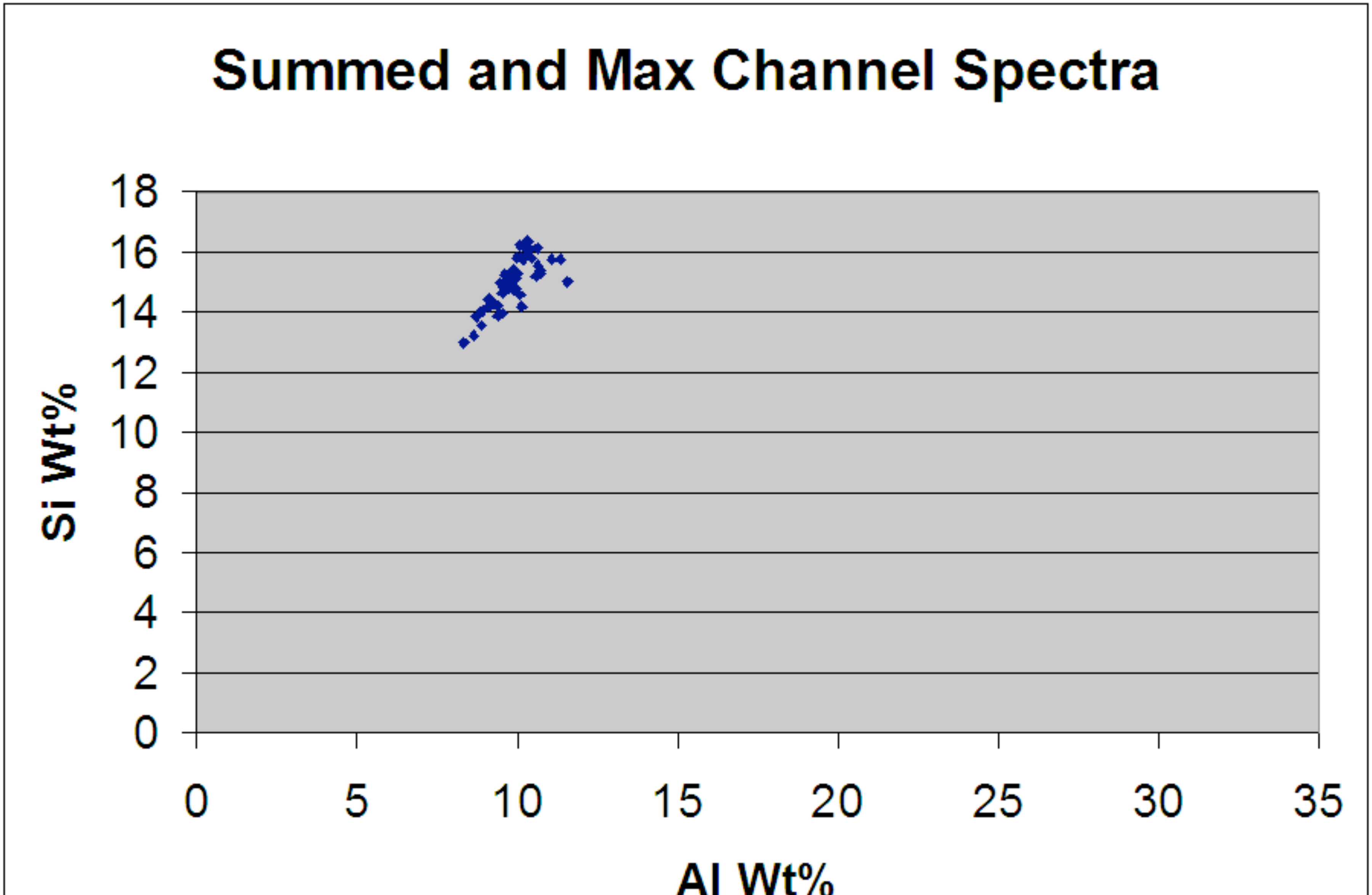

Al Si Atomic%
10.4 16.1
1.1 1.1
10.0 15.3
10.4 16.1
Upper
Lower
Sum
Max
1
2
3
4
5
6
7
8
9
10
11
SE1
50µm

Improved Quant with Multiple Detectors



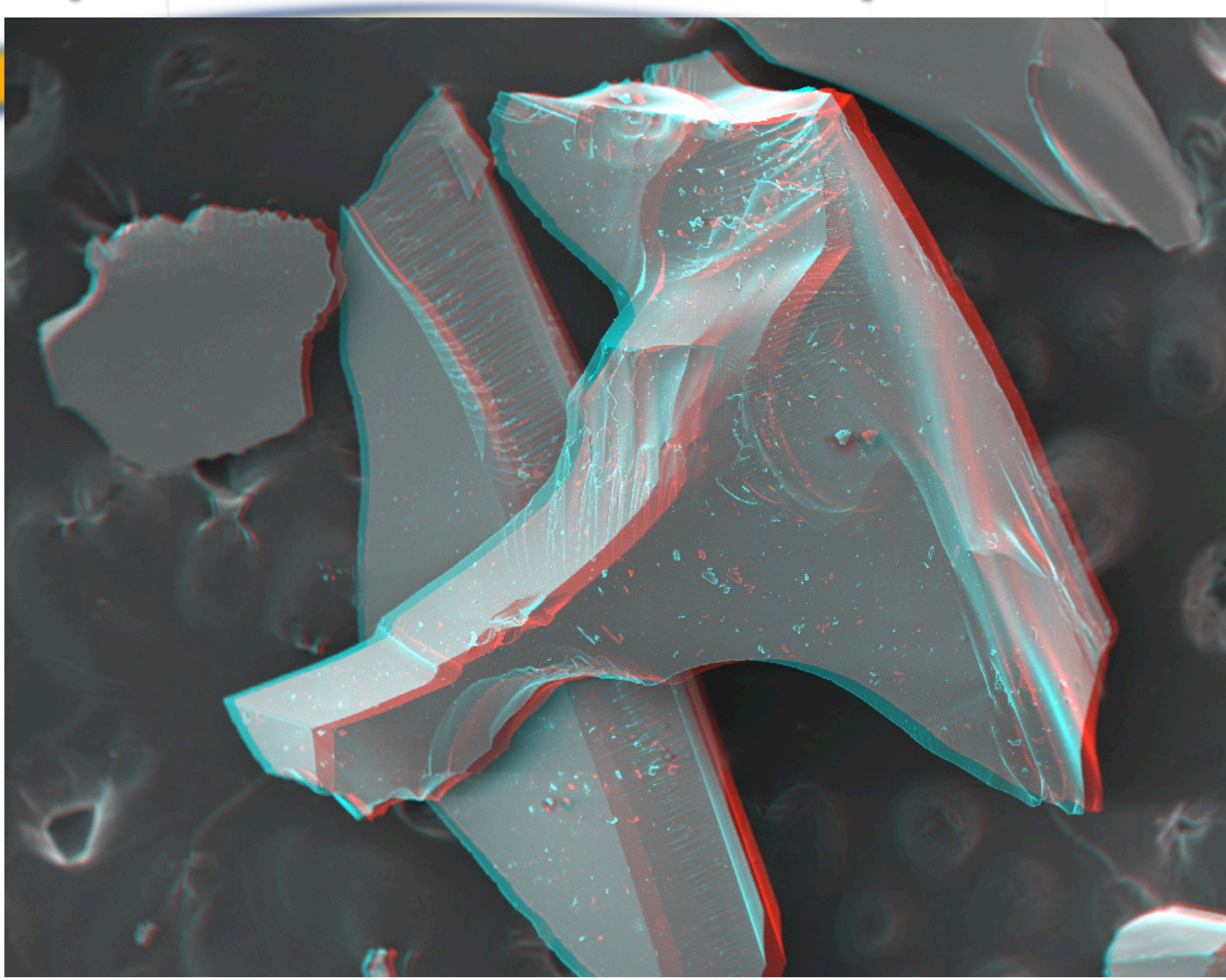
At% not Wt%

Improved Quant with Multiple Detectors



At% not Wt%

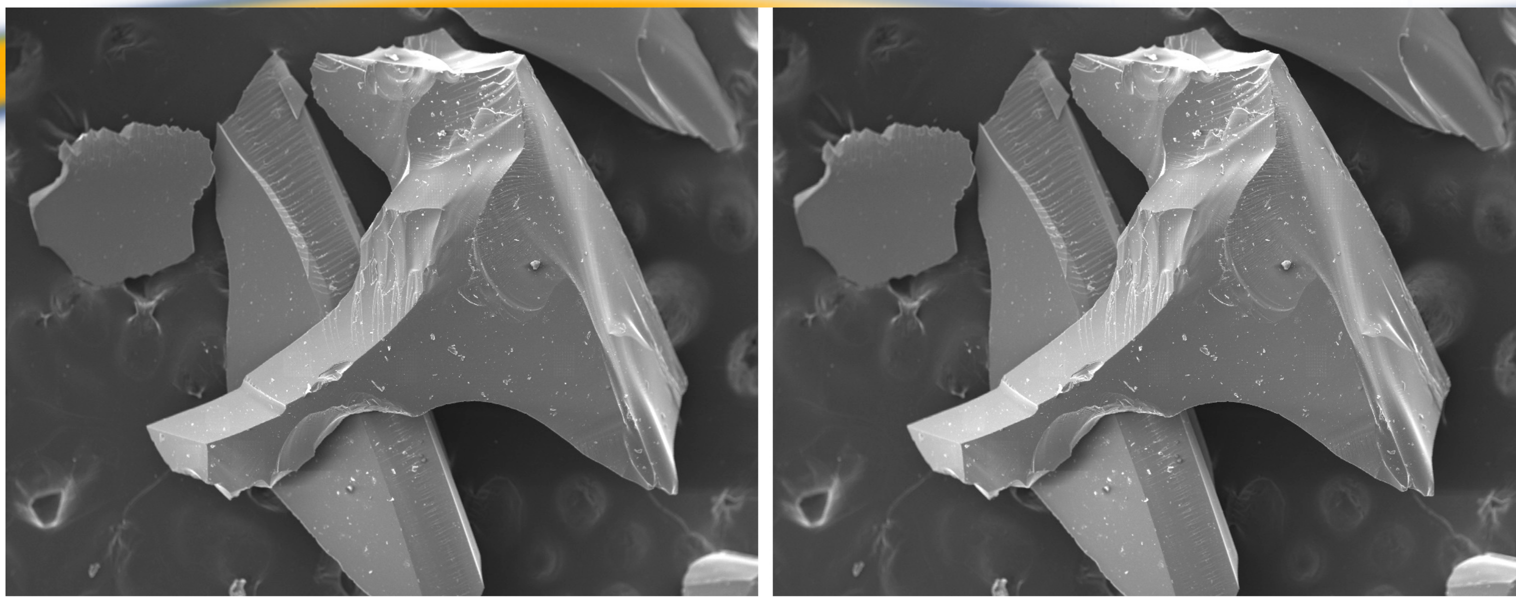
Improved Quant with Multiple Detectors



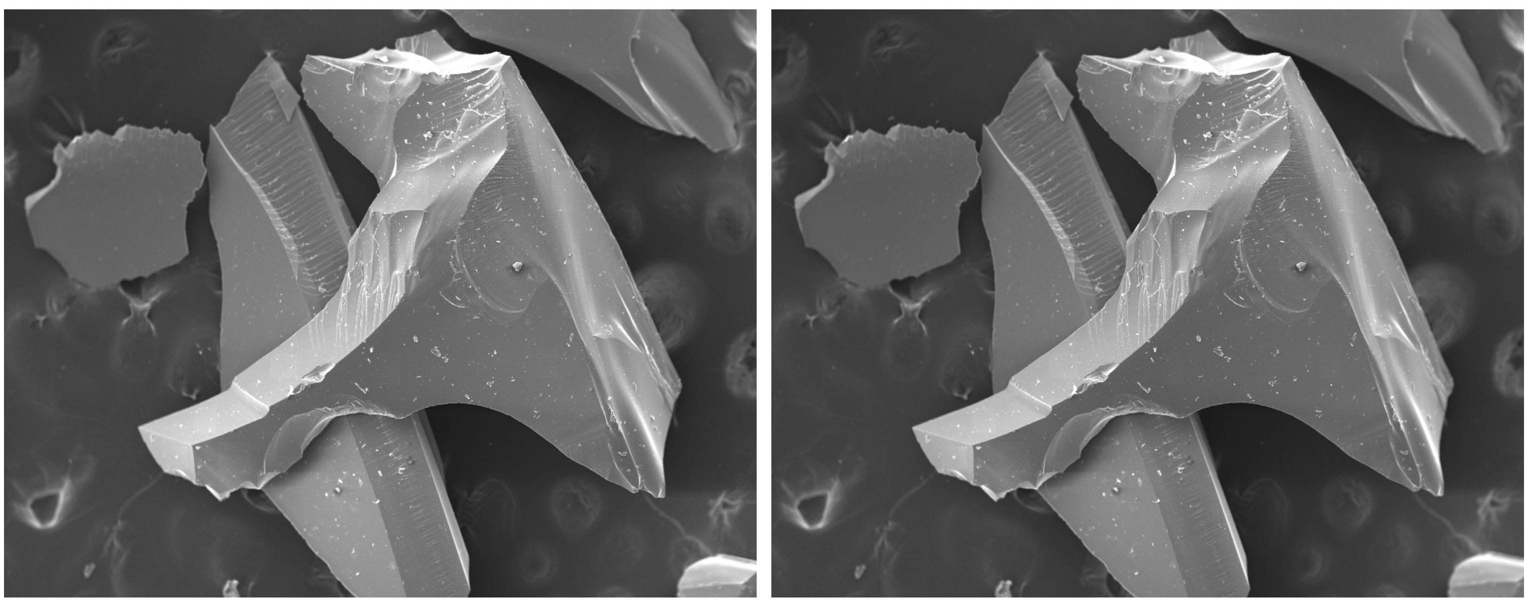
HFW= 2 mm

Improved Quant with Multiple Detectors

Stereo Pairs

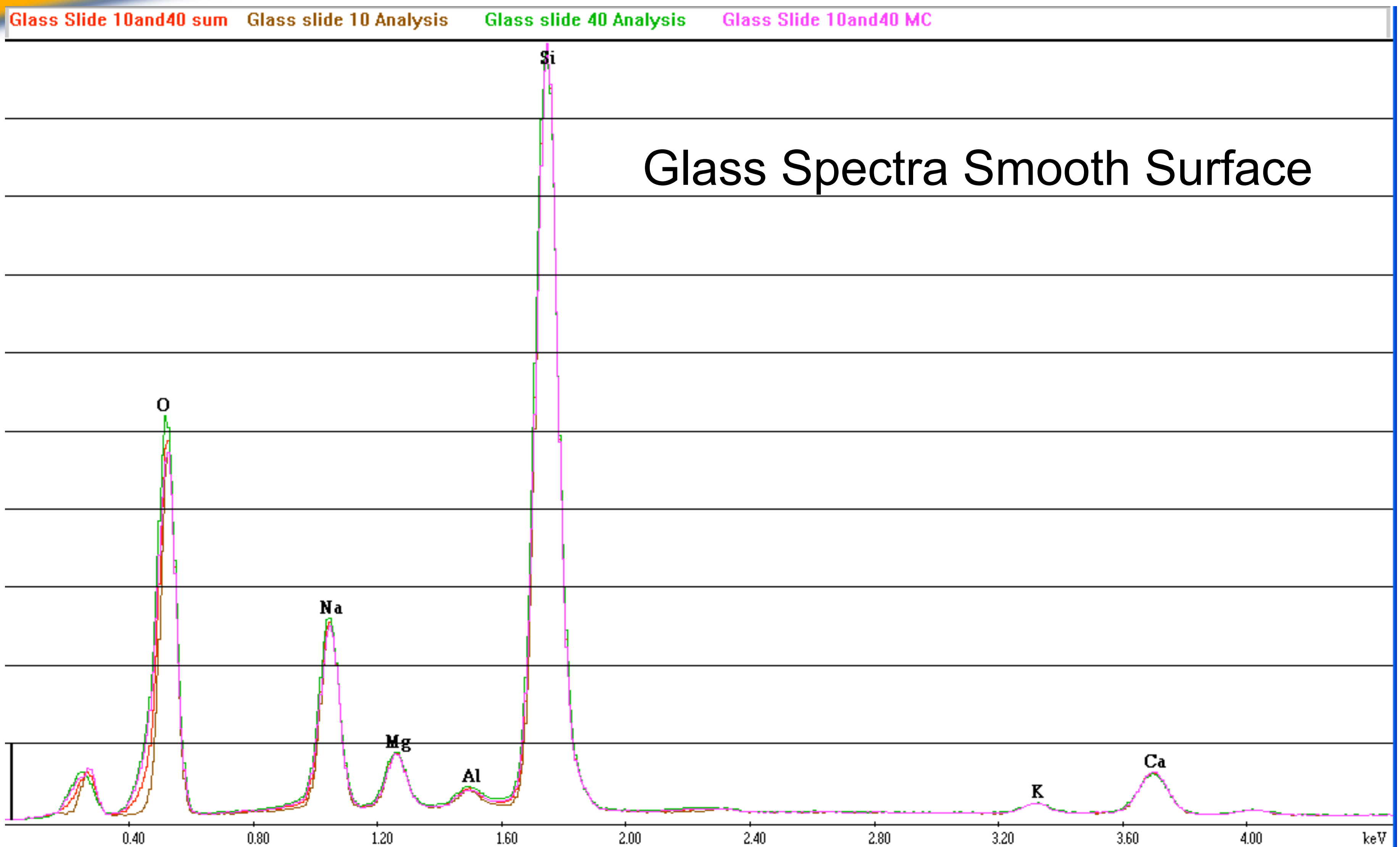


Straight-eyed

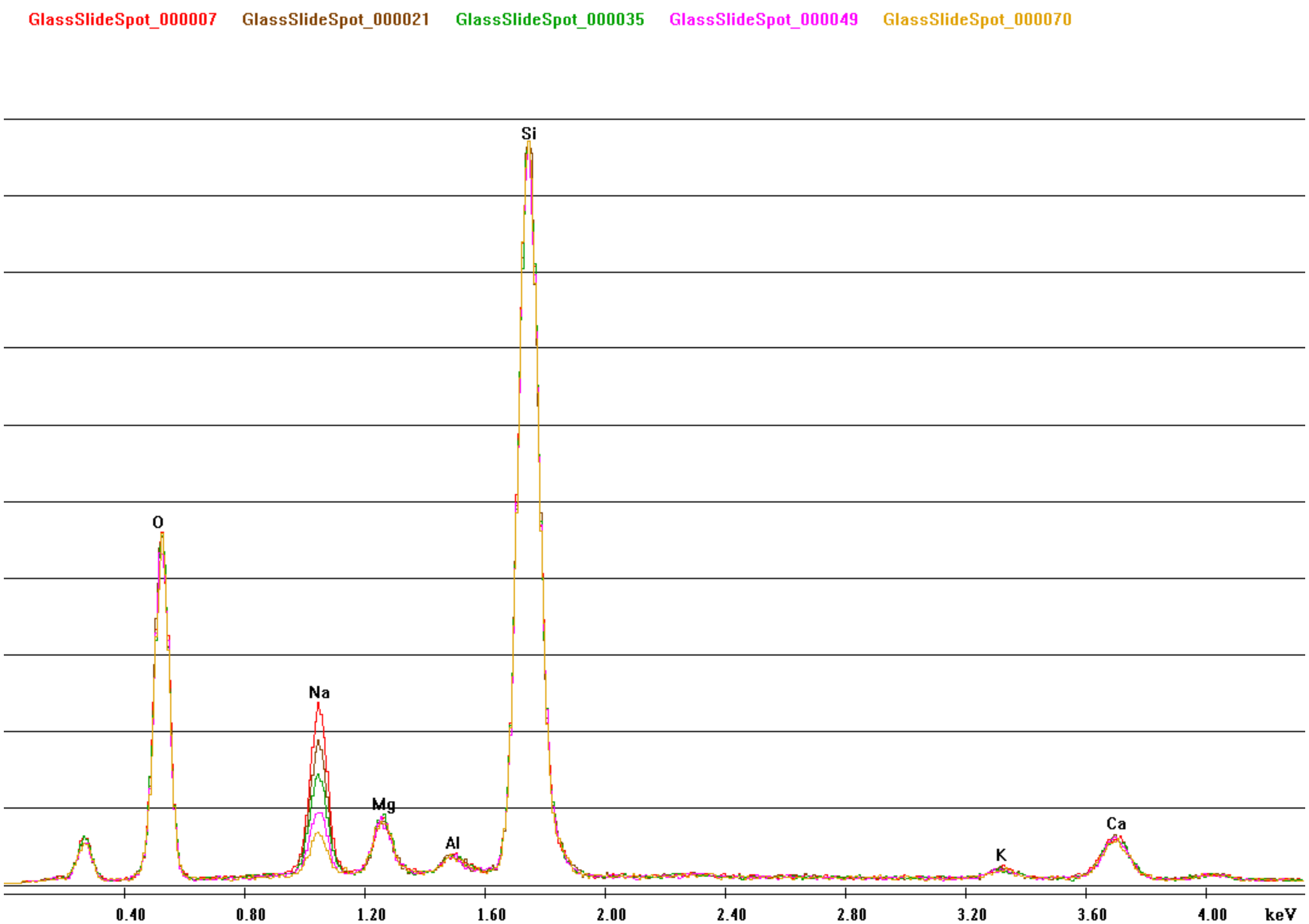


Cross-eyed

Improved Quant with Multiple Detectors



Improved Quant with Multiple Detectors

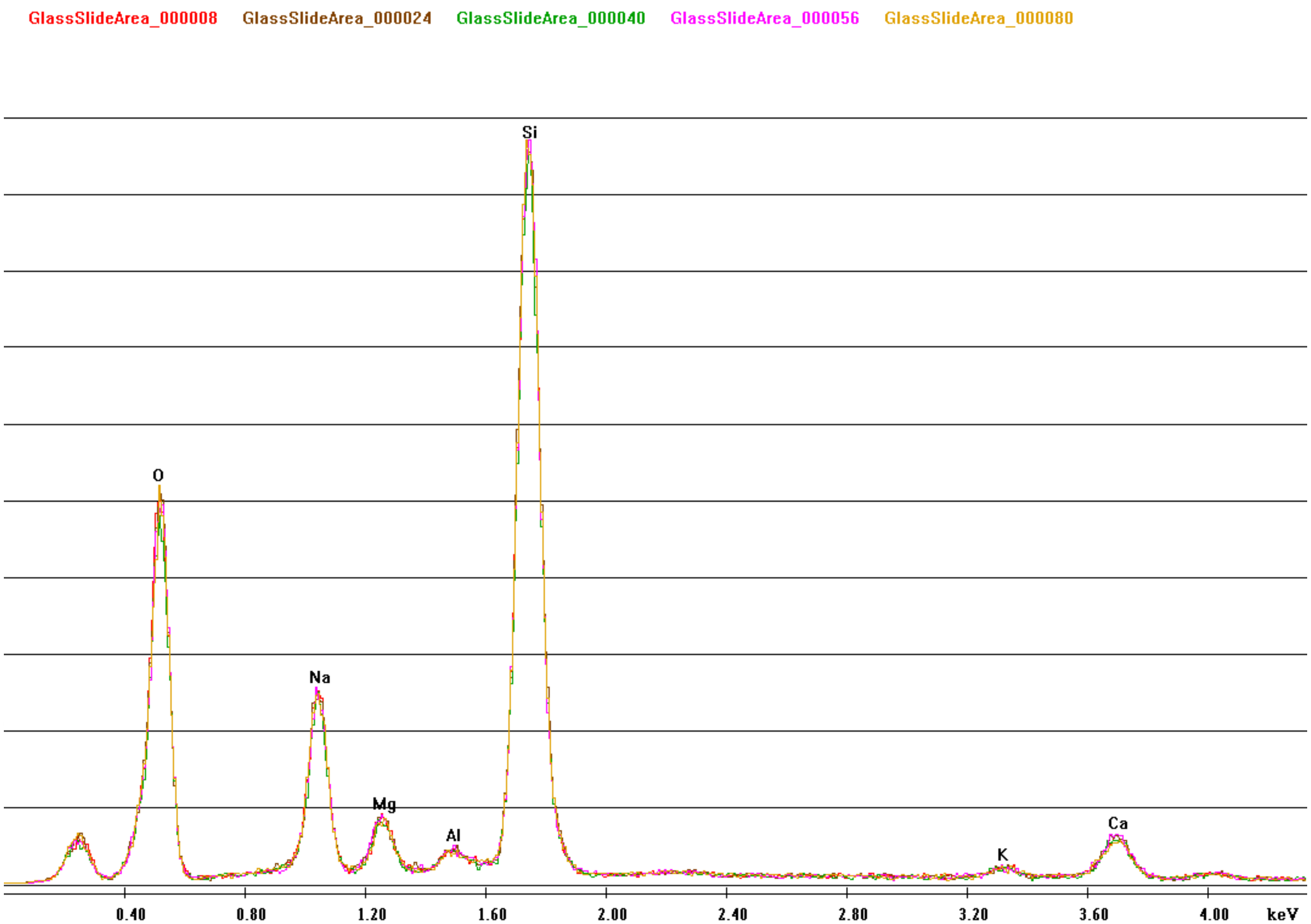


Spot Mode Very Bad!

Note the volatility of the Na.

15 kV, 2-3 nA.

Improved Quant with Multiple Detectors

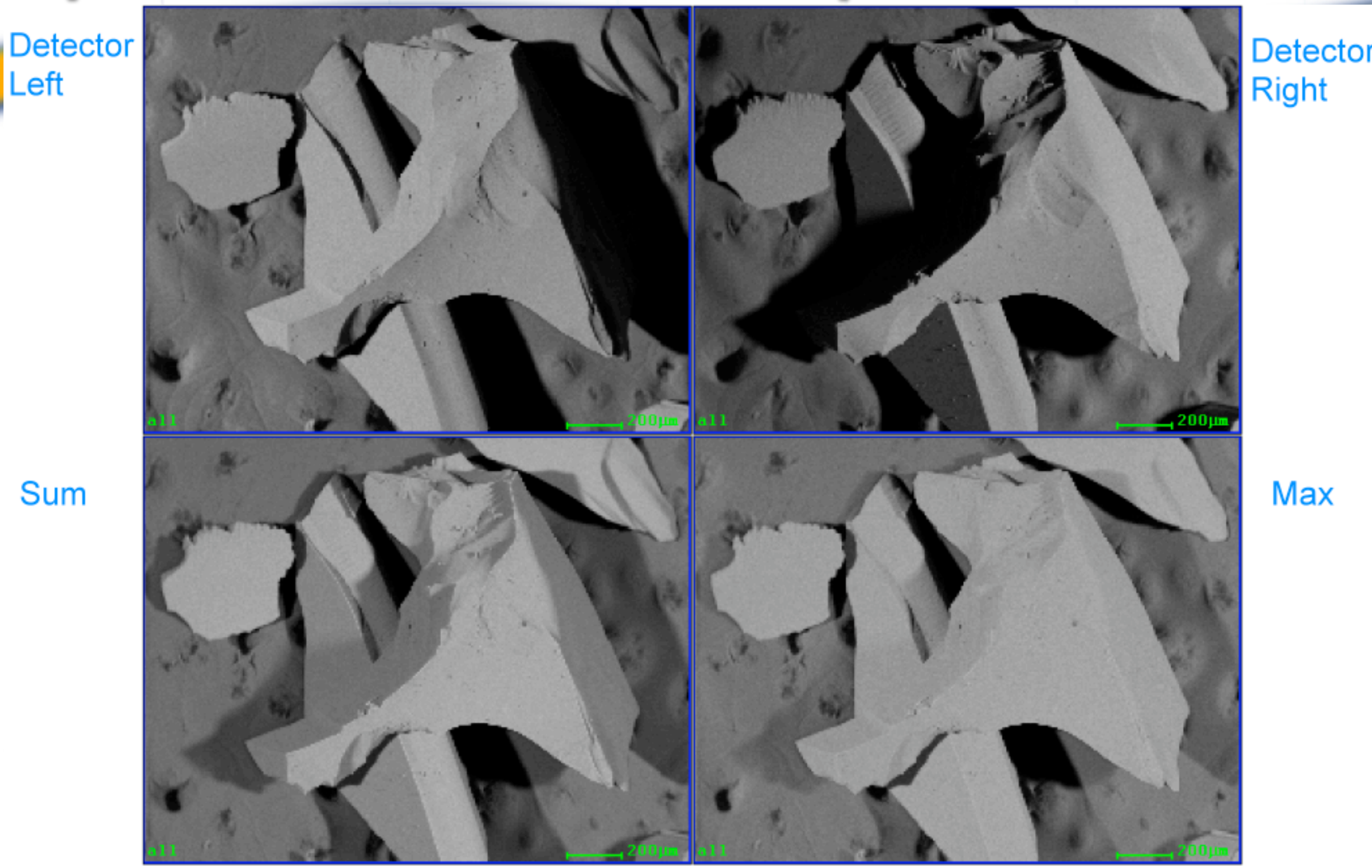


Selected Area Not So Bad!

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

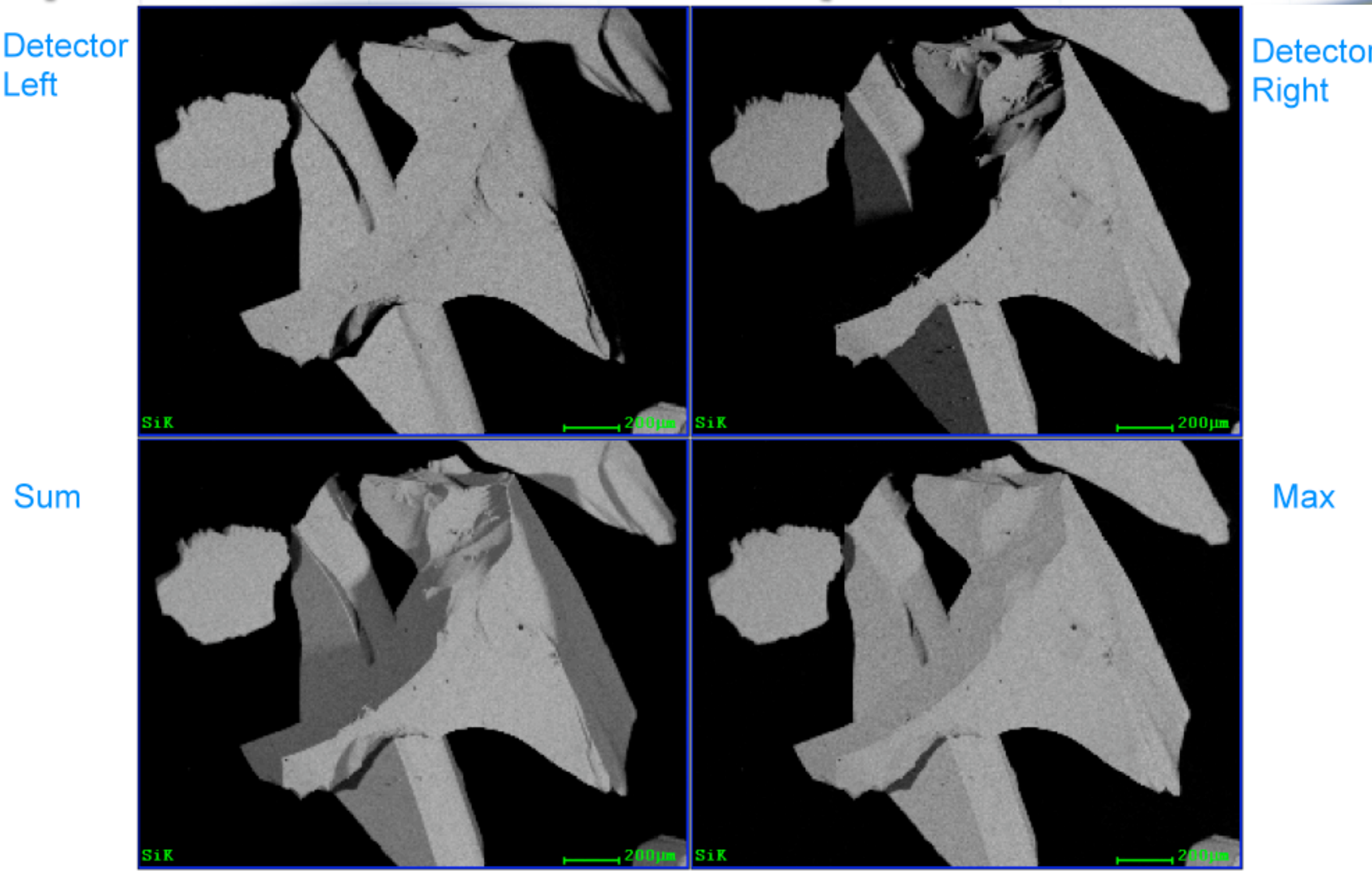
--Albite

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

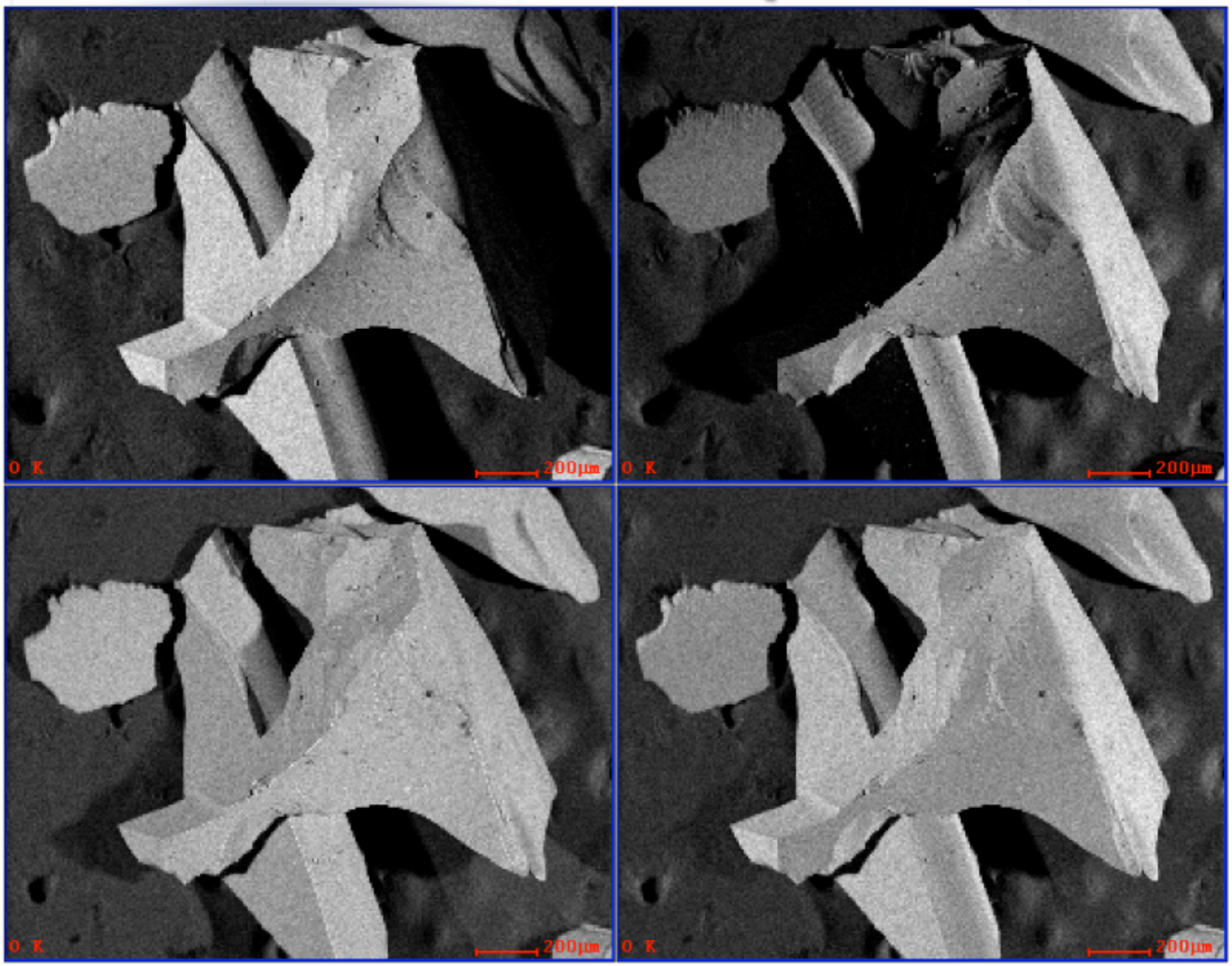


Maps of the Si K ROI.

Improved Quant with Multiple Detectors

Detector Left

Detector Right



Sum

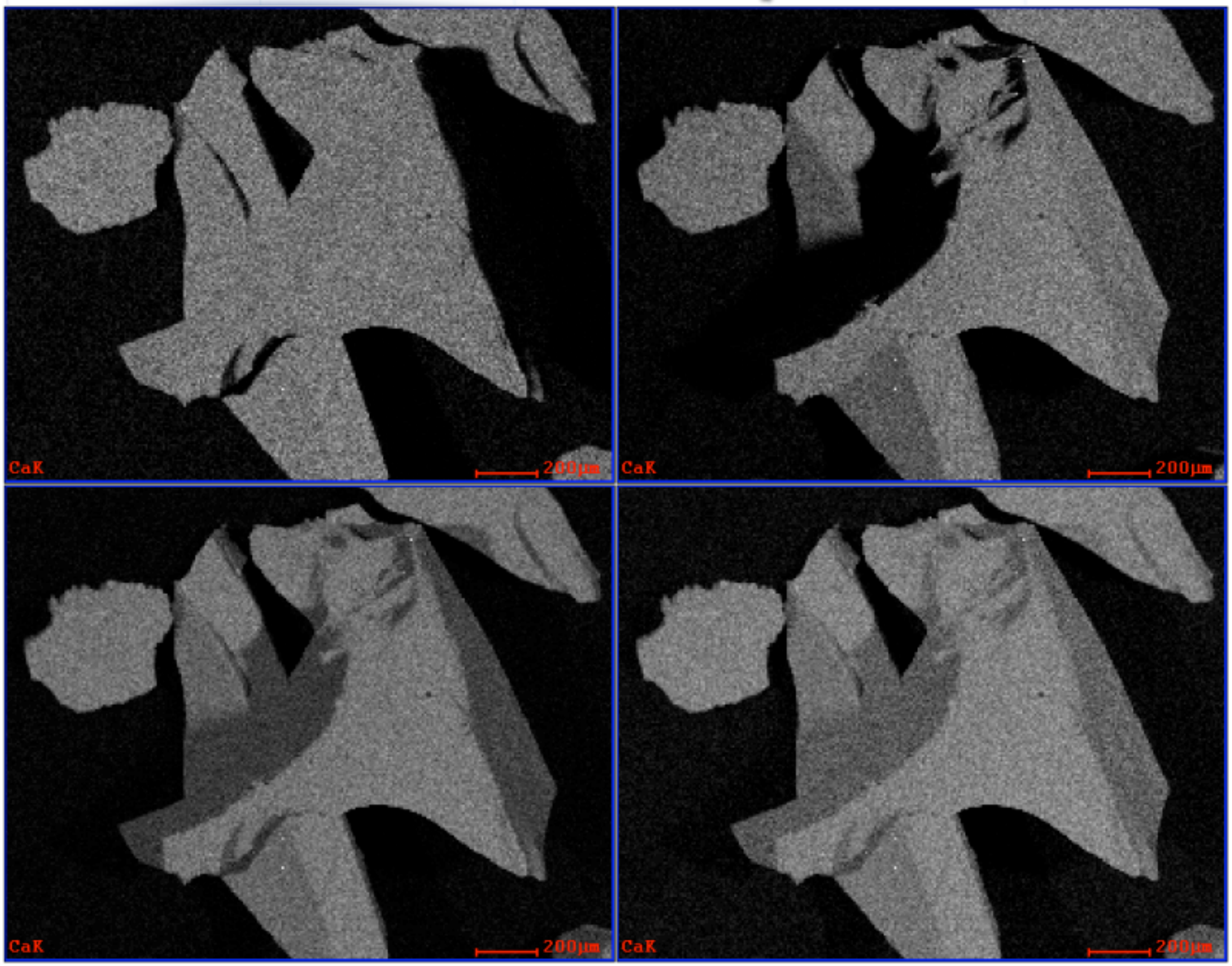
Max

Maps of the O K ROI.

Improved Quant with Multiple Detectors

Detector Left

Detector Right

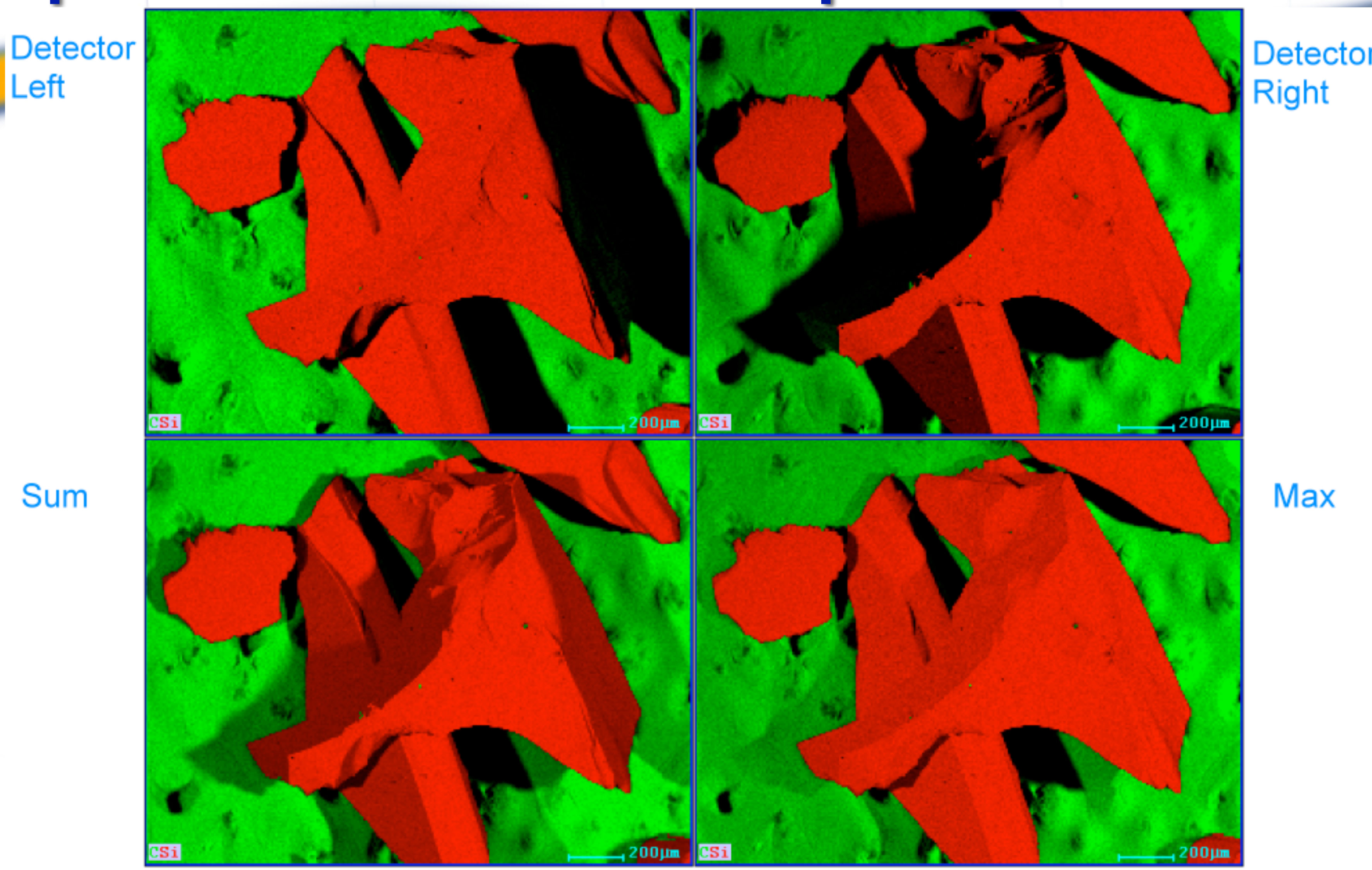


Sum

Max

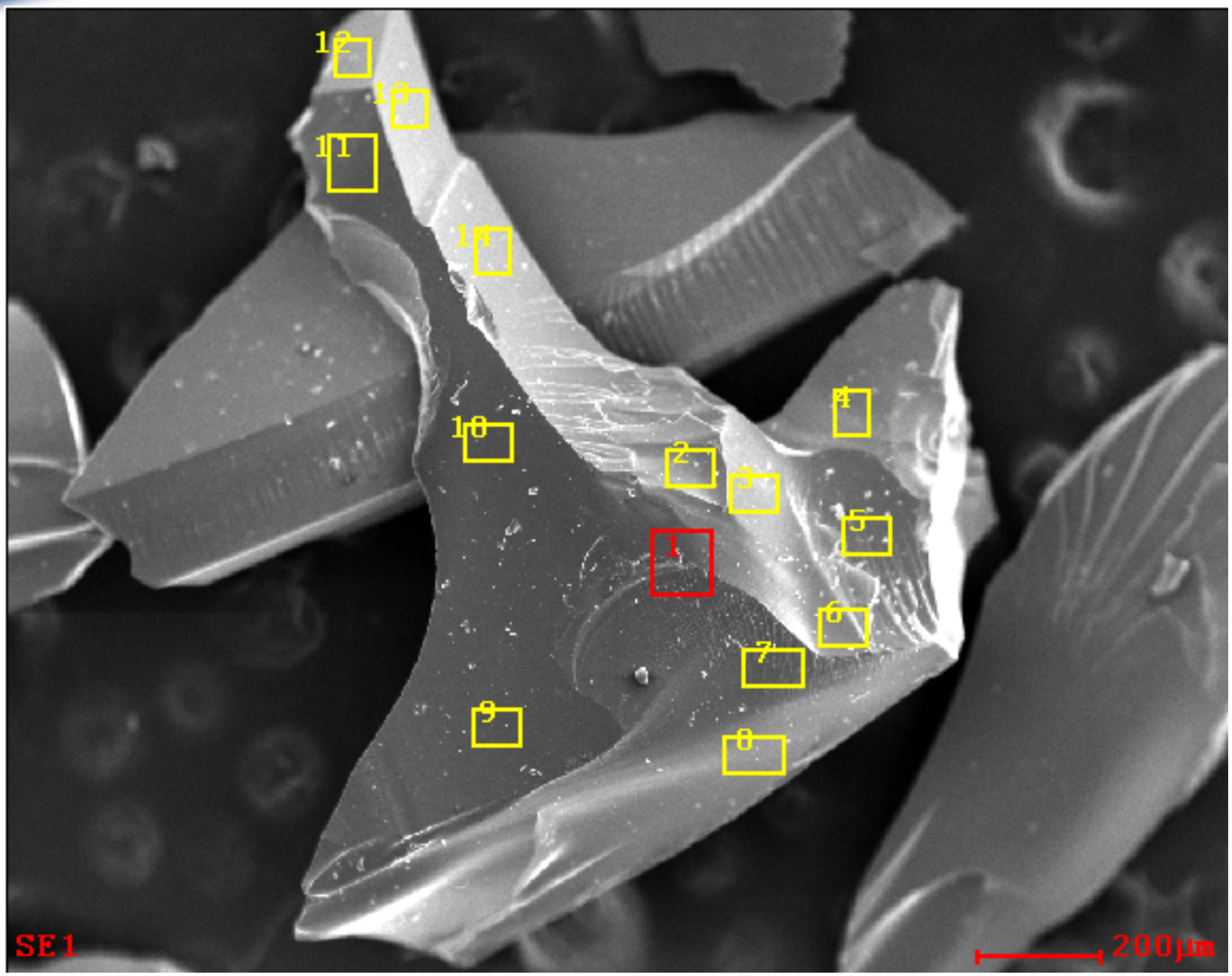
Maps of the Ca K ROI.

Improved Quant with Multiple Detectors



Overlay of the C and Si (ROI) maps.

Improved Quant with Multiple Detectors

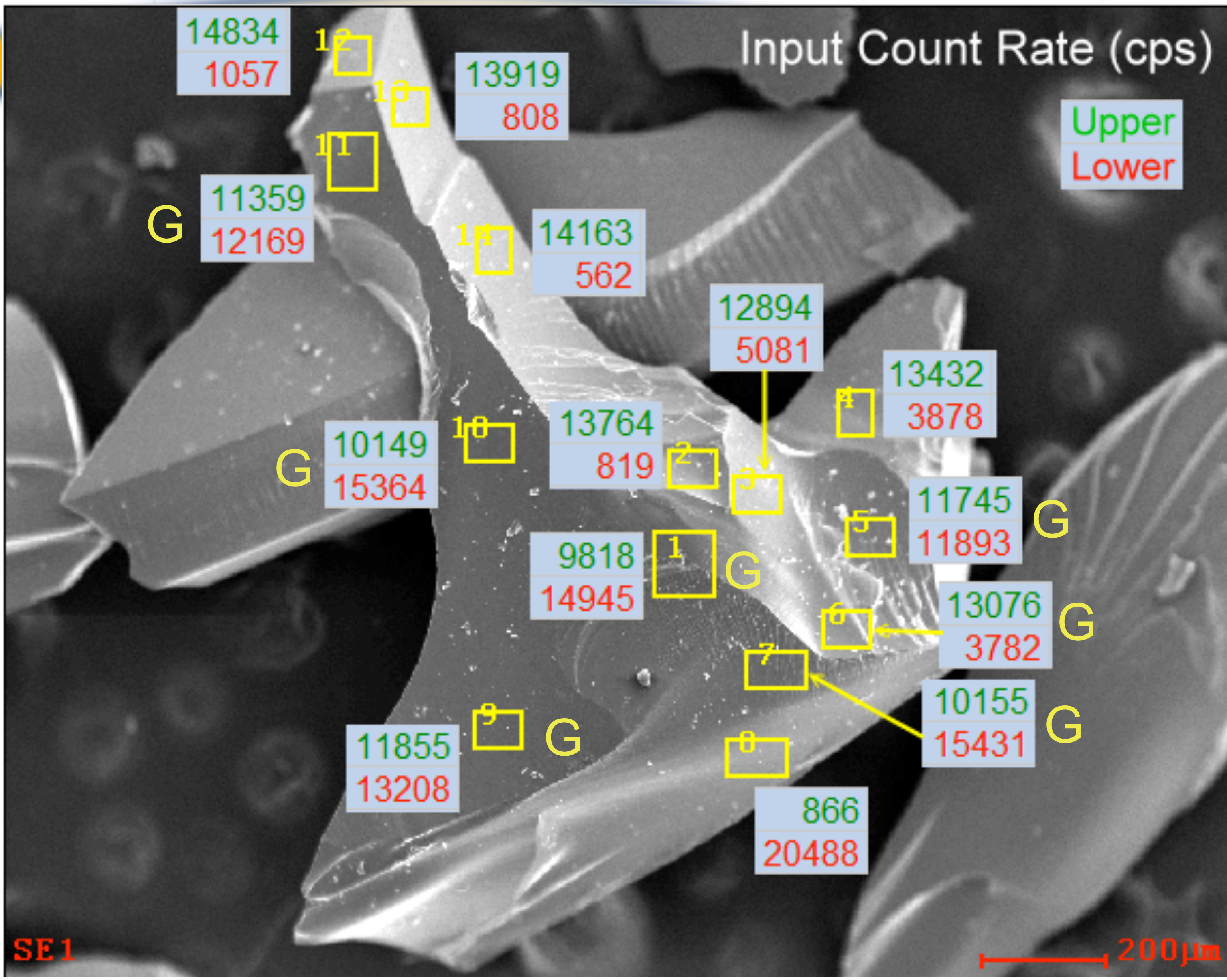


Good Sum Spectra

1, 5, 6, 7, 9, 10, 11

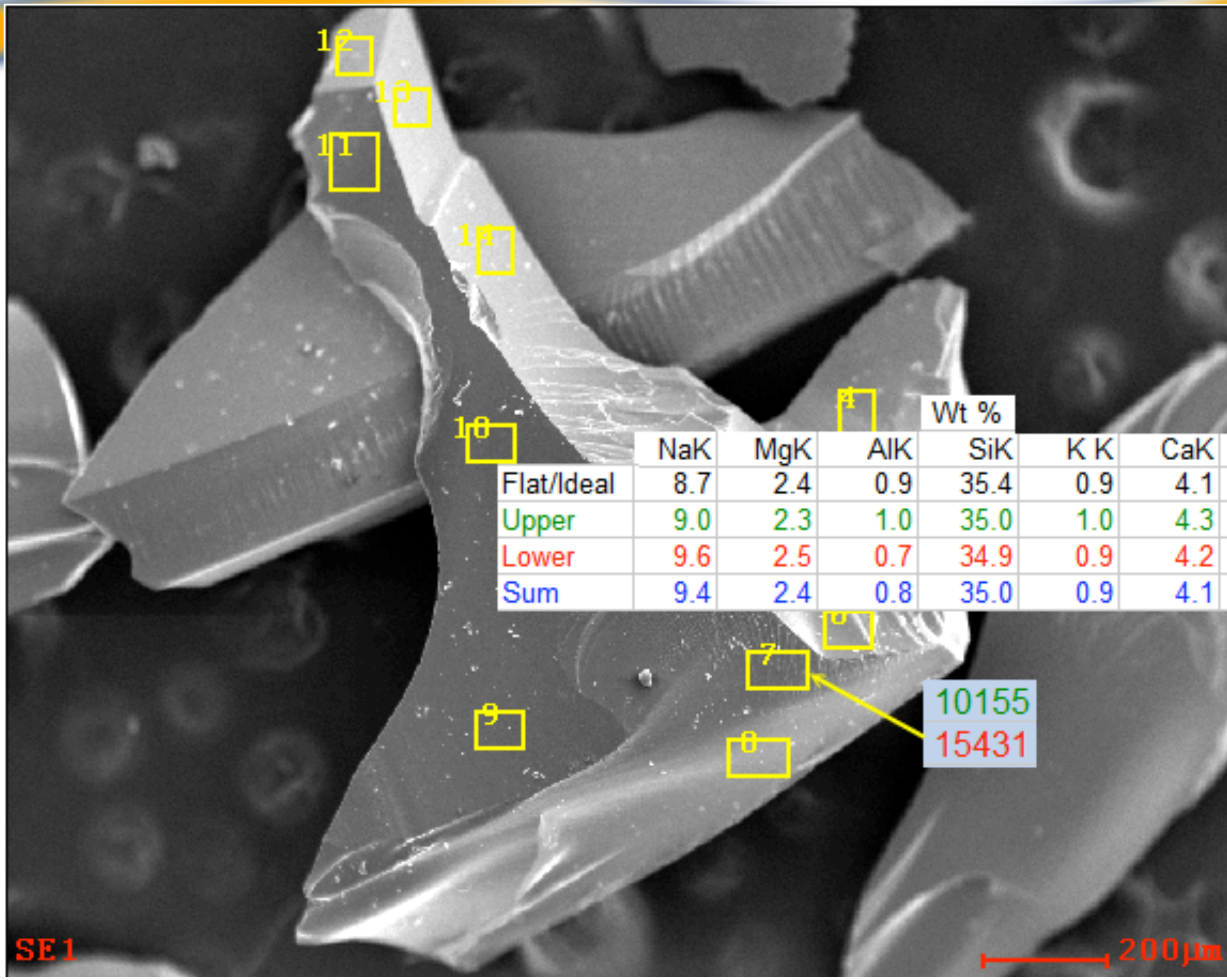
Also, note the rotation of the image from the maps. The detectors are now above and below.

Improved Quant with Multiple Detectors



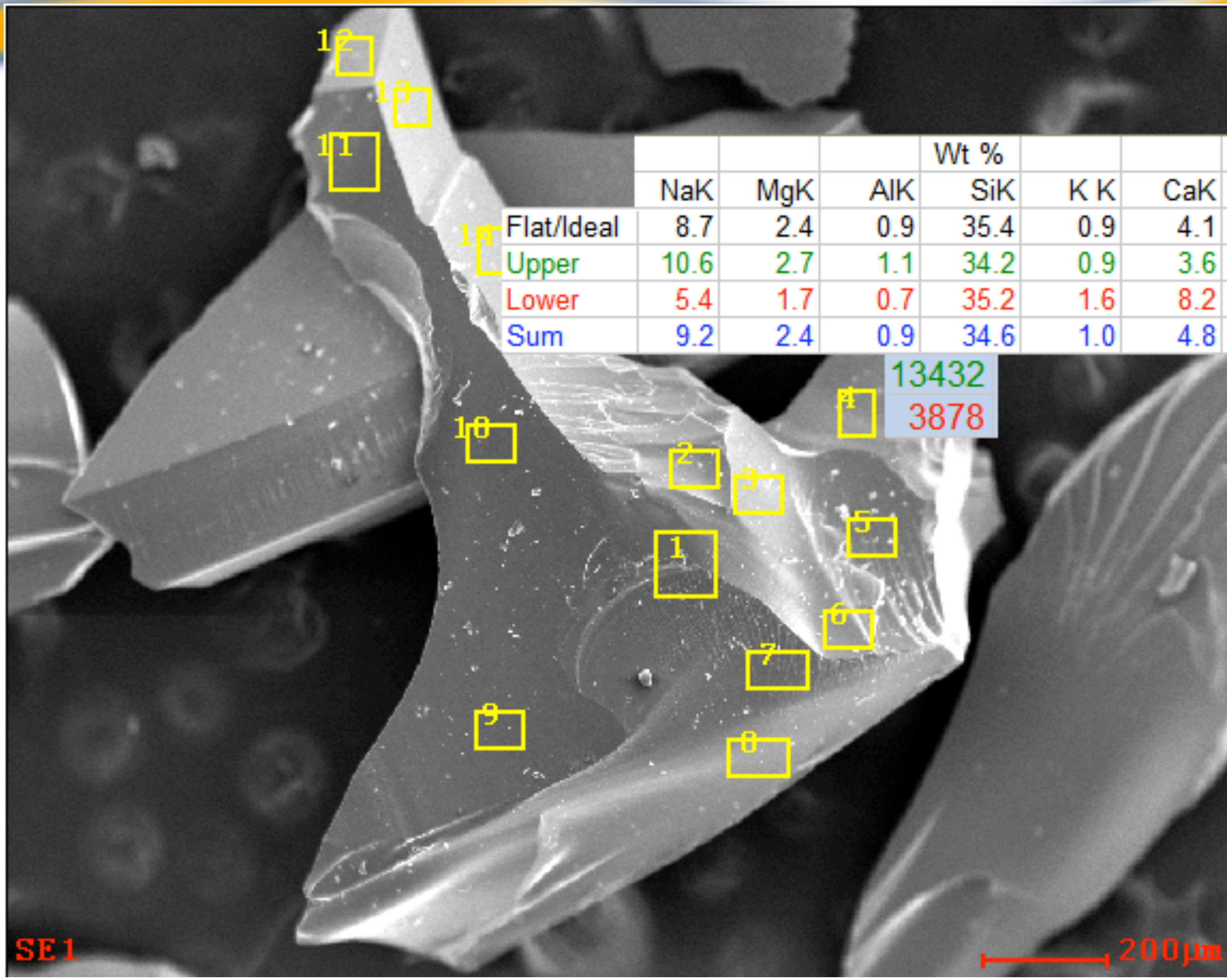
G=good sum spectrum

Improved Quant with Multiple Detectors



	NaK	MgK	AlK	SiK	K K	CaK
Wt %						
Flat/Ideal	8.7	2.4	0.9	35.4	0.9	4.1
Upper	9.0	2.3	1.0	35.0	1.0	4.3
Lower	9.6	2.5	0.7	34.9	0.9	4.2
Sum	9.4	2.4	0.8	35.0	0.9	4.1

Improved Quant with Multiple Detectors



	NaK	MgK	AlK	SiK	K K	CaK
				Wt %		
Flat/Ideal	8.7	2.4	0.9	35.4	0.9	4.1
Upper	10.6	2.7	1.1	34.2	0.9	3.6
Lower	5.4	1.7	0.7	35.2	1.6	8.2
Sum	9.2	2.4	0.9	34.6	1.0	4.8

Improved Quant with Multiple Detectors

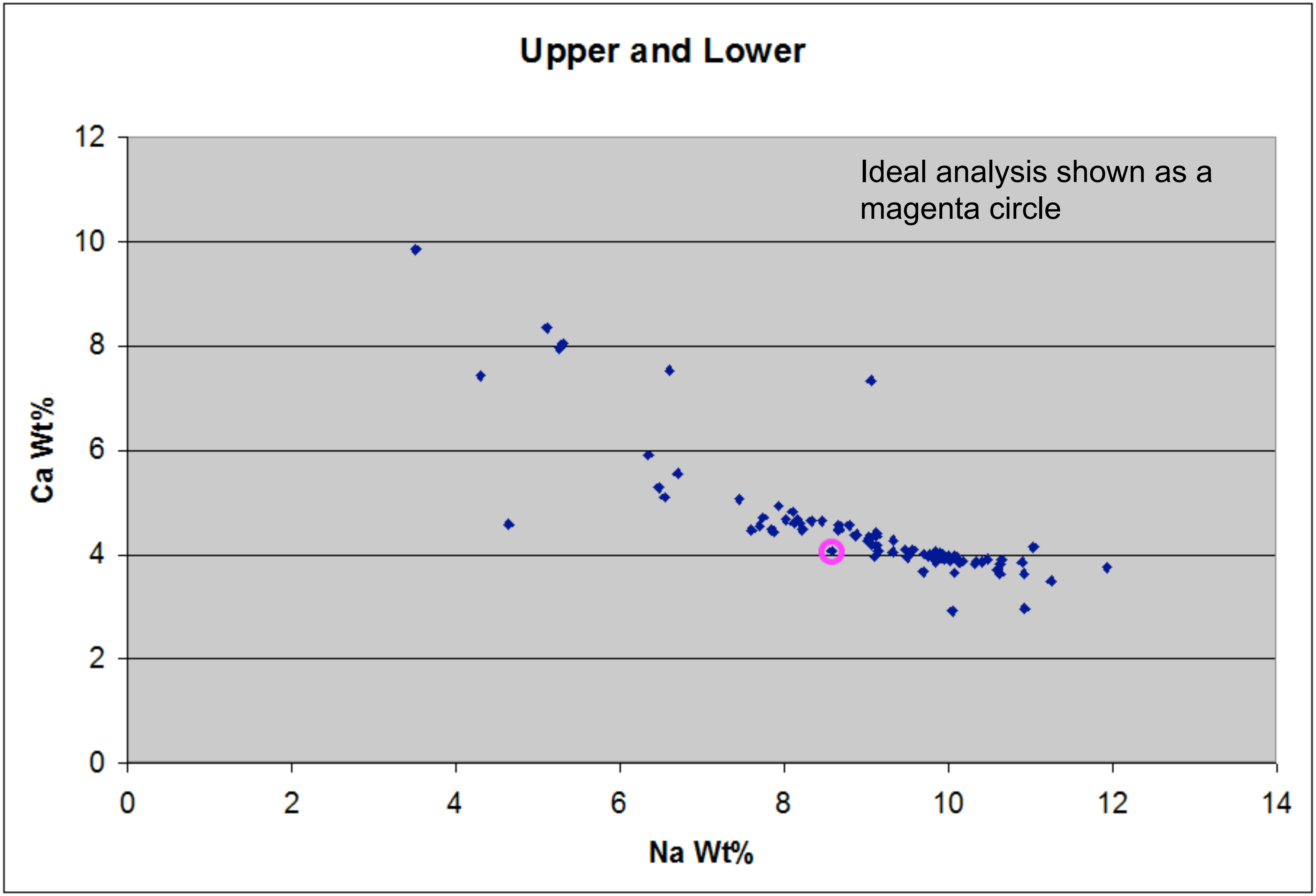
Weight % of Glass Analyses

	NaK	MgK	AlK	SiK	K K	CaK	O K*	
Ideal Flat	8.6	2.4	0.8	35.6	0.9	4.1	47.7	
Single	8.9	2.4	1.4	34.5	1.0	4.5	47.2	n=82
Analysis	1.7	0.5	1.7	1.5	0.3	1.2	0.4	
Sum	9.2	2.4	1.1	34.9	0.9	4.2	47.4	n=41
	0.7	0.2	0.7	0.6	0.1	0.4	0.2	
Max	9.6	2.5	1.1	34.7	0.9	4.0	47.2	n=41
	0.8	0.2	0.8	0.7	0.1	0.4	0.2	

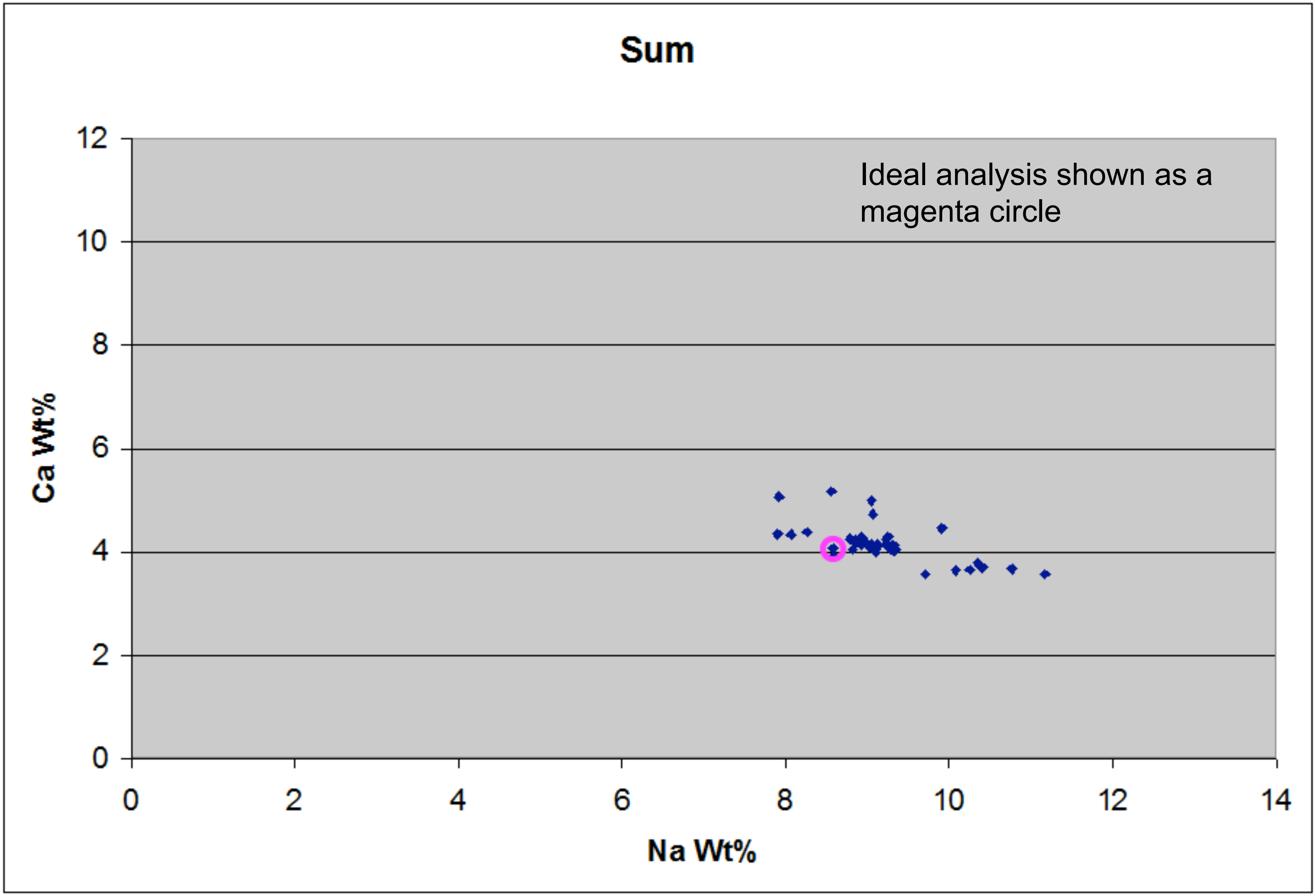
*Oxygen determined by stoichiometry

Mean
Std. Dev.

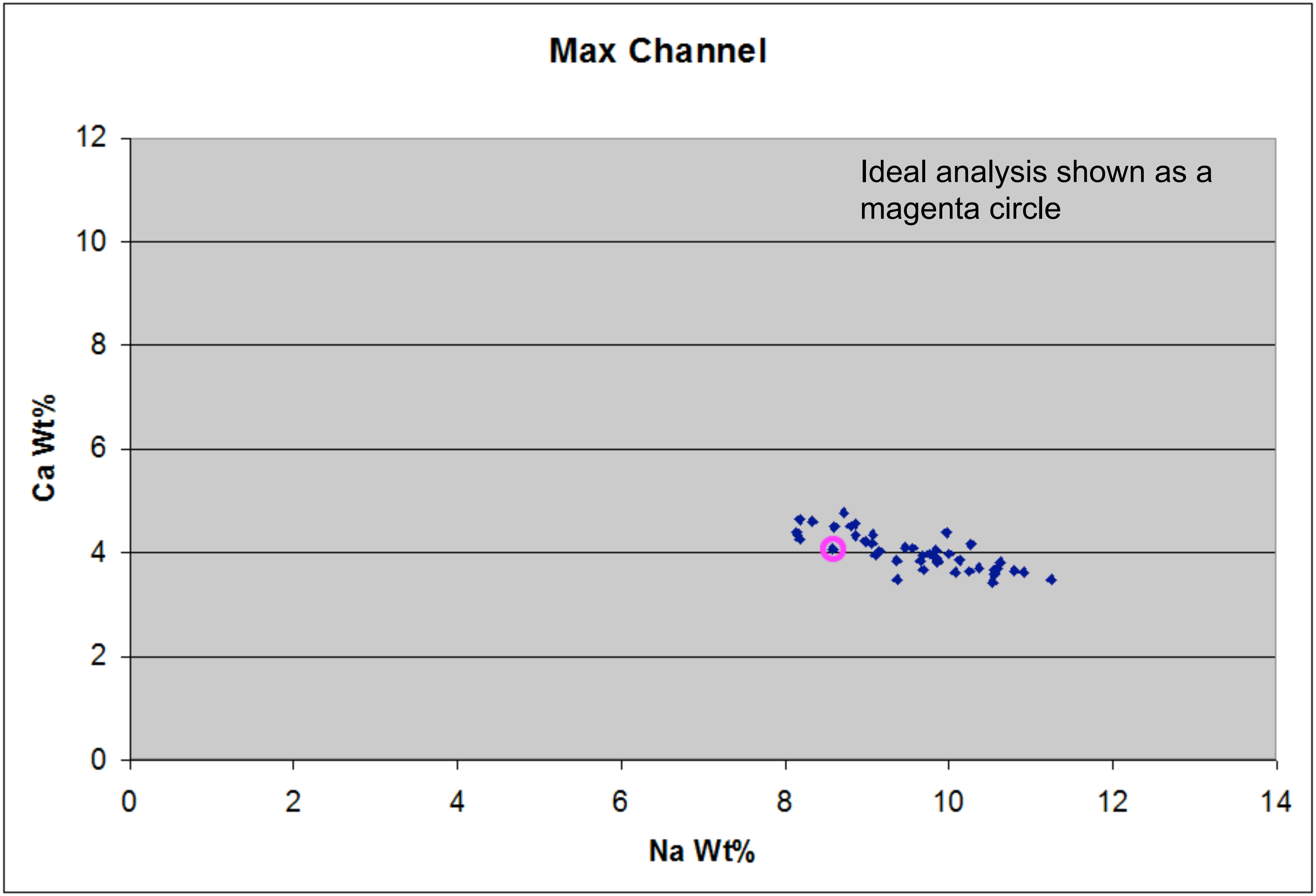
Improved Quant with Multiple Detectors



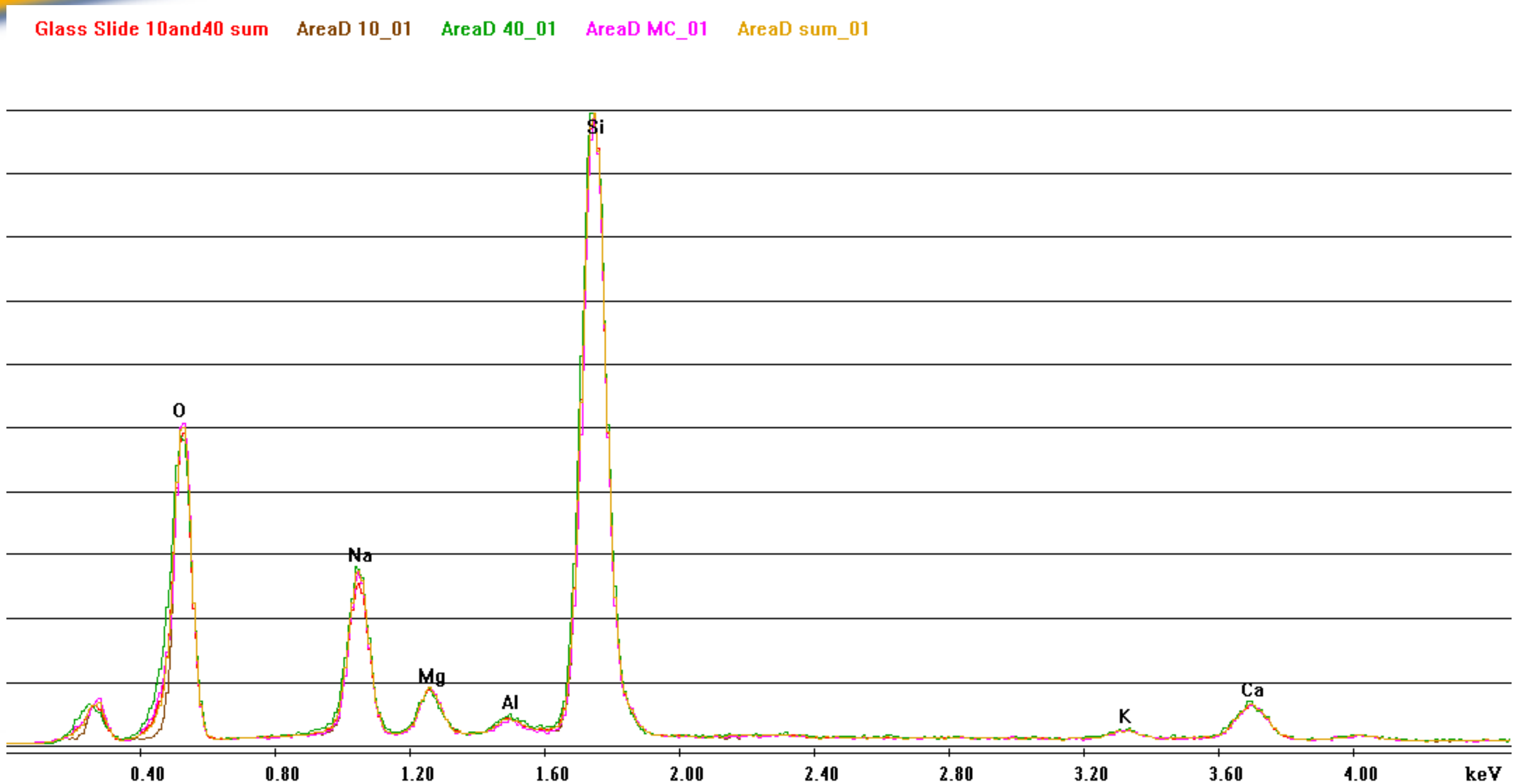
Improved Quant with Multiple Detectors



Improved Quant with Multiple Detectors

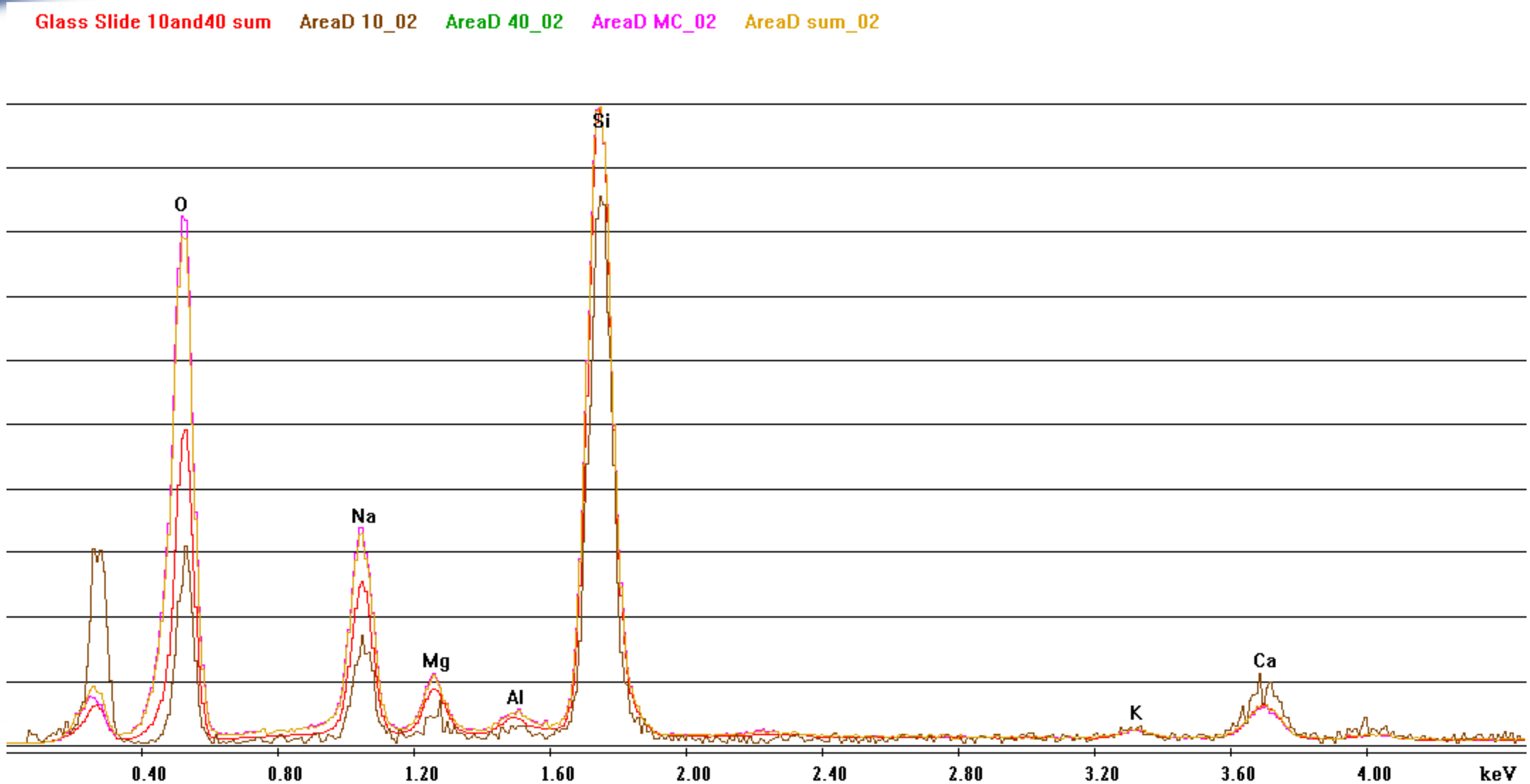


Improved Quant with Multiple Detectors



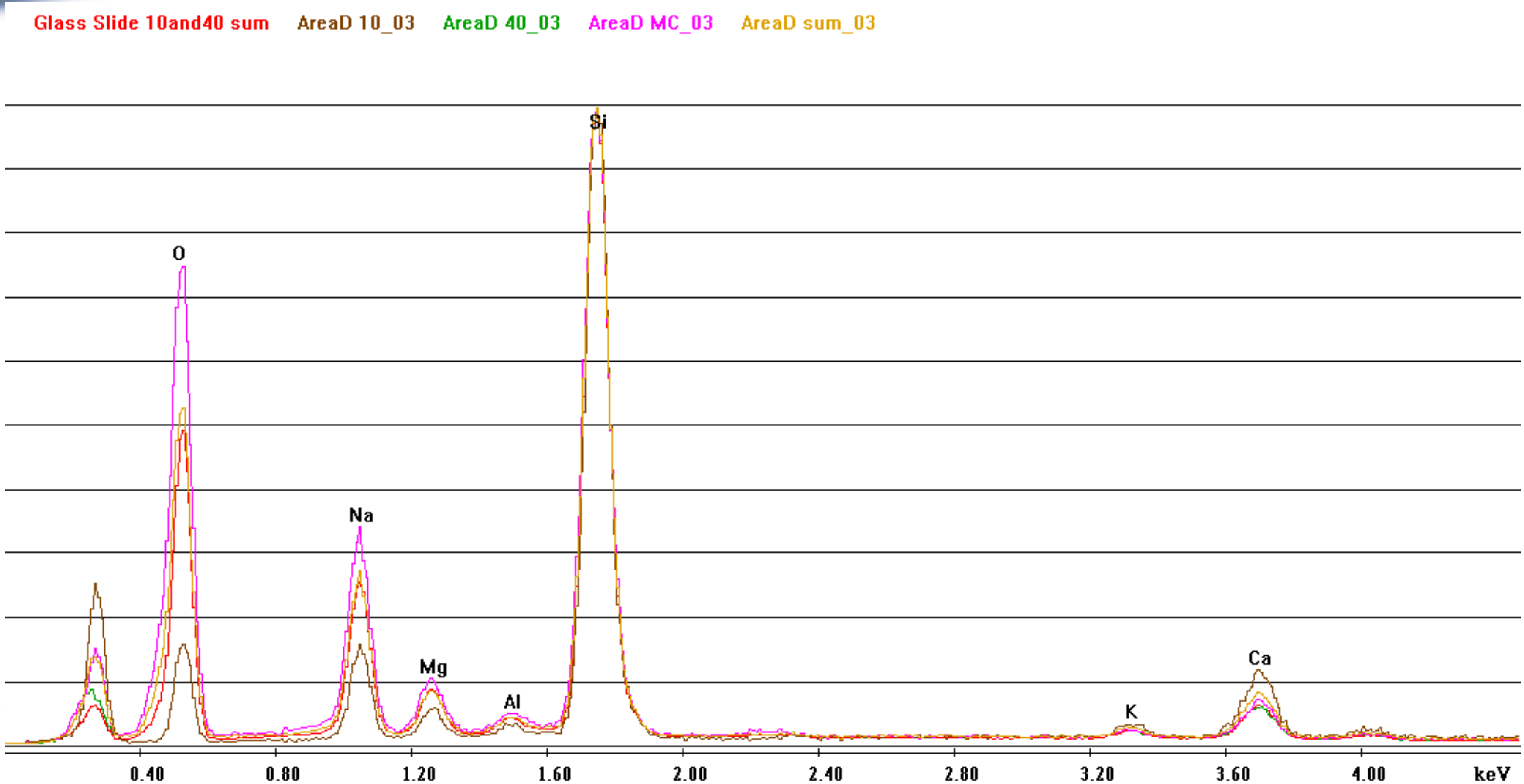
Spectra collected from, or derived from point 1. The count rates were 10 kcps vs 15 kcps but the spectra are very similar.

Improved Quant with Multiple Detectors



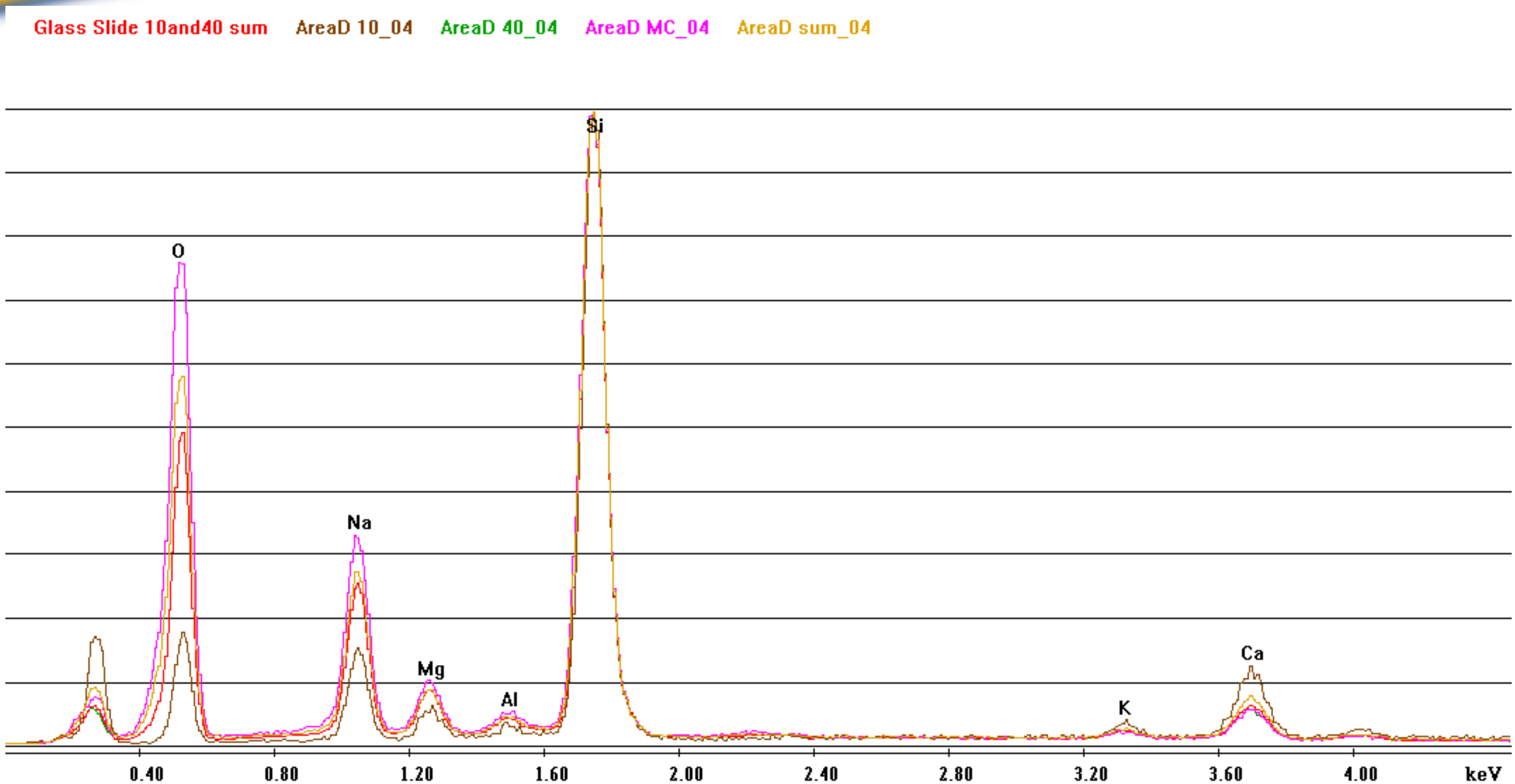
Spectra collected from, or derived from point 2. The count rates were 1 kcps vs 14 kcps and the spectra are very dissimilar.

Improved Quant with Multiple Detectors



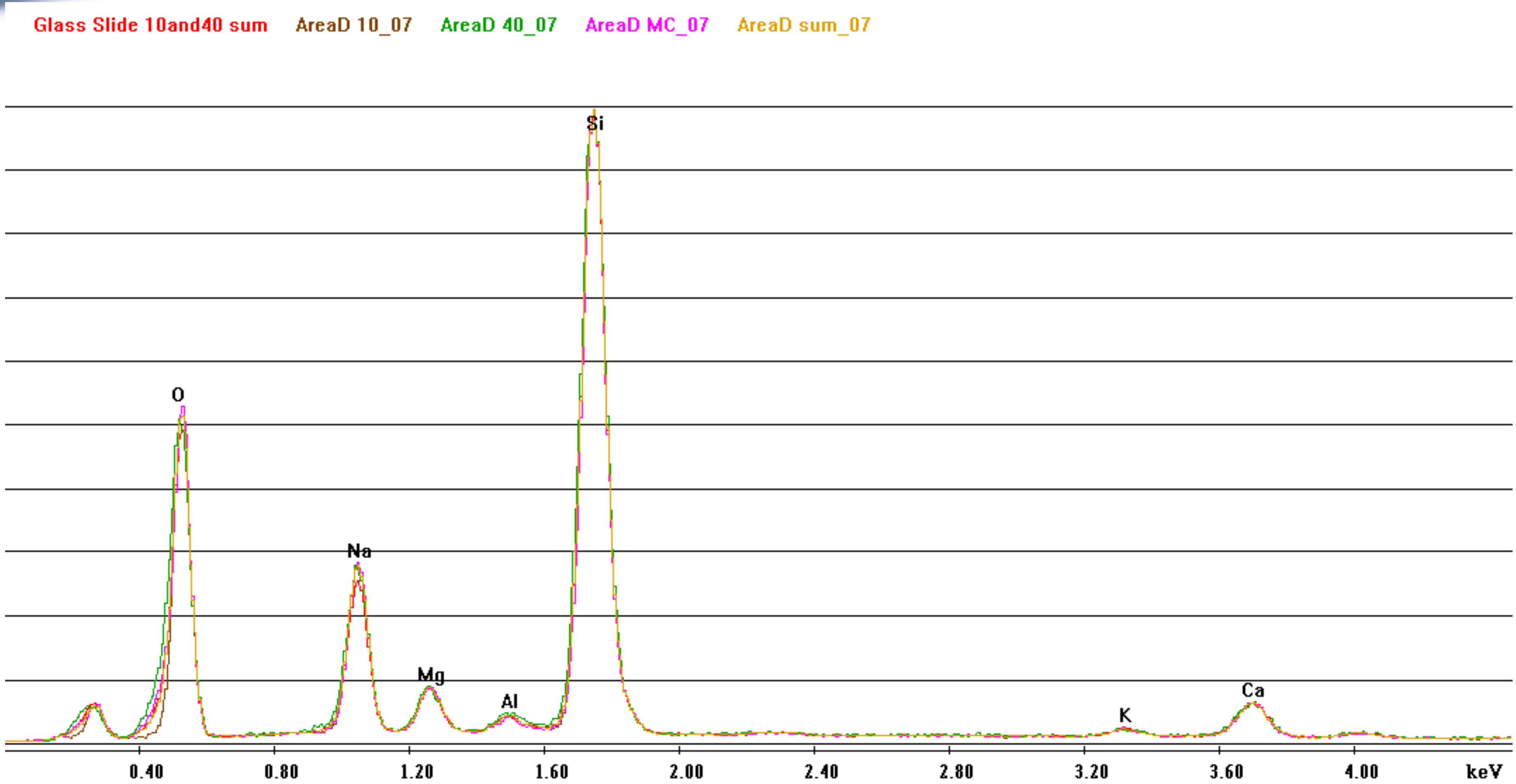
Spectra collected from, or derived from point 3. The count rates were 5 kcps vs 13 kcps and the spectra are very dissimilar.

Improved Quant with Multiple Detectors



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.

Improved Quant with Multiple Detectors

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).