COVALENT METROLOGY

CM000015583 – Nanoshell SEM/EDS-COSEVA Advanced TRS

30 September, 2020

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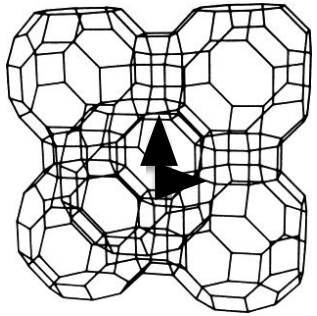
Contents

Job Description

- Determine the elemental composition of particles in liquid suspension
  - Make special note of similarity or dissimilarity to zeolite, especially clinoptilolite
  - If clinoptilolite is found, the sample is to be compared to a reference

Clinoptilolite

Clinoptilolite:
$(Na,K,Ca)_{2\text{-}3}Al_3(Al,Si)_2Si_{13}O_{36}\cdot 12H_2O$



Dimensions of void space
0.7-0.9 nm

Range of Possible Stoichiometries for Clinoptilolite:

$(Na,K,Ca)_{2\text{-}3}Al_3Si_{15}O_{36}\cdot 12H_2O$

$(Na,K,Ca)_{2\text{-}3}Al_4Si_{14}O_{36}\cdot 12H_2O$

$(Na,K,Ca)_{2\text{-}3}Al_5Si_{13}O_{36}\cdot 12H_2O$

Typical reference compositional % for Clinoptilolite:

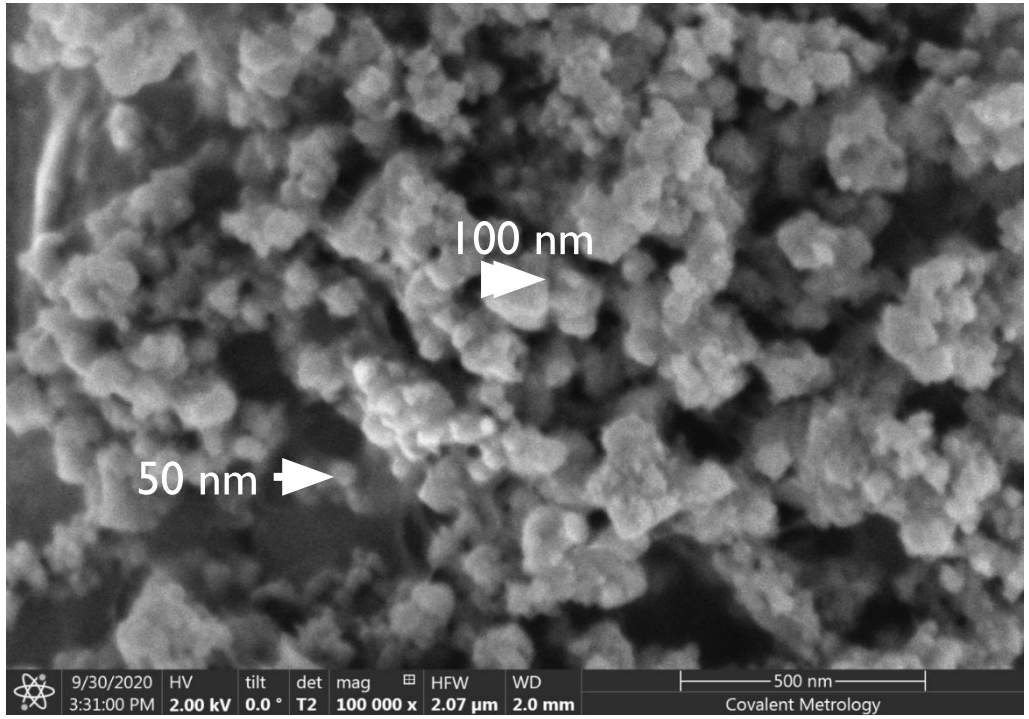
*Na, K, Ca, Al, and Si can (slightly) vary.

Element	Clinoptilolite
O	64
Na	2
Mg	0
Al	7
Si	25
S	0
K	2
Ca	2

Summary

- 3 sites are analyzed via EDS for composition with 5 kV incident beam
- After corrections, all display a reasonable match to clinoptilolite
- All particles show trace levels of Mg (used in processing buffer)
- Particles are clumped together due to drying on the Cu/C specimen plate.
- Particles range in size from 20 to 100 nm

Note: Corrections were applied post-measurement and therefore are not represented in the visualized spectra of this presentation.



Composition*, Atomic %

Element	Part. 1	Part. 2	Part. 3	Average	Clinoptilolite
O	63	62	62	62	64
Na	3	2	3	3	2
Mg	1	1	1	1	0
Al	6	6	6	6	7
Si	26	27	27	26	25
S	0	0	0	0	0
K	0	0	0	1	2
Ca	1	1	1	2	2

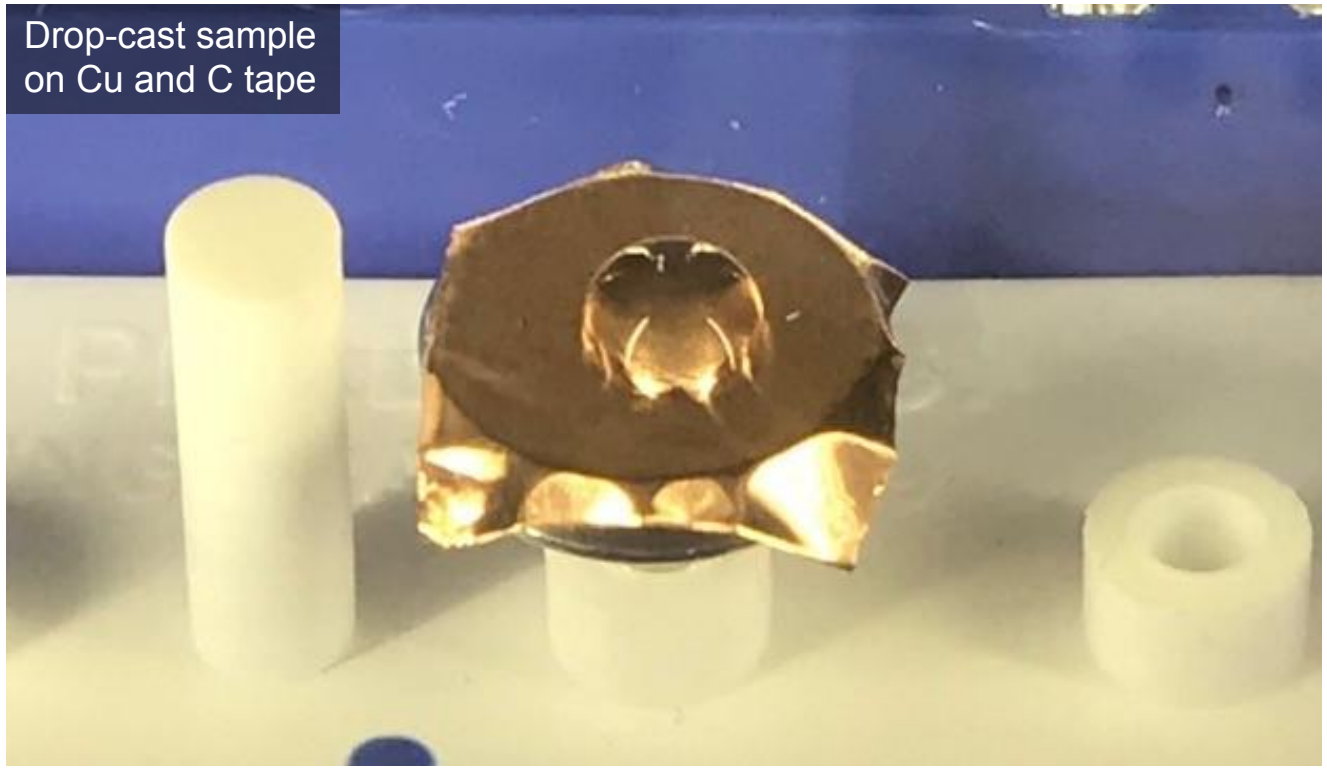
*Calculation includes background subtraction (Cu and C) and carbon subtraction (~22%)

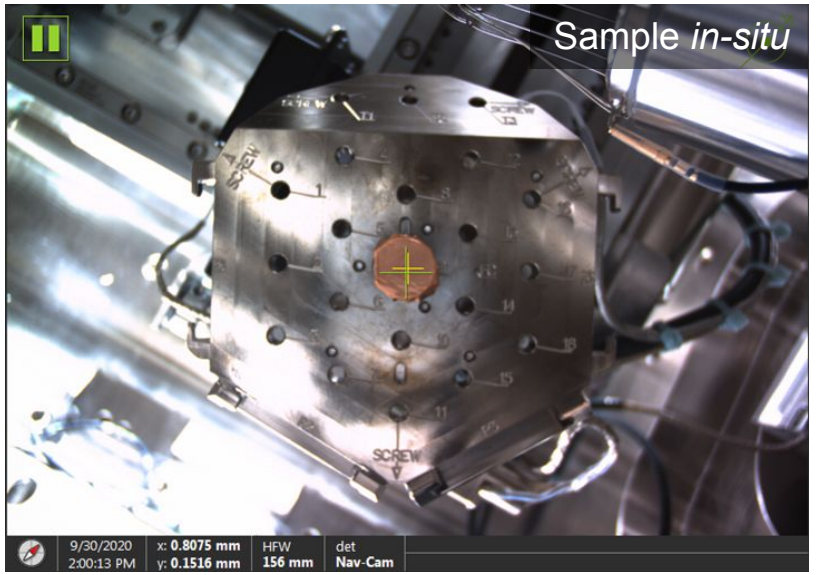
Approximate Stoichiometry for Product:

$$(Na,K,Ca)_{2\text{-}3}Al_3Si_{13}O_{31}\cdot 17H_2O$$

Sample Description

- Liquid sample is drop-cast on Cu tape
  - Chosen for its dissimilarity to target Zeolite and Clinoptilolite
- Dried under vacuum at room temperature
- Sample is not coated prior to measurement





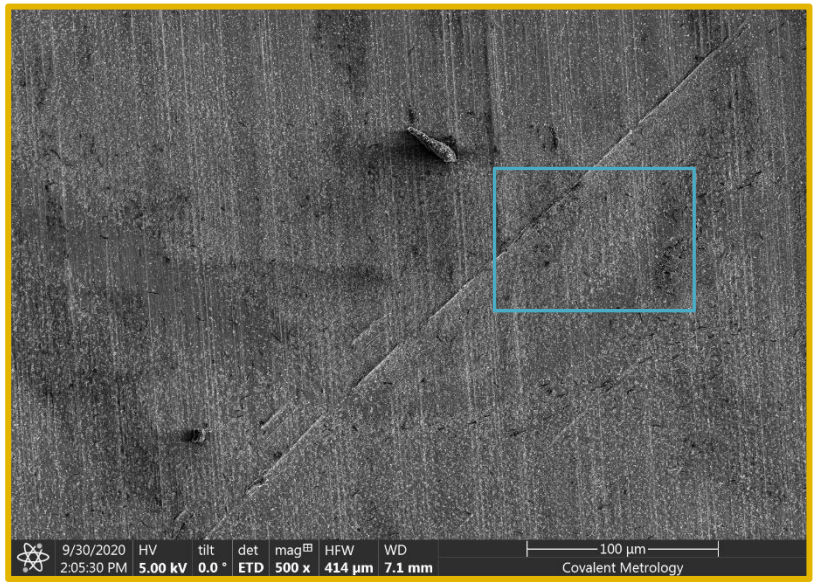
System Used

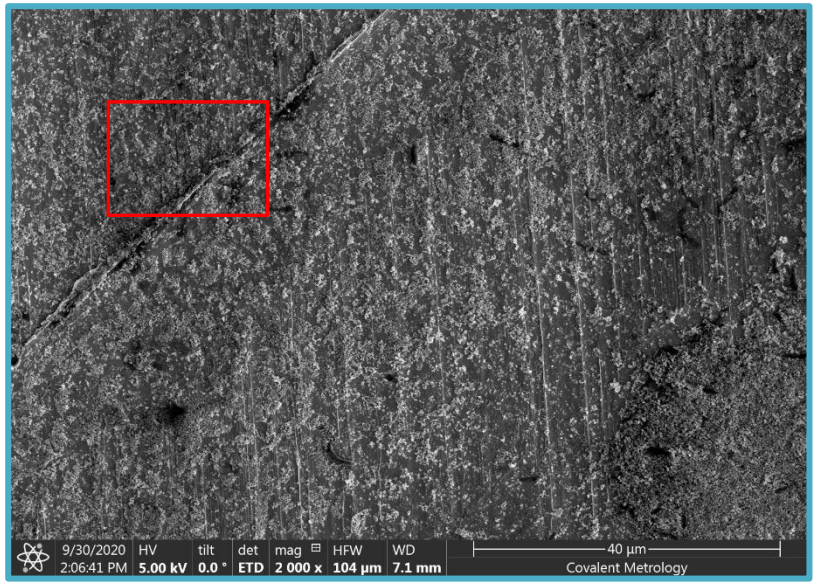
SEM images and EDS maps were obtained on a FEI Scios Dualbeam with an Oxford 150mm^2 detector.

Results and Analysis

SEM Images

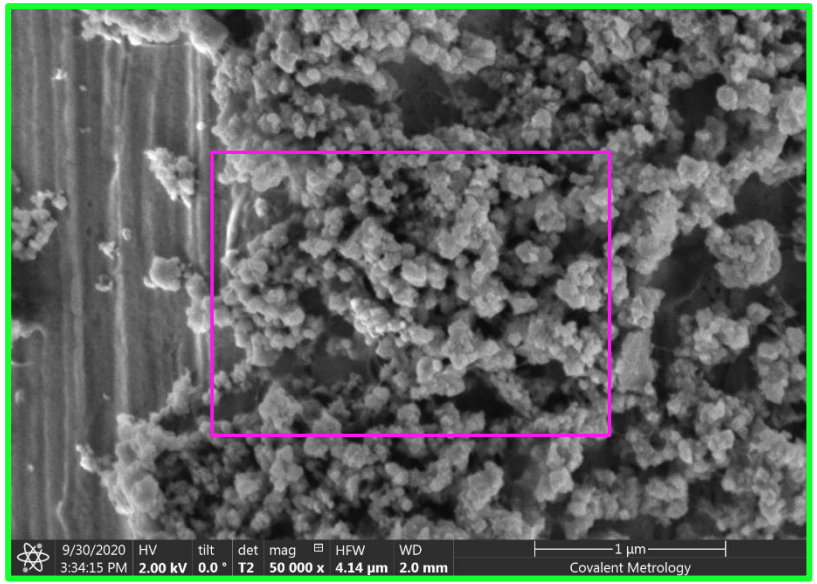

Drop
area
9/30/2020
2:03:50 PM
HV
5.00 kV
tilt
0.0 °
det
ETD
mag
52 x
HFW
4.00 mm
WD
7.0 mm
1 mm
Covalent Metrology


9/30/2020
2:05:30 PM
HV
5.00 kV
tilt
0.0 °
det
ETD
mag
500 x
HFW
414 µm
WD
7.1 mm
100 µm
Covalent Metrology


9/30/2020
2:06:41 PM
HV
5.00 kV
tilt
0.0 °
det
ETD
mag
2 000 x
HFW
104 µm
WD
7.1 mm
40 µm
Covalent Metrology

SEM Images


9/30/2020 3:32:04 PM
HV 2.00 kV
tilt 0.0 °
det T2
mag 10 000 x
HFW 20.7 µm
WD 2.0 mm
5 µm
Covalent Metrology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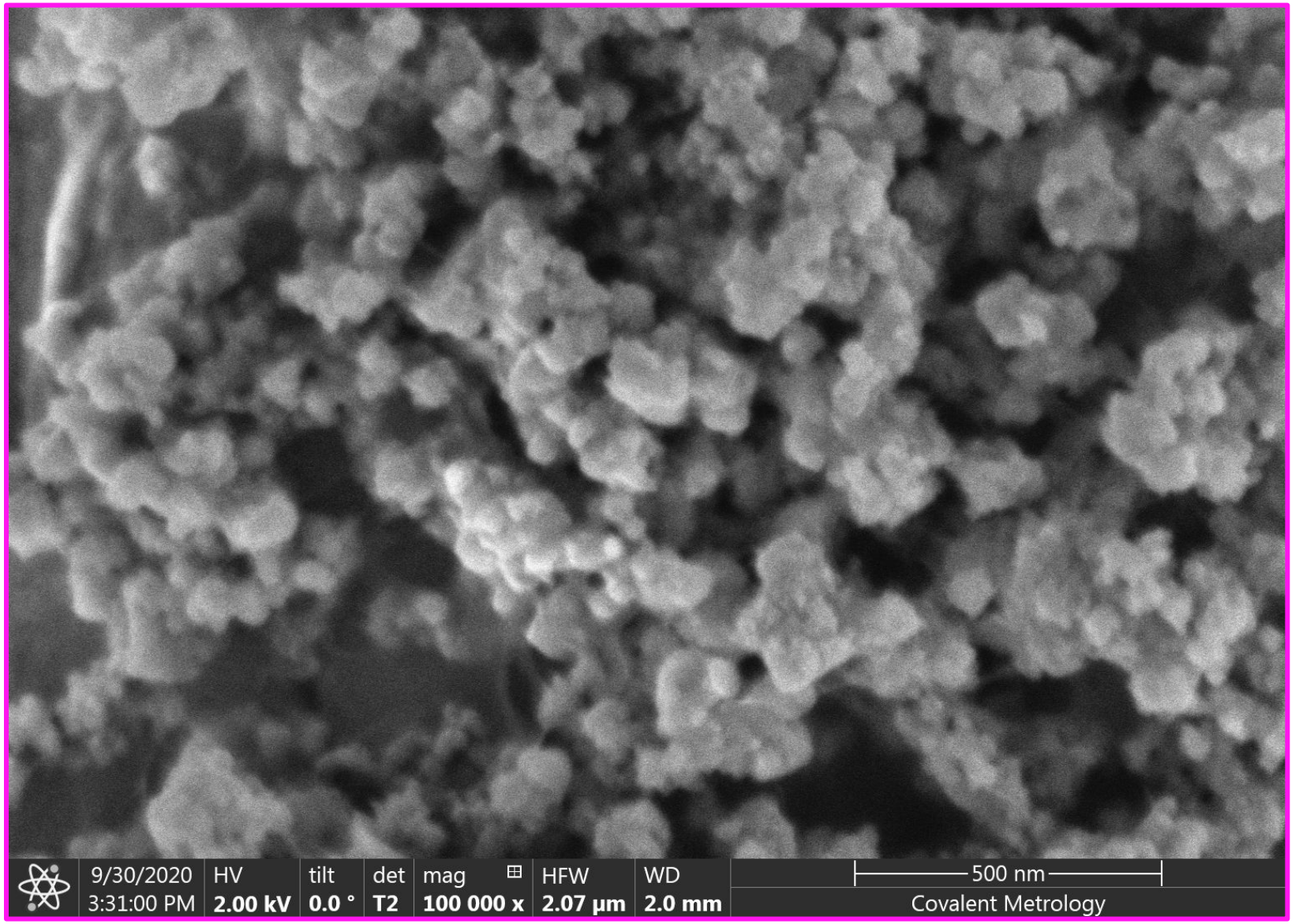
WD 2.0 mm
1 µm
Covalent Metrology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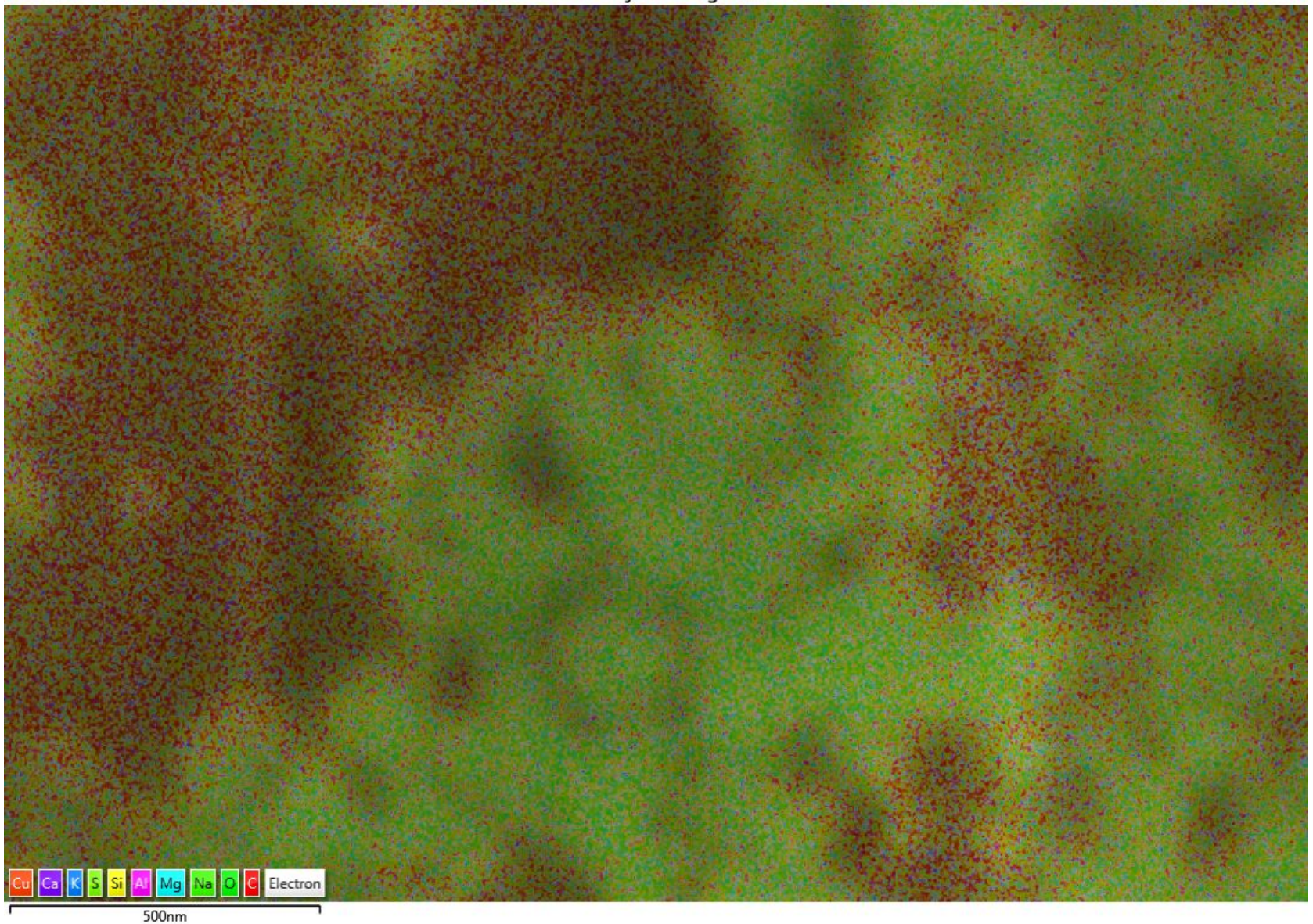
tilt 0.0 °
det T2
mag 100 000 x
HFW 2.07 µm
WD 2.0 mm
500 nm
Covalent Metrology

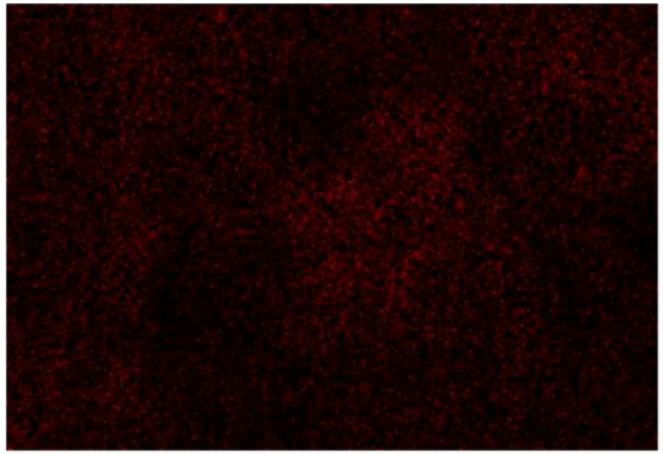
Site 1 – Results

EDS Layered Image 7

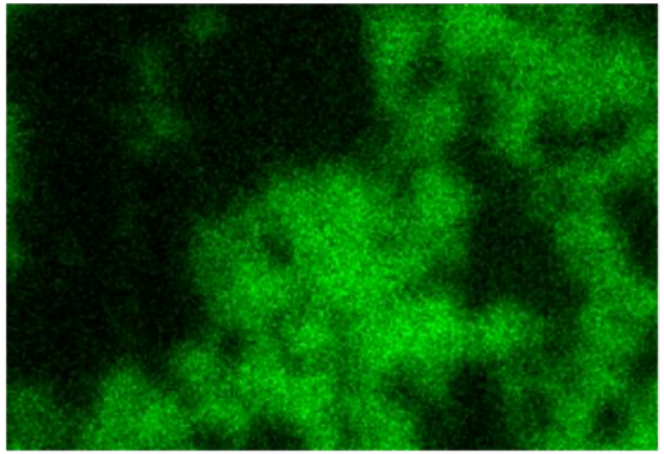


Site 1 – EDS Maps

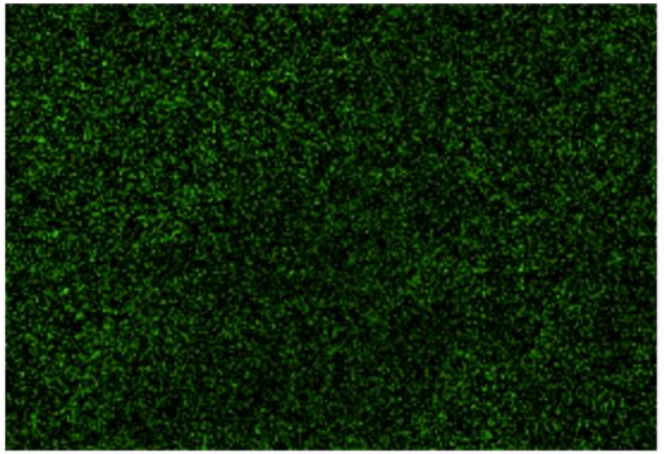
C K series



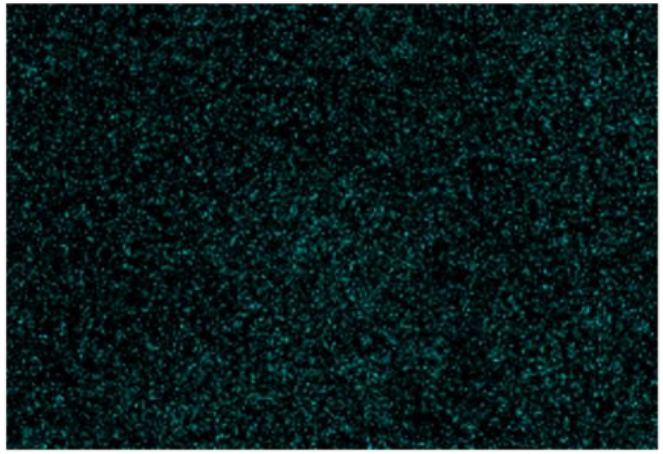
O K series



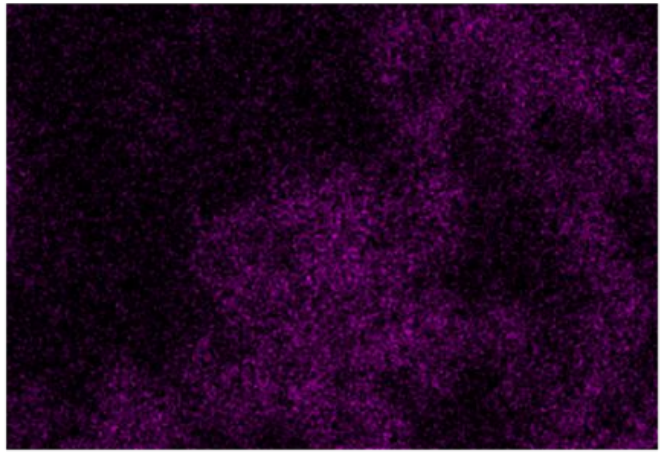
Na K series



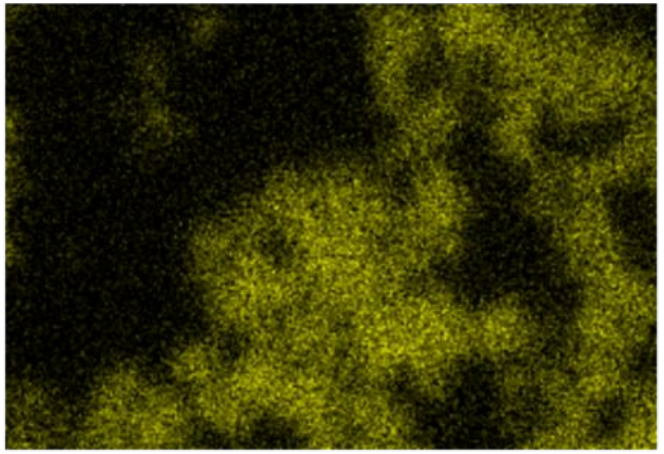
Mg K series



Al K series

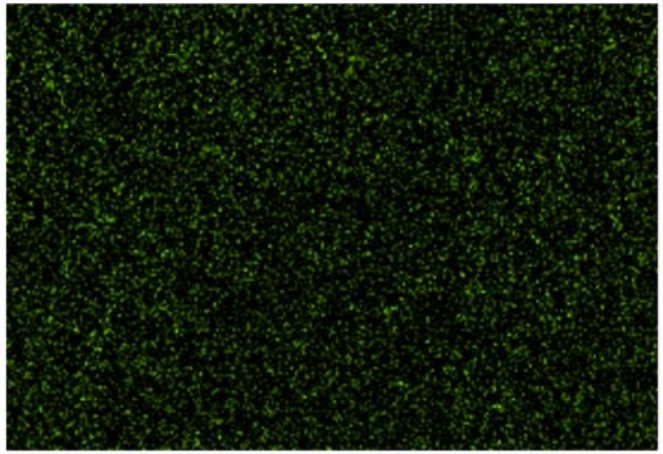


Si K series

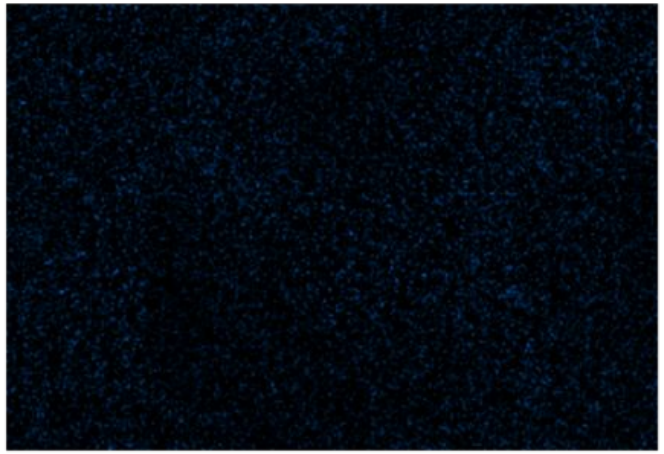


Site 1 – EDS Maps

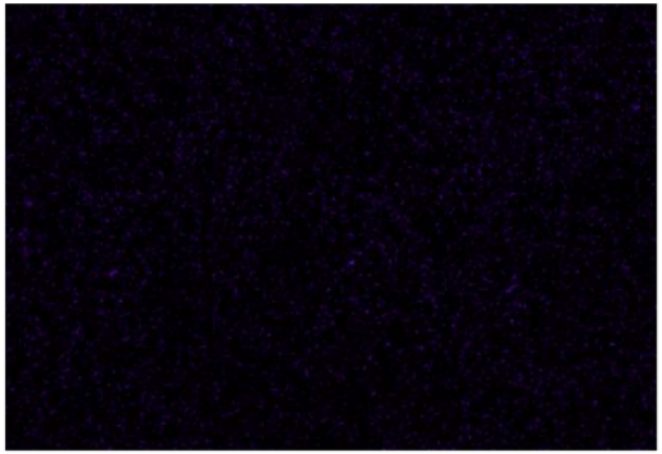
S K series



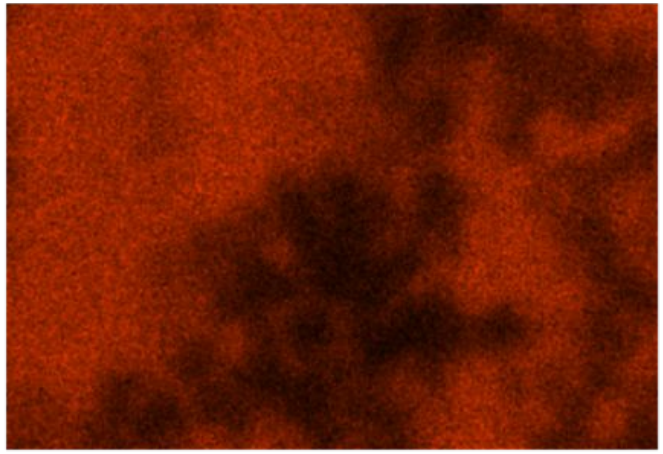
K L series



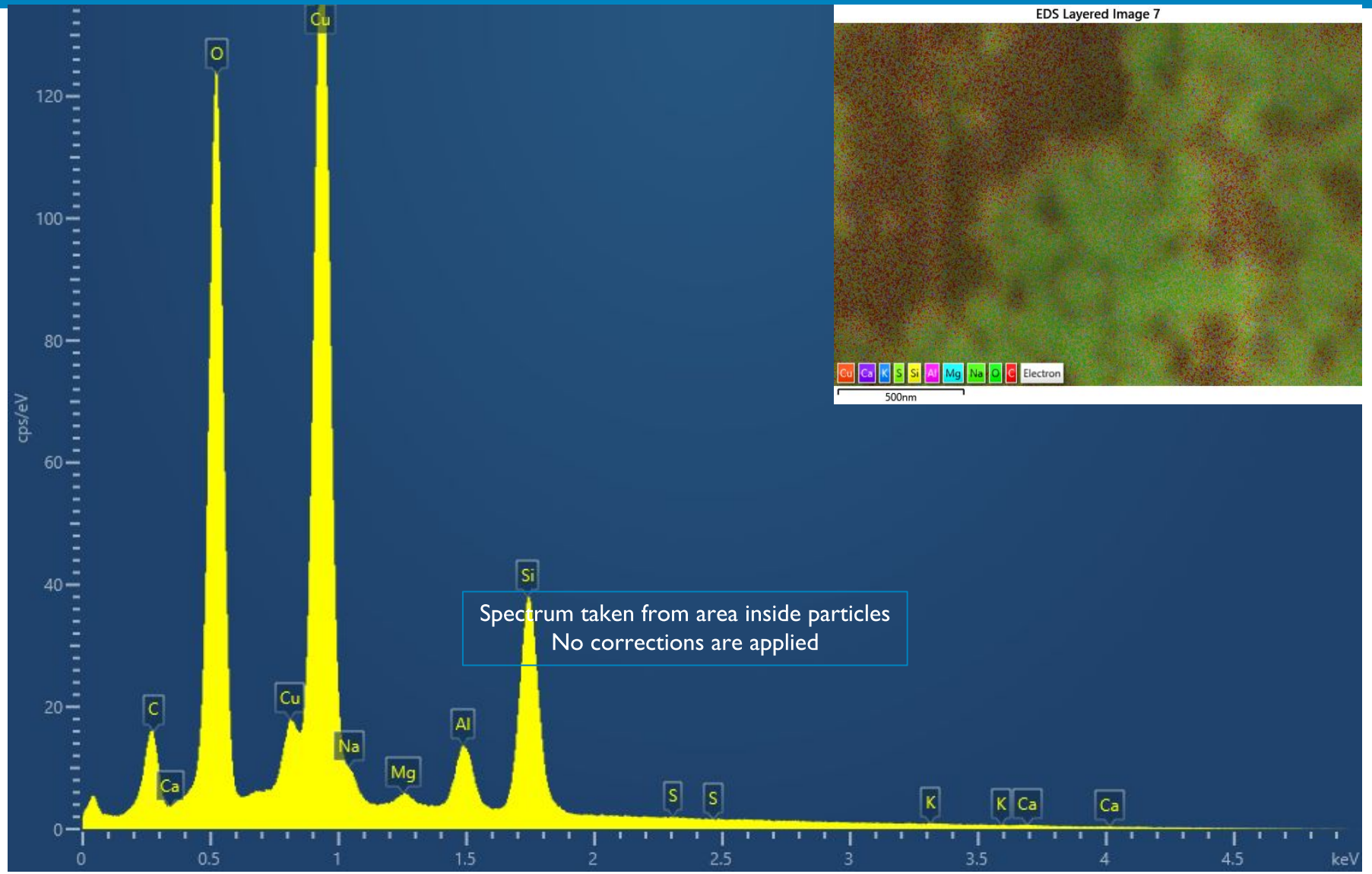
Ca L series



Cu L series

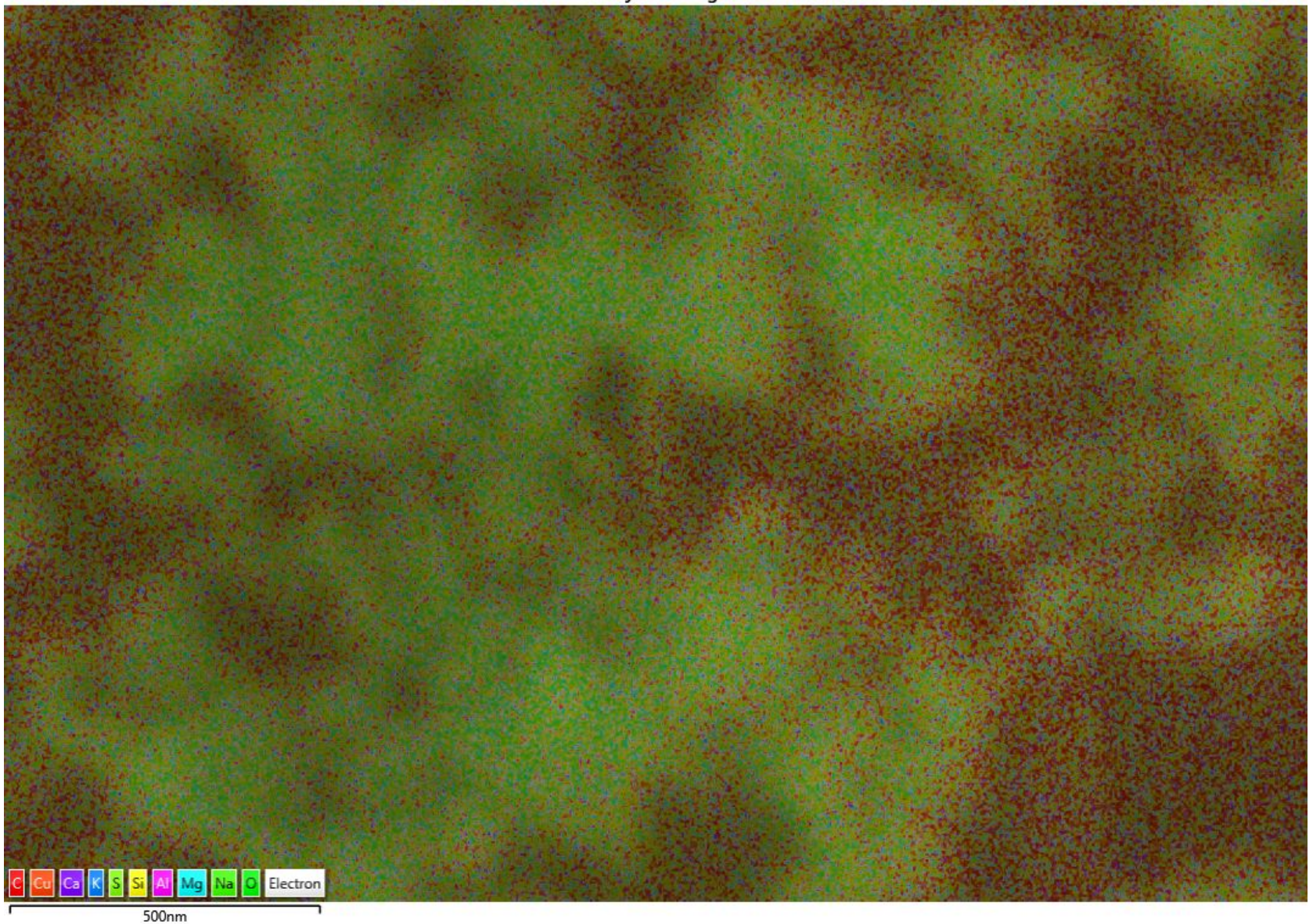


Site 1 – Spectrum



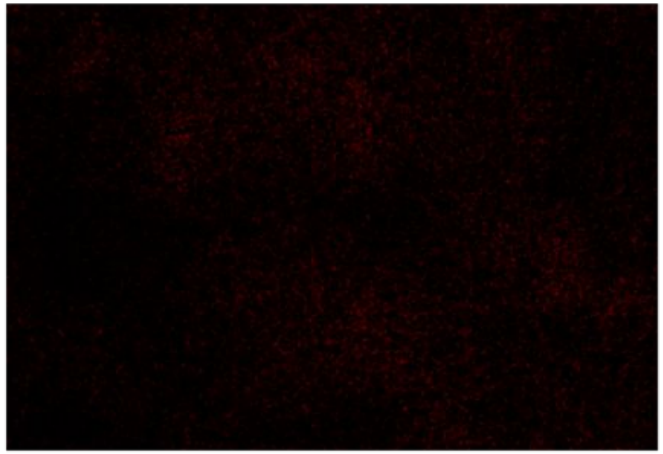
Site 2 – Results

EDS Layered Image 8

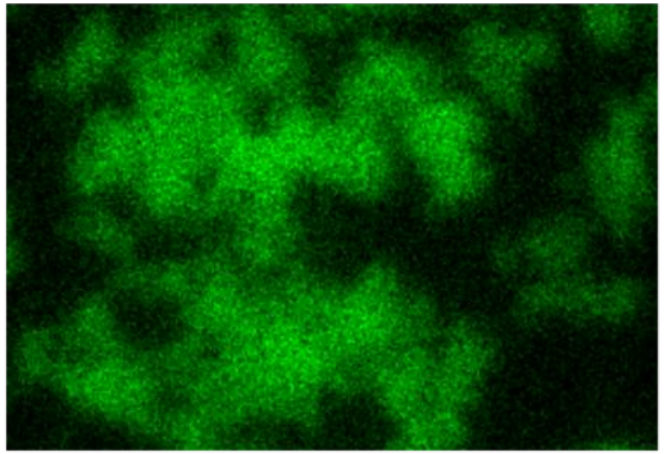


Site 2 – EDS Maps

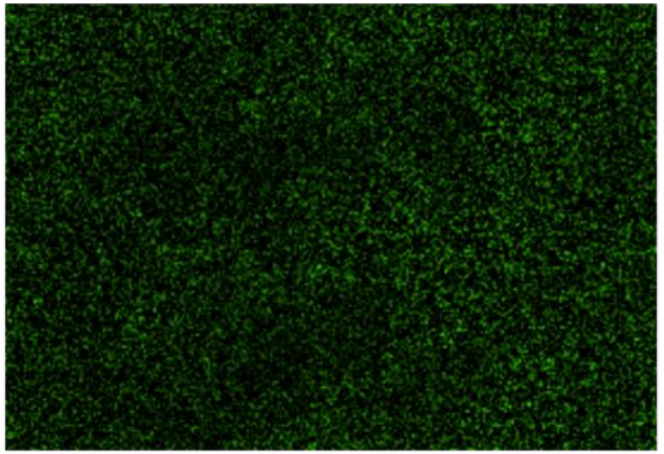
C K series



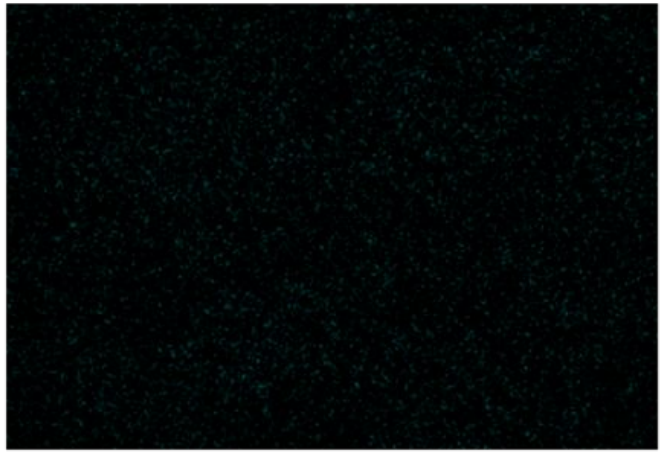
O K series



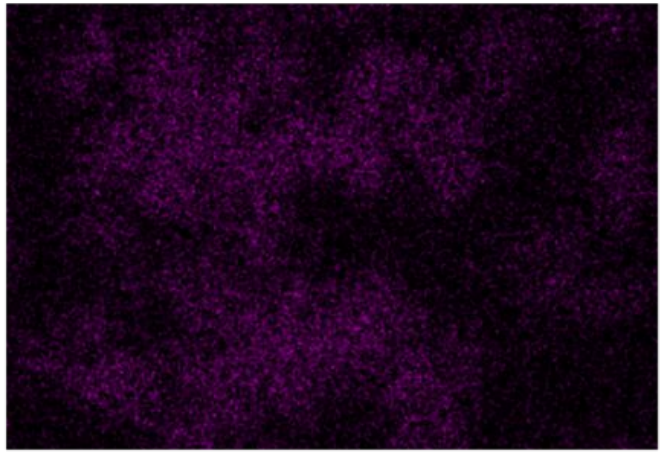
Na K series



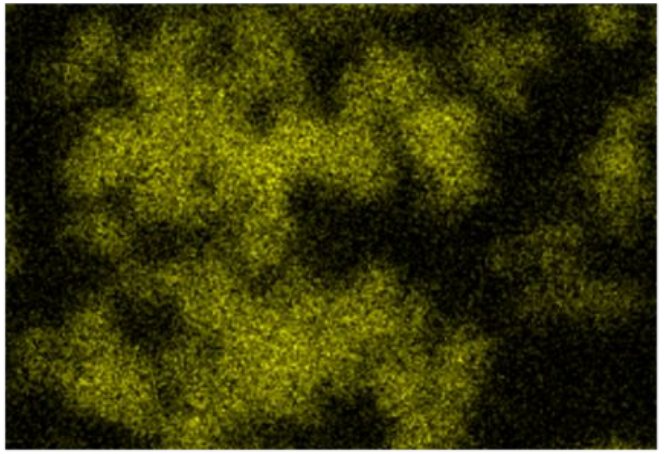
Mg K series



Al K series

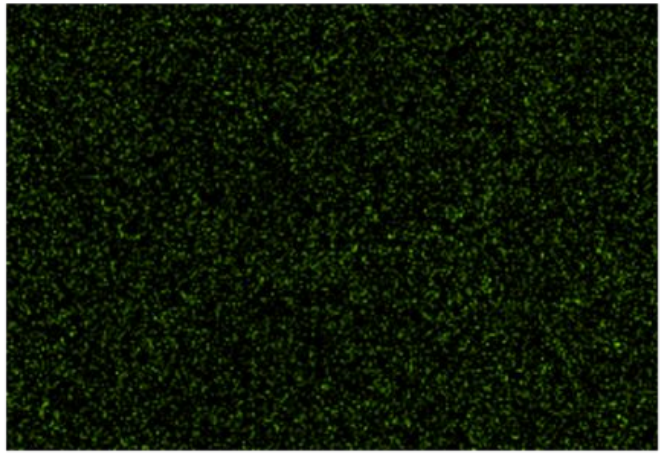


Si K series

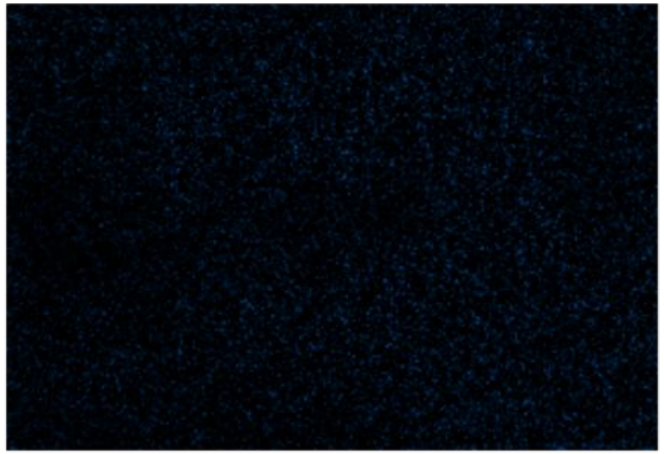


Site 2 – EDS Maps

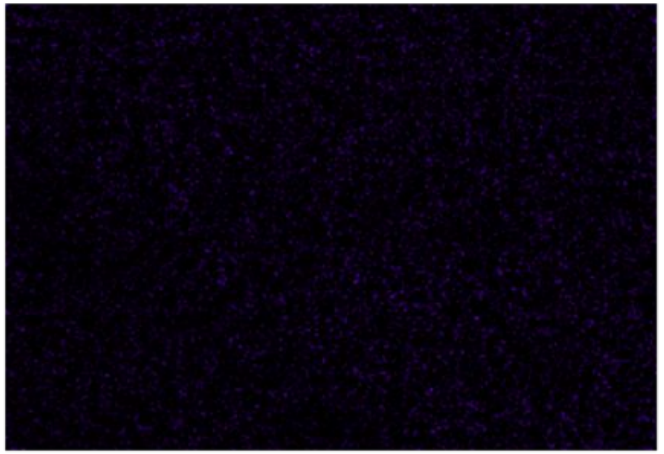
S K series



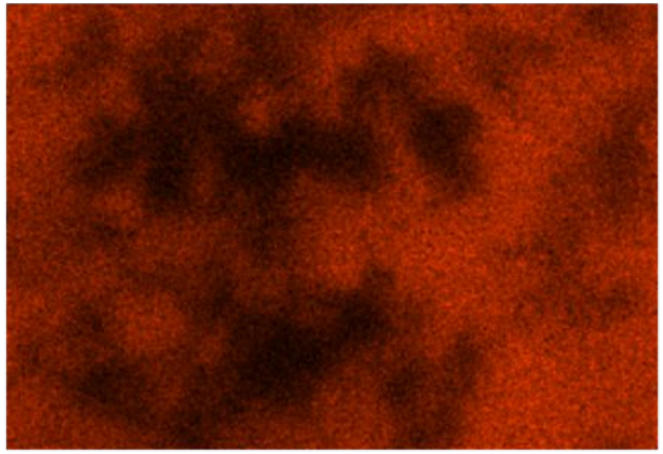
K L series



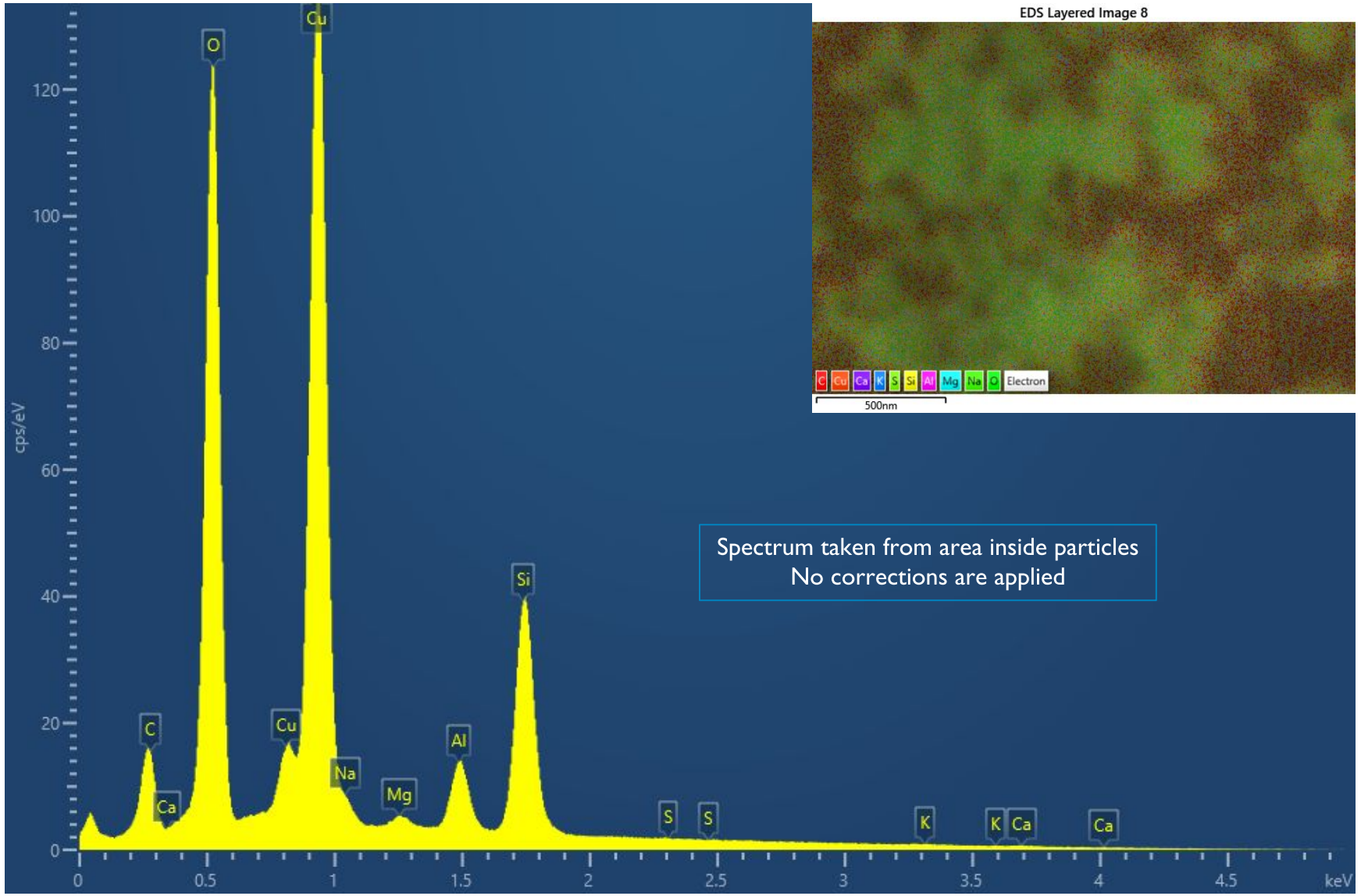
Ca L series



Cu L series



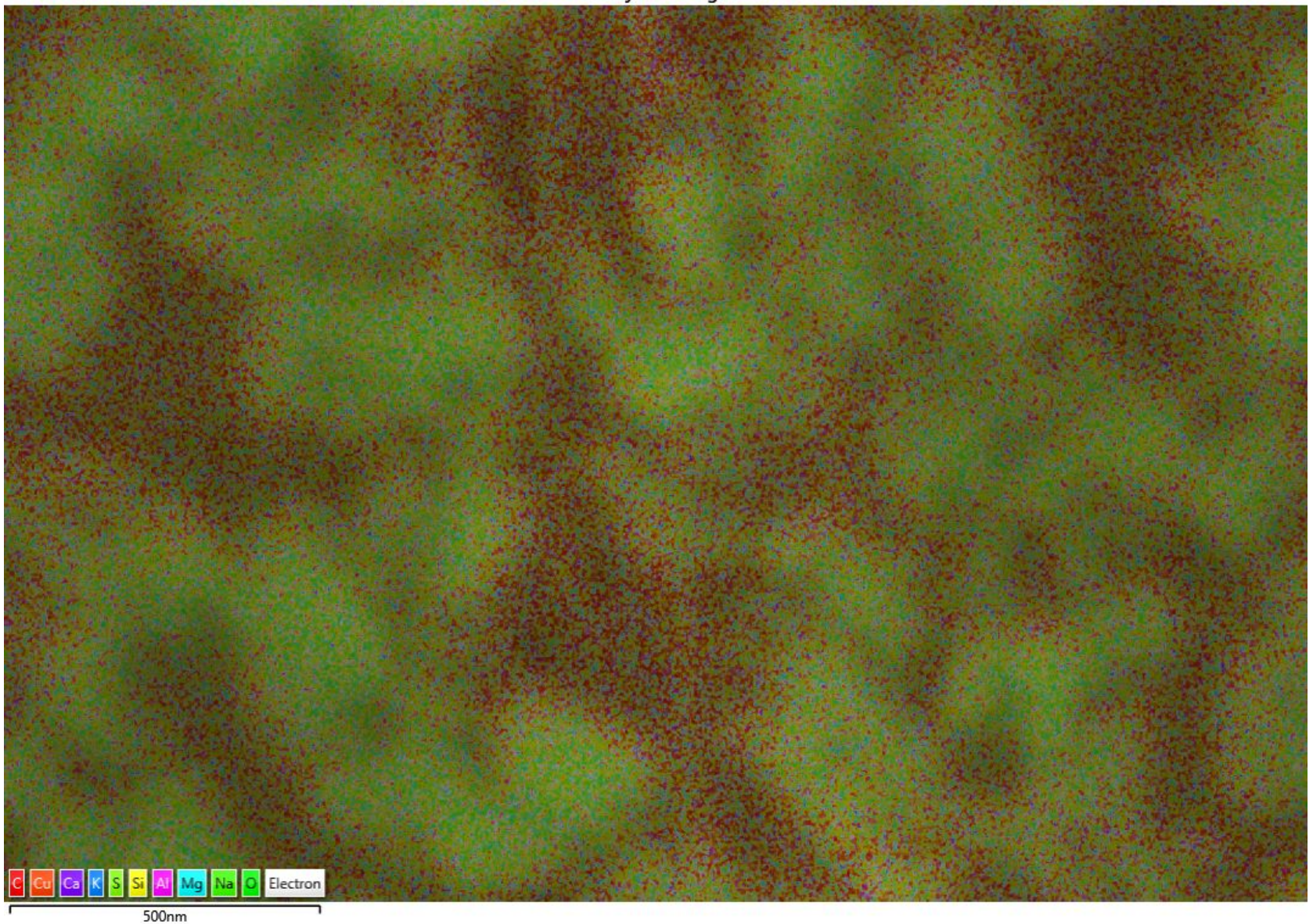
Site 2 – Spectrum



Spectrum taken from area inside particles
No corrections are applied

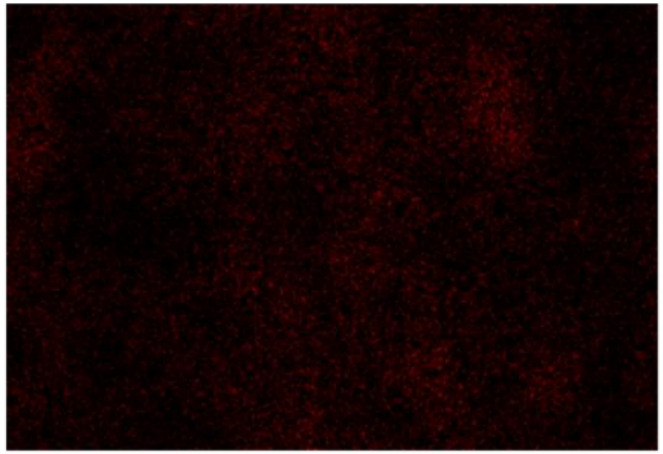
Site 3 – Results

EDS Layered Image 9

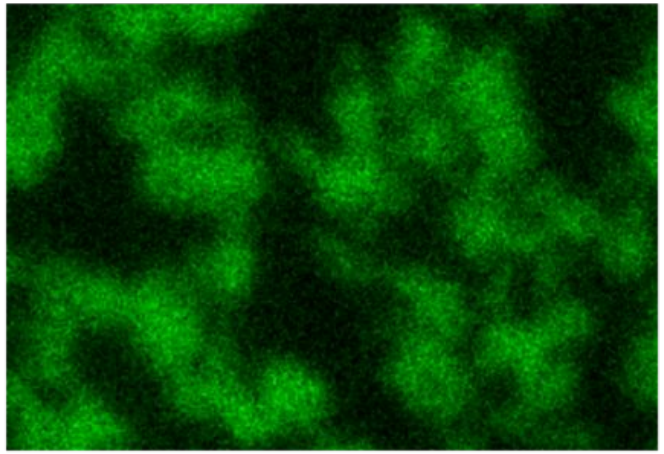


Site 3 – EDS Maps

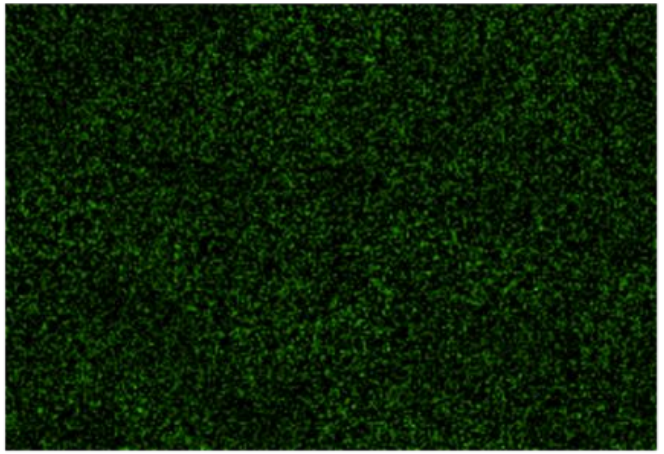
C K series



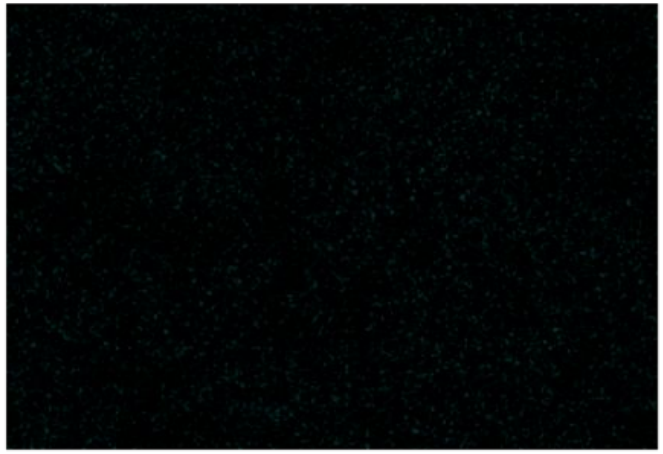
O K series



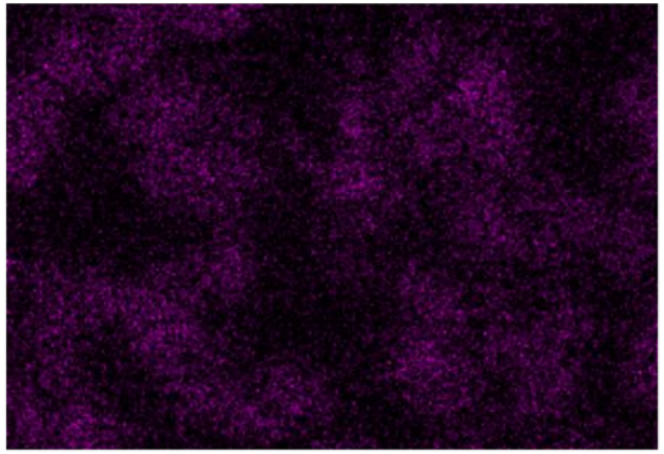
Na K series



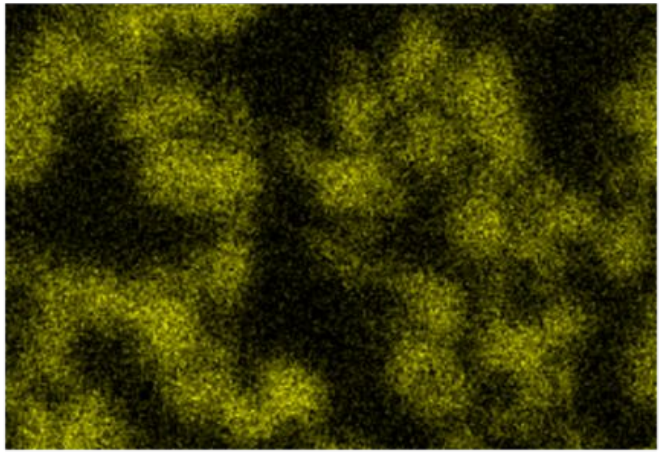
Mg K series



Al K series

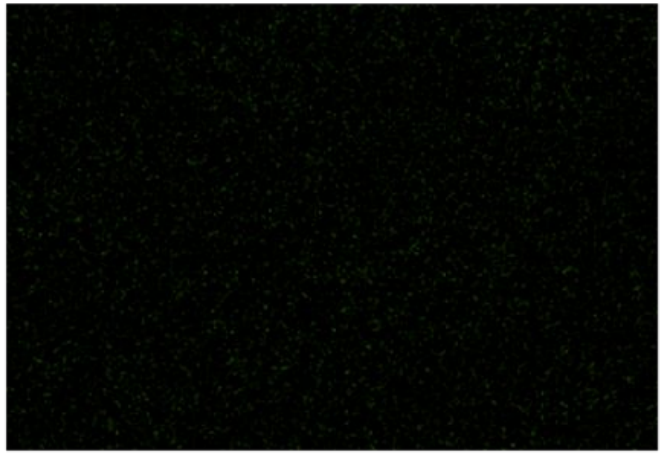


Si K series

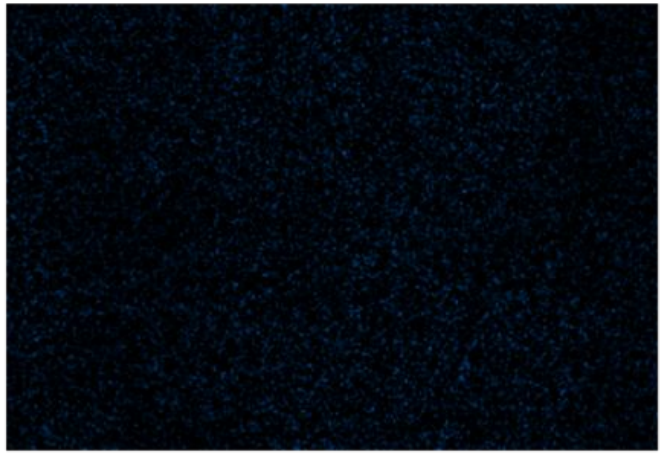


Site 3 – EDS Maps

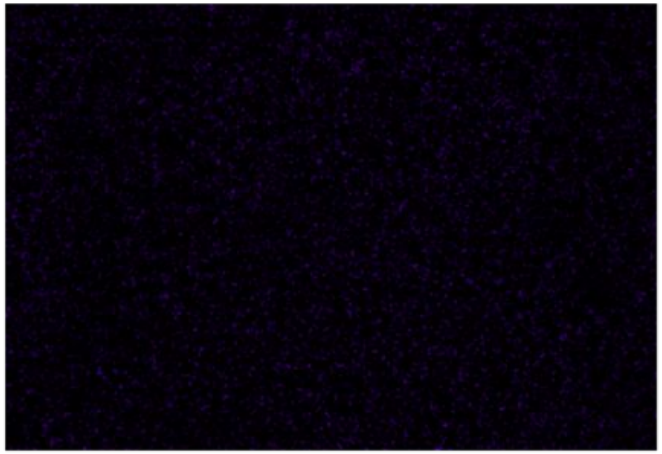
S K series



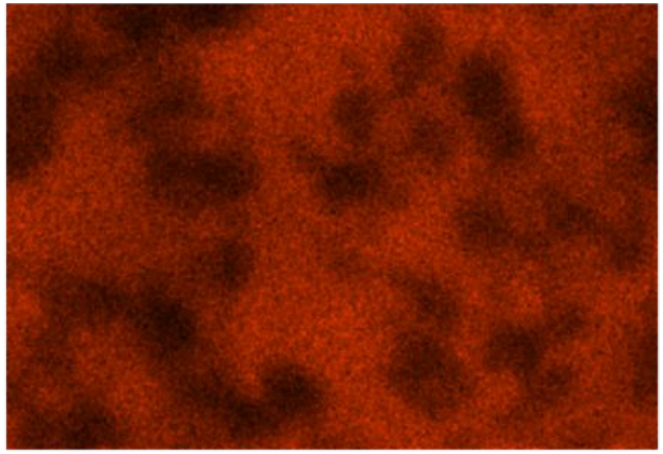
K L series



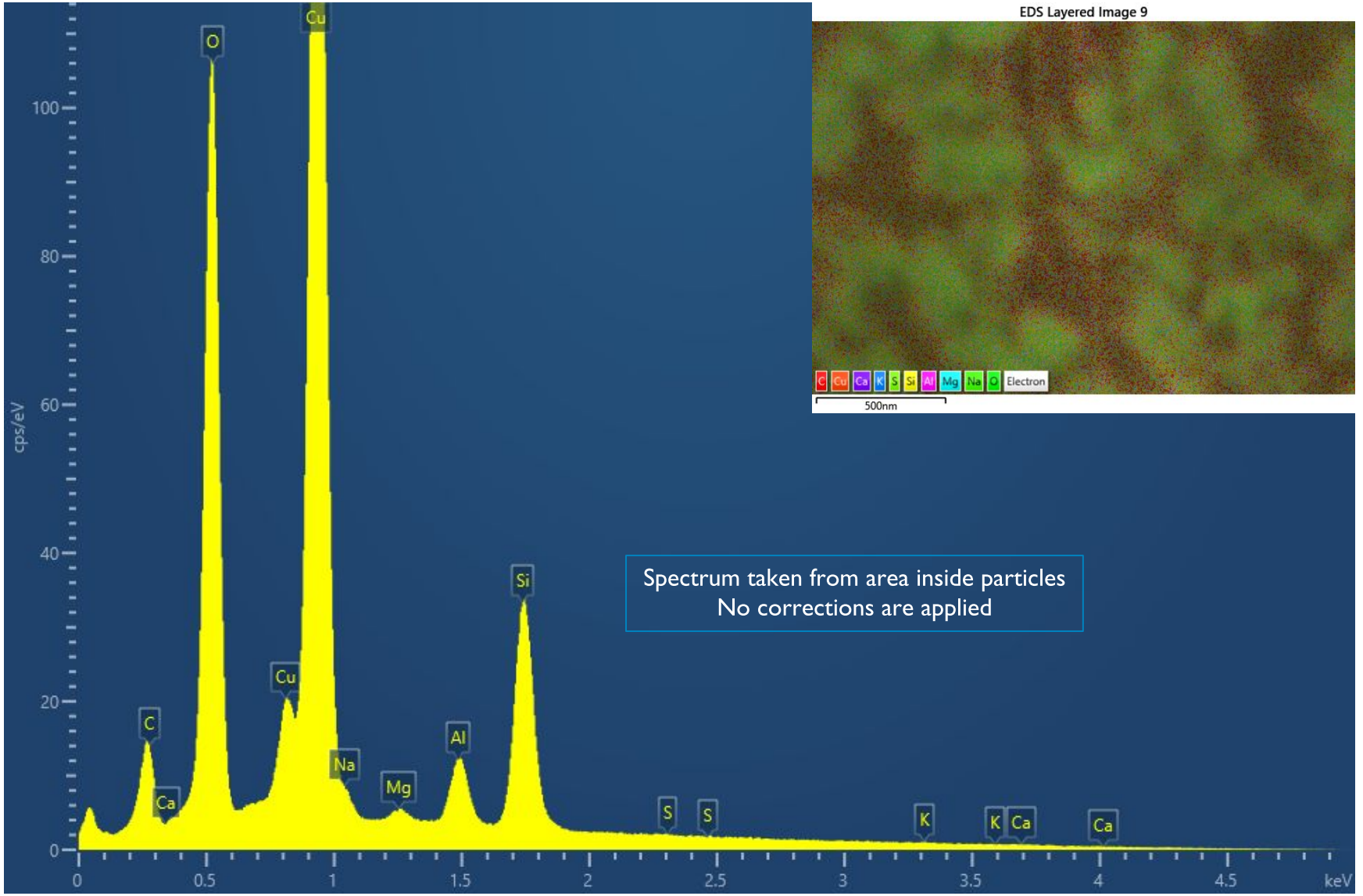
Ca L series



Cu L series



Site 3 – Spectrum



Spectrum taken from area inside particles
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