BRUKER NANO ANALYTICS

Microanalysis Of Battery Materials

Bruker KOREA
Application Team
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Presenters

Sungji Choi

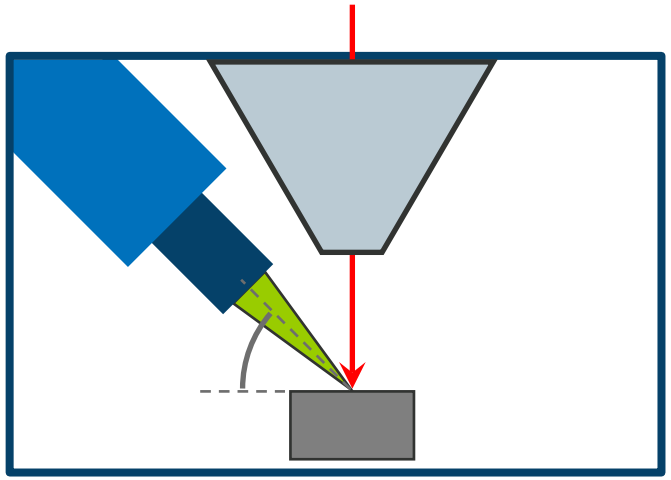
- Application specialist
- **EDS / EBSD / FlatQUAD / Micro-XRF / WDS**
- Bruker Nano Analytics, Korea

How to get the most information – use FlatQUAD for better detection

	XFlash® 760	XFlash® FlatQUAD
		
SDD geometry		
HV	Up to 30 kV	up to 20 kV
WD (min)	~ 4 mm	~ 8 mm
Window	optional	multiple
Solid angle (up to)	0.1 sr	1.1 sr

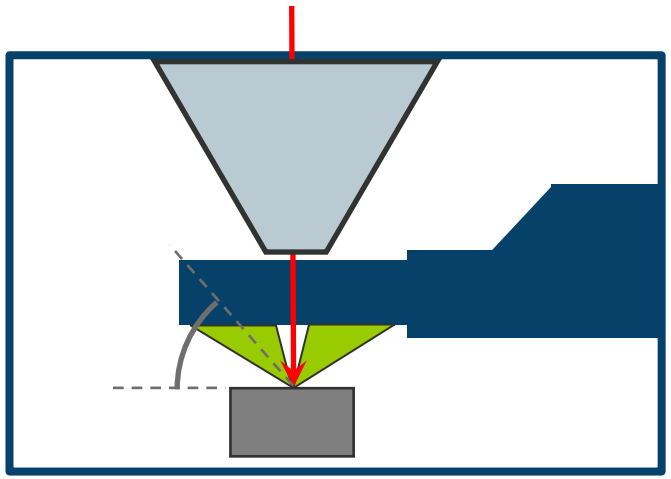
How to get the most information – use FlatQUAD for better detection

Conventional detector
60 mm² EDS detector
@WD= 10mm



take off angle=35°
solid angle=0.043 sr

FlatQUAD detector
60 mm² (4x15mm²)
@WD=10mm

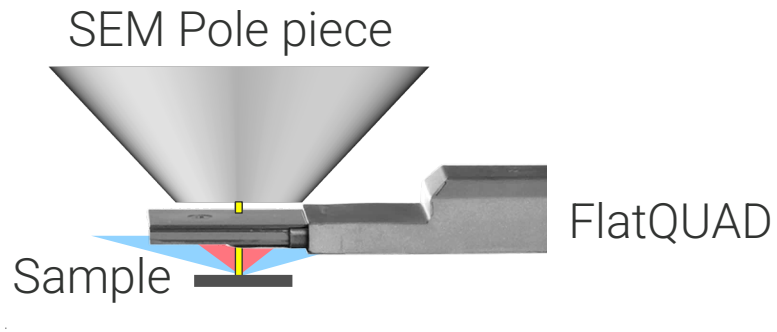


take off angle=60-70°
solid angle = 1.1 sr

→ **Better view of sample topography**

→ **x15-30 more X-ray signal:**
less measurement time needed

Bruker XFlash® FlatQUAD detector
Features and advantages

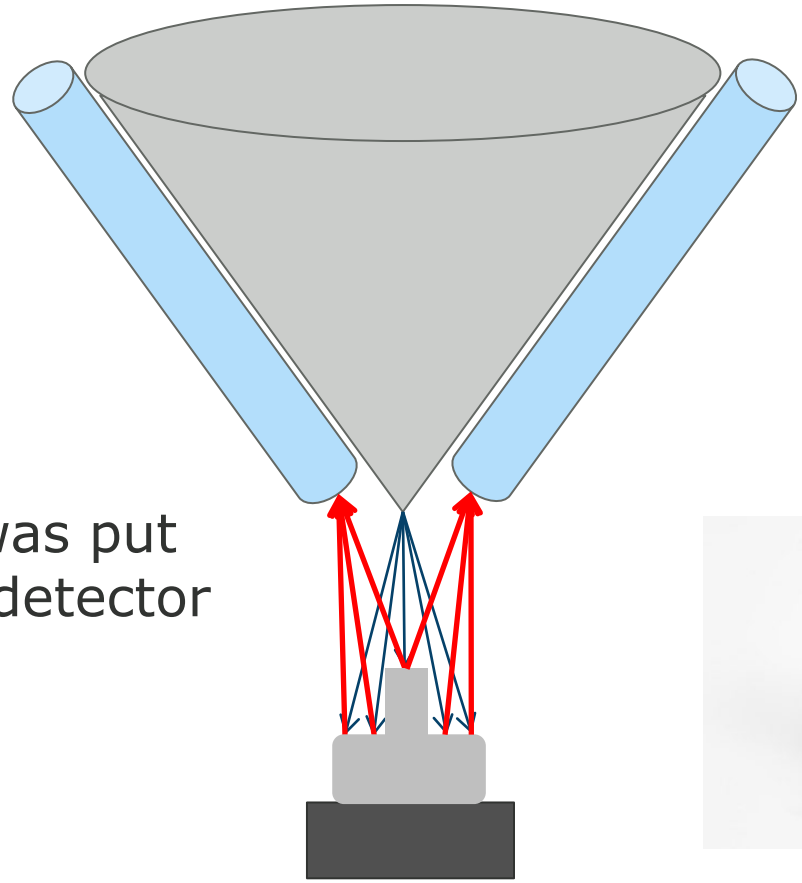


- Annular 4-segment (4x) SDD geometry, central ap.
- Side entry EDS (STEM/BSE like)
- Large solid angle of 1.1 sr
- High take-off angle (~60˚)
- Optimal signal collection geometry

- High sensitivity at very low probe currents ~few pA
- Minimize sample charging/damage/C-deposition at low PC
- High vacuum conditions EDS – high resolution
- Low vacuum capability
- Moderate probe currents for high-speed EDS mapping
- Low x-ray yield samples: Low PC – High resolution
- Nanoparticles, Thin lamellae, beam sensitive materials

Shadow effect

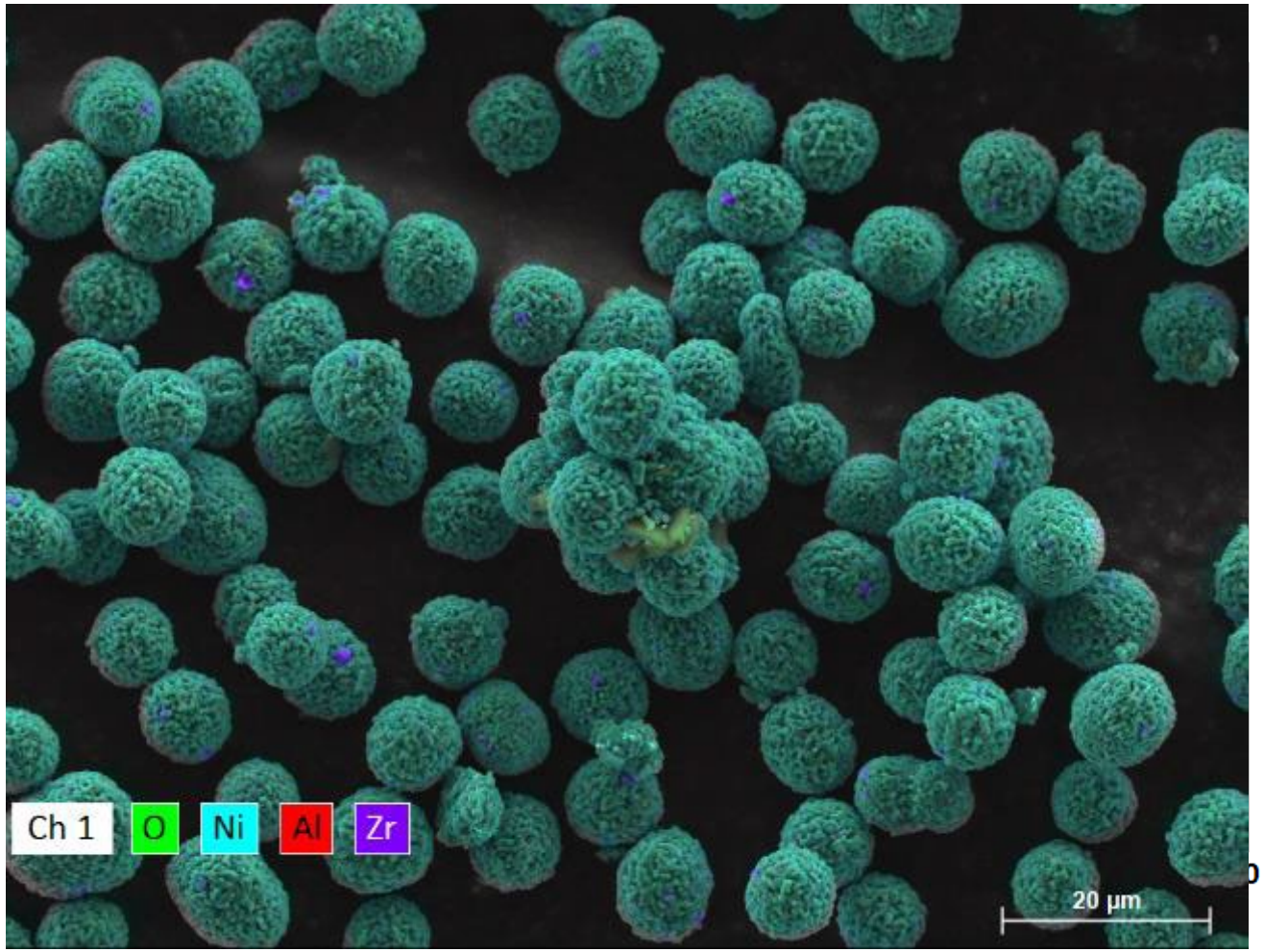
SEM-EDS system was put on a just one EDS detector at common



If your samples having a roughness at surface,
Easy to get a shadow effect by one way detection

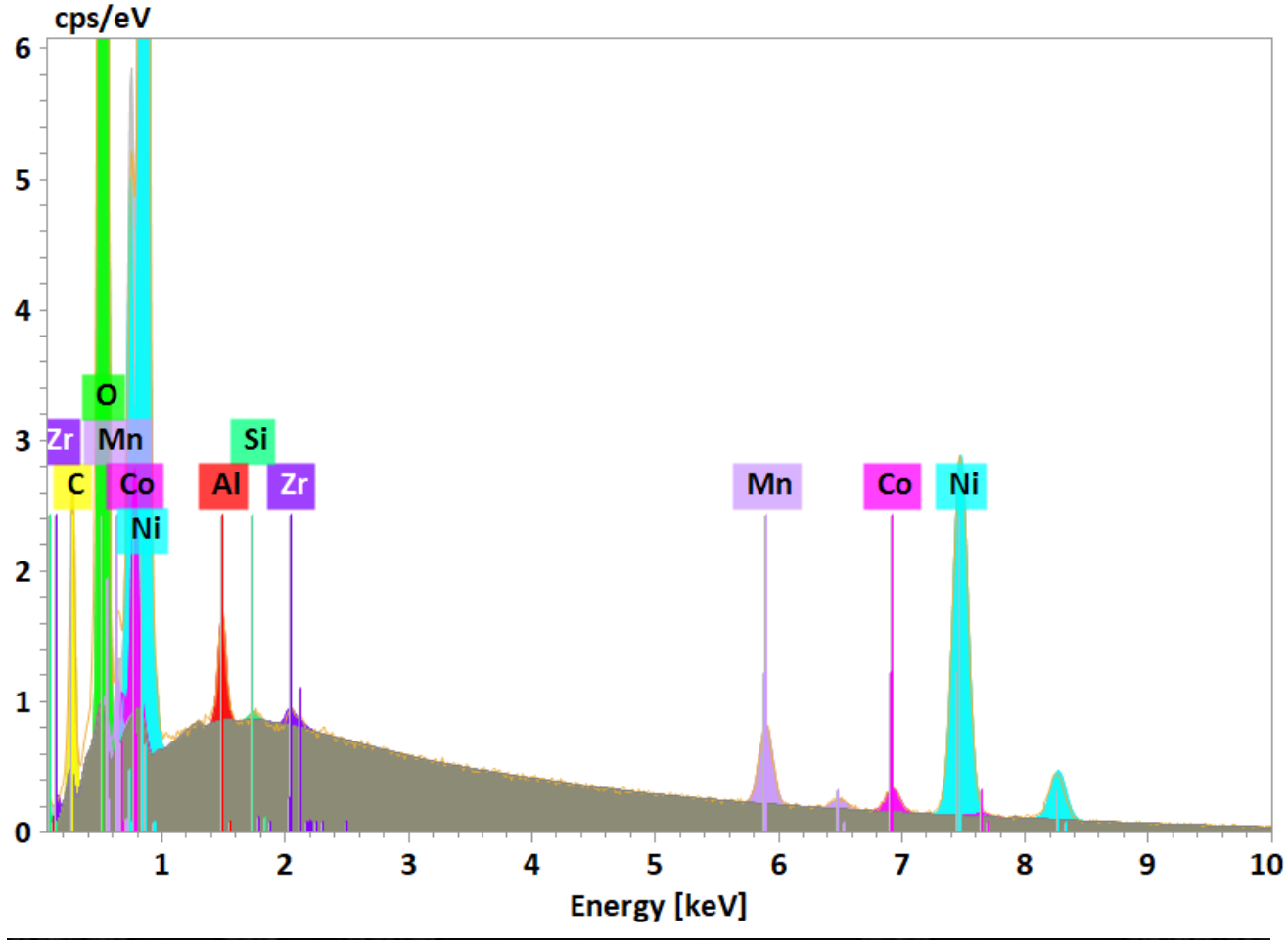
Pre-screening of NCM particles

- Electron microscopy: SE imaging gives morphological information.
- Flatquad EDS detector allows for very fast mapping of spherical particles without shadowing effect (full particle is mapped)
- High sensitivity allows detection of contaminants in short time.
- No low vacuum, no inert gas transfer and no sample coating needed
- Sample: NCM particles (raw material) loosely distributed on sticky carbon pad



Pre-screening of NCM particles

- Sum spectrum: elements are identified
- Individual element maps:
  - C: sample substrate
  - Ni, Co, Mn: NCM main components
  - O: inhomogeneity of oxide material, oxidation, contamination?
  - Al: sintering agent or dopant?
  - Zr: sintering agent or dopant?



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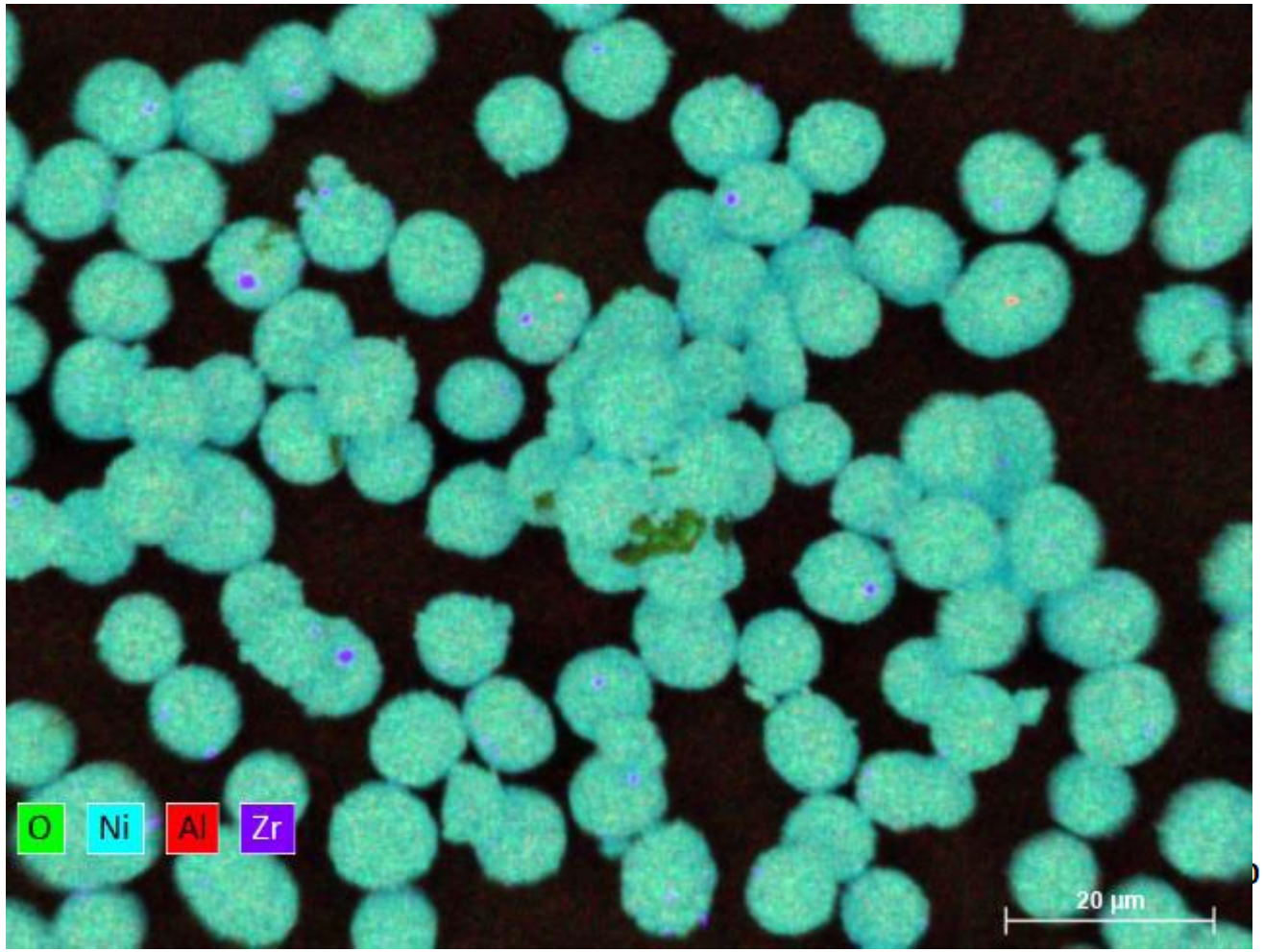

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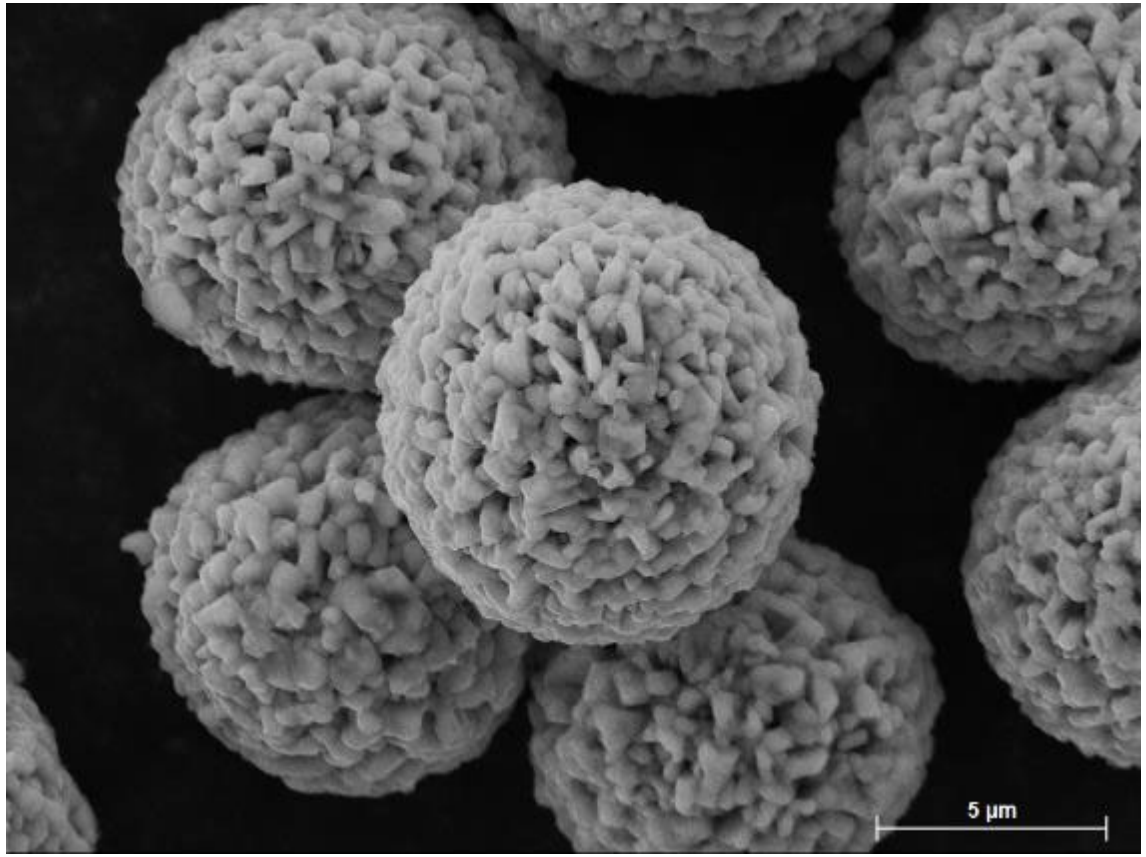
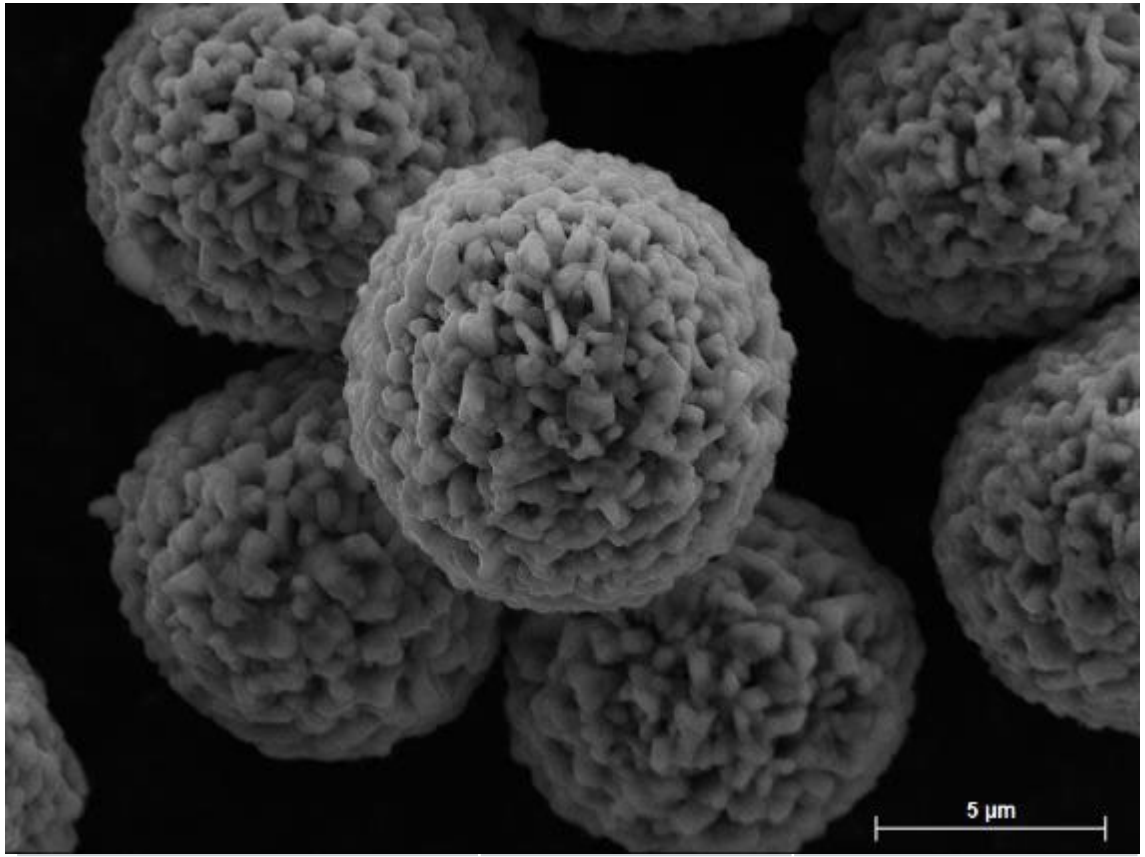




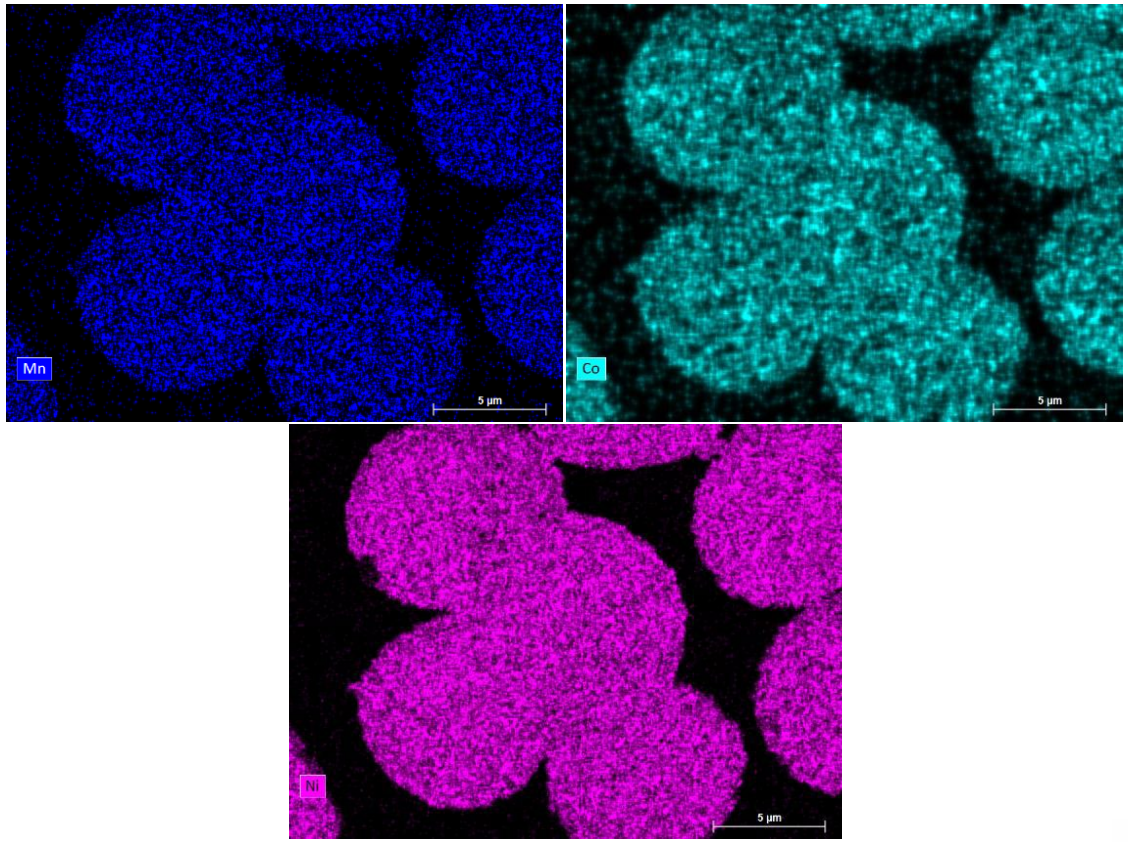
Conventional EDS

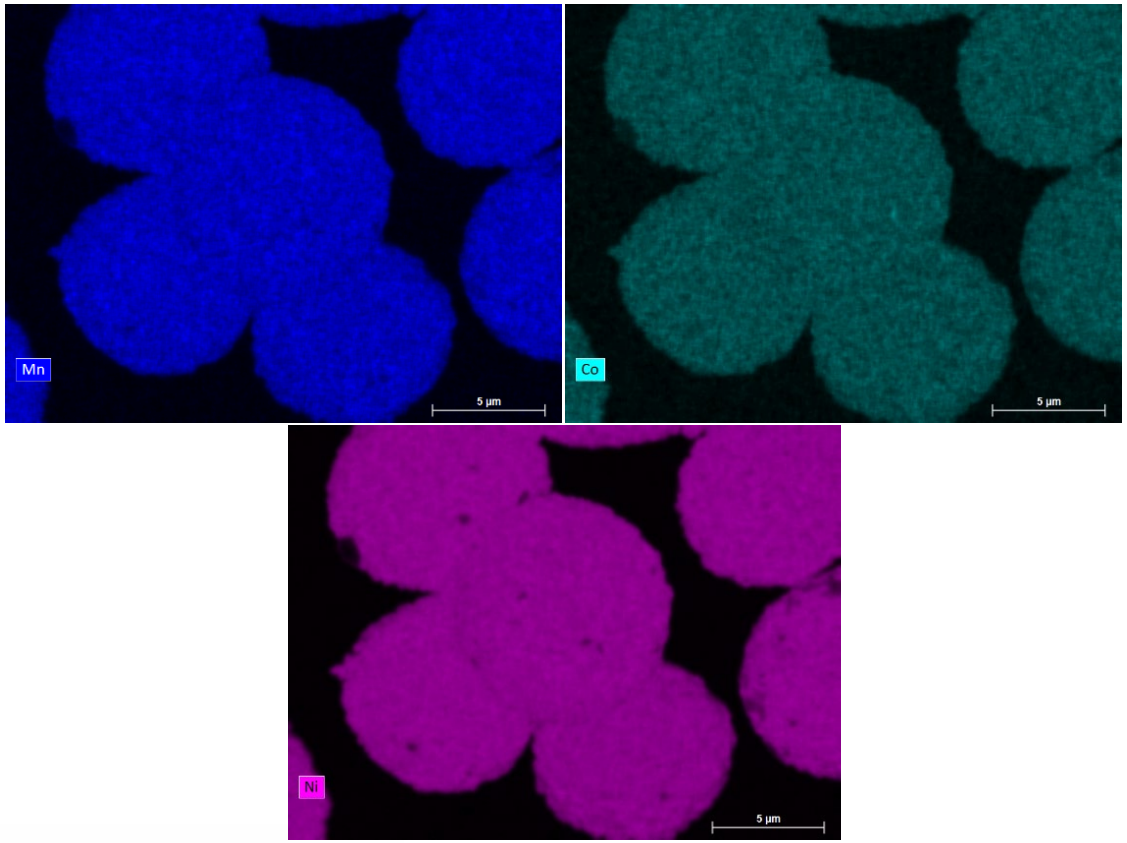
12 kV / 510pA

XFlash® FlatQUAD

12 kV / 510pA

Pre-screening of NCM particles





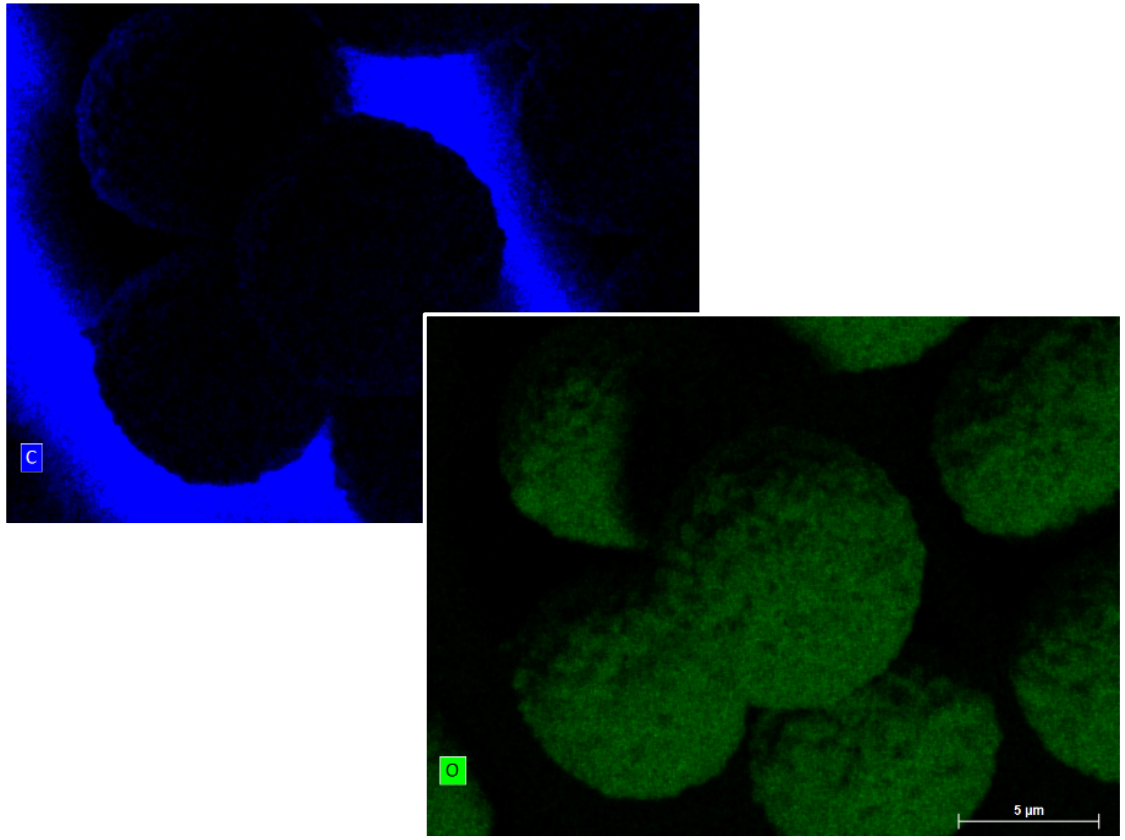
Conventional EDS

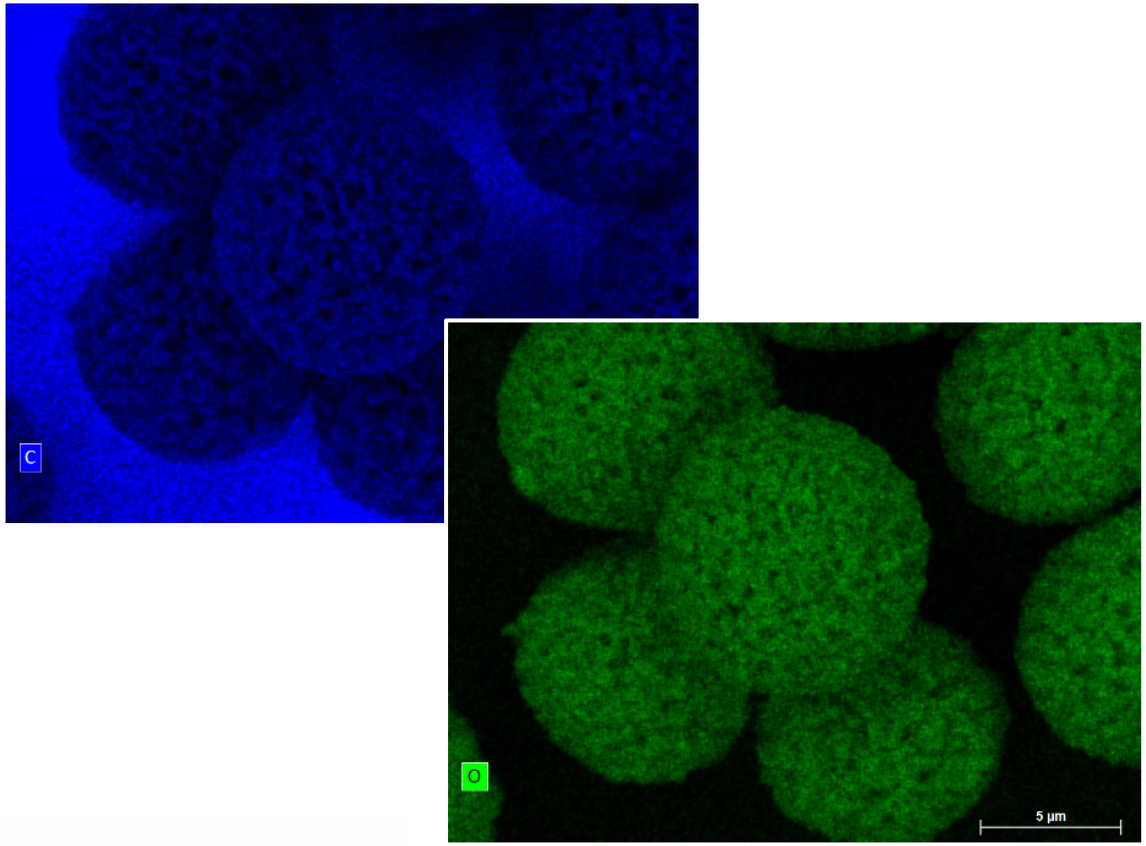
12 kV / 510pA / 600s / **7,190 cps**

XFlash® FlatQUAD

12 kV / 510pA / 600s / **133,900 cps**

Pre-screening of NCM particles





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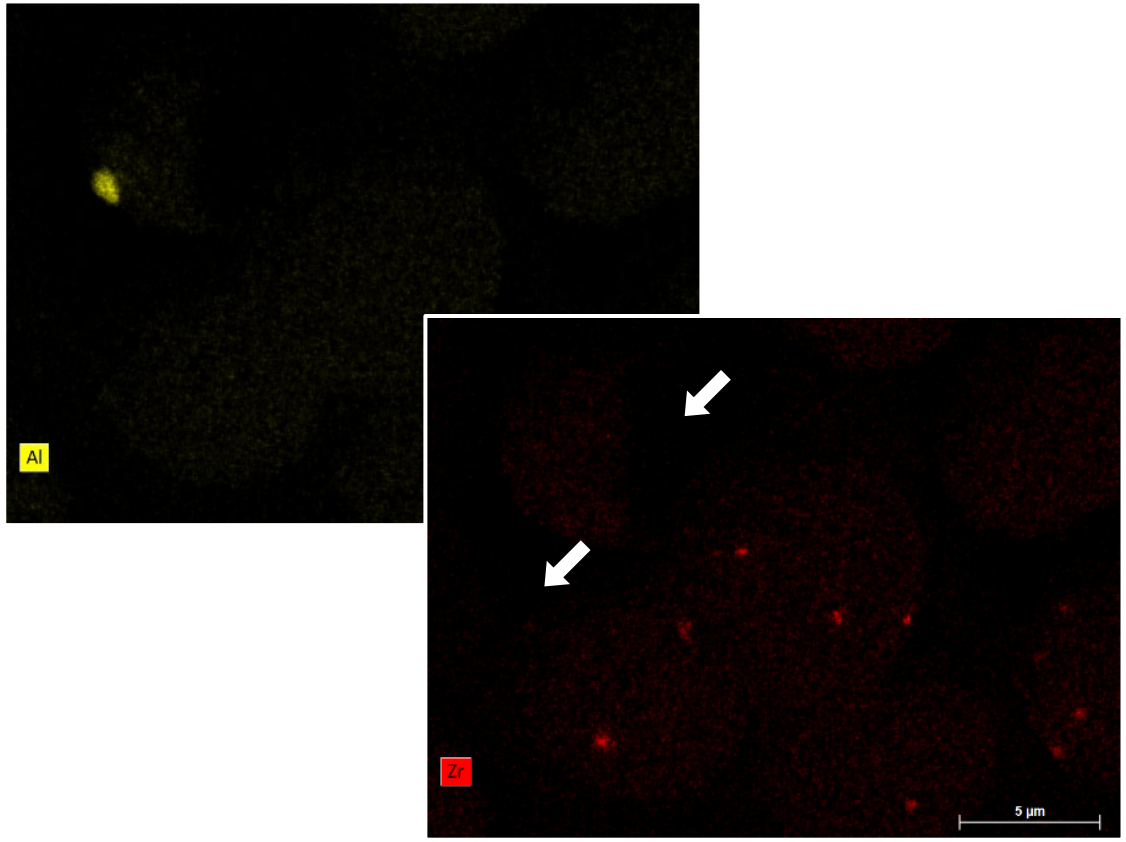

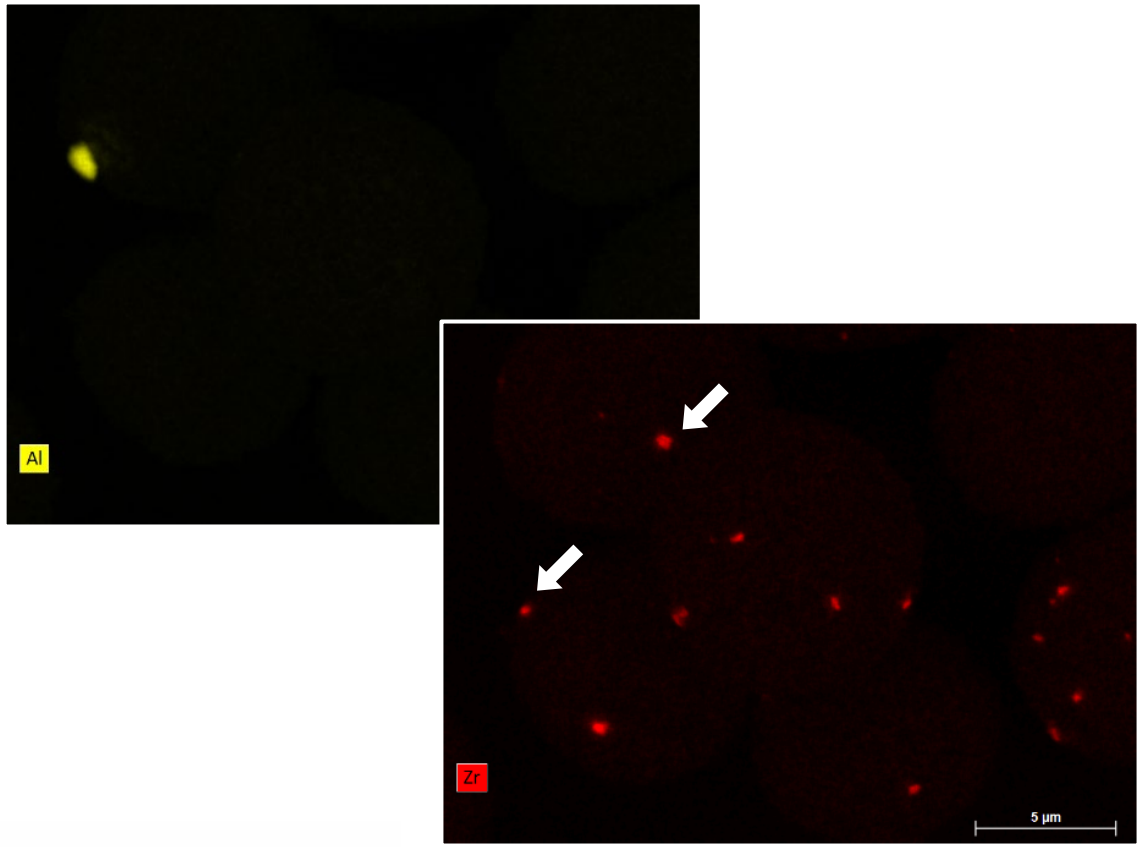

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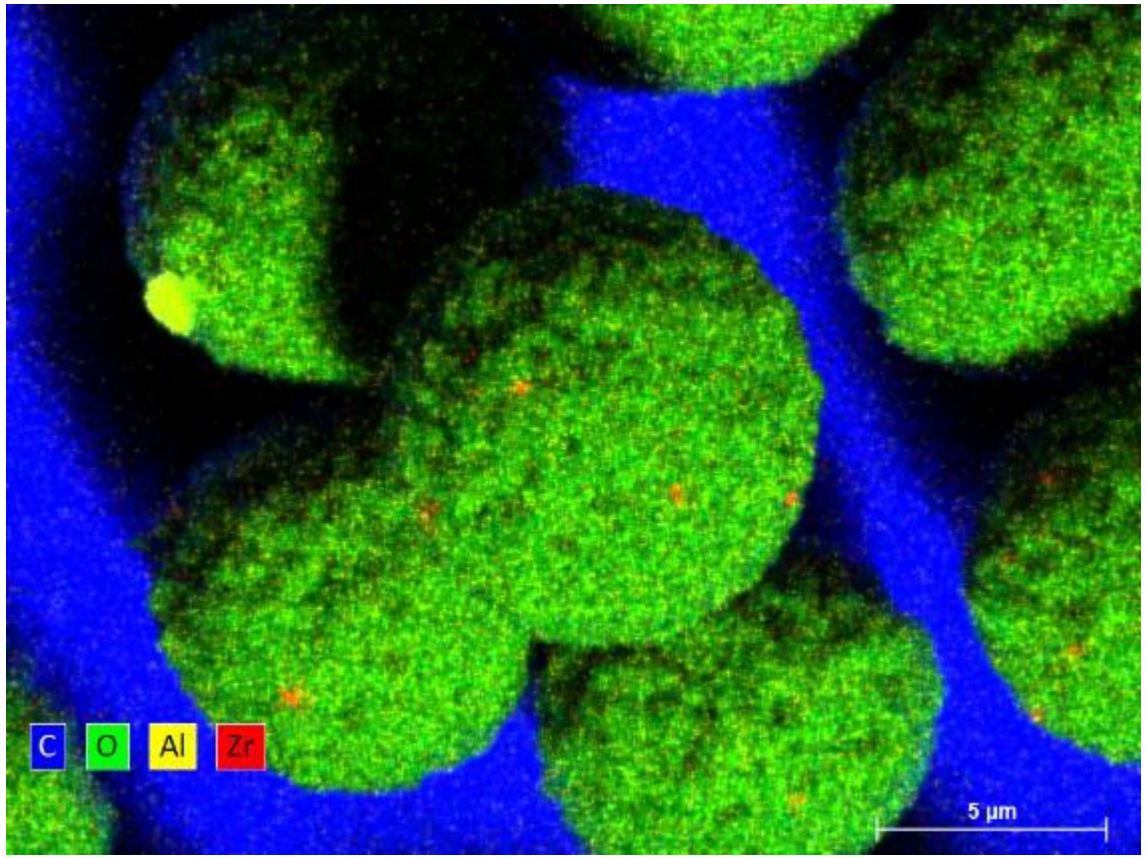

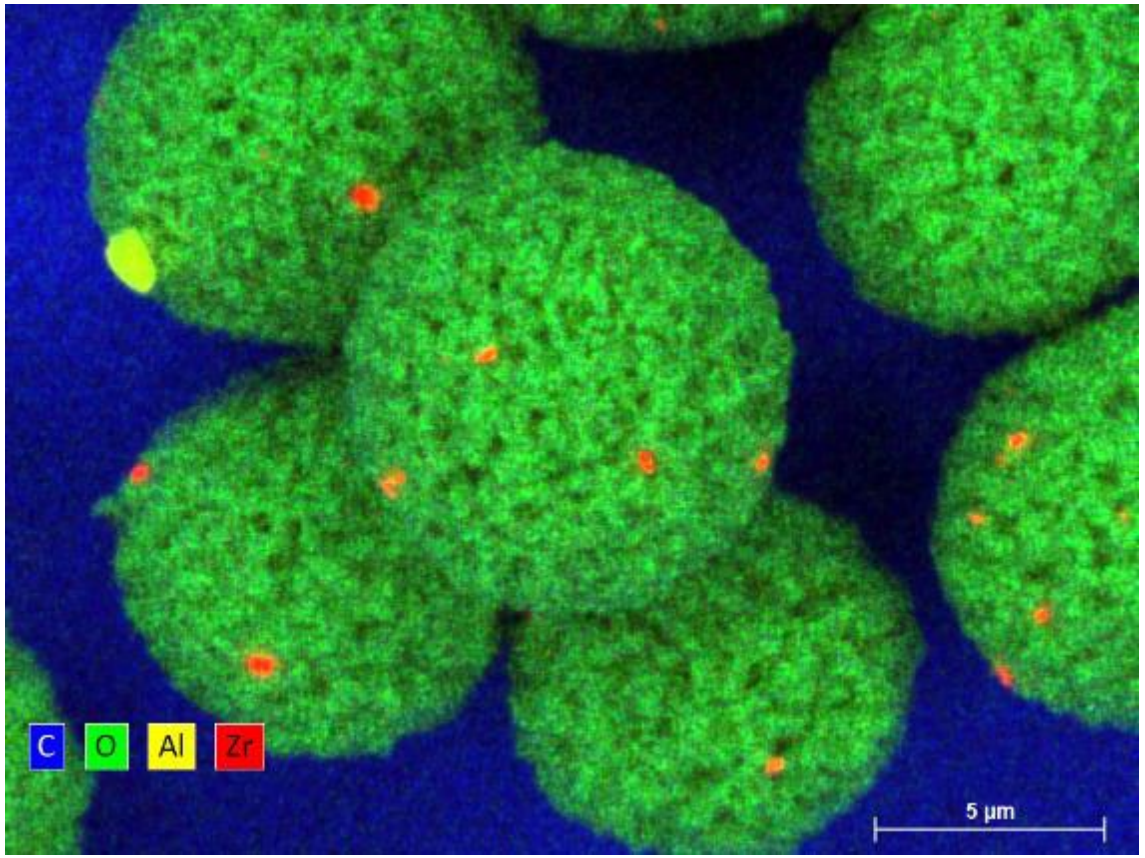

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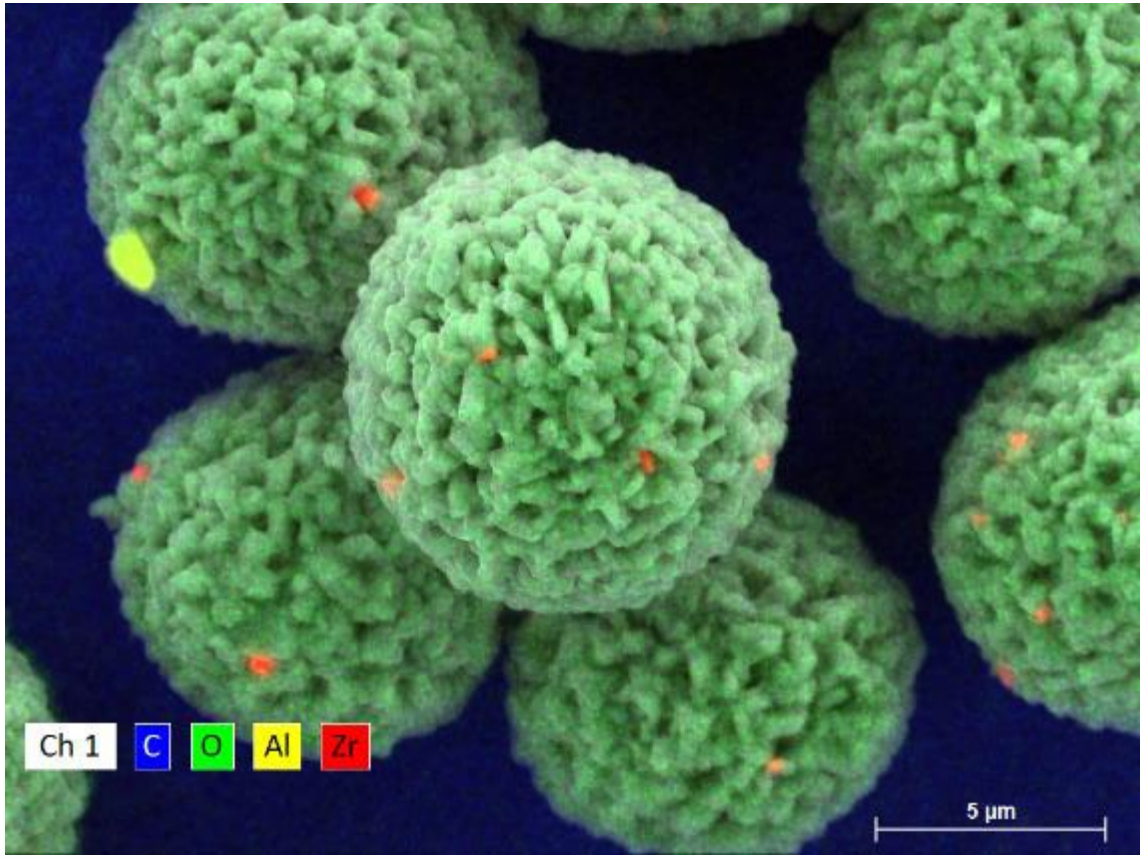


XFlash® FlatQUAD

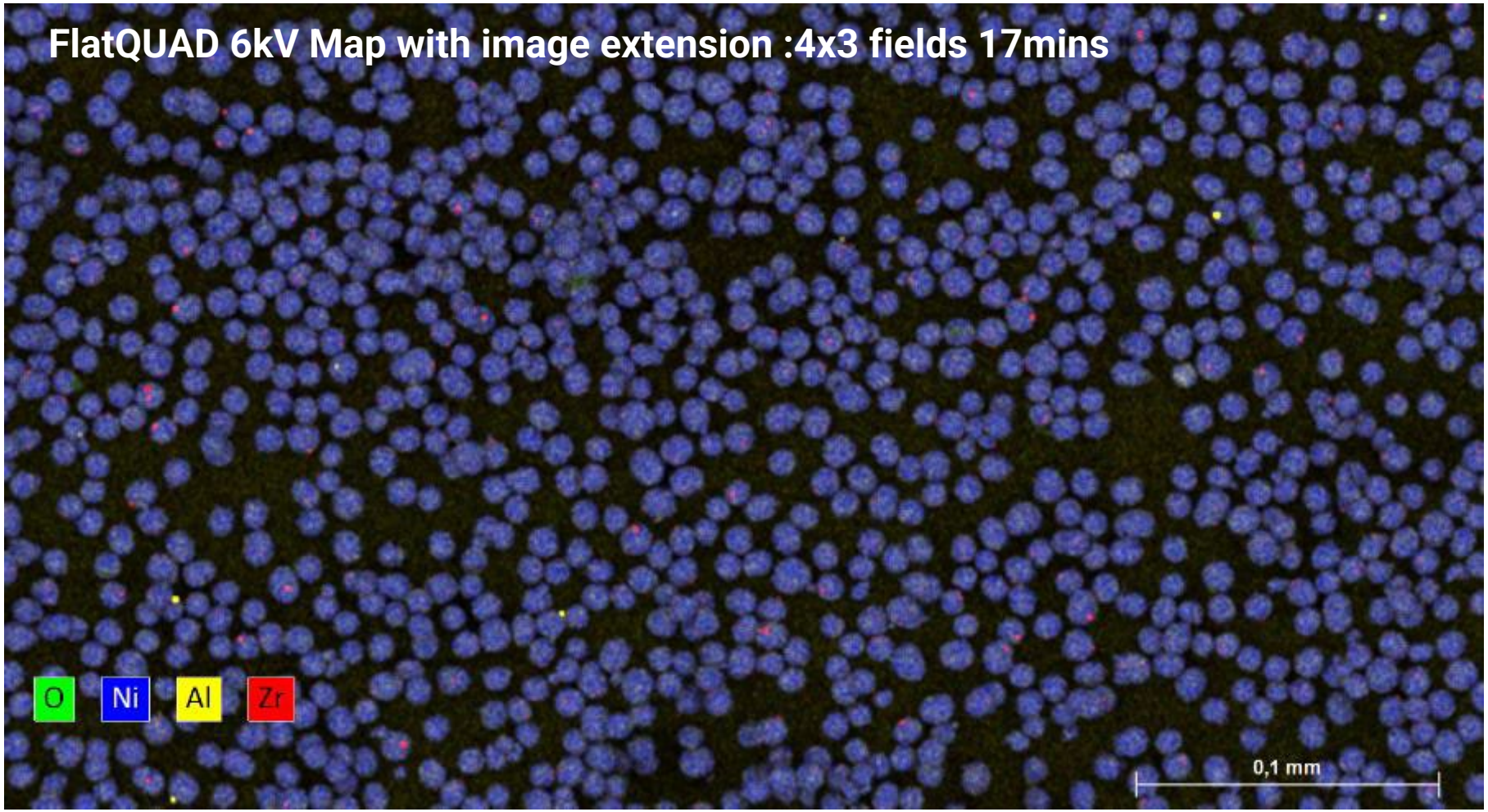
12 kV / 510pA / 600s / **133,900 cps**

Pre-screening of NCM particles: large area scans

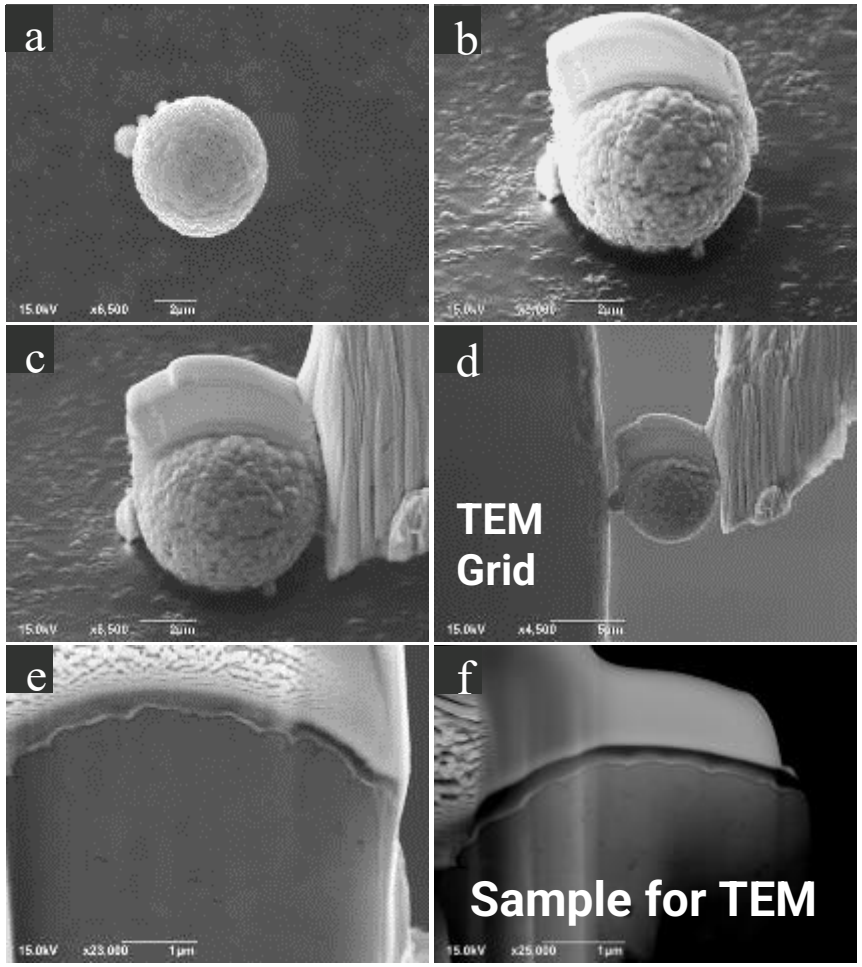
- FlatQUAD makes fast element distribution maps possible: ->survey of large areas, particle screening with microscope stage automation:

  Use higher beam current to maximize count rate

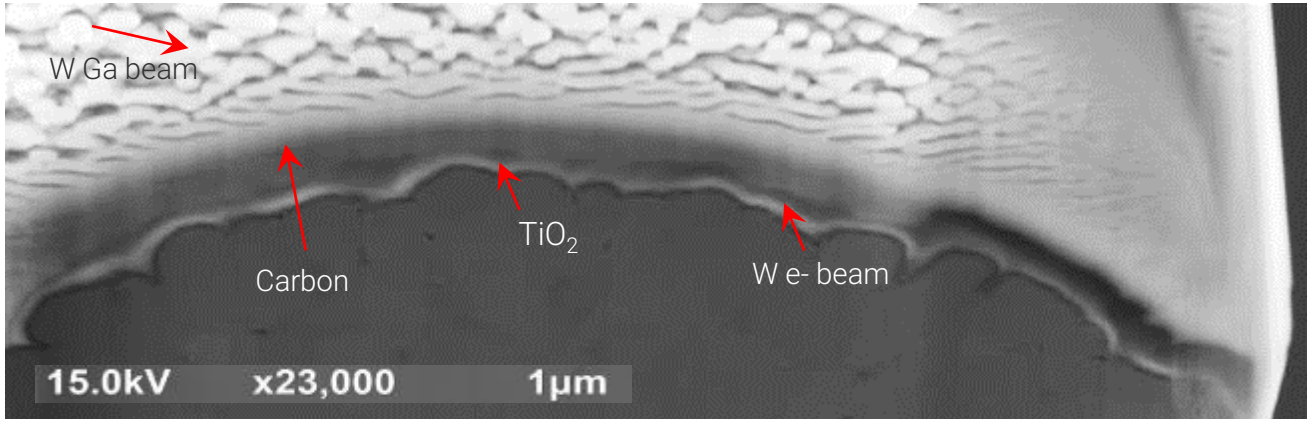
  –> minimize analysis time



ALD coated NCM particle - FIB lamella



Motivation: Ti-Coating to enhance capacity
Challenge: HAADF contrast can not distinguish between W protection layer and Ti coating –> measure with EDS

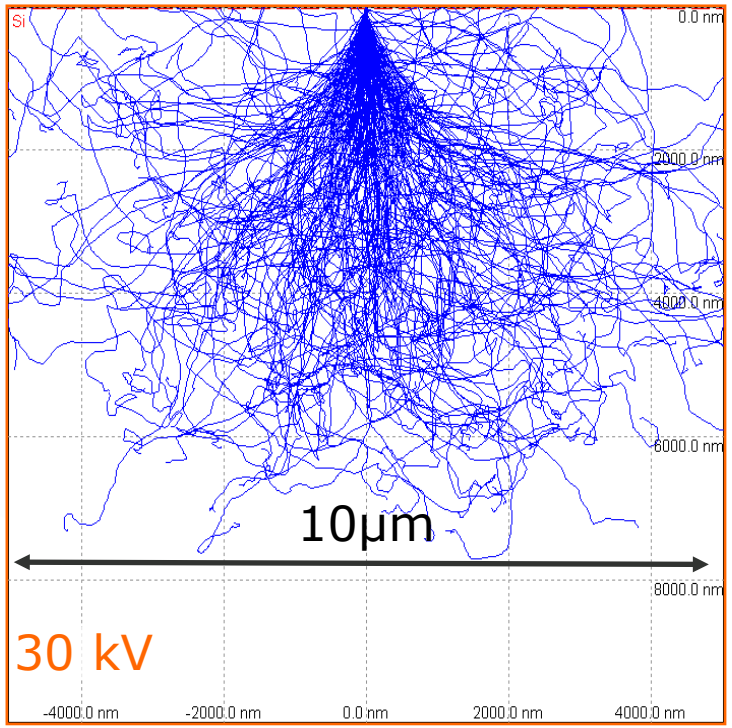


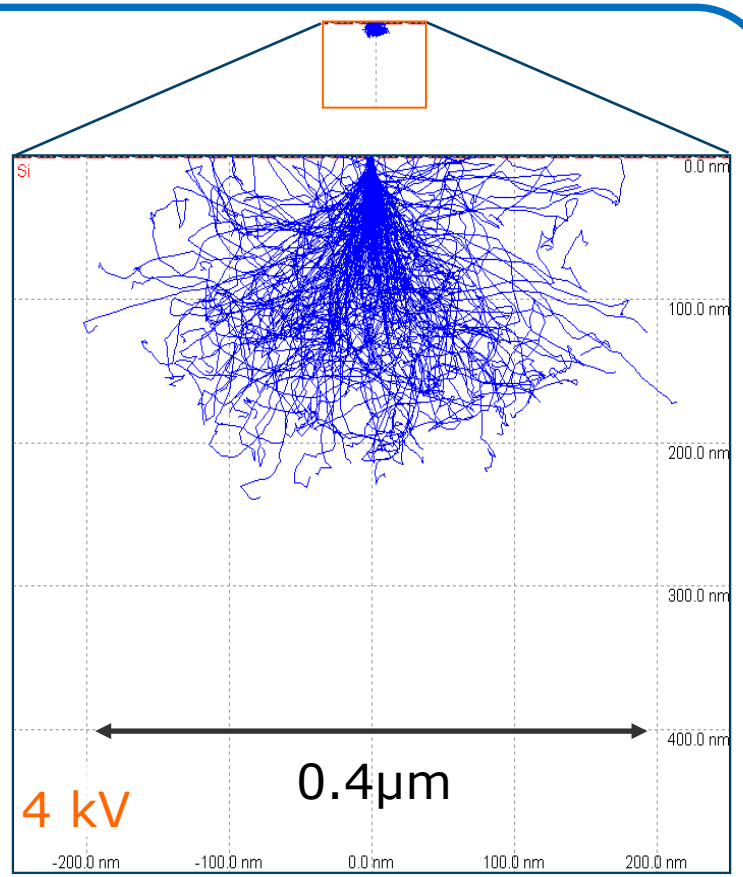
Watch on-demand joint webinar on on demand:
[Significance of STEM-EDXS Analysis in the Characterization of Rechargeable Battery Components]

Data courtesy:
M. Malaki, S. Ahmed, Philipps University Marburg, Germany

Spatial resolution of X-rays analysis

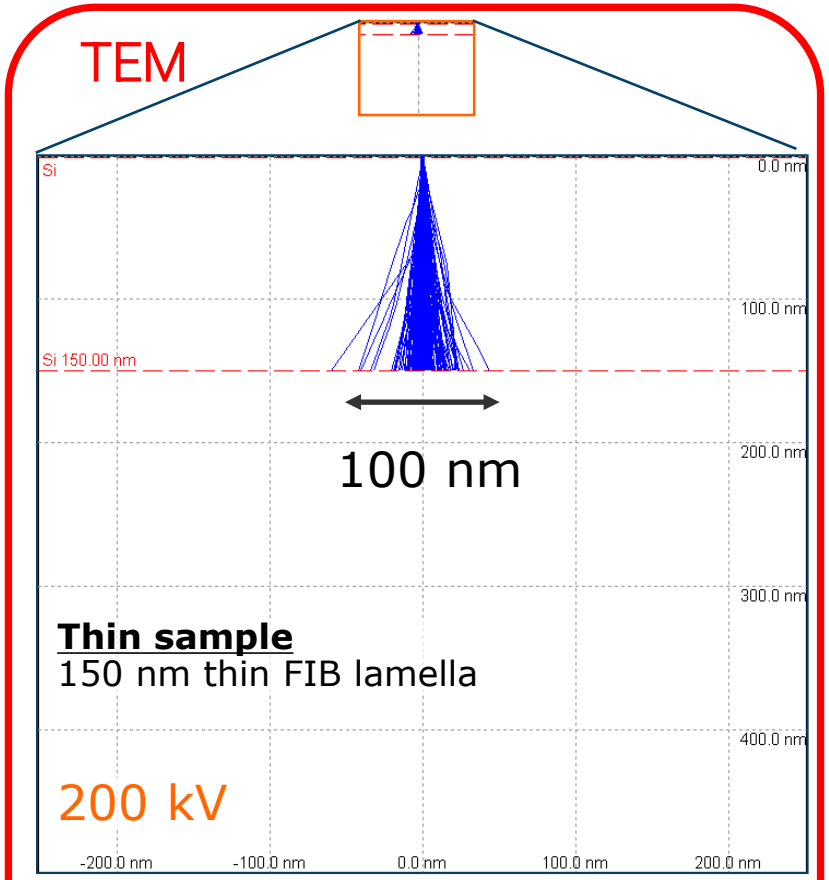
SEM





Si bulk sample

TEM

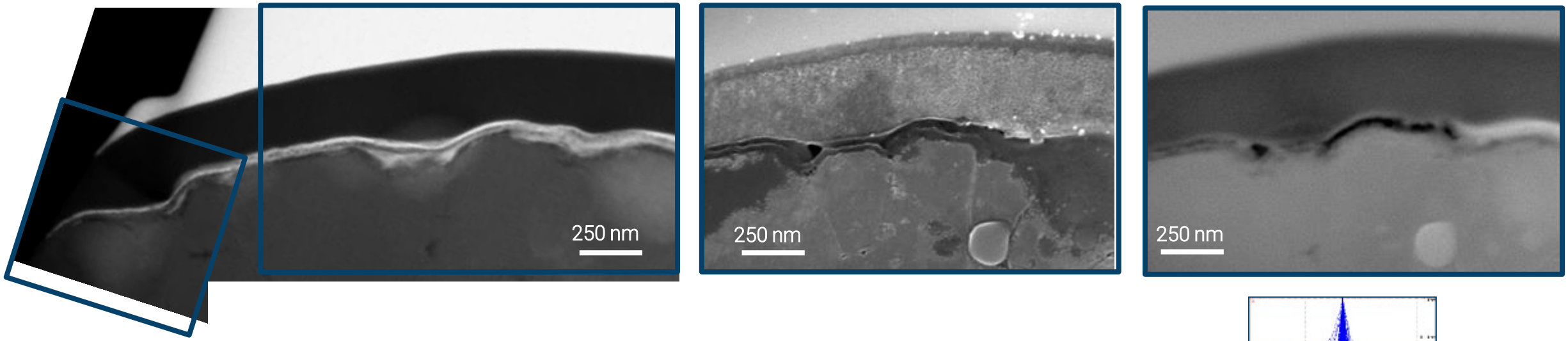


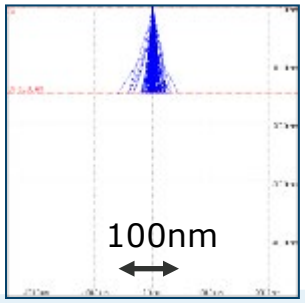
ALD coated NCM particle sample: STEM/EDS-SEM/EDS comparsion

STEM 200kV
HAADF image

SEM 20kV
In-lens image

SEM 20kV
SE image with
inserted FlatQUAD





Images taken under measurement conditions optimized for EDS analysis
Image quality does not affect EDS resolution on this scale!

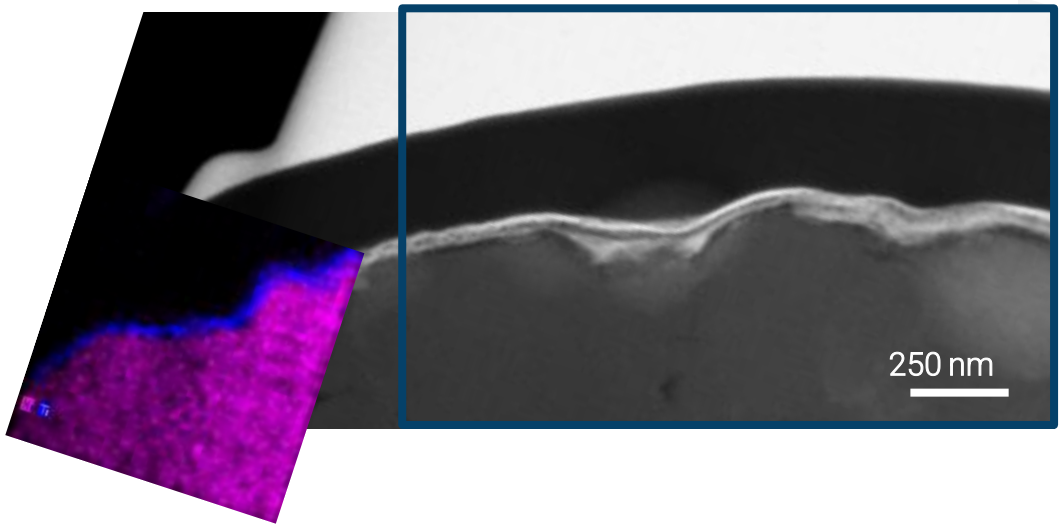
Sample and data courtesy: Michael Malaki, Shamail Ahmed, Materials Sciences Center, Philipps University Marburg

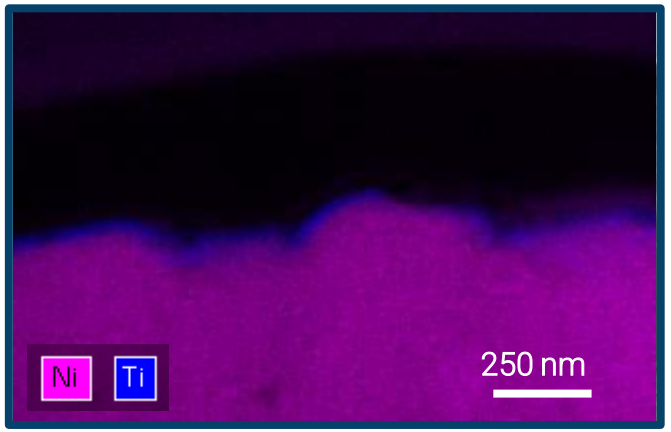
ALD coated NCM particle sample: STEM/EDS-SEM/EDS comparsion

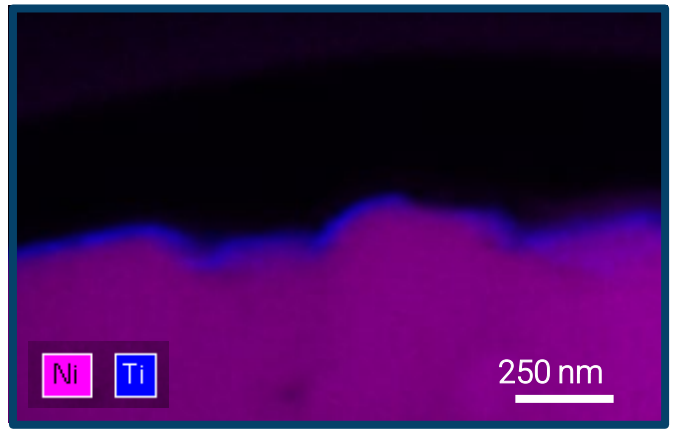
STEM 200kV
60 mm^2 EDS detector

SEM 20kV
60 mm^2 EDS detector

SEM 20kV
FlatQUAD detector







Total measurement time= 8 min
Beam current= 0.2 nA
Input count rate ~ 1,000 cps

Total measurement time= 34 min
Beam current=2 nA
Input count rate ~ 30,000 cps

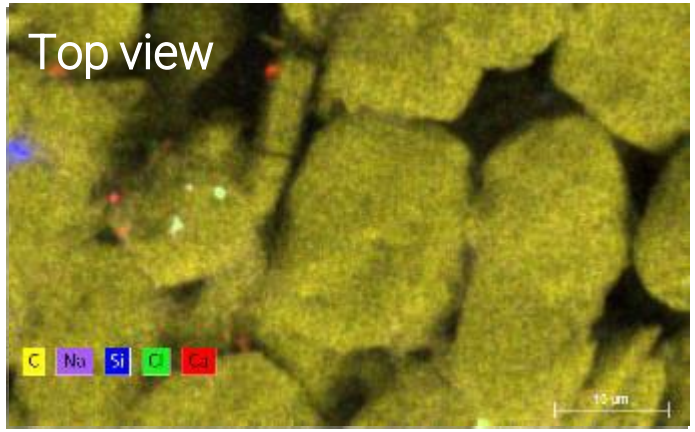
Total measurement time= 5 min
Beam current= 2 nA
Input count rate ~ 460,000 cps

Sample and data courtesy: Michael Malaki, Shamail Ahmed, Materials Sciences Center, Philipps University Marburg

Ex-situ contamination analysis of anode/cathode material

Anode sample:

graphite on copper (not cycled)



Cathode sample:

LiPO4 on aluminum (not cycled)

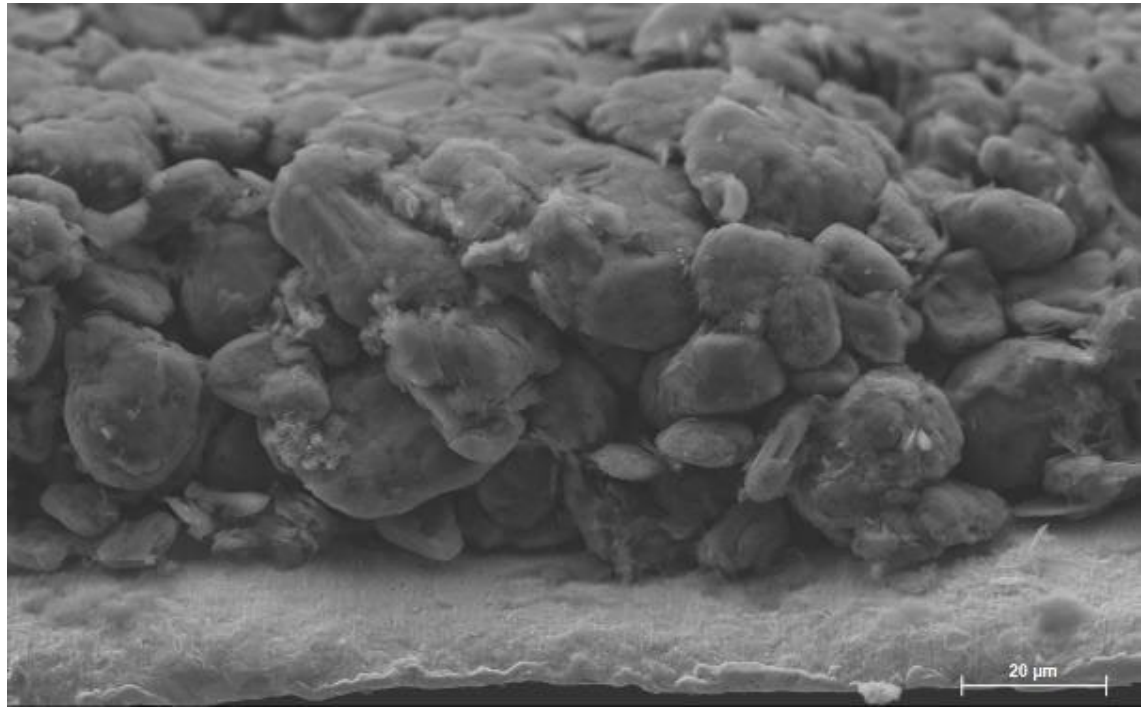
Join our joint webinar on 2nd November 2023!

Elemental Mapping (EDS) for the Optimization of Battery Materials and Processes

Register under:

https://www.bruker.com

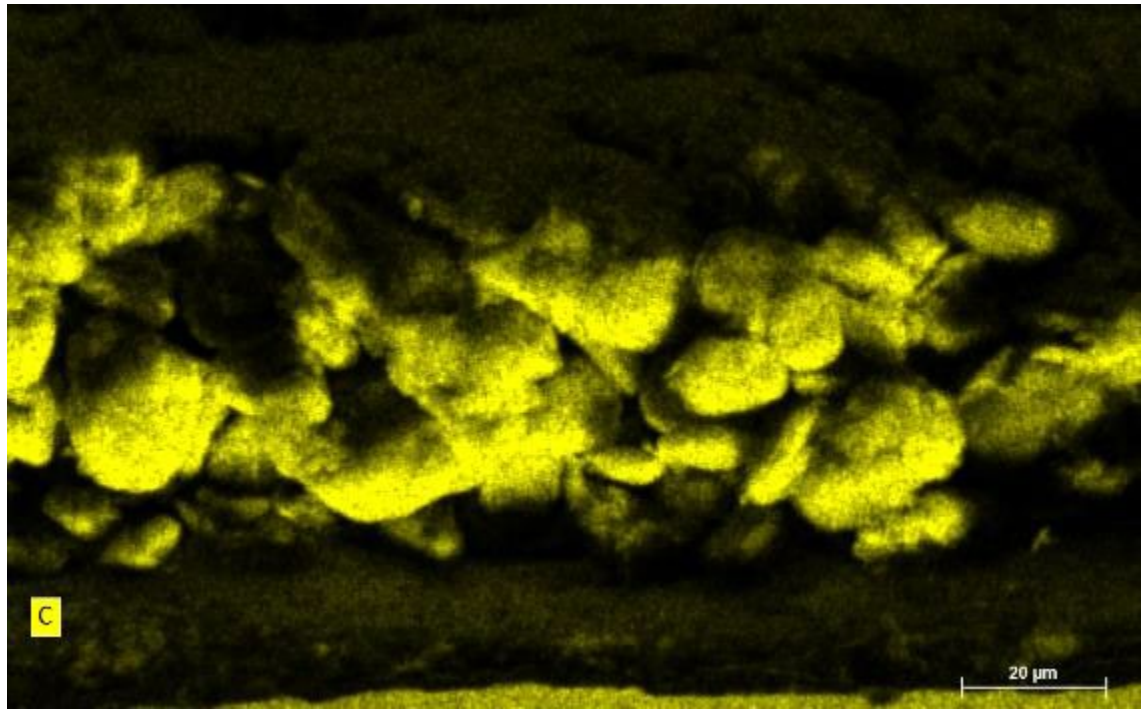
ANODE cross section – XFlash® 760 vs FlatQUAD

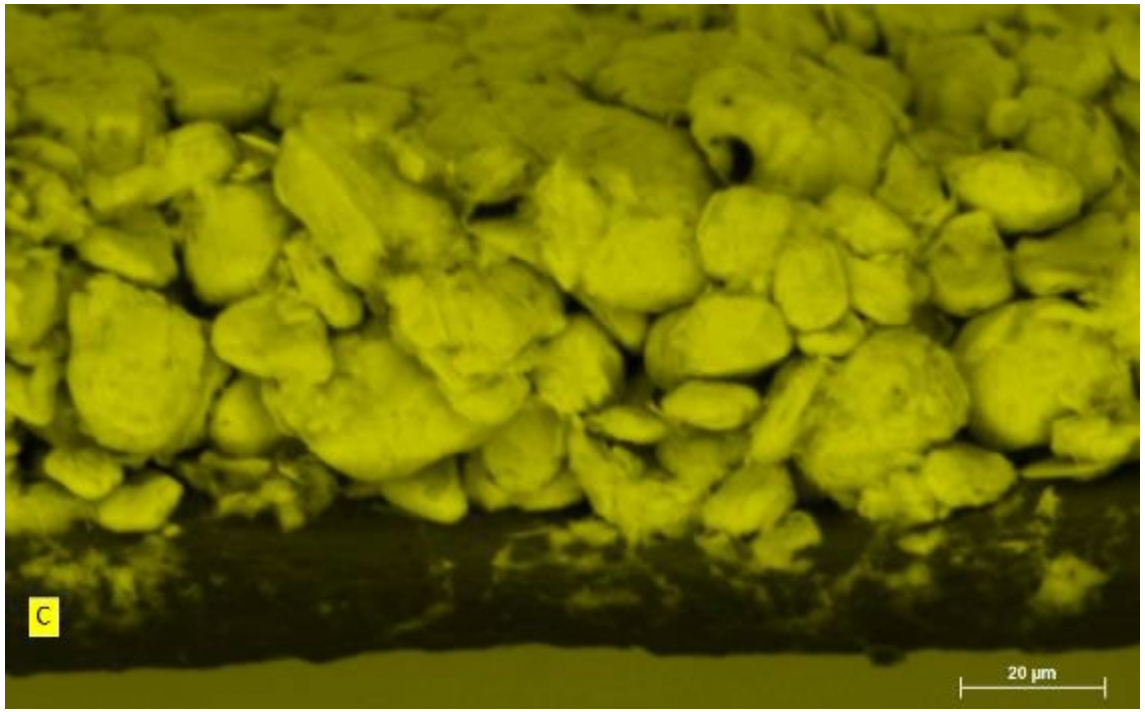


Analysis parameters		
Detector	XFlash® 760	XFlash® FlatQUAD
High voltage	12 kV	12 kV
Beam current	1.2 nA	1.2 nA
Mapping time	30 min	7 min
Input count rate (ICR)	19,500 cps	667,000 cps

ANODE cross section – XFlash® 760 vs FlatQUAD

Shadowed areas





XFlash® 760

XFlash® 760

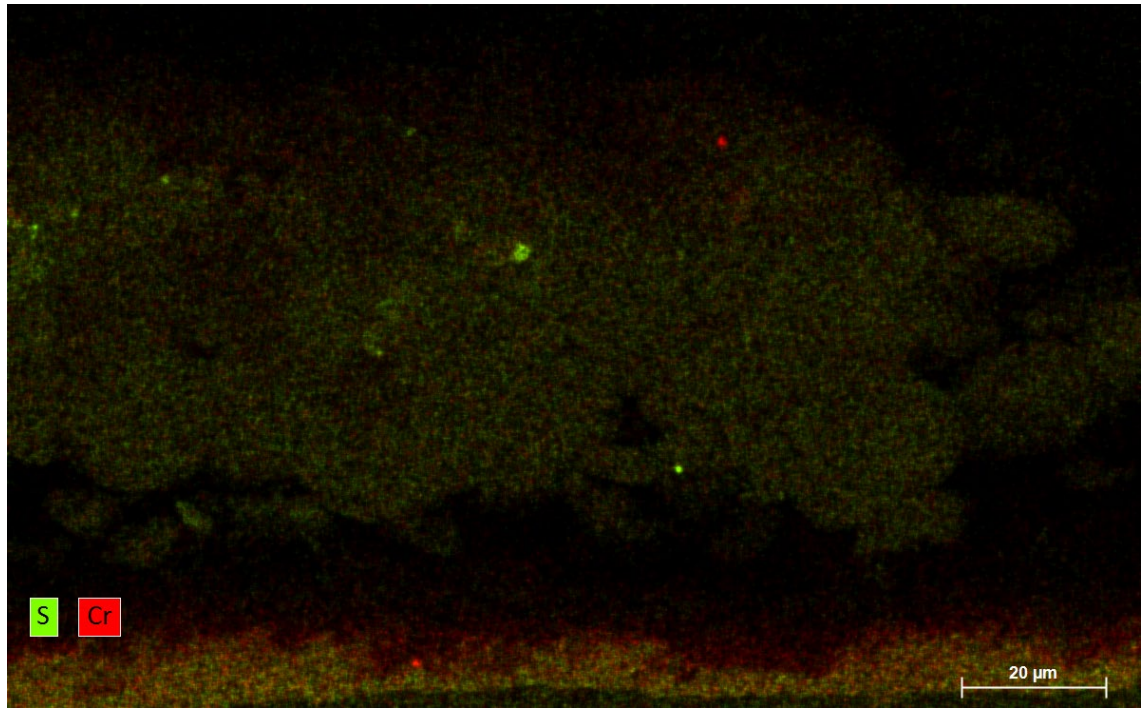
12 kV / 1.2 nA / 30m / **19,500 cps**

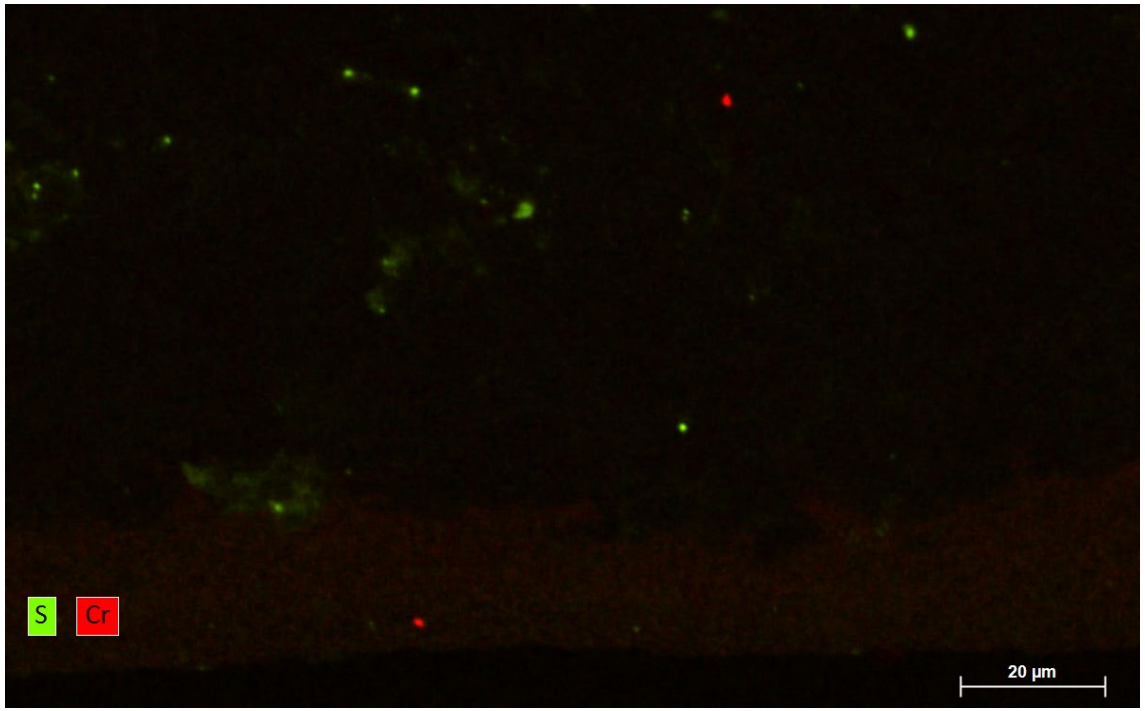
XFlash® FlatQUAD

XFlash® FlatQUAD

12 kV / 1.2 nA / 7m / **667,400 cps**

ANODE cross section – XFlash® 760 vs FlatQUAD:
Finding “hiding elements”: S, Cr





XFlash® 760

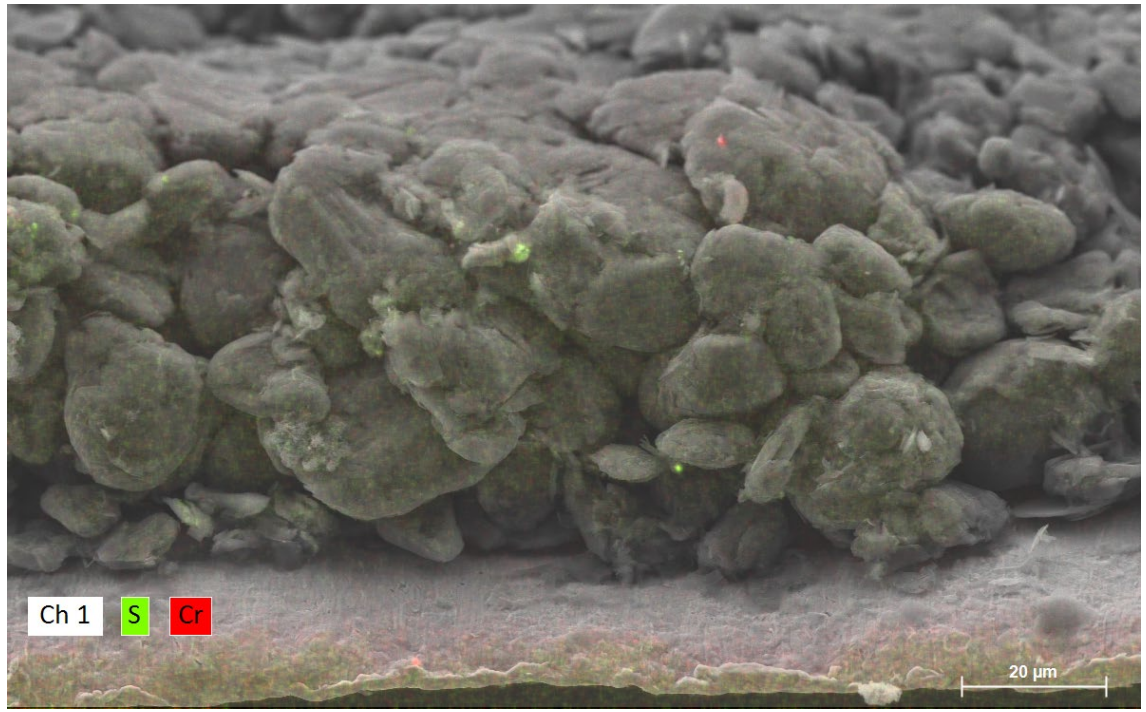
Noisier maps,
Shadowed areas

XFlash® FlatQUAD

Less noise in maps,
Access to deeper /shadowed areas

Webinar link: Elemental Mapping (EDS) for the Optimization of Battery Materials and Processes

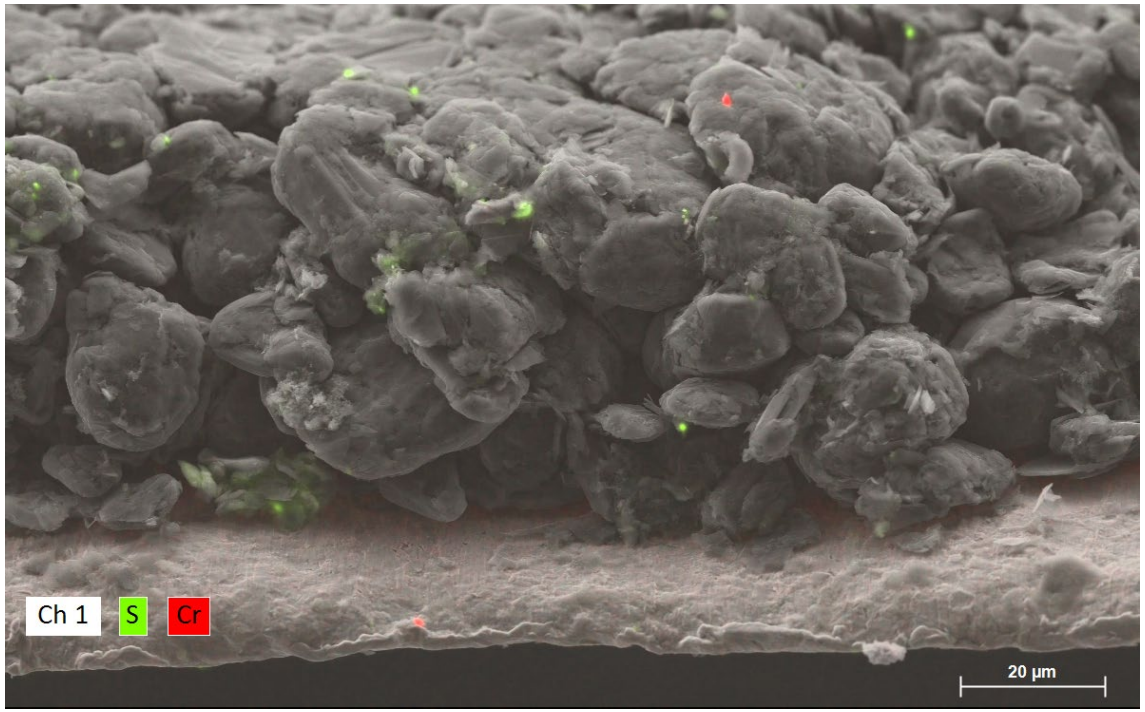
ANODE cross section – XFlash® 760 vs FlatQUAD:
Finding “hiding elements”: S, Cr



XFlash® 760

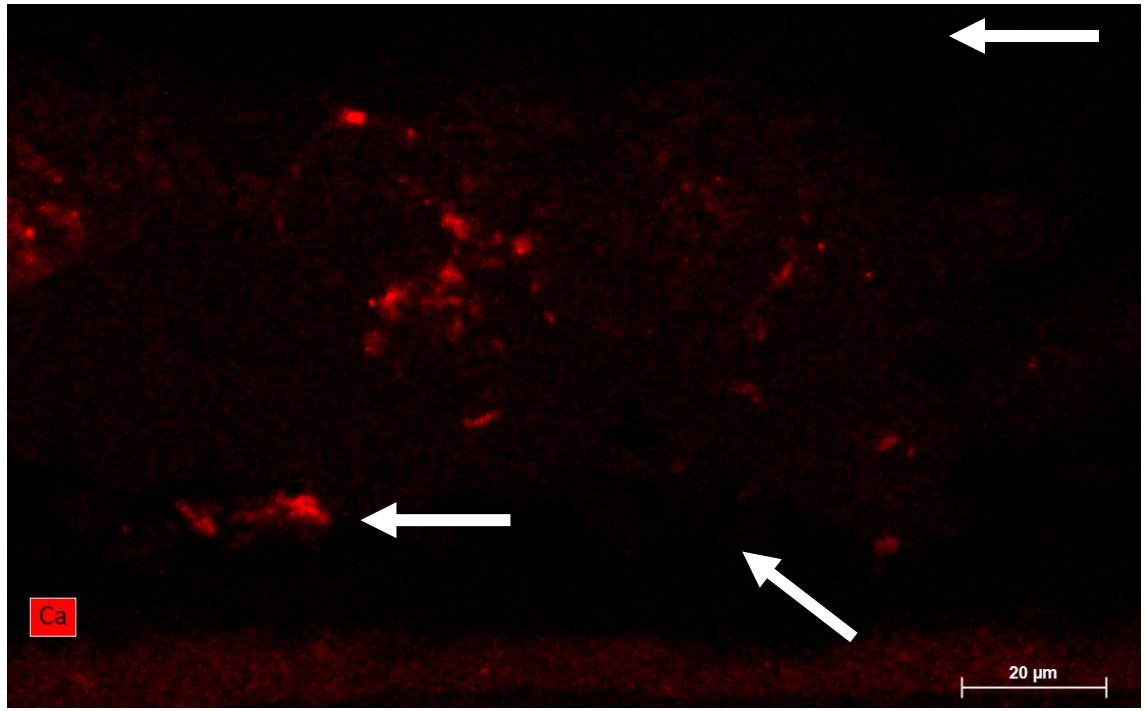
Noisier maps,
Shadowed areas

XFlash® FlatQUAD



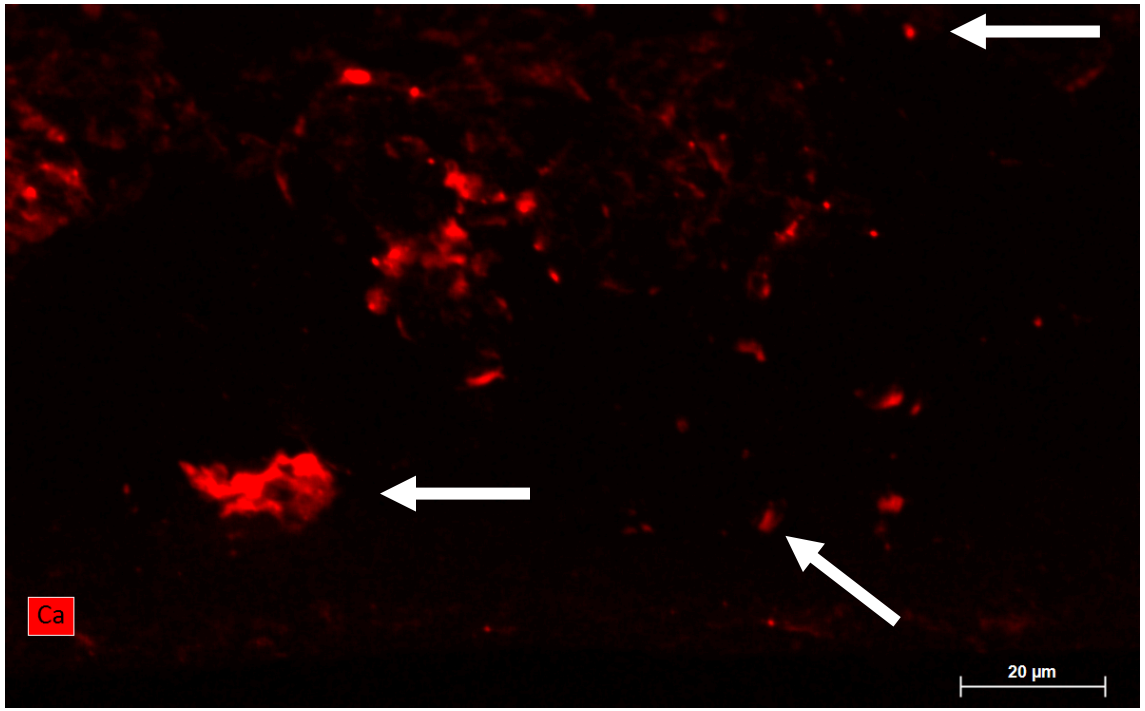
Less noise in maps,
Access to deeper /shadowed areas

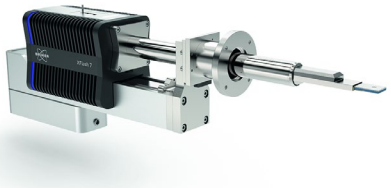
ANODE cross section – XFlash® 760 vs FlatQUAD:
Finding “hiding elements”: Ca



XFlash® 760

Noisier maps,
Shadowed areas





XFlash® FlatQUAD

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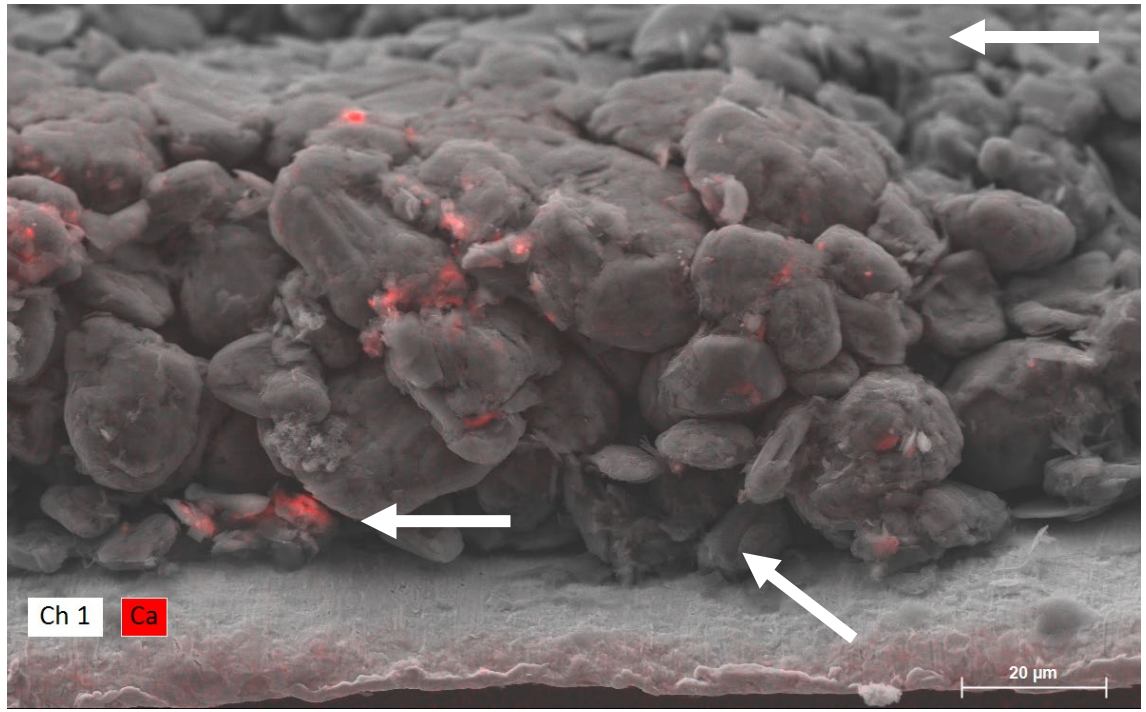
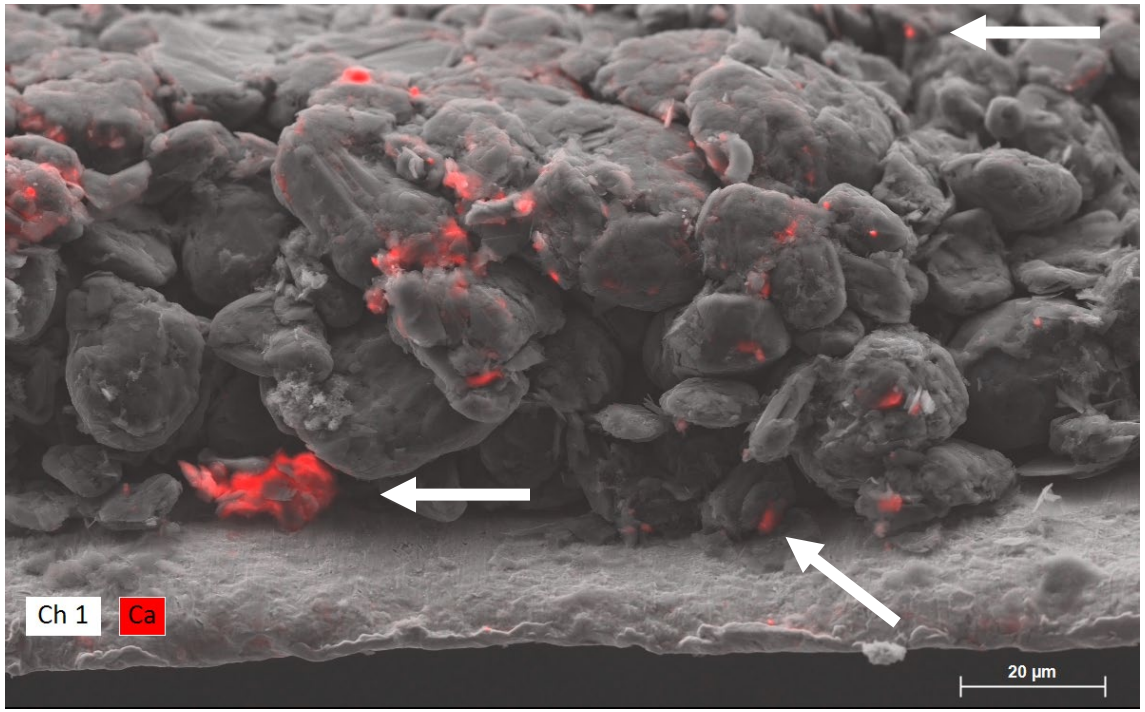
ANODE cross section – XFlash® 760 vs FlatQUAD:
Finding “hiding elements”: Ca





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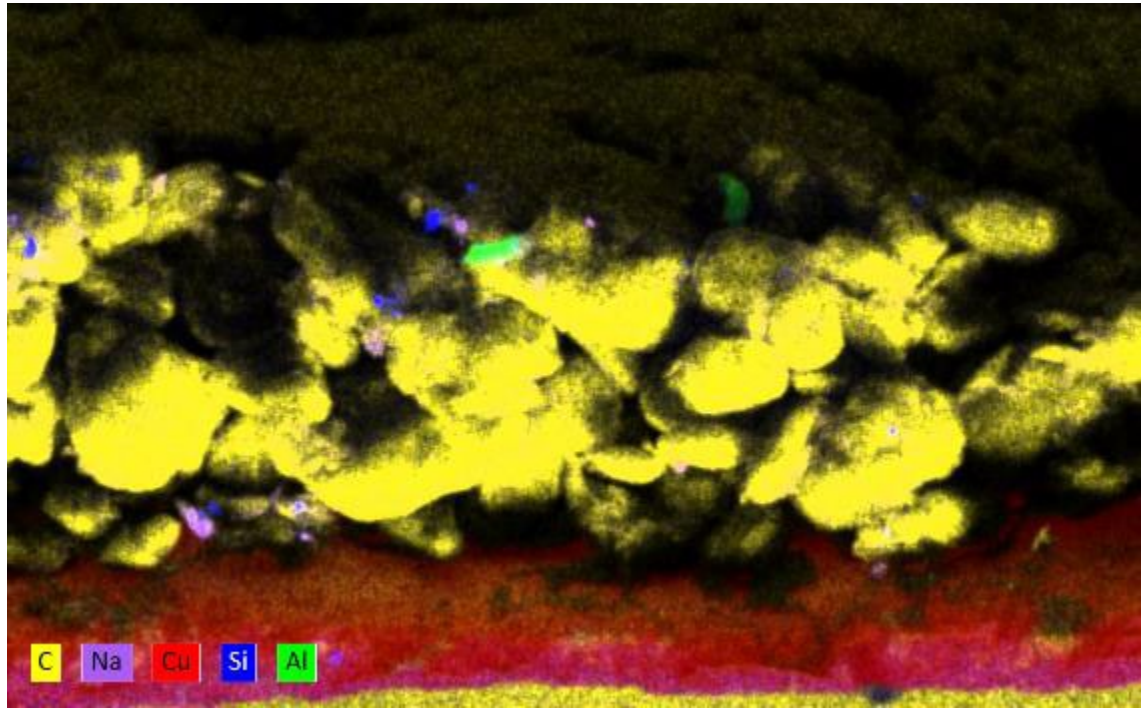

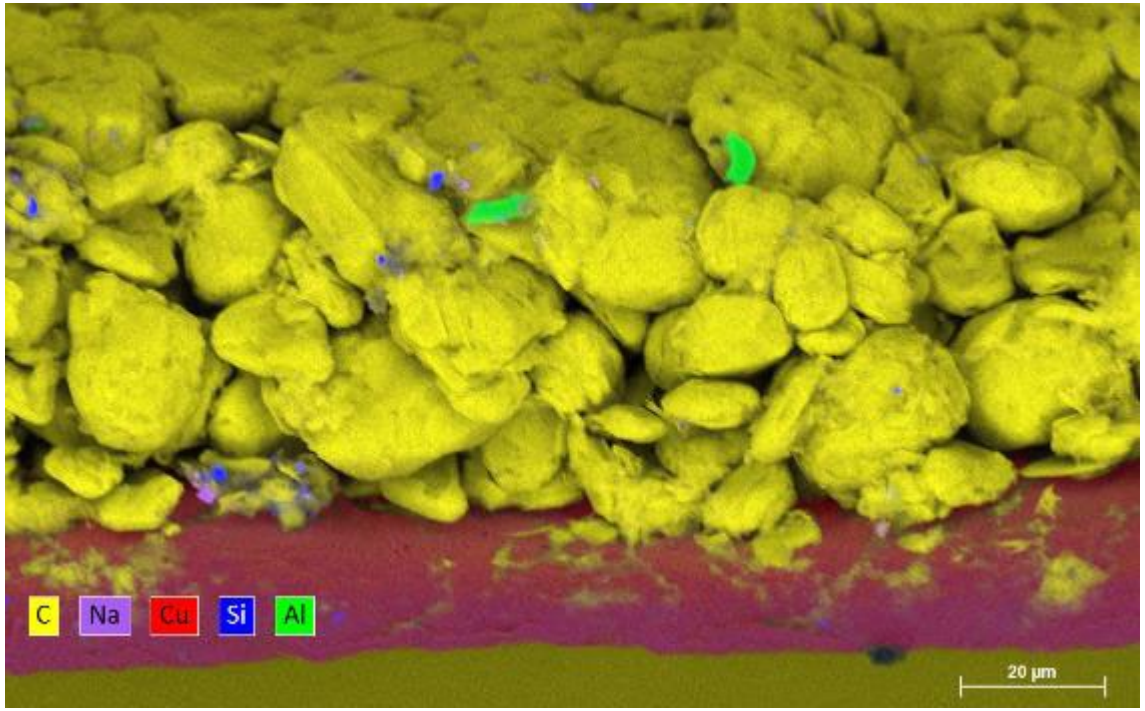

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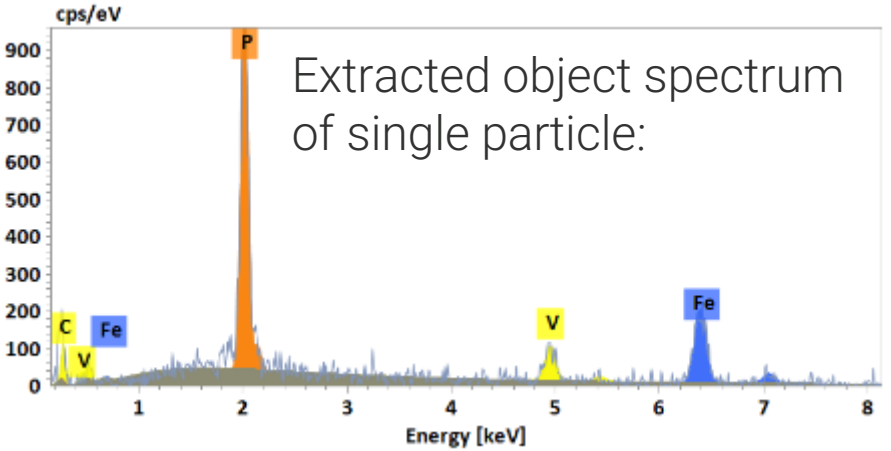
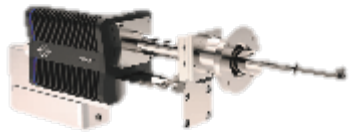
Webinar link: Elemental Mapping (EDS) for the Optimization of Battery Materials and Processes

CATHODE - Identify and locate contaminants in very low concentration



Extracted object spectrum of single particle:

Element	At. No.	Line series	Mass Norm. [%]	Atom [%]	abs. error [mass%] (3 σ)
C	6	K	6,02	19,63	2,40
P	15	K	24,35	30,79	6,66
V	23	K	11,43	8,79	4,78
Fe	26	K	58,19	40,80	25,64
			100,00	**100,00**	



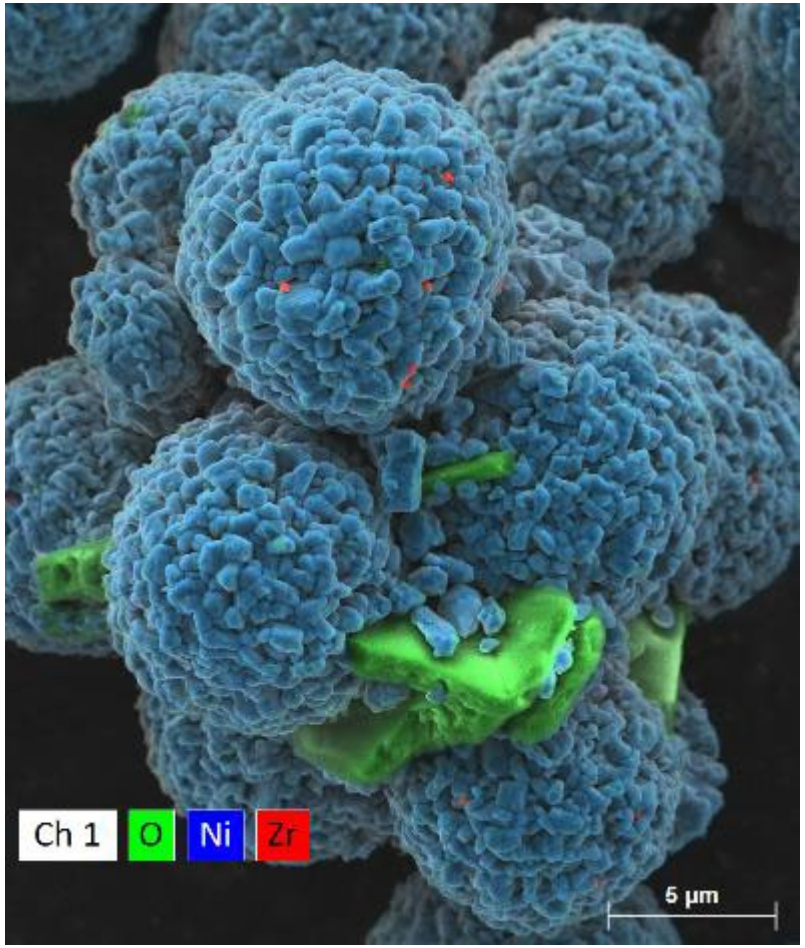
XFlash® FlatQUAD

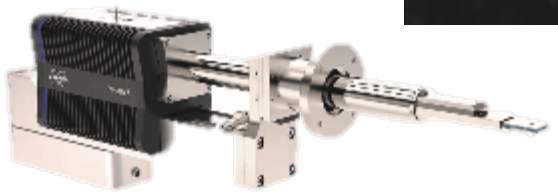
Area coverage= 0.01%

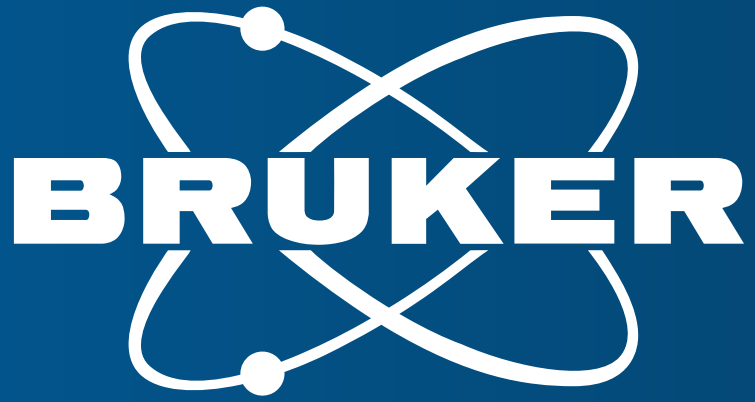
Local concentration of V: 11% -> **11 ppm V** detected within **3 minutes** of measurement time!

Summary

- XFlash® FlatQUAD: High sensitivity, high speed at low probe currents
- Faster chemical analysis of battery materials at high resolution / large area analysis
- Battery materials highly topographic: Better view of topography, hidden elements/contaminants visible
- 4 segment, side entry EDS detector, detection from Boron, ideal for battery applications







Innovation with Integrity

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