BRUKER WEBINAR WITH DRAGONFLY ENERGY CORP.

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

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- Dr. Igor Nemeth

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Outline

01 EDS and multi-model analytical applications in Battery Cell R&D

02 EDS analysis for detecting and identifying contaminants in Cathode and Anode

03 Conclusions

04 Question & Answer session

01

EDS and multi-model analytical applications in Battery Cell R&D

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Revolutionizing Green Energy Storage.

EDS Applications in Battery Cell R&D

Presenter:

Emily Litt,
Research & Development Scientist
& Group Lead

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DRAGONFLY ENERGY

- Comprehensive Lithium Battery Company
  - Proven Market Penetration Through Battle Born Batteries Product Line
  - Strong Vision for Future Domestic Cell Production and Technology

Over 225,000
Batteries
Sold Since 2020

$86
Million
2022 Revenue

10+
Years
of Research & Development in Battery Technologies

85+
Patents
Filed & Pending

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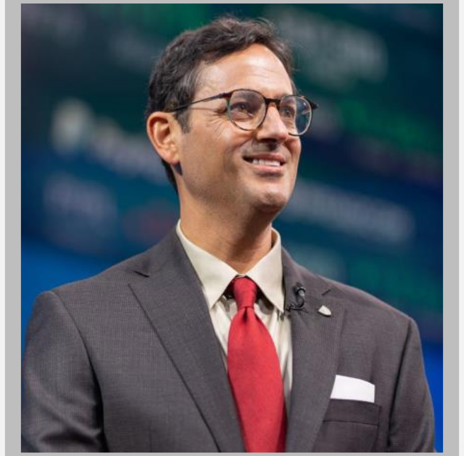
THE TEAM



Denis Phares, Ph.D

Chief Executive Officer

- 30+ years of experience in the fields of Energy, Nanotechnology, Fluid Mechanics, and powder processing
- B.S. in Physics from Villanova University
- M.S. and Ph.D in Environmental Engineering Science from California Institution of Technology
- MBA from the University of Nevada, Reno

Vick Singh, Ph.D

Director of Research and Development

- 10+ years of experience in various research functions within private & public sector firms
- Postdoctoral Research Fellow at the Lawrence Livermore National Laboratory's Center for Global Security Research
- B.S. in Chemical Engineering from the University of Tennessee, Knoxville
- Ph.D in Materials Science & Engineering from the University of Nevada, Reno

Emily Litt

Research & Development Scientist & Group Lead

- B.S. in Material Science and Engineering from the University of Nevada, Reno
- Leads team in systematic development of innovative methods to enhance lithium-ion batteries
- Conducts electrochemical experiments and materials analysis, aiming to optimize battery performance and efficiency

15+ Engineers and Scientists Supporting Research Efforts

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Dragonfly Energy's approach to energy technology development can be described in one word:

Comprehensive

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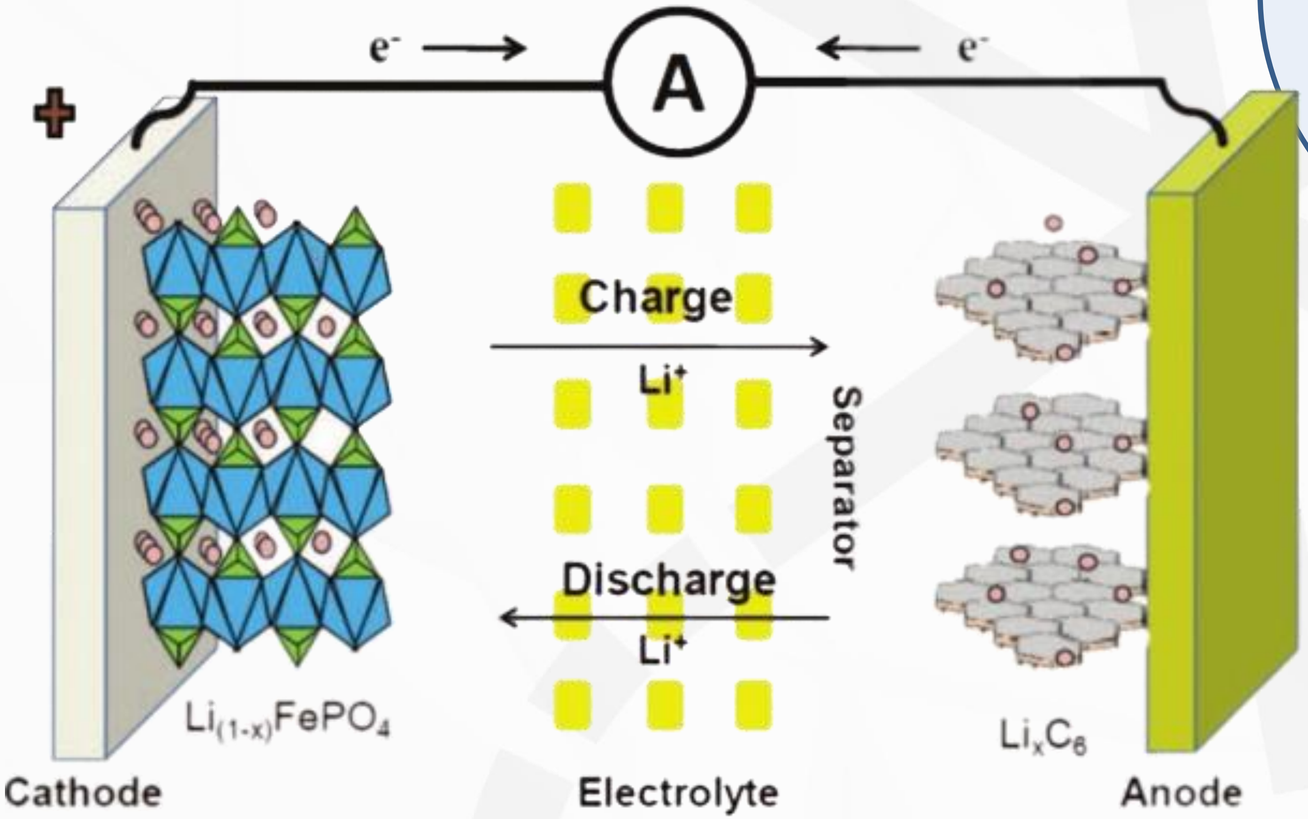
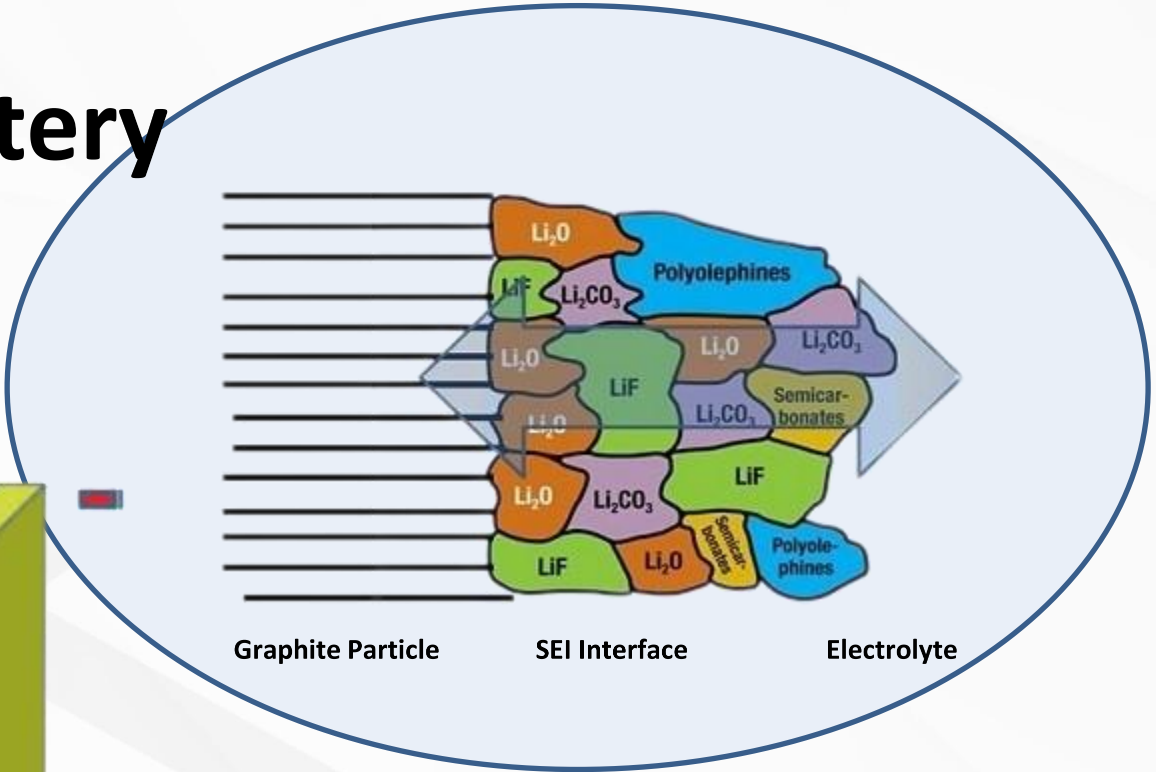
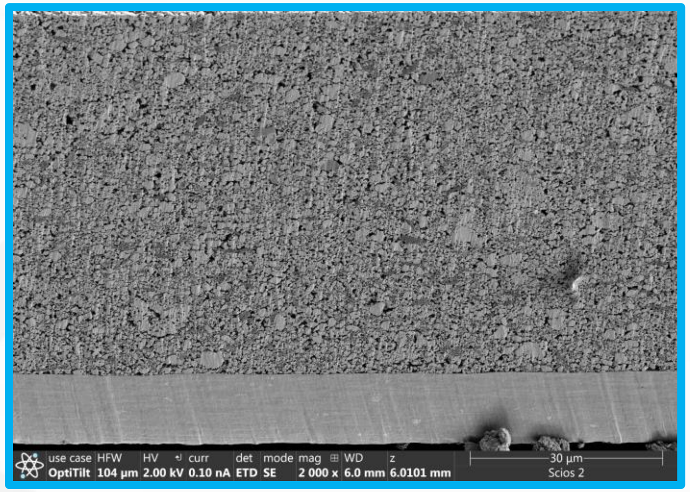
How Does a Lithium-Ion Battery Work?

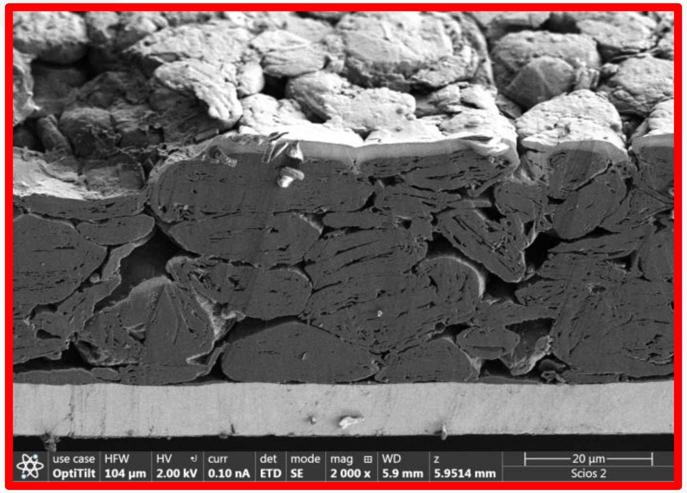




How are Lithium-ion Batteries Made?



Lithium Iron Phosphate Cathode



Graphite Anode

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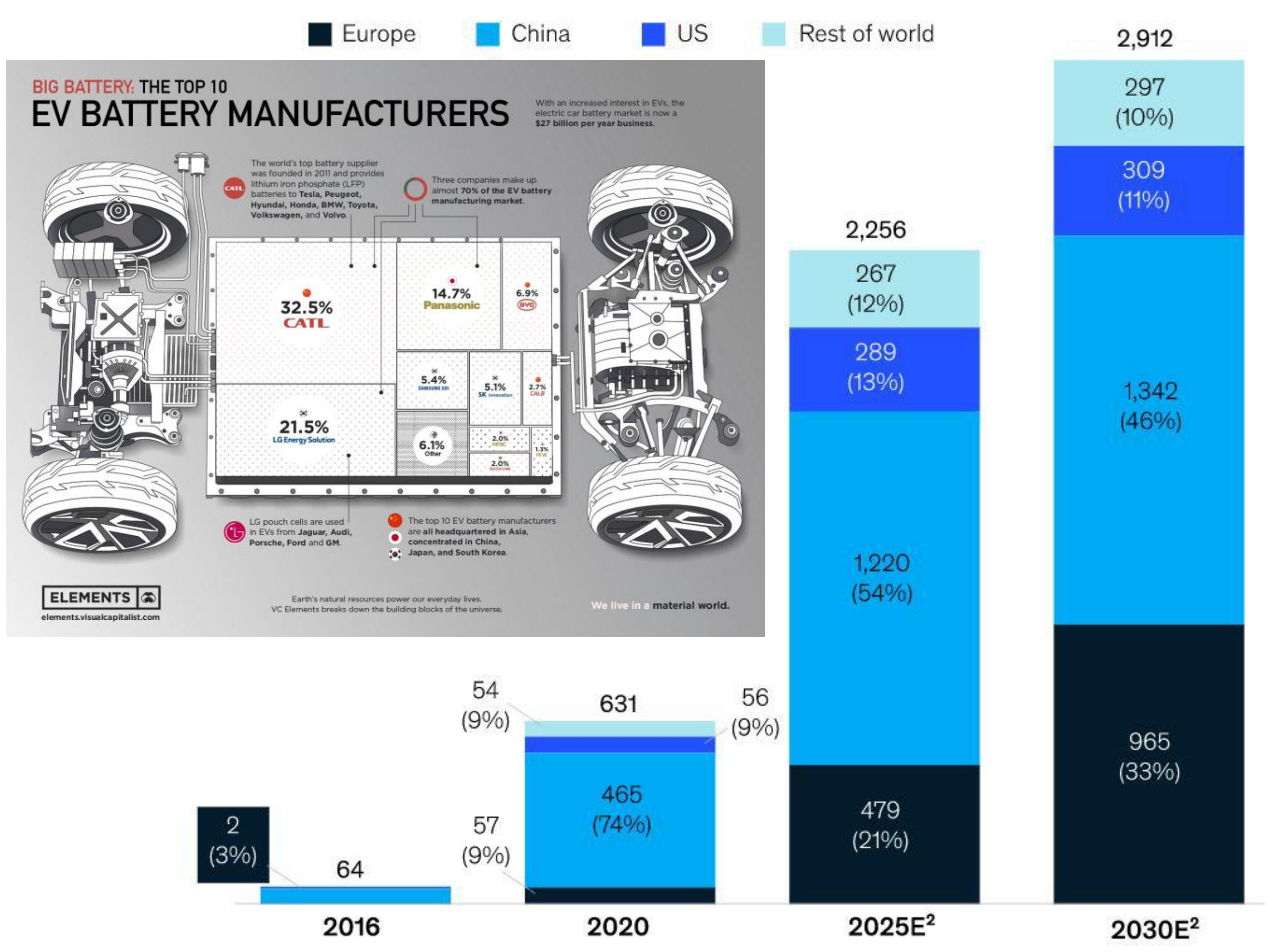
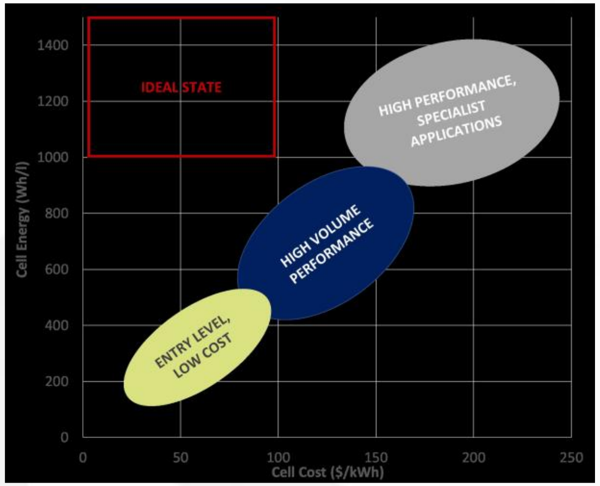
What’s the current status of lithium-ion?

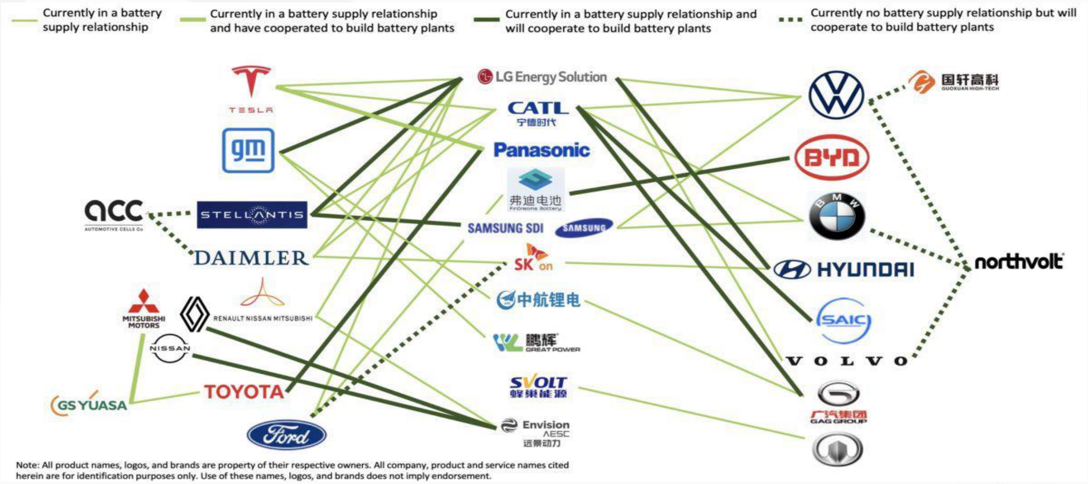




ENTRY LEVEL LOW COST				
LFP	Na-ion	Mn-rich	Na-Ni-Cl	NFA

HIGH VOLUME PERFORMANCE					
NCA	NMC	NCMA	eLNO	Gr-Si Blend Anodes	Mn-rich

HIGH PERFORMANCE, SPECIALIST APPLCIATION			
Ultra-High Ni Blends	Silicon Anodes	Lithium Metal Anodes	Solid State Electrolyte
Li-S	HV-Spinel Cathodes	Rapid Charge Anodes	Lithium Air



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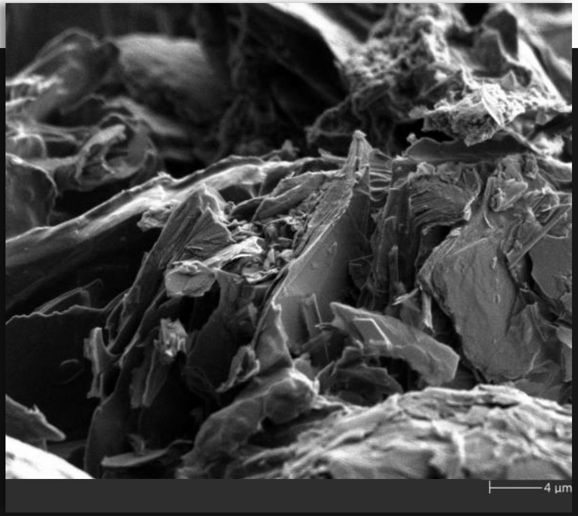
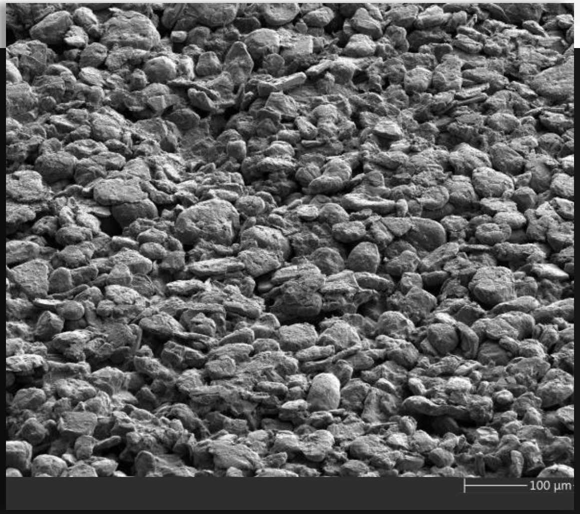
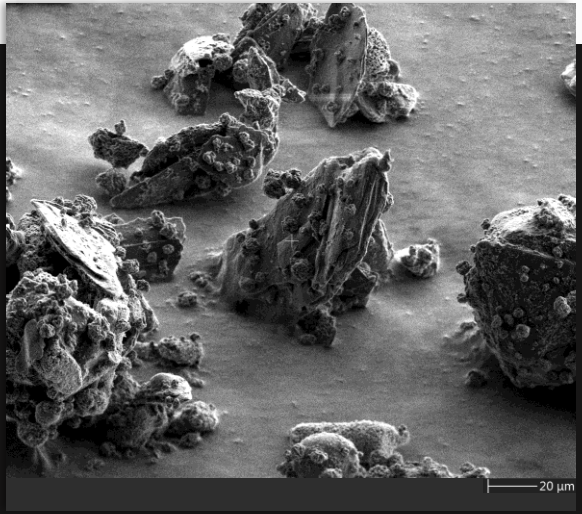
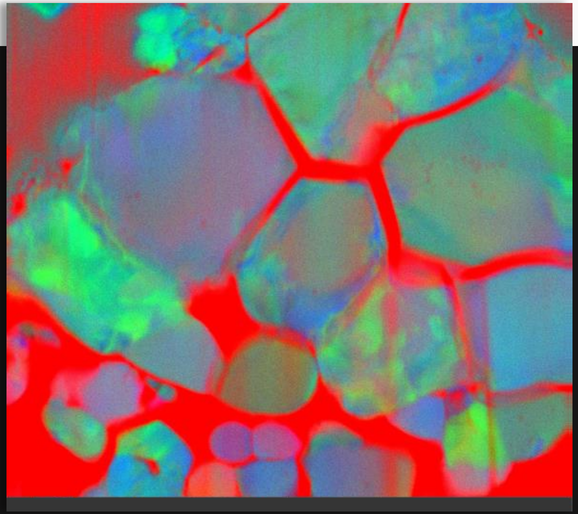
FUNDAMENTAL MATERIALS AND PROCESSES

- Founded as a manufacturing technology company in 2012
- Applying sustainable manufacturing processes to current and next-generation cell chemistries









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FUNDAMENTAL MATERIALS AND PROCESSES

- Fundamental Capabilities:
  - Infrastructure
    - +$15 million investment with Bruker and Tescan for scientific instruments
  - Highly-Skilled R&D Team and Thrust Areas
    - Battery Cell Science
    - Manufacturing Innovation
    - Materials Characterization
    - Data Science

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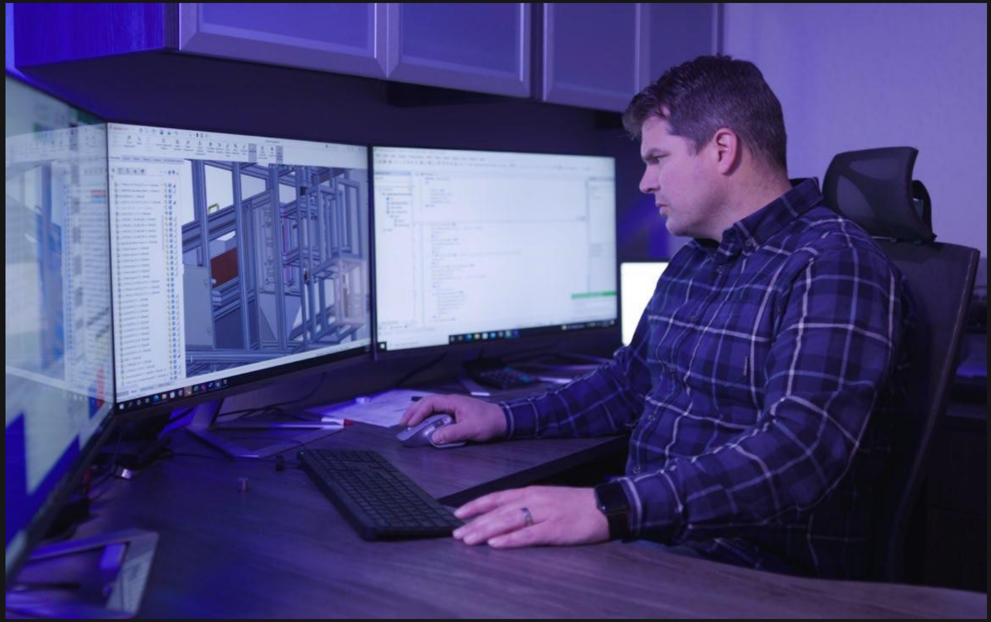
Streamlined Cell Manufacturing & Battery Pack Assembly

- **Optimization of Manufacturing Processes**
  - Extensive Automation Experience
  - Designed & Built in House
  - Cost Effective
- **Dry Room**
  - Coming Soon for Conventional and Solid-State Cell Prototyping
- **Decreased Manufacturing Footprint**
- **Up to 150 MWh in Cell Production Capacity via Pilot Line**



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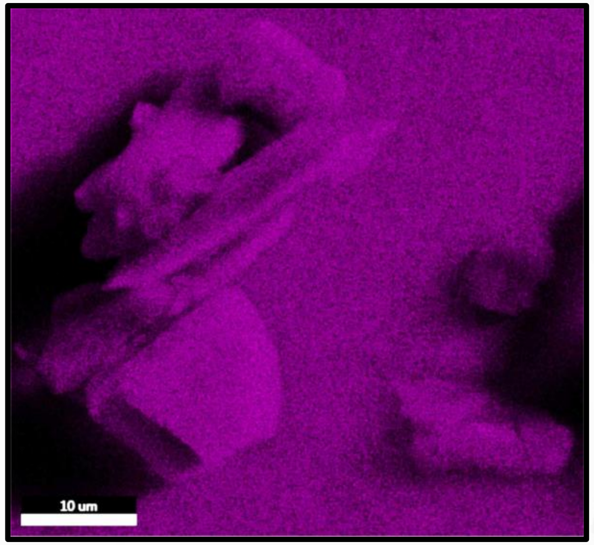
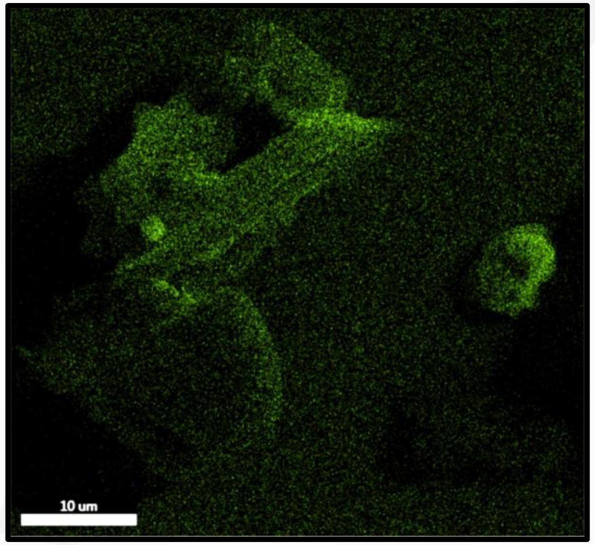
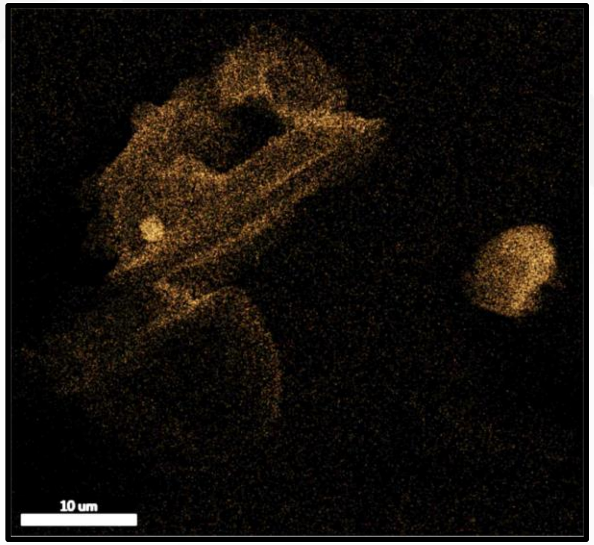
Powders

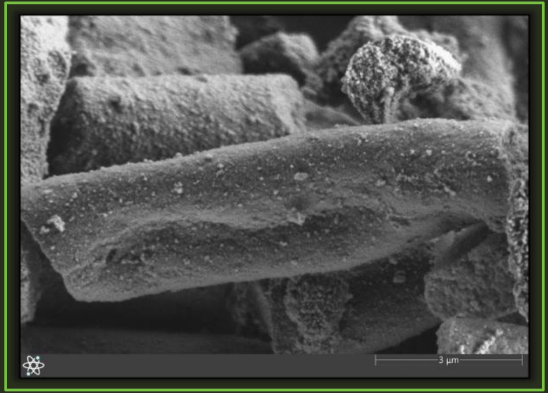
Analysis & Findings Using EDS:

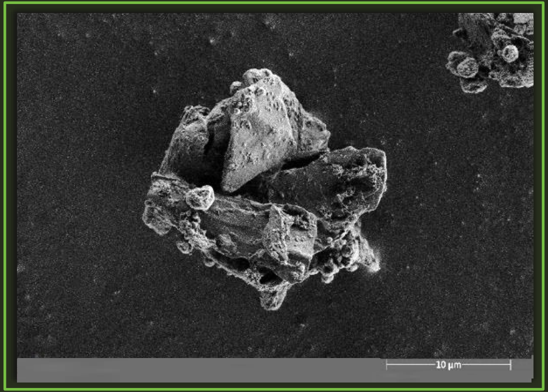
- ❑ Coating
- ❑ Composition
- ❑ Homogeneity & Distribution

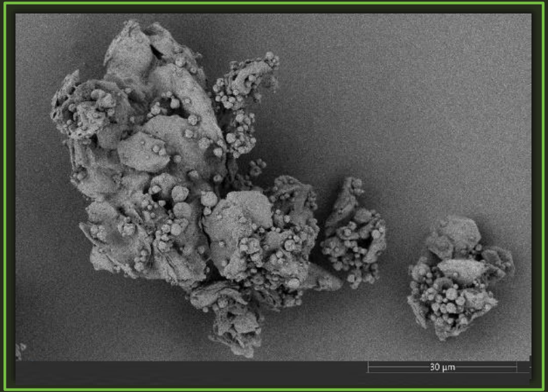












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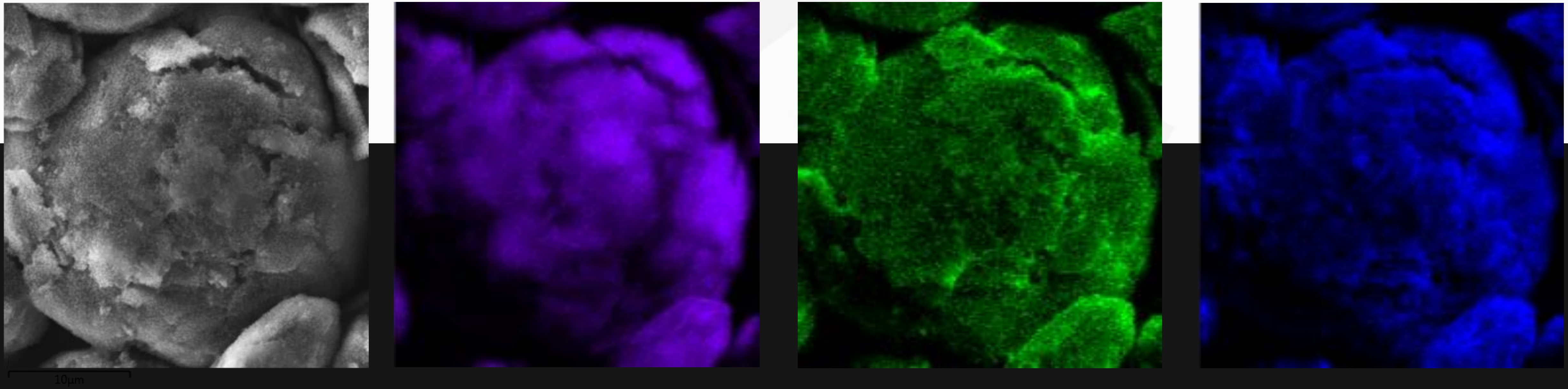
ANODE ANALYSIS

Analysis & Findings Using EDS:

- General Construction & Quality
- Post-Mortem Analysis
- Interface Investigation



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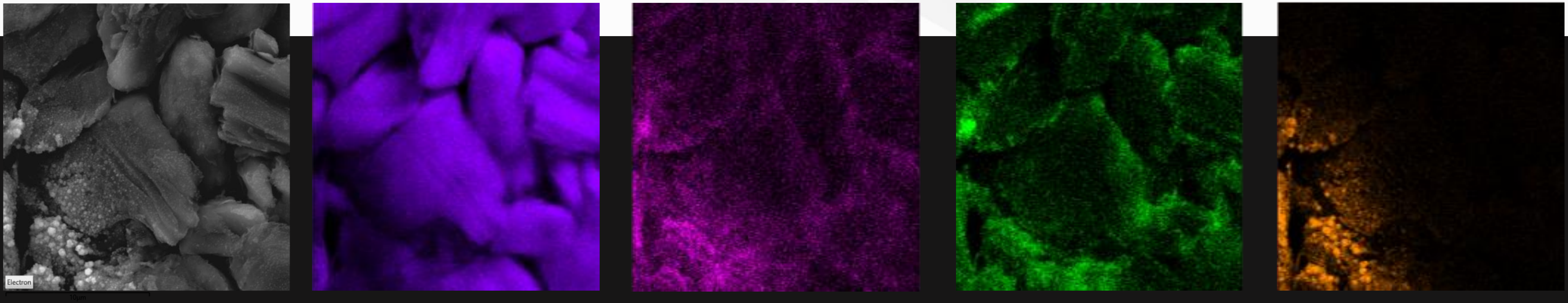
CATHODE ANALYSIS

Analysis & Findings Using EDS:

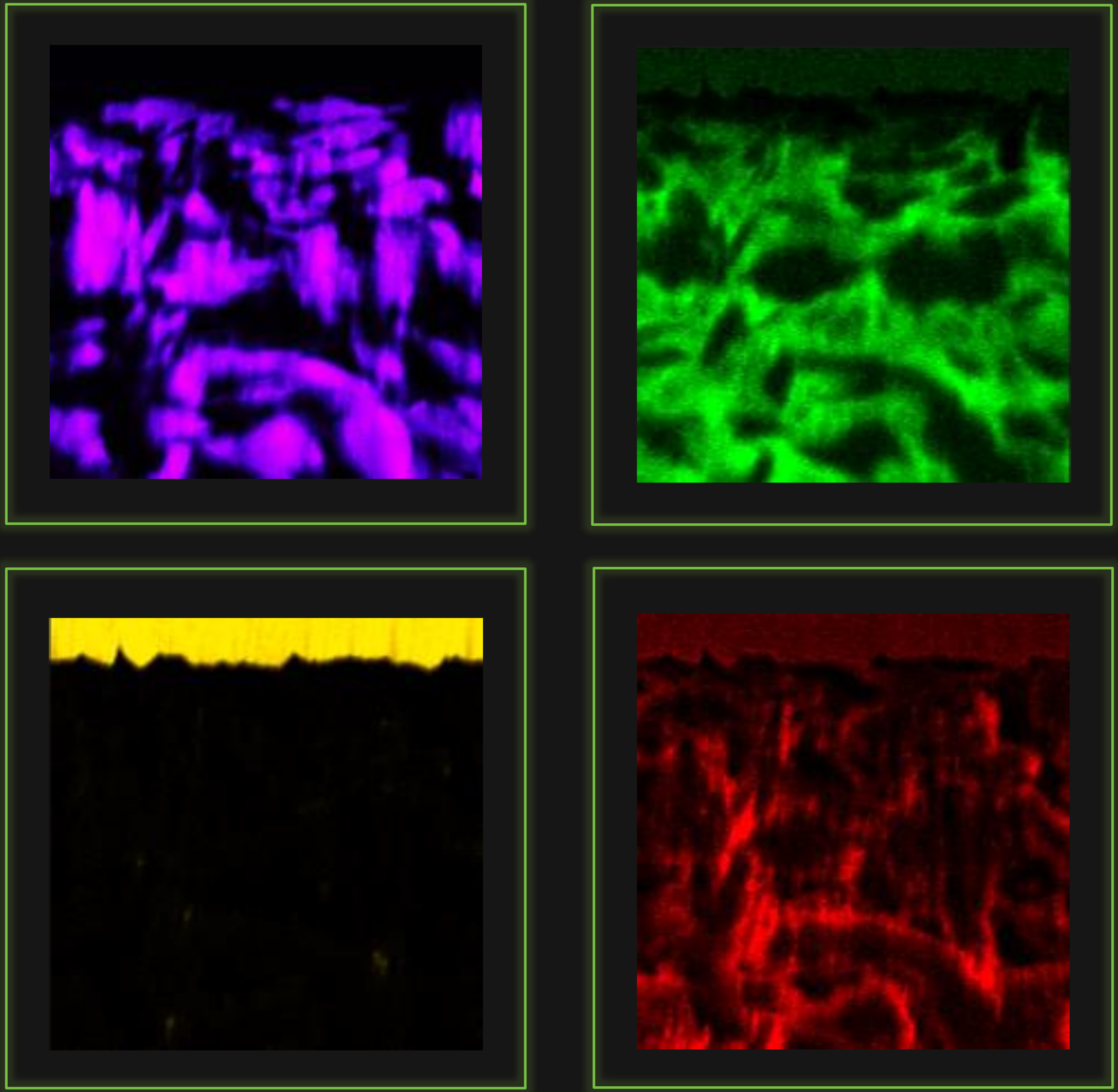
- General Construction & Quality
- Post-Mortem Analysis
- Interface Investigation



SOLID STATE ANALYSIS

Analysis & Findings Using EDS:

- Electrolyte Wetting
- Homogeneity & Distribution
- Speciation Using EDS
- Interface Investigation



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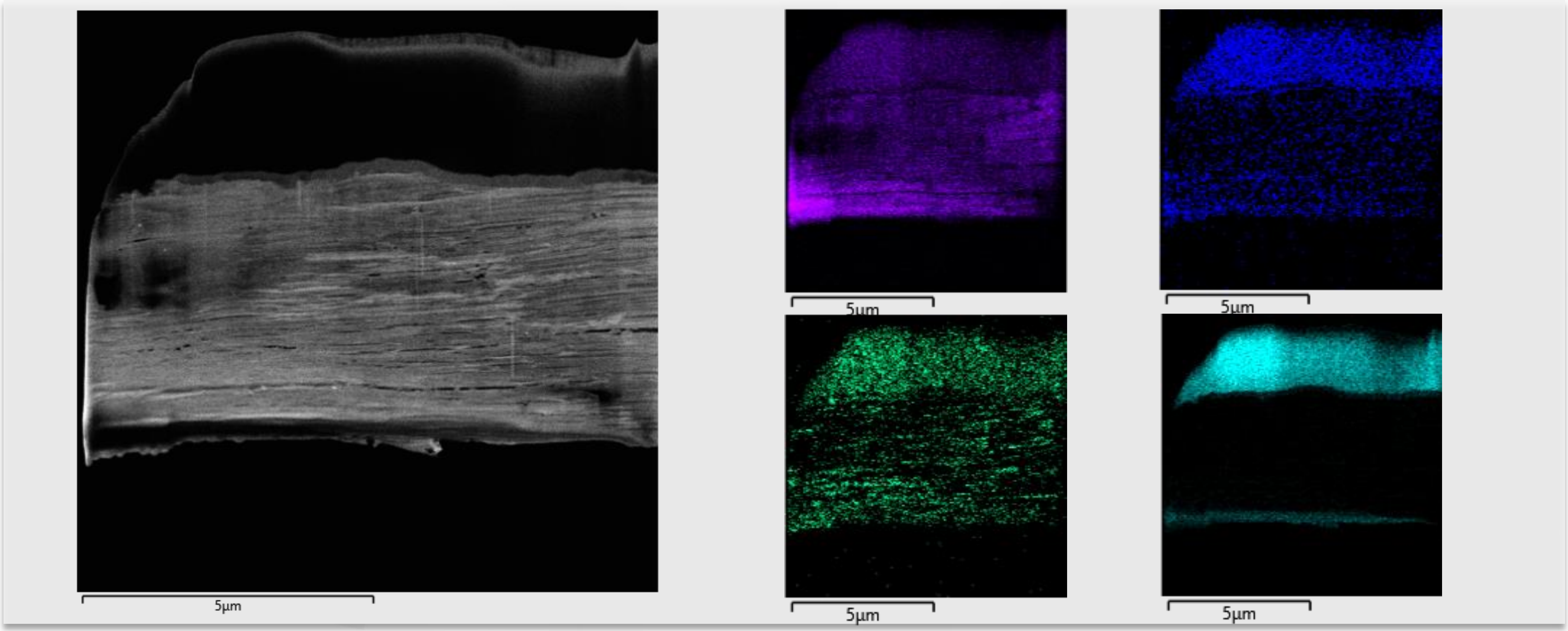
ADVANCED TECHNIQUES

- High Resolution Elemental Mapping
- Complementary Multimodal Techniques



Dragonfly is Positioned to Sit at the Nexus of Energy Technology

Solid State Li-ion Cells

QuantumScape

Solid Power

Conventional Li-ion Battery Manufacturers

Samsung
Samsung SDI

Microvast

CATL

LG Energy Solution

Enovix

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Vertically Integrated Energy

Tesla

BYD

Li-Cycle

Technology-Focused Energy Storage

eos

stem

ESS INC

Displacing Lead Acid Today, Replacing Coal Tomorrow

	TODAY	TOMORROW
Technology	Leader in non-toxic lithium-ion batteries	Proprietary non-flammable, non-toxic solid state cells
Products	Extensive line of batteries and accessories	Vertically integrated energy storage system integrator
Operations	99k sq. ft. battery assembly facility	Fully automated battery assembly and solid state cell manufacturing
Sales Channels	DTC + OEMs	DTC + OEMs + Fleets + Utilities

Thank You

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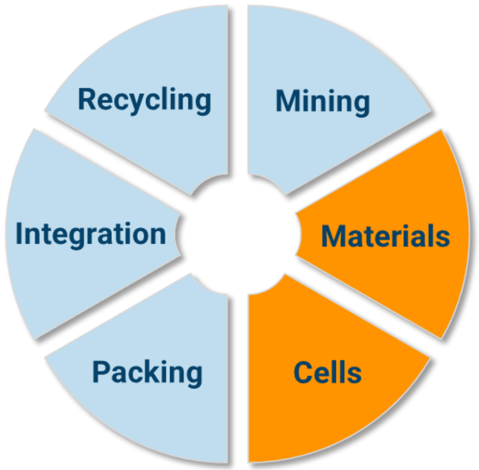
02

EDS analysis for identifying and detecting contaminants in cathode and anode

Outline

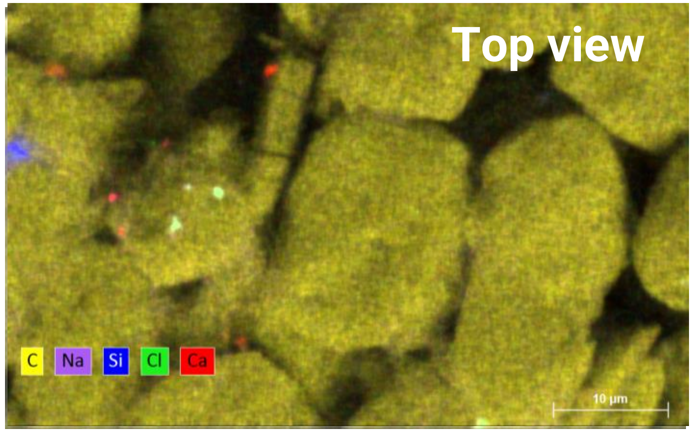
- Introduction of investigated samples and materials
- Introduction to EDS - what signal is being measured, how to interpret, what questions can be answered
- Measurement conditions
  - Choice of acceleration voltage and SEM parameters
- Results of anode and cathode material
- Introduction and comparison of EDS detectors of conventional geometry and Bruker FlatQUAD
- Detection limit of EDS: How can very low concentration contamination be detected and located?
- Comparison of anode/cathode results with EDS detectors of conventional geometry and Bruker FlatQUAD

Investigated samples



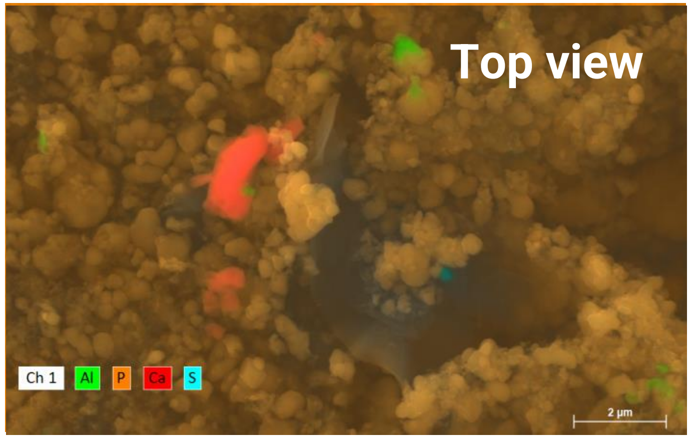
Anode sample:

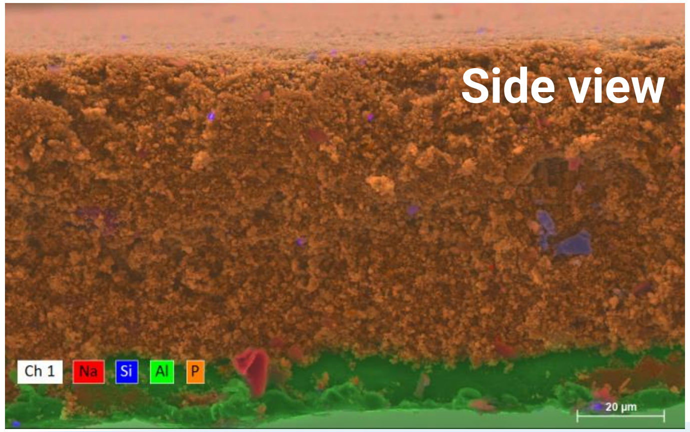
graphite on copper (pristine, not cycled)



Cathode sample:

LiPO4 on aluminum (pristine, not cycled)

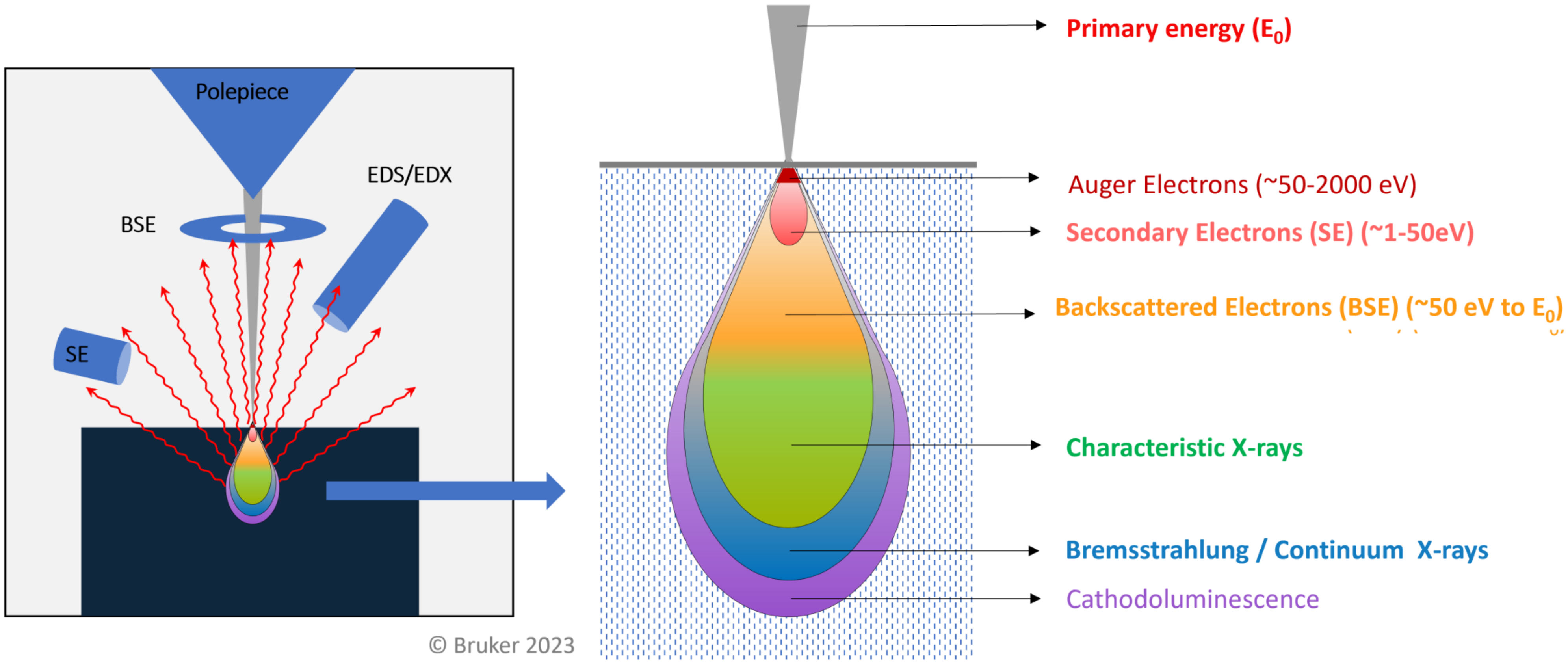




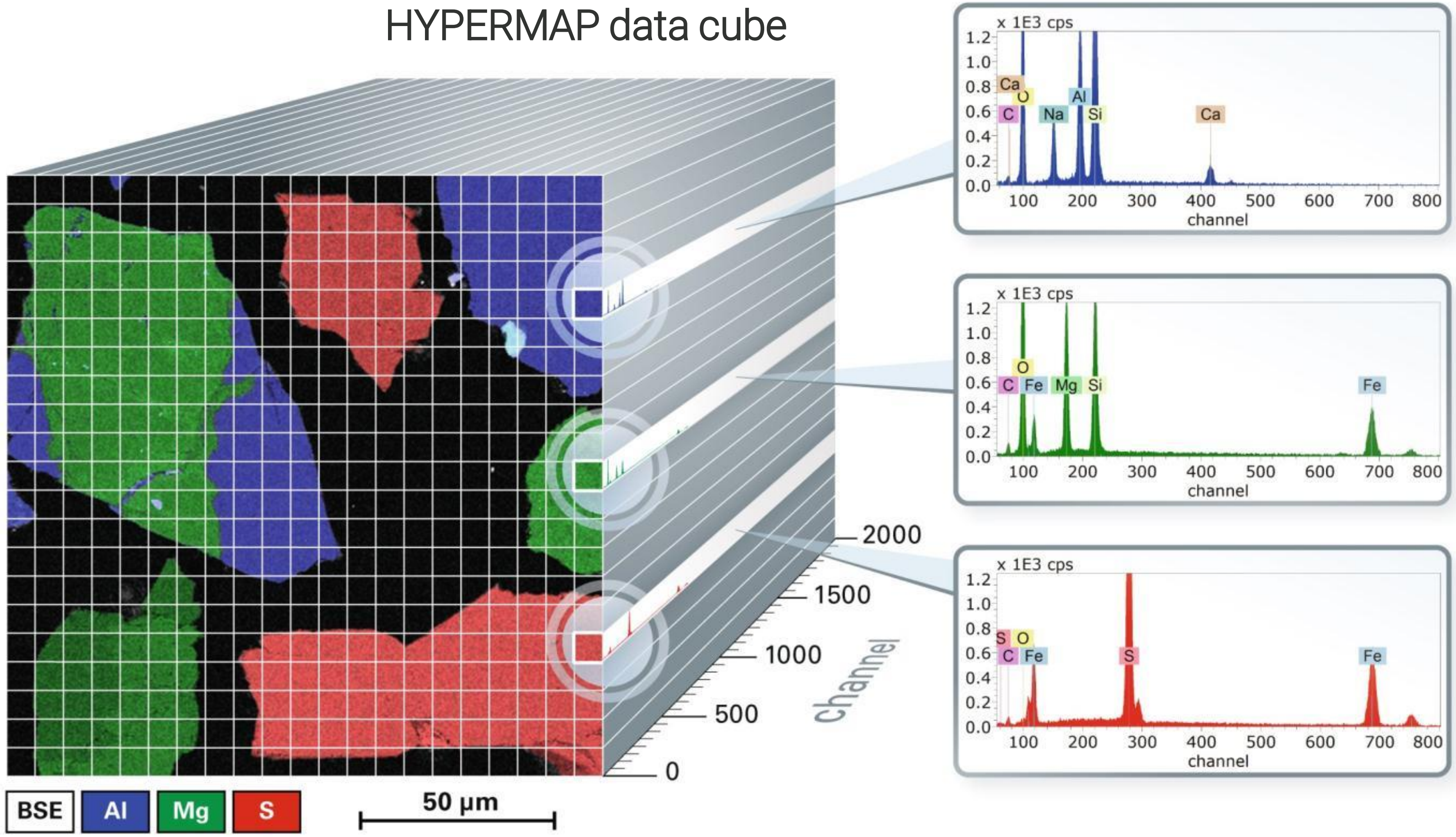
What is EDS and what questions can it answer?

- Element detection based on X-ray emitted due to the excitation of primary electron beam of a scanning electron microscope
- How is element distribution interpreted?
- What elements are in the sample?
- How are they distributed? (-> where do they originate from in the battery manufacturing/cycling/life process)
- In what quantity are these elements present in the sample?
- What is the lowest amount of material EDS can detect?

Introduction to EDS – what signal is being measured



Introduction to EDS – how mapping works



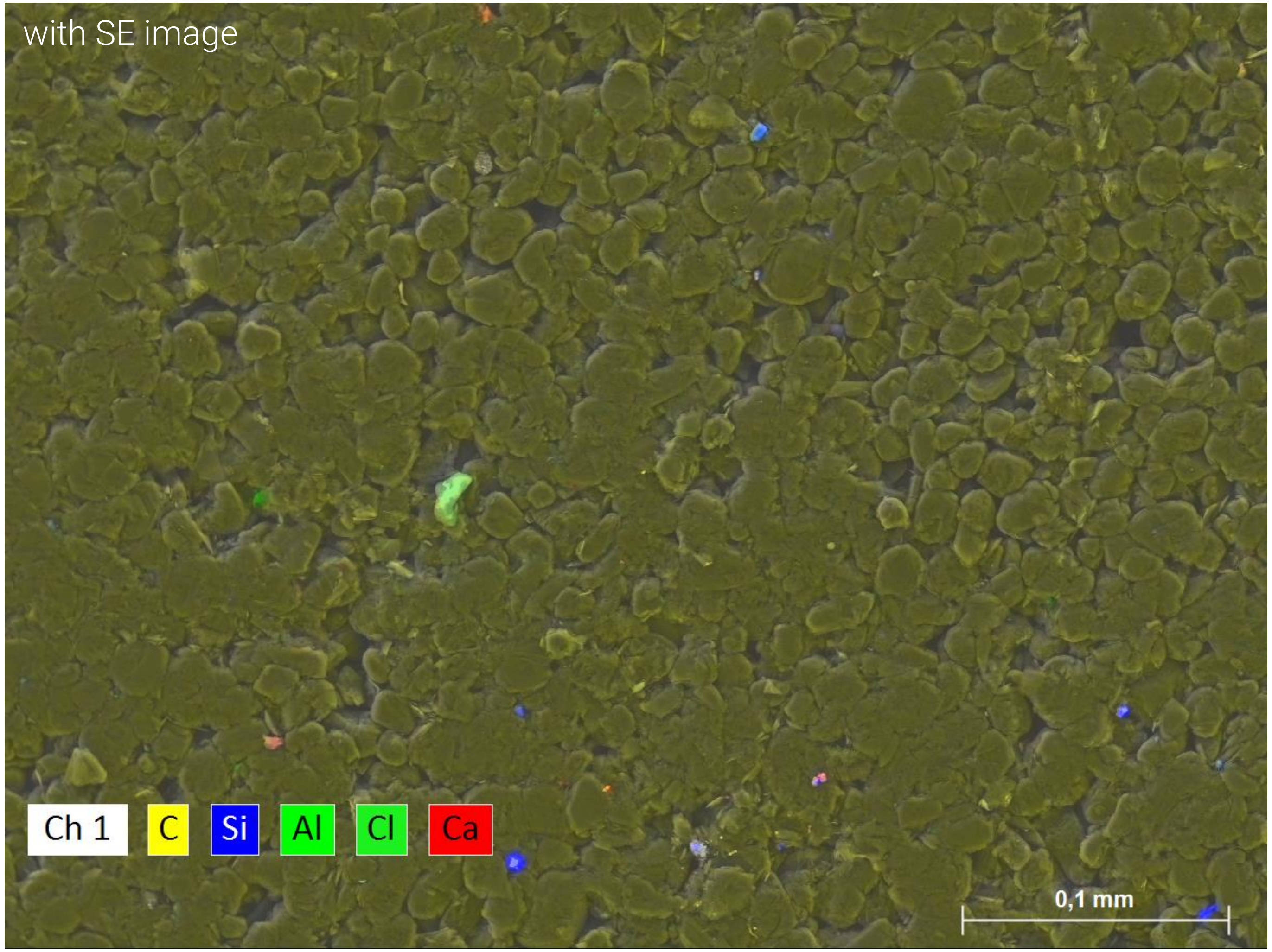
Measurement key points

- Samples: Manufactured electrode tapes:
  - Pristine Anode: Copper foil coated with carbon on a single side.
  - Pristine Cathode: Aluminum foil coated with LiFePO4 on a single side.
- Sample preparation: Samples mounted on carbon tape. Mechanical cut for crossection

  The samples are stable in room conditions, no glovebox or inert gas transfer was necessary
- EDS /SEM Measurement conditions:

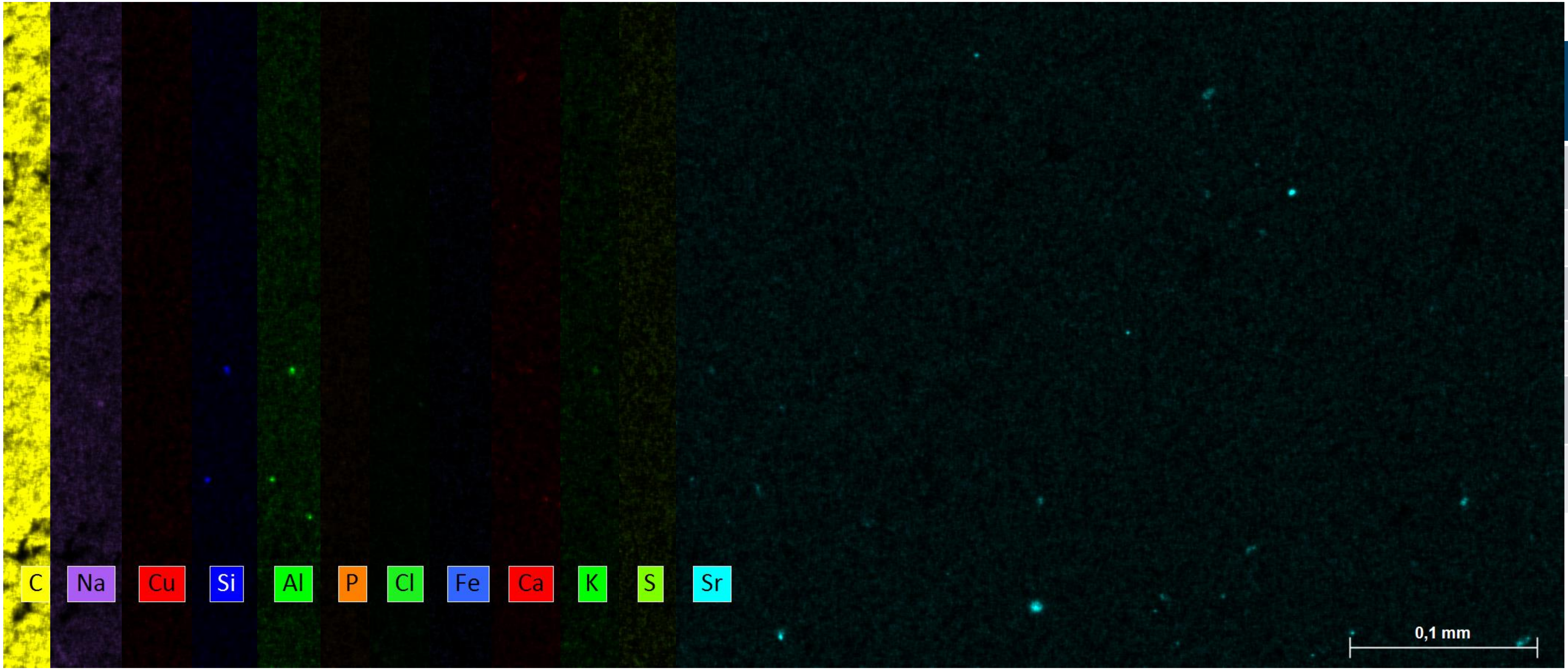
  high vacuum SEM conditions. No low vacuum/variable pressure needed
- EDS setup: Choice of acceleration voltage: 12 or 15kV is sufficient to cover all elements
  - Lower kV: minimizing interaction volume-> maximizing spatial resolution. (not necessary)
  - Higher kV: access K-Lines of heavier elements (not necessary as deconvolution works fine)

Graphite ANODE - top overview

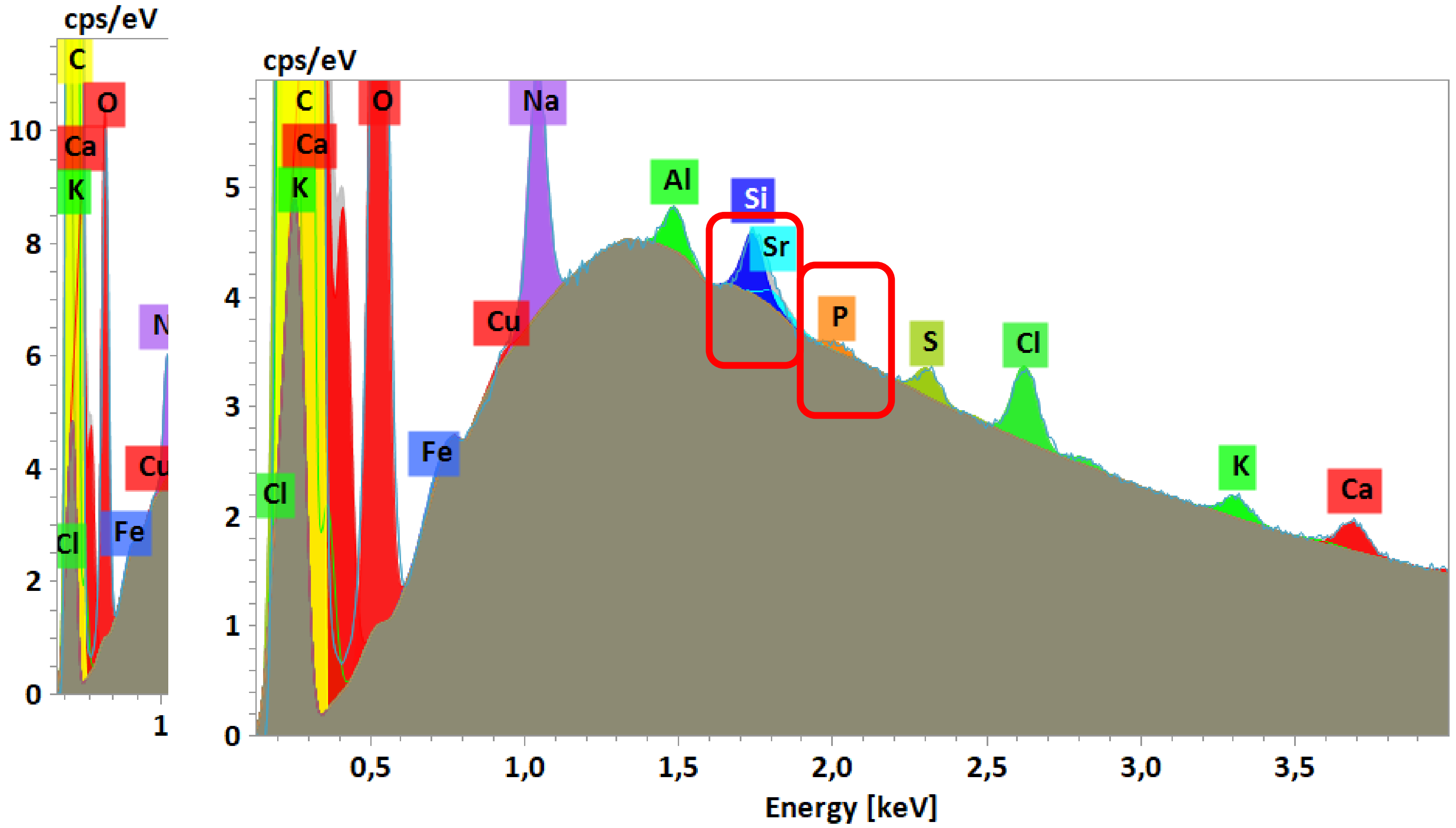


Analysis parameters	
Detector	XFlash® 760
High voltage	15 kV
Magnification	250
Beam current	~5 nA
Mapping time	12 min
Input count rate (ICR)	**92 kcps**

Graphite ANODE - top overview



Element identification – how to find all elements



Elements identified by deconvolution of sum spectrum.

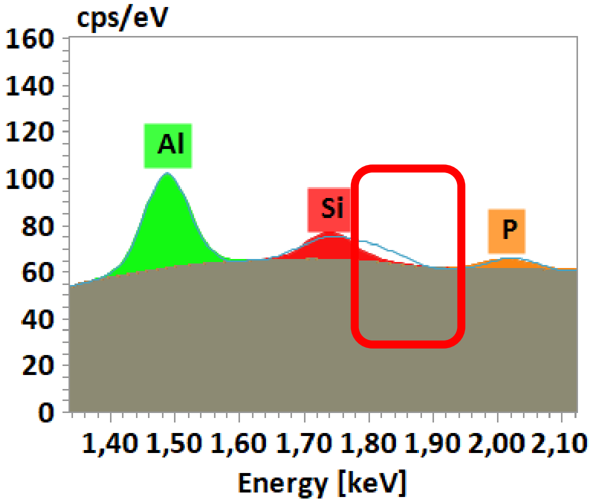
Element maps are visualized based on the deconvolved net coutns

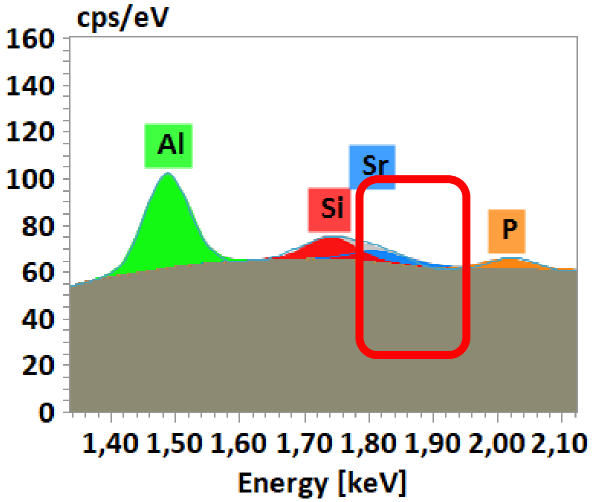
Element identification – presence of Sr or is it Si?

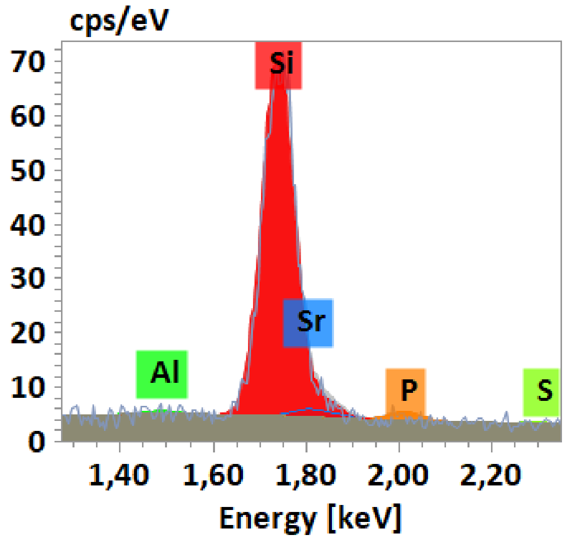
Are the Sr maps only the misinterpretation of Si due to the peak overlap?

15kV measurements: only Sr-L lines available which overlap with Si-K!
Sr-K lines at 14-15kV are not excited

Peakform of a pure Si-particle:







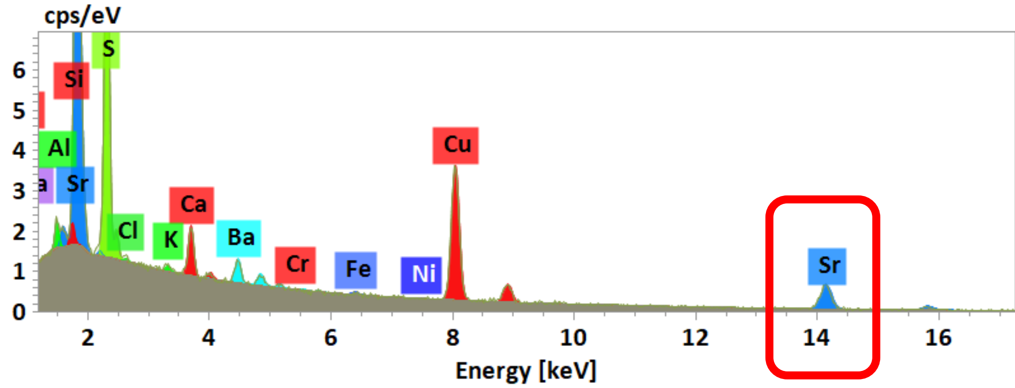
Deconvolution with Si only
-> „bad match“

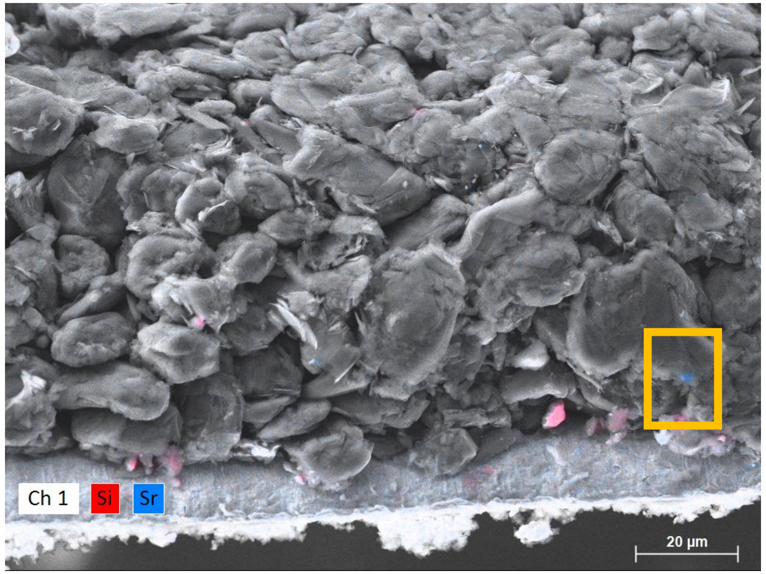
Deconvolution with Si and Sr:
Good match!

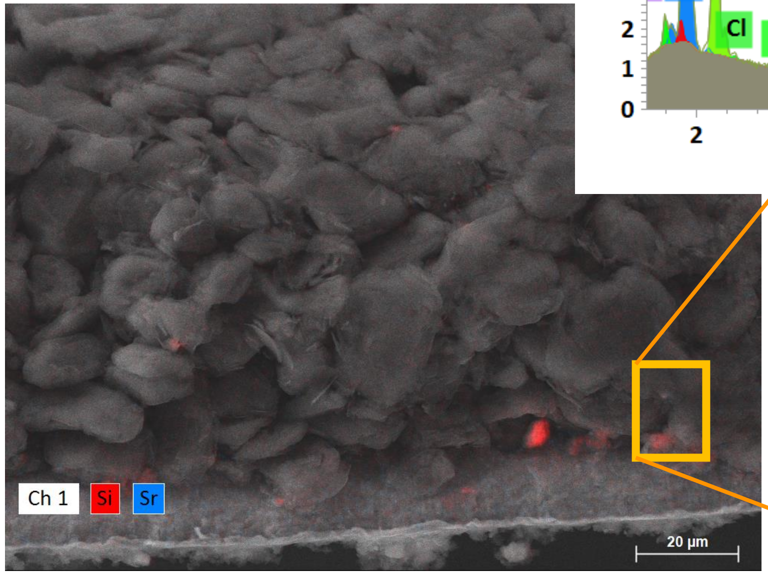
Element identification – presence of Sr or is it Si?

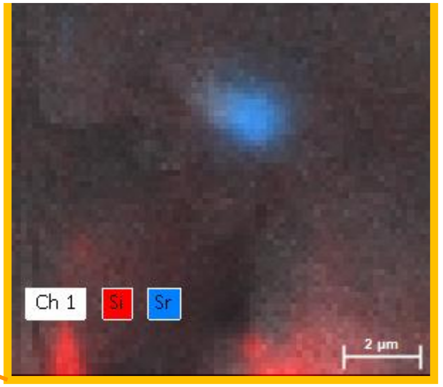
Are the Sr counts only the misinterpretation of Si?

25kV measurements: Sr-K lines properly excited and present









Sr L-line map
15kV
XFlash760
30 minutes

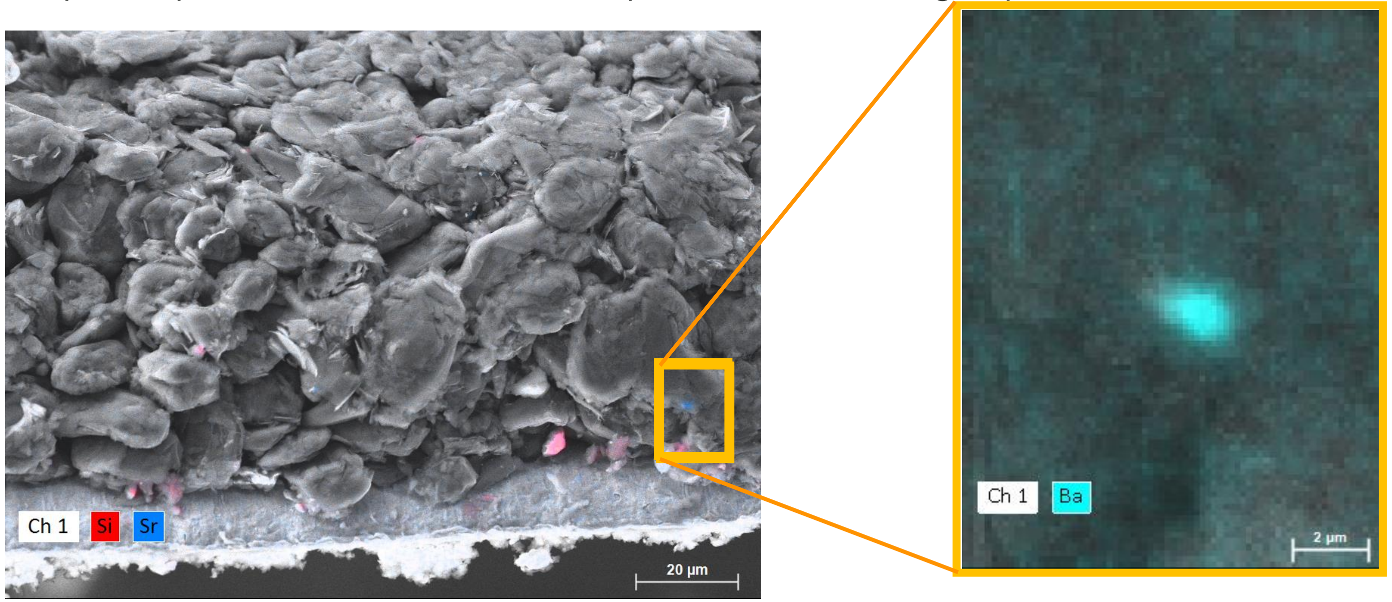
Sr K-line map
25kV
XFlash760
13 minutes

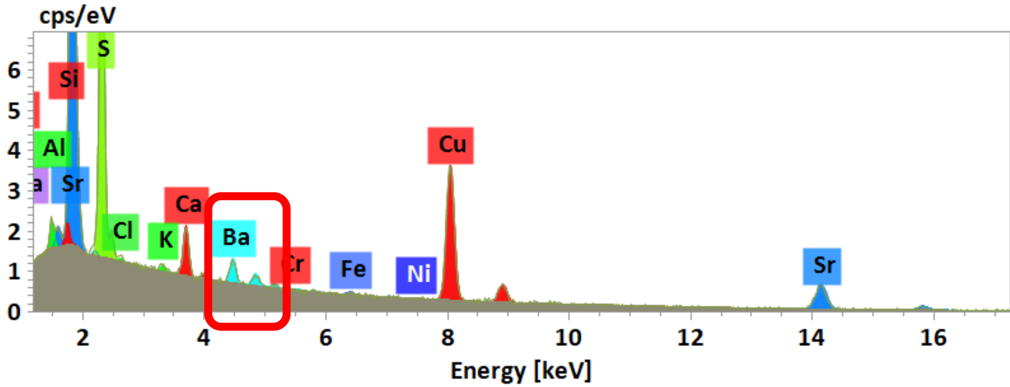
Very low Sr-L signal!-> longer measurement needed

Sr K- line map
25kV
XFlash760
90 minutes

Element identification – this particle contains also Ba

Ba-peak present in extracted spectrum of single particle!





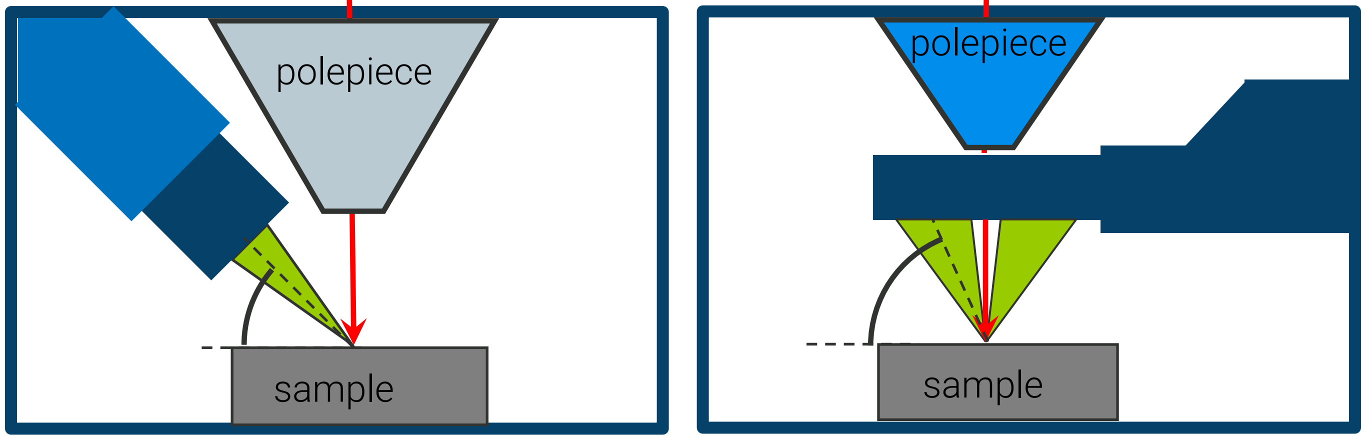
Element	Line series	Mass Norm. [%]	Atom [%]	abs. error [mass%] (3 σ)
C	K	0,00	0,00	0,00
O	K	10,81	33,18	1,10
Na	K	1,41	3,01	0,25
Cu	K	21,17	16,37	1,18
Si	K	0,22	0,38	0,08
Al	K	0,73	1,33	0,10
P	K	0,55	0,88	0,09
Fe	K	0,20	0,18	0,09
Ca	K	1,57	1,93	0,13
Sr	K	52,34	29,34	4,85
S	K	8,07	12,36	0,43
Ba	L	2,92	1,05	0,25
		100,00	**100,00**	

Do the math:
quantification of particle spectrum and dividing with area coverage:
3w% Ba * 3% area coverage * 1% area coverage of original map:
= 9*10E-6
-> ppm sensitivity!

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

Conventional detector
60 mm² EDS detector
@WD= 10mm

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



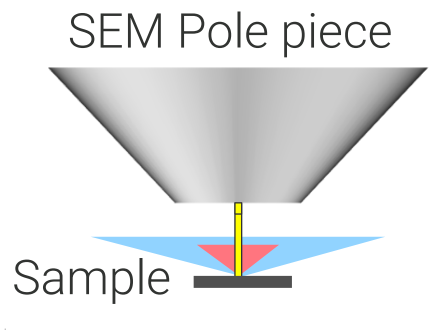
take off angle=35°
solid angle=0.043 sr

take off angle=60-70° → Better view of sample topography
Less absorption of X-ray signal

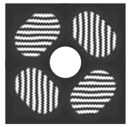
solid angle = 0.7-1 sr → x15-30 more X-ray signal:
less measurement time needed

Bruker XFlash® FlatQUAD EDS detector
Features and advantages

XFlash ® FlatQUAD



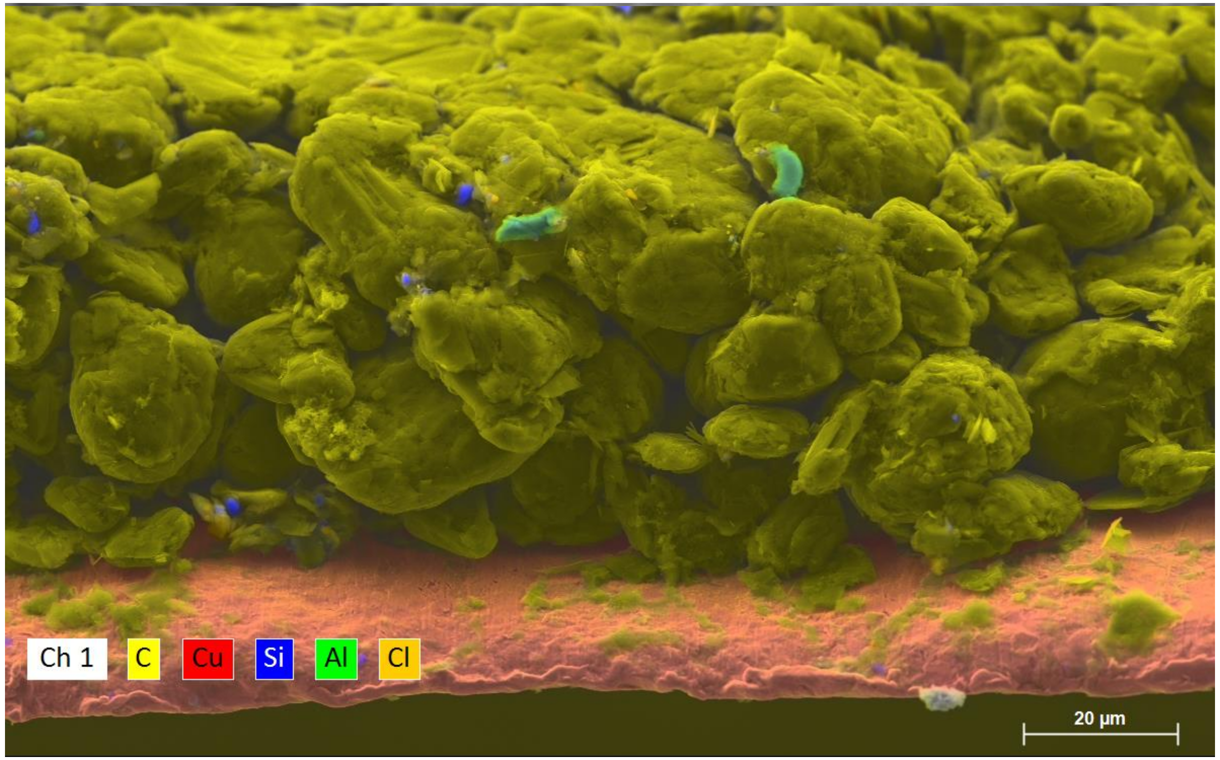
FlatQUAD



- 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

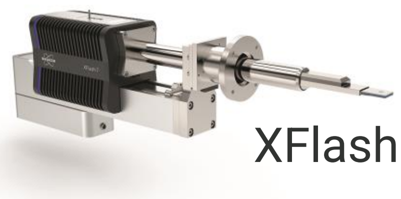
- High sensitivity at very low probe currents ~few pA
- Minimize sample charging/damage/C-deposition at low PC
- High vacuum conditions EDS – high resolution
- Low vacuum capability
- Moderate probe currents for high-speed EDS mapping
- Low x-ray yield samples: Low PC – High resolution
- Nanoparticles,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

ANODE - cross section: comparison of conventional detector and FlatQUAD



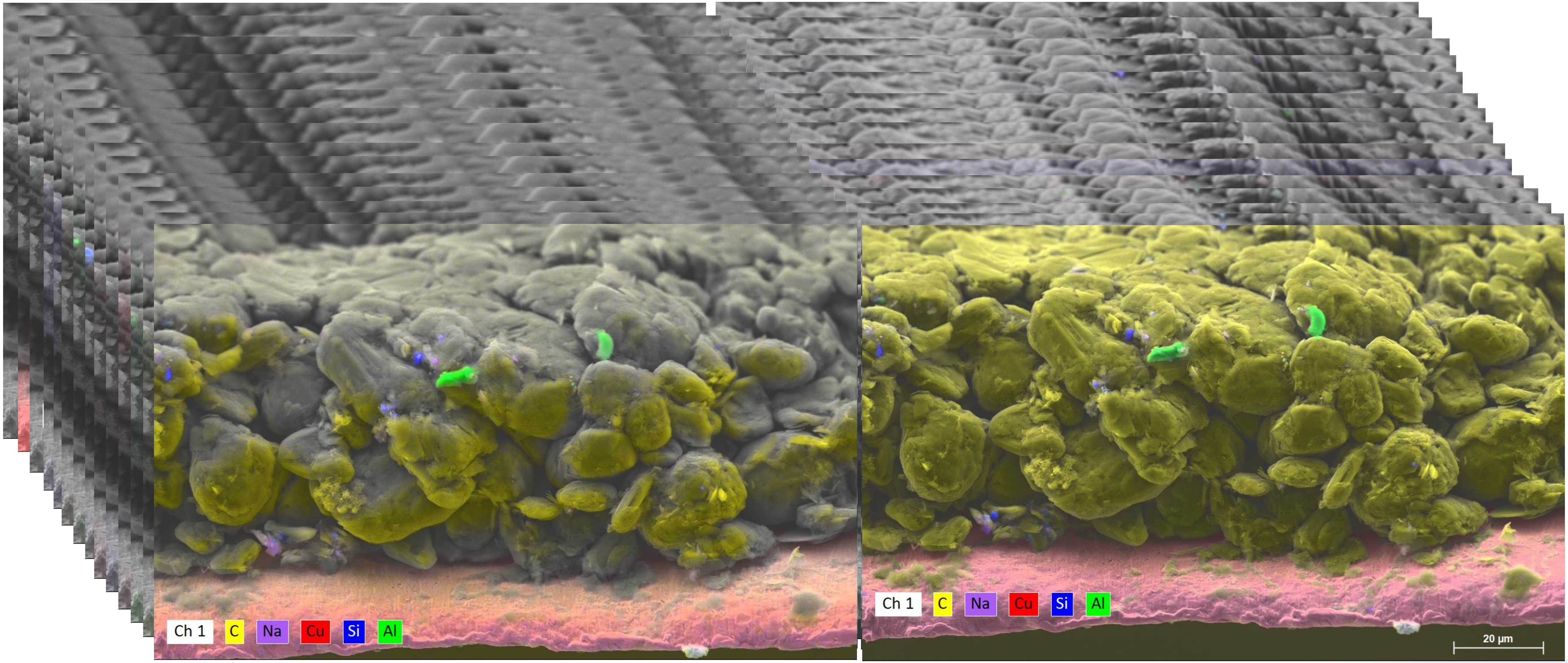
Analysis parameters		
Detector	XFlash® 760	XFlash® FlatQUAD
High voltage	12 kV	12 kV
Beam current	~1nA	~1nA
Mapping time	30 min	30 min
Input count rate (ICR)	**19,5 kcps**	**667 kcps**
Total counts in map	**2.7 x10^7**	**8.2 x10^8**

XFlash® 760

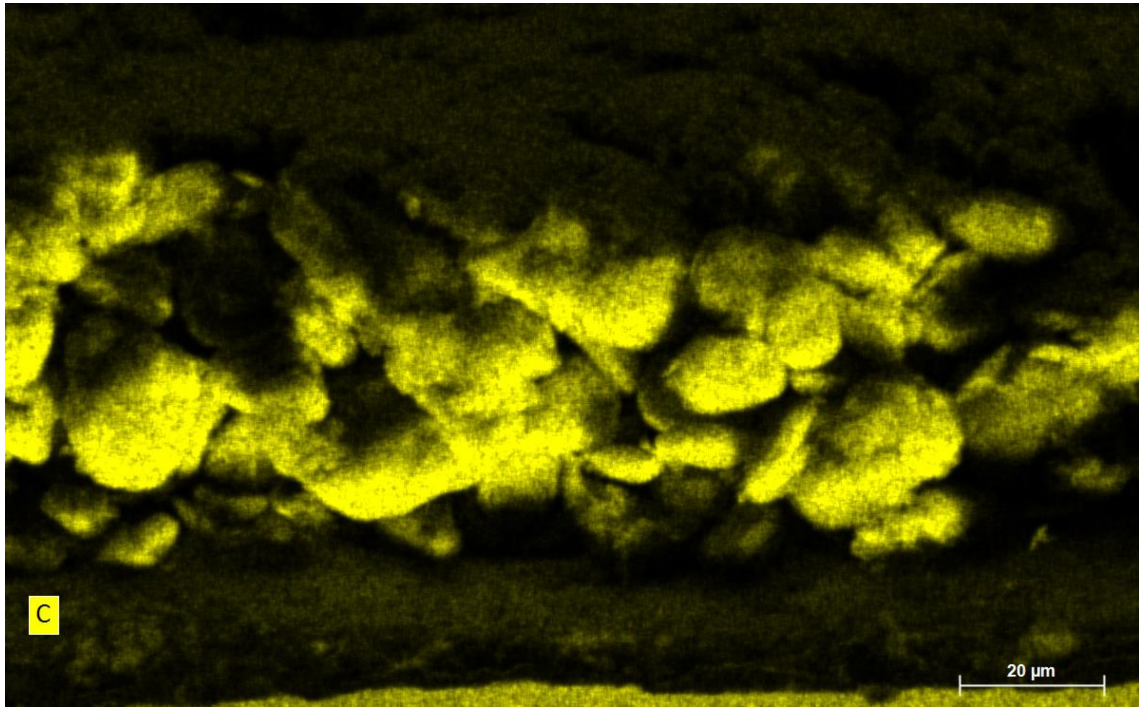


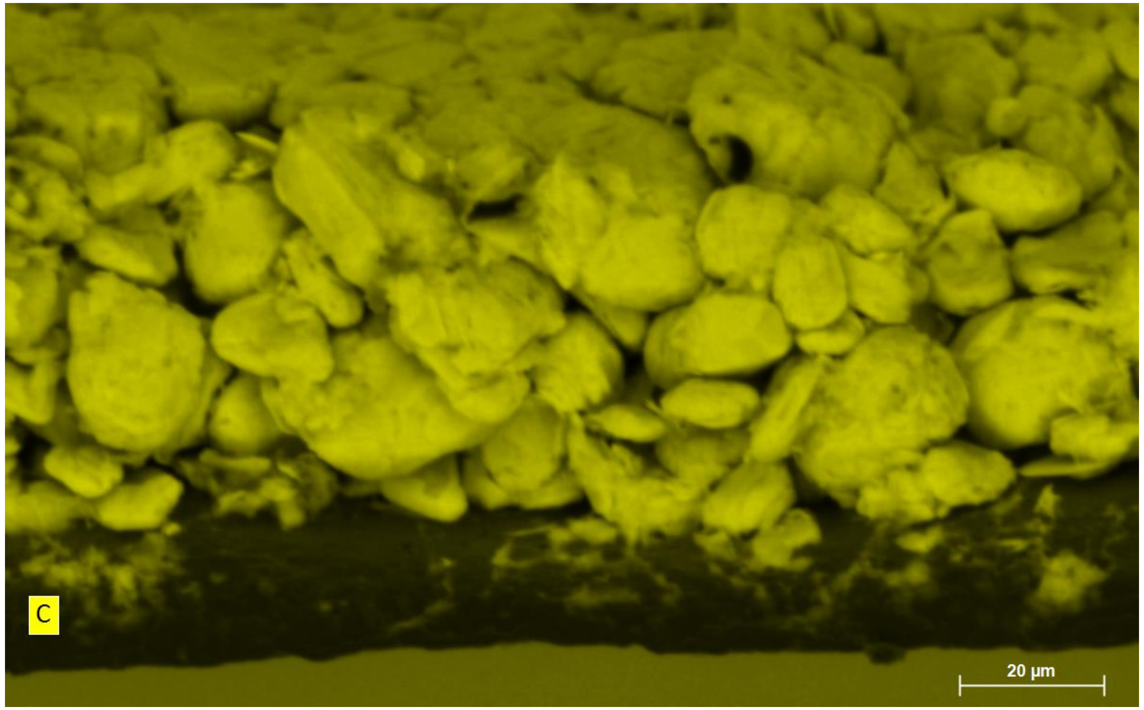
XFlash® FlatQUAD

ANODE crossection – XFlash® 760 vs FlatQUAD



ANODE crossection – XFlash® 760 vs FlatQUAD

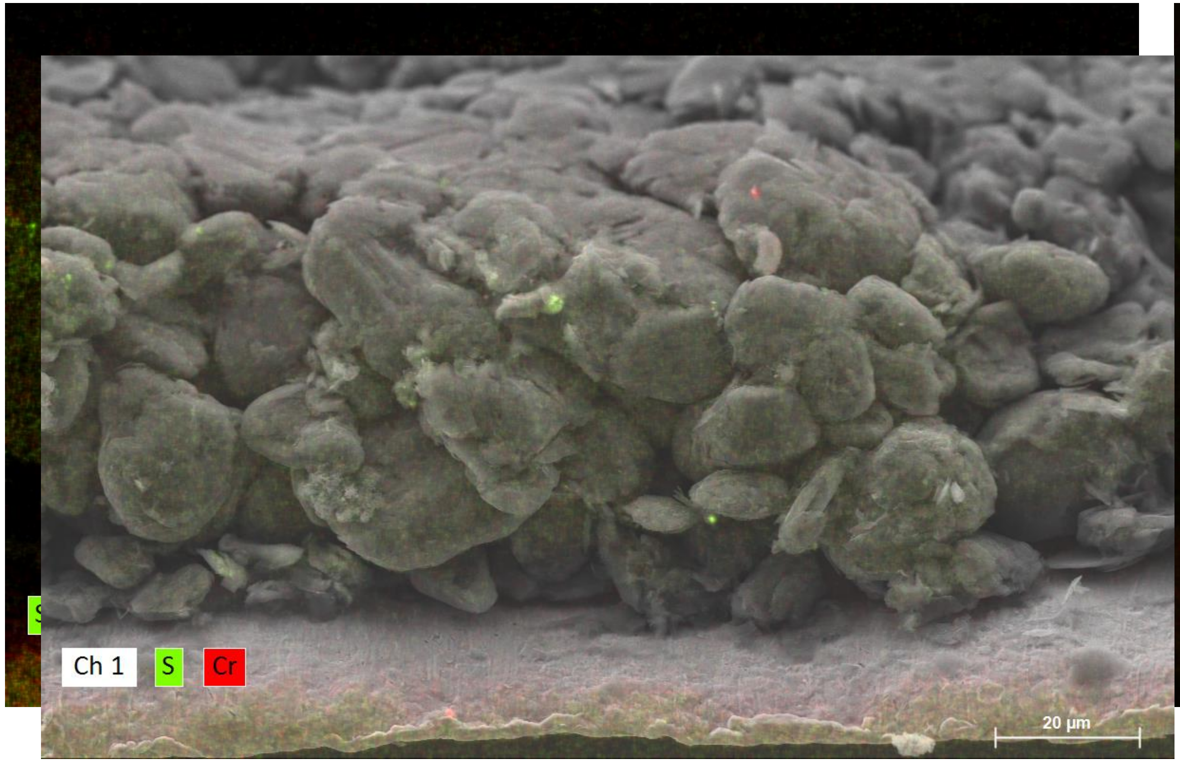


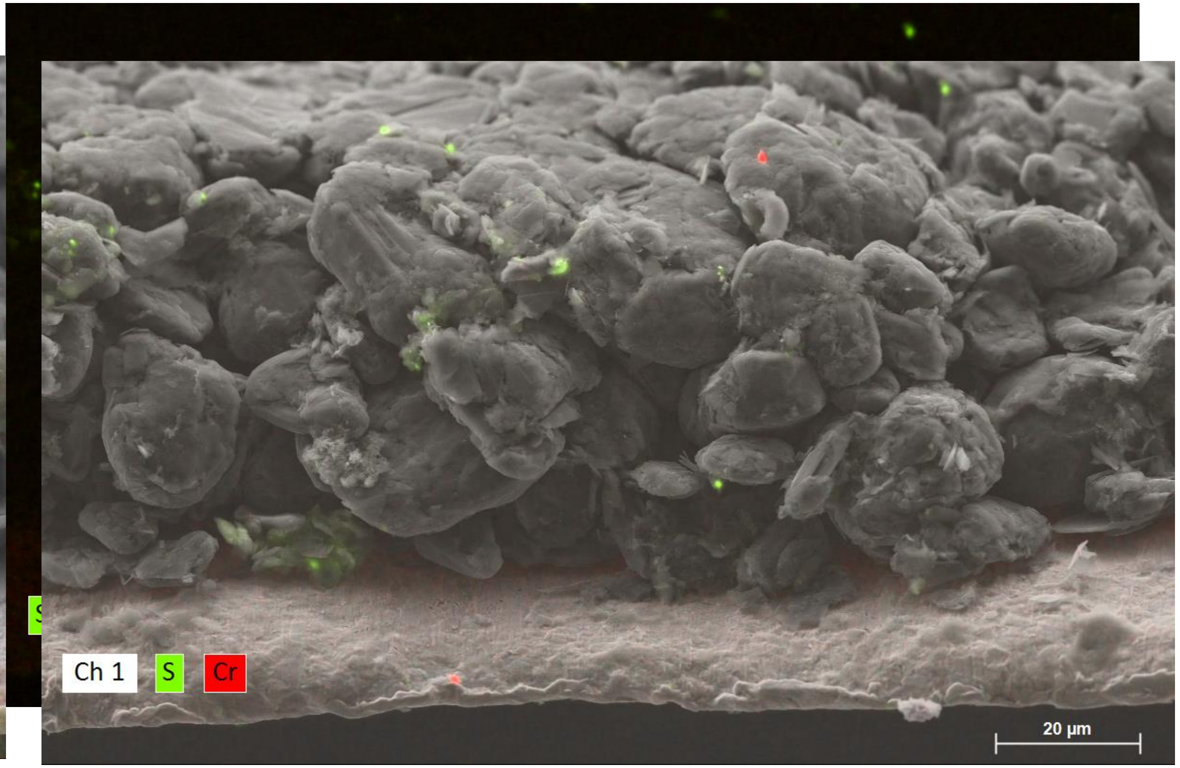


XFlash® 760

XFlash® FlatQUAD

ANODE crossection – XFlash® 760 vs FlatQUAD: finding "hiding elements": S, Cr





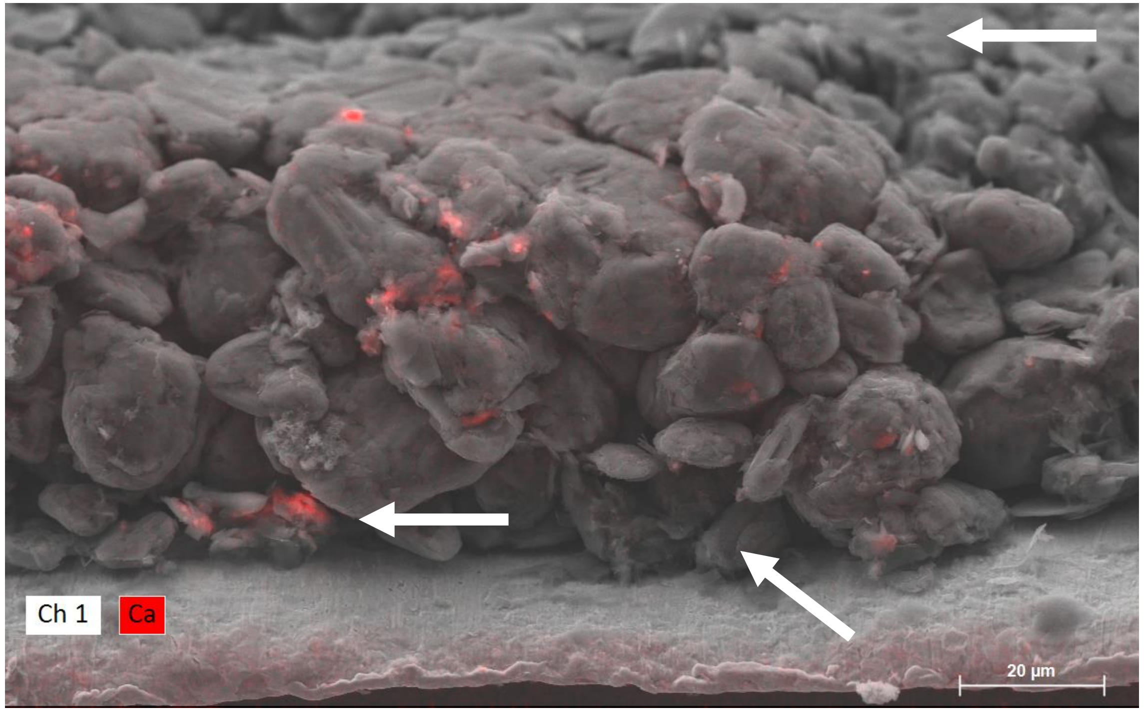
XFlash® 760

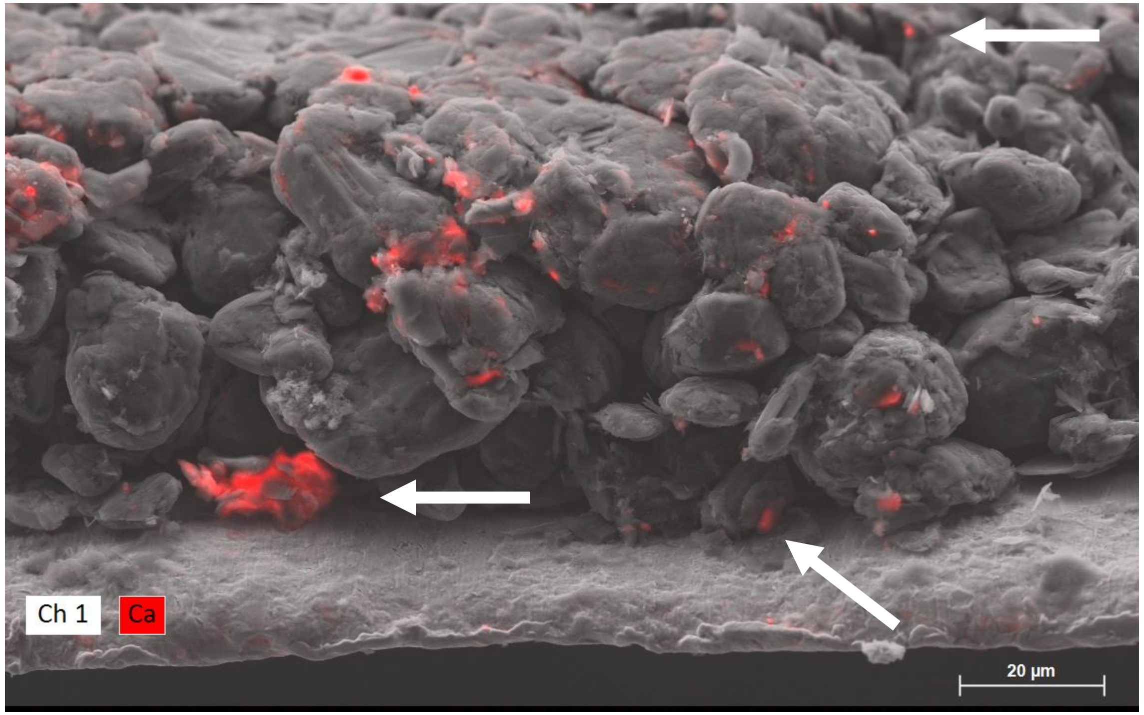
Noisier maps,
Shadowed areas

Less noise in maps,
Access to areas with high topography

XFlash® FlatQUAD

ANODE crossection – XFlash® 760 vs FlatQUAD: finding “hiding elements”: Ca





XFlash® 760

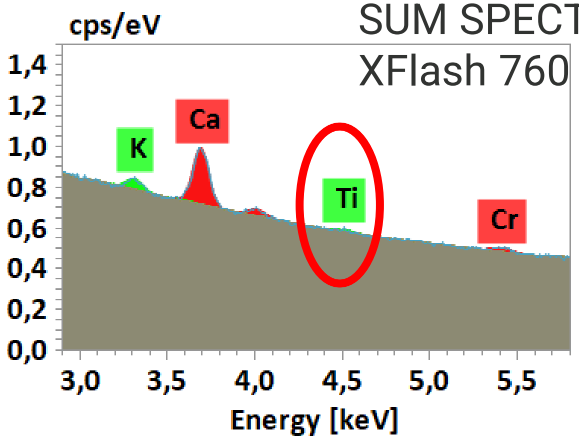
Noisier maps,
Shadowed areas

XFlash® FlatQUAD

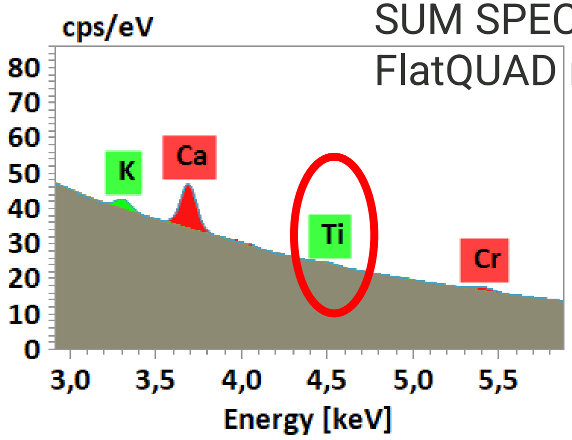
Less noise in maps,
Access to deeper /shadowed areas

ANODE crossection – XFlash® 760 vs FlatQUAD: finding "hiding elements": Ti

SUM SPECTRUM of XFlash 760 map

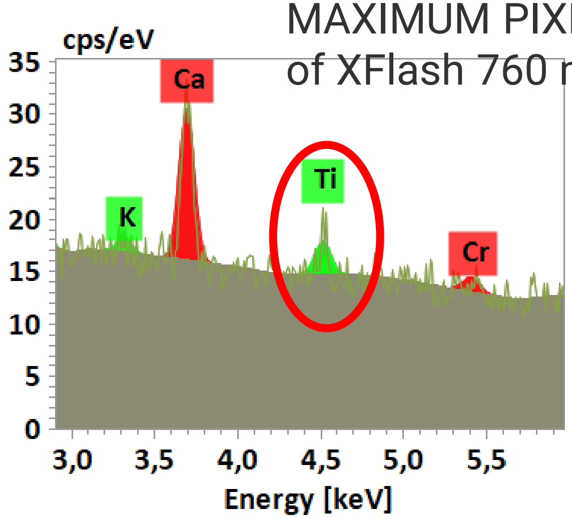


SUM SPECTRUM of FlatQUAD map

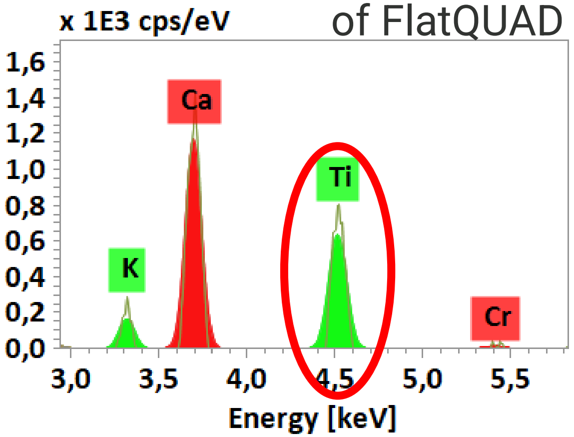


Elements identified by deconvolution of sum spectrum:
No Ti found
Ti is below detection level!

MAXIMUM PIXEL SPECTRUM of XFlash 760 map

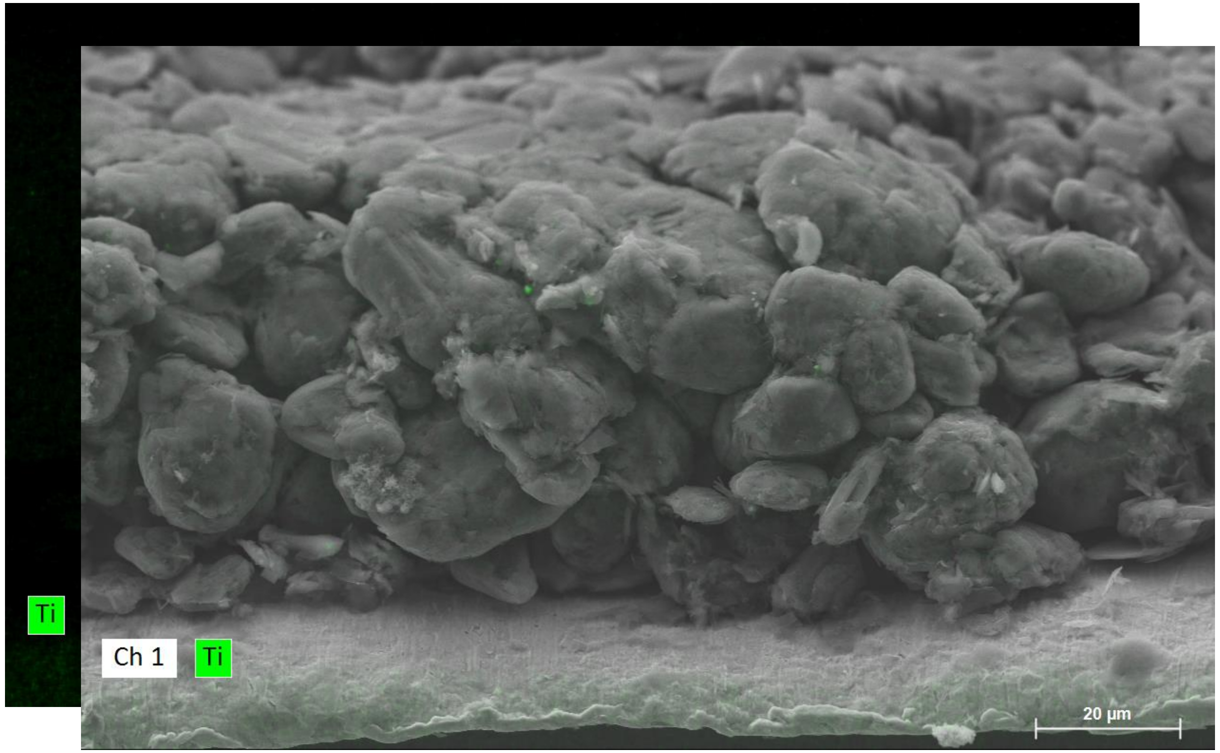


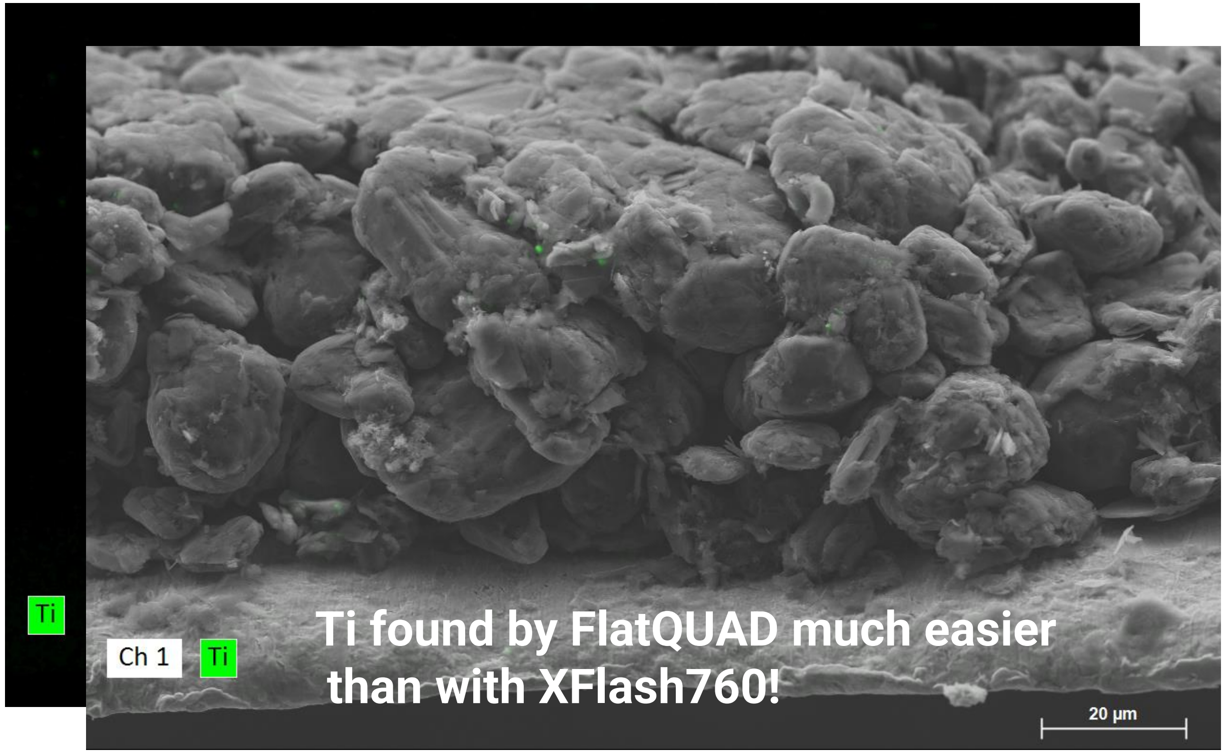
MAXIMUM PIXEL SPECTRUM of FlatQUAD map



Elements identified by deconvolution of MAXIMUM PIXEL SPECTRUM
Ti identified

ANODE crossection – XFlash® 760 vs FlatQUAD: finding “hiding elements”: Ti



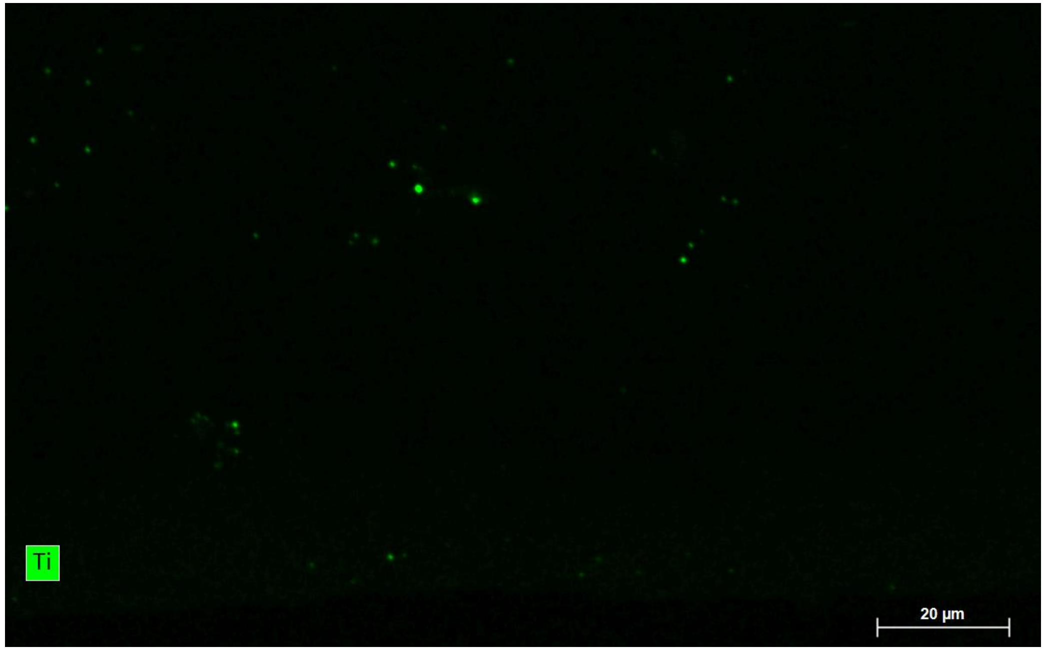


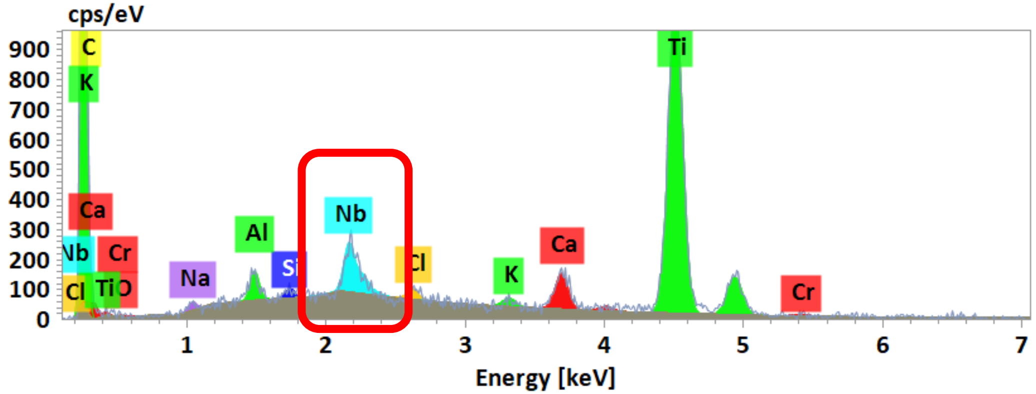
XFlash® 760

XFlash® FlatQUAD

Ti identified by deconvolution of MAXIMUM PIXEL SPECTRUM
Ti is below detection level

ANODE crossection – Ti: trace element below EDS detection limit





Do the math:
Use AUTOPHASE function to calculate
Area coverage= **0.01%**
Extract object spectrum of single particle:
Local concentration of Ti: 85%
-> **85 ppm** Ti detected

Nb also identified in the particles!

Element	Line series	Mass Norm. [%]	Atom [%]	abs. error [mass%] (3 σ)
C	K	0,00	0,00	0,00
O	K	0,41	1,19	0,88
Na	K	1,07	2,18	0,54
Si	K	0,53	0,87	0,25
Al	K	1,59	2,75	0,41
Cl	K	0,55	0,73	0,22
Ca	K	4,41	5,13	0,84
K	K	0,71	0,84	0,26
Cr	K	0,80	0,72	0,72
Ti	K	85,35	83,26	14,70
Nb	L	4,58	2,30	0,77
		100,00	100,00	

Area coverage= **0.01%**
Extract object spectrum of single particle:
Local concentration of Nb: ~5%
-> presence of **Nb** on the concentration level of **5ppm** detected!

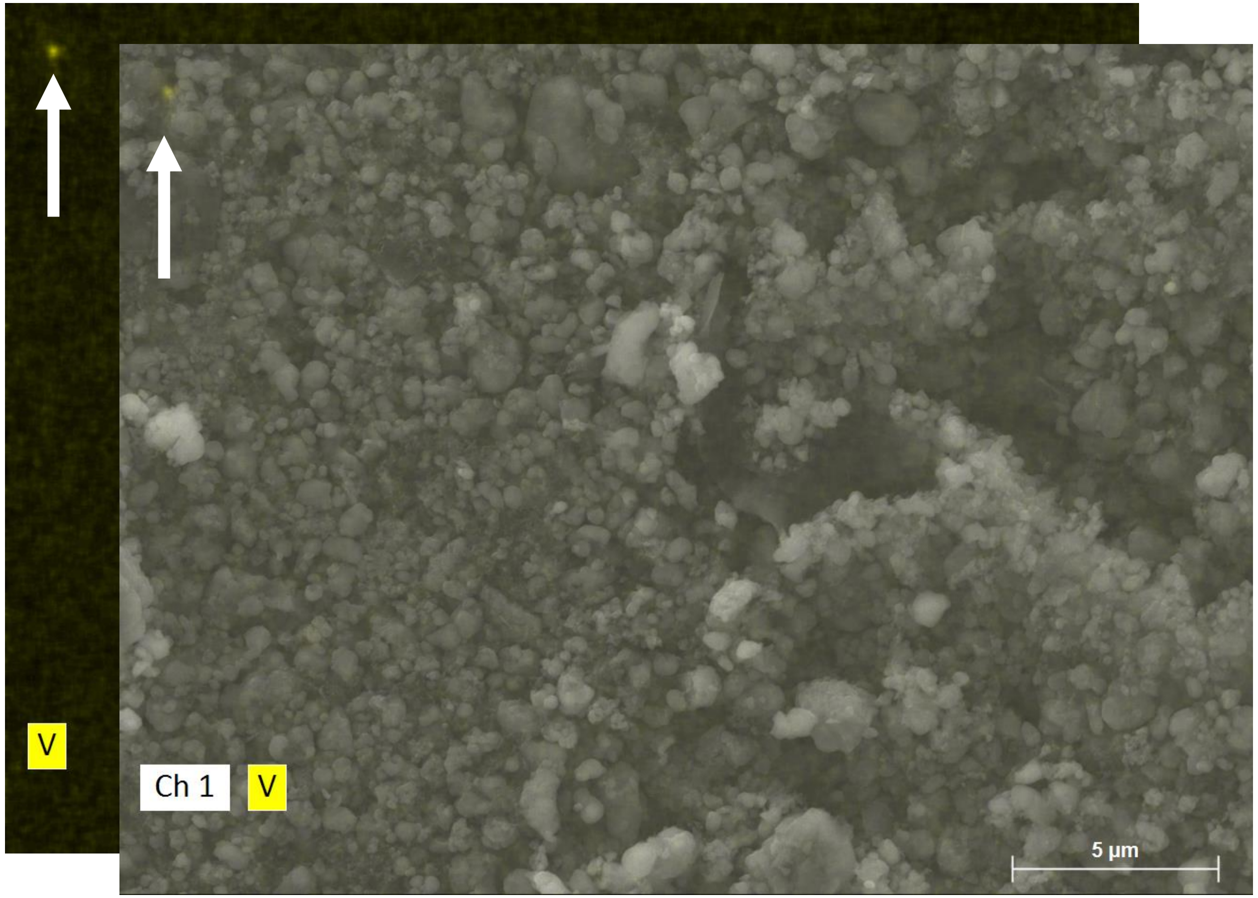
Detected main elements and contaminants – Graphite anode material on Cu

Element	Coexist with	Detection with EDS	Detection at 12/15kV	Origin	Comment
Na		easy	K–line	Polymer binder	Assists in mapping polymer distribution.
Cu		easy	K–line	Anode substrate	
Si	Partly with Sr	easy	K–line	Contaminant	Common contaminant in anode manufacturing.
Al		easy	K–line	Contaminant	Common contaminant in anode manufacturing.
Fe, Cr, Ni		Low signal	K–line	Contaminant	Common contaminant in anode manufacturing.
P		Low signal, could be found with 760	K–line	Contaminant	Common contaminant in anode manufacturing.
Cl		easy	K–line	Contaminant	Common contaminant
S		easy	K–line	Contaminant	Common contaminant
Ti		Better with FQ Found with MaxPix	K–line	Contaminant	Common contaminant in anode manufacturing.
K		easy	K–line	Contaminant	Common contaminant
Sr	S, Ba?	Better with FQ	L–line + doublecheck with K-Line @25kV	Contaminant	Sr-sulphate?
Ba	Sr, S	Hard to find. ppm	L-line	Contaminant	?
Nb	Ti	Hard to find. ppm	L-line	Contaminant	?

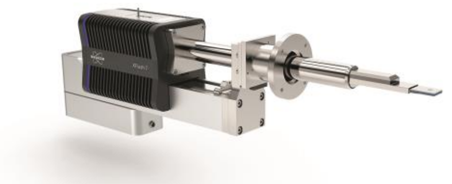
Main elements and contaminants – LiPO4 cathode material on Aluminium

Element	Coexist with	Detection with EDS	Origin	Comment
Fe		easy	Cathode	LiFePO4
O		easy	Cathode	LiFePO4
P		easy	Cathode	LiFePO4
C		easy	Cathode/Contaminant	Carbon is added to cathodes, however graphite may be occosionally viewed in cathodes as a contaminant from manufacturing.
Al		easy	Current Collector	Aluminum comes from current collector.
Ti		easy	Contaminant	Common contaminant in cathode manufacturing.
Na	Partly with Cl	easy	Contaminant	Common contaminant in cathode manufacturing.
Cl	Partly with Na	easy	Contaminant	Common contaminant in cathode manufacturing.
Si		easy	Contaminant	Common contaminant in cathode manufacturing.
Ca		easy	Contaminant	Common contaminant in cathode manufacturing.
K		easy	Contaminant	Common contaminant in cathode manufacturing.
S	Partly with Sr	easy	Contaminant	Common contaminant in cathode manufacturing.
Cu		easy	Contaminant	Common contaminant in cathode manufacturing.
Cr	Ni	with MaxPixSpectrum	Contaminant	Common contaminant in cathode manufacturing.
Ni	Cr	with MaxPixSpectrum	Contaminant	Common contaminant in cathode manufacturing.
Sr			Contaminant	
V		FlatQUAD; with MaxPixSpectrum	Contaminant	

CATHODE - Top view with FlatQUAD

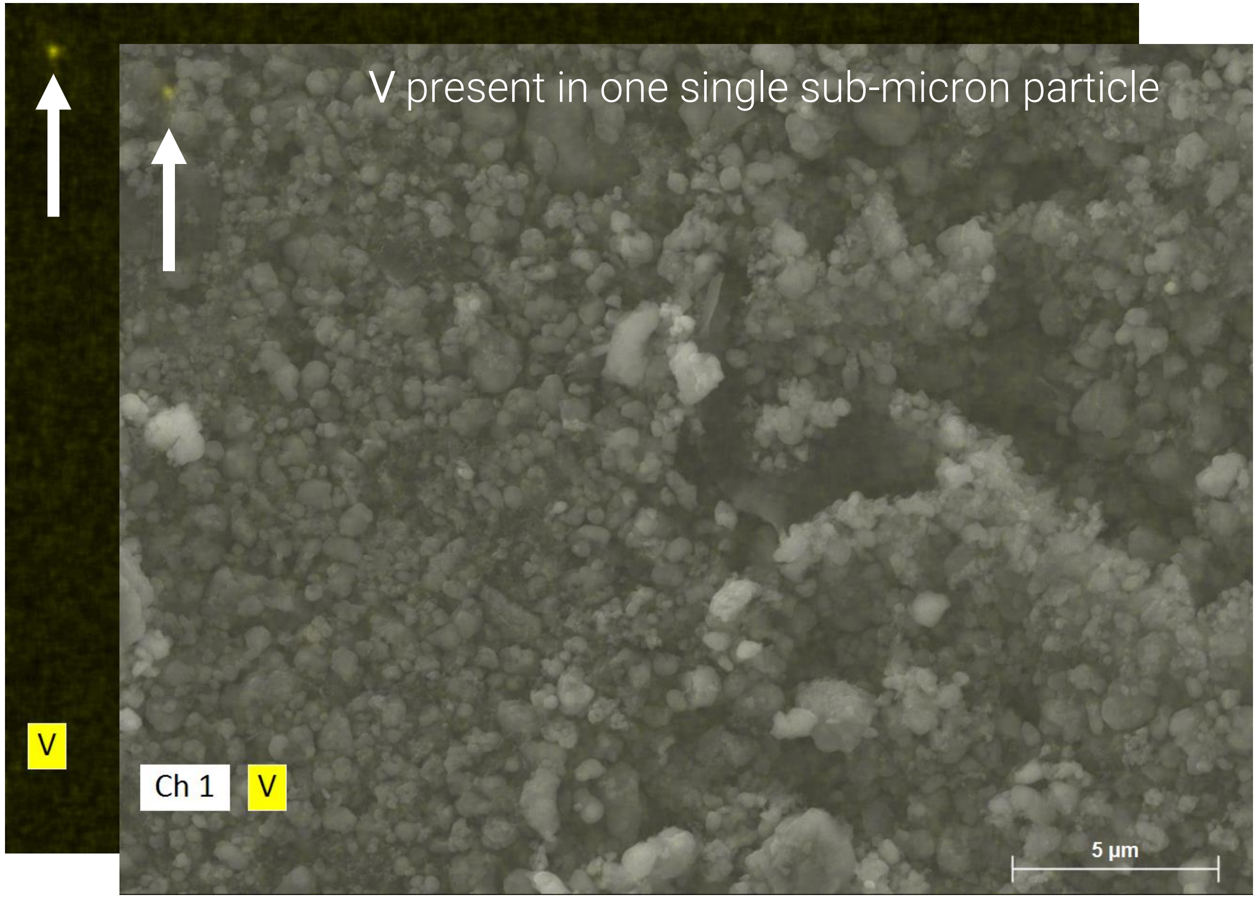


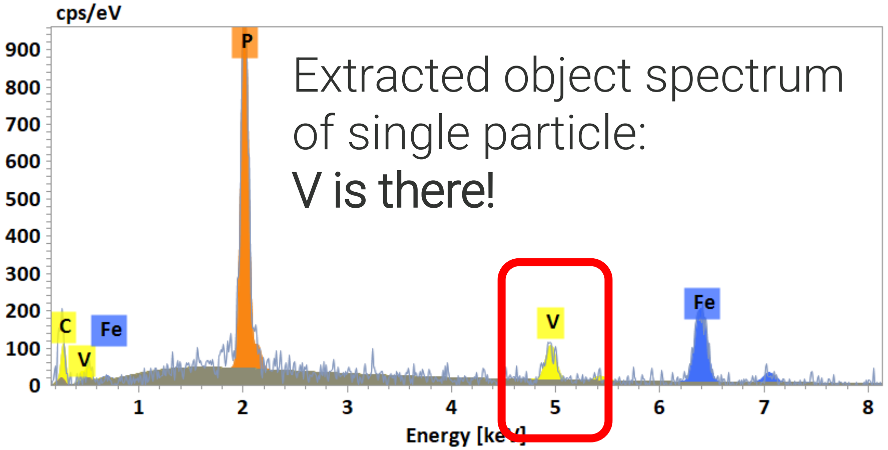
Analysis parameters	
Detector	FlatQUAD
High voltage	12 kV
Beam current	~0,5 nA
Input count rate (ICR)	640 kcps
Total counts (per map)	$7x10^7$
Mapping time	3 min



XFlash® FlatQUAD

CATHODE - Top view with FlatQUAD





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** Ti detected within **3 minutes** of measurement time!

Summary

- Introduction to EDS - what signal is being measured, how to interpret, what questions can be answered
- Measurement conditions
  - Choice of acceleration voltage and SEM parameters
- Results of anode and cathode material in top and side view
- Introduction and comparison of EDS detectors of conventional geometry and Bruker FlatQUAD
- Detection limit of EDS: How can very low concentration contamination be detected and visualized?
- Comparison of results of EDS detectors of conventional geometry and Bruker FlatQUAD
  - Shorter measurement times for trace element detection
  - No shadowing effect (easier localization of hiding elements on samples with rough topography)

03

Conclusions

04

Questions & Answers

please type your questions in the Q&A window and press *SEND*

Thank you!

BRUKER

Innovation with Integrity