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BENEATH THE SURFACE: X-RAY ANALYSES OF BATTERY MATERIALS AND STRUCTURES

A Battery Webinar Series by Rigaku

Non-destructive Inspection of Batteries Using X-ray Computed Tomography

Starting at 1 pm CDT

- *You will be muted during the webinar.*
- *You can ask questions using the Q&A tool.*
- *You should hear music if your sound is working.*

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Non-destructive Inspection of Batteries Using X-ray Computed Tomography

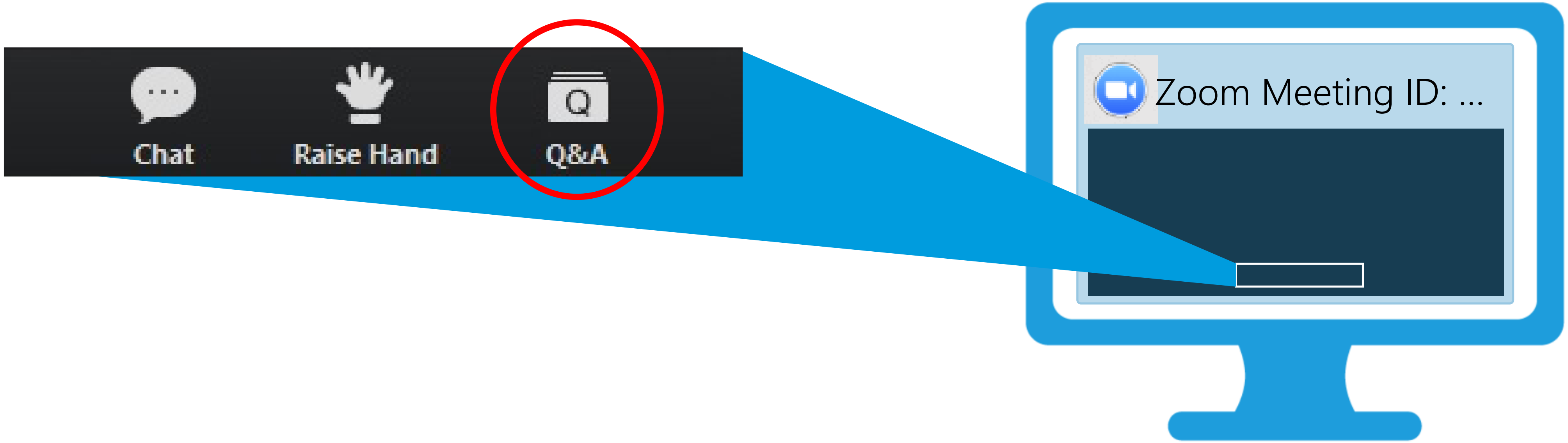
Starting at 1 pm CDT

We are starting now…

Presenter: **Angela Criswell** | Director of X-ray Imaging

Co-presenter: **Tim Bradow** | Sr. Business Development Manager

Host: **Aya Takase** | Head of Global Marketing



You can ask questions following the presentation.

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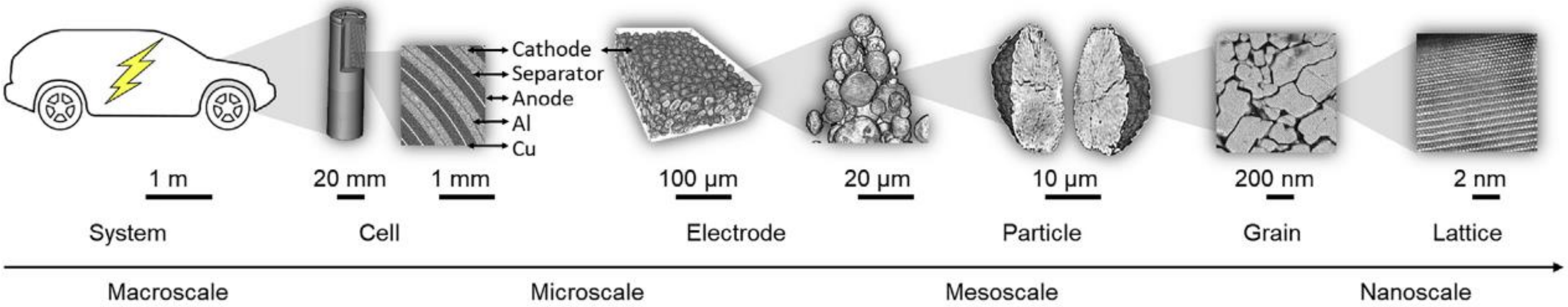
Recording will be available tomorrow.

Non-destructive inspection of batteries using X-ray computed tomography



Qian, G., *et. al.*, 2021. *Cell Reports Physical Science* 2, 100554.

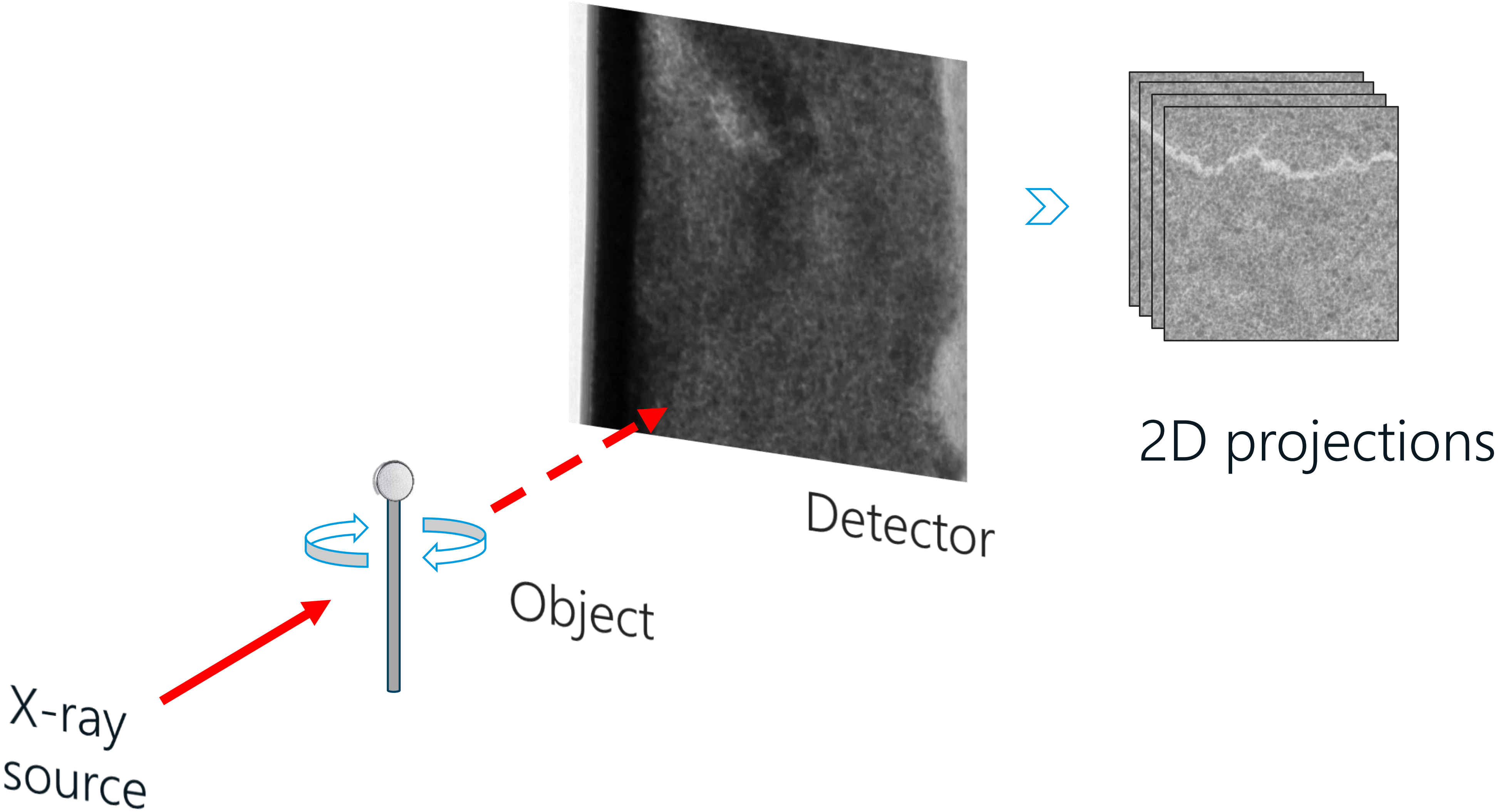
We will discuss:

- What is X-ray CT?
- What are the considerations when using X-ray CT for battery research?
- What information can we extract from CT data about batteries?
- Battery analysis examples

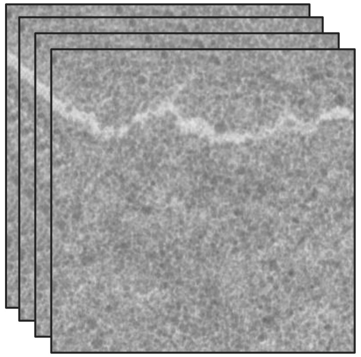
Polling Question #1

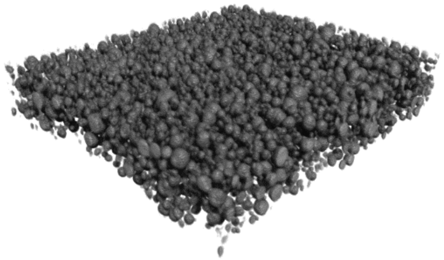
Microsoft Stock

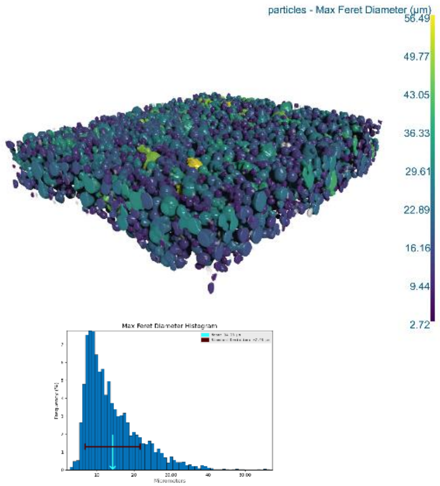
What is X-ray CT?


Detector
2D projections
Object
X-ray source

Rigaku







CT scan

Reconstruction

3D Data

Segmentation & data analysis

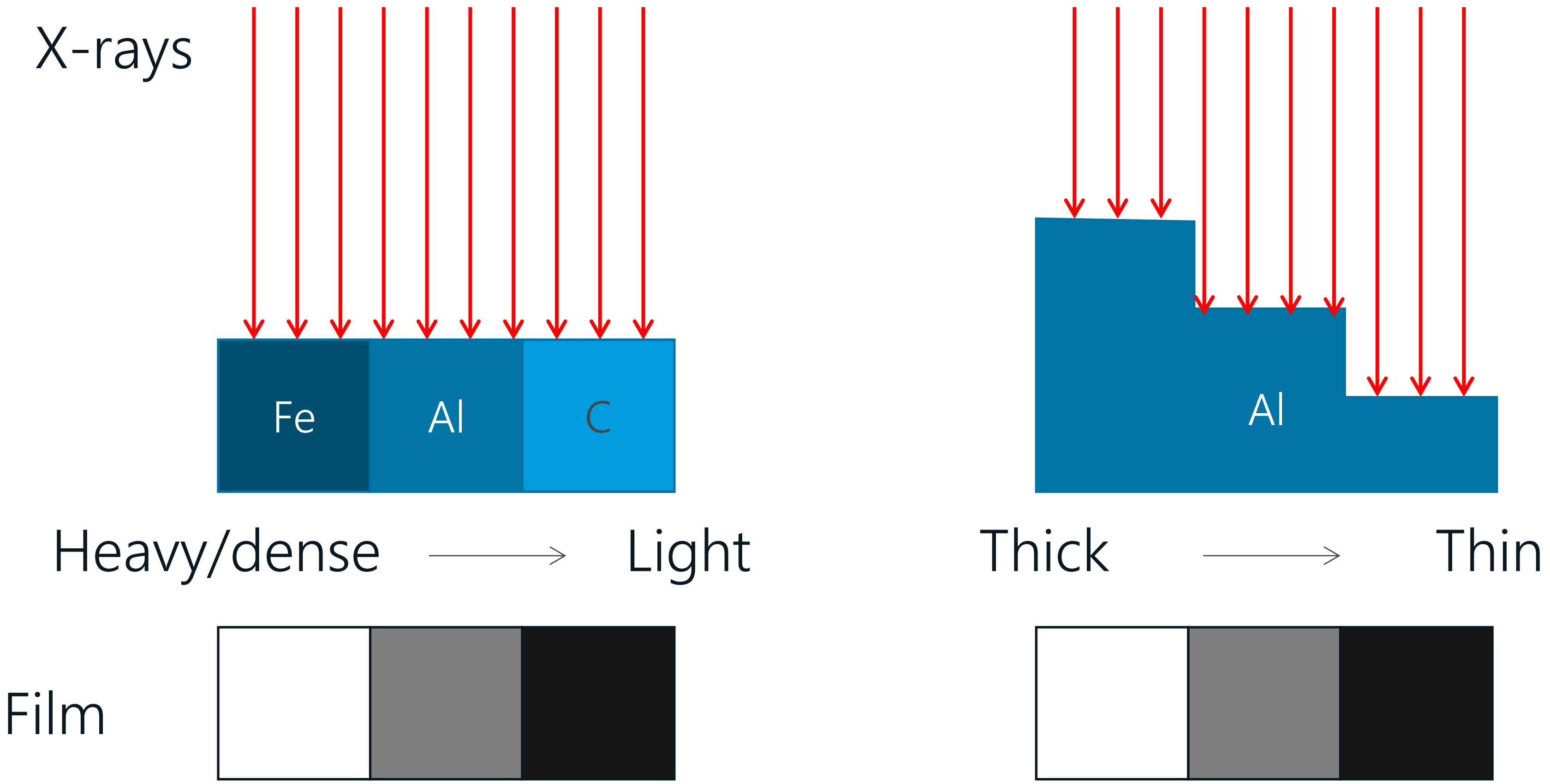
What are the considerations when using X-ray CT for battery research?

X-ray CT experiment considerations

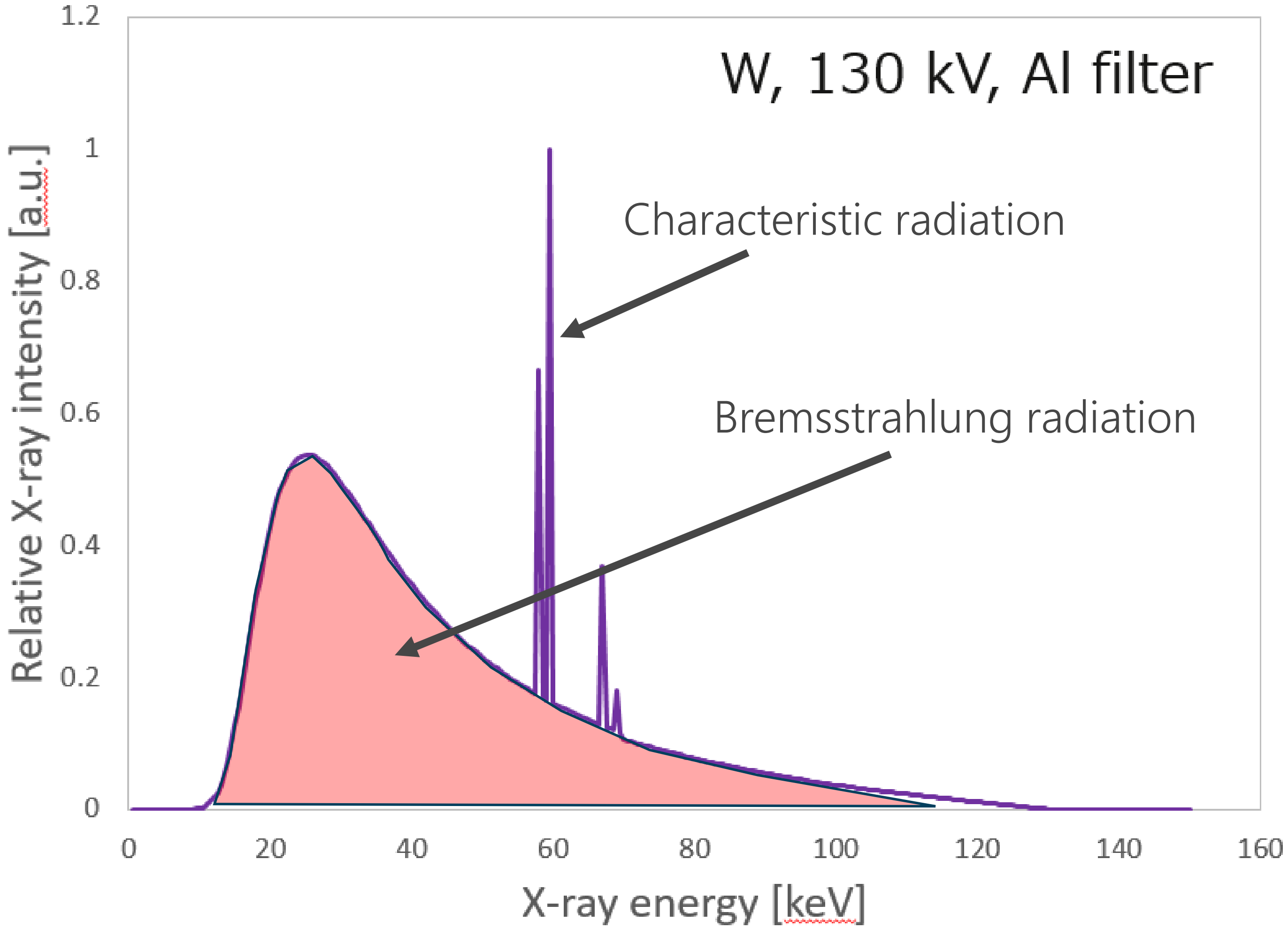
- X-ray energy
- Image contrast
- Spatial resolution
- Field of view (FOV)
- Signal-to-noise
- Experiment type

X-ray energy & image contrast

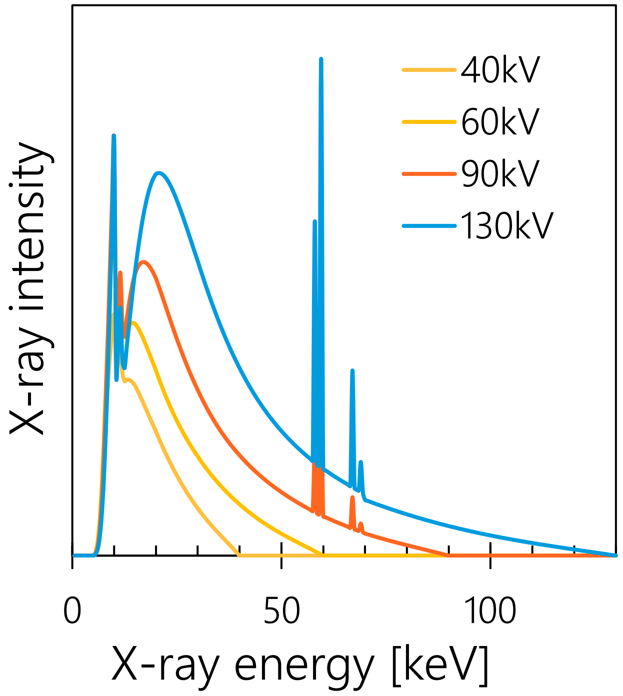
X-ray CT is an X-ray absorption technique

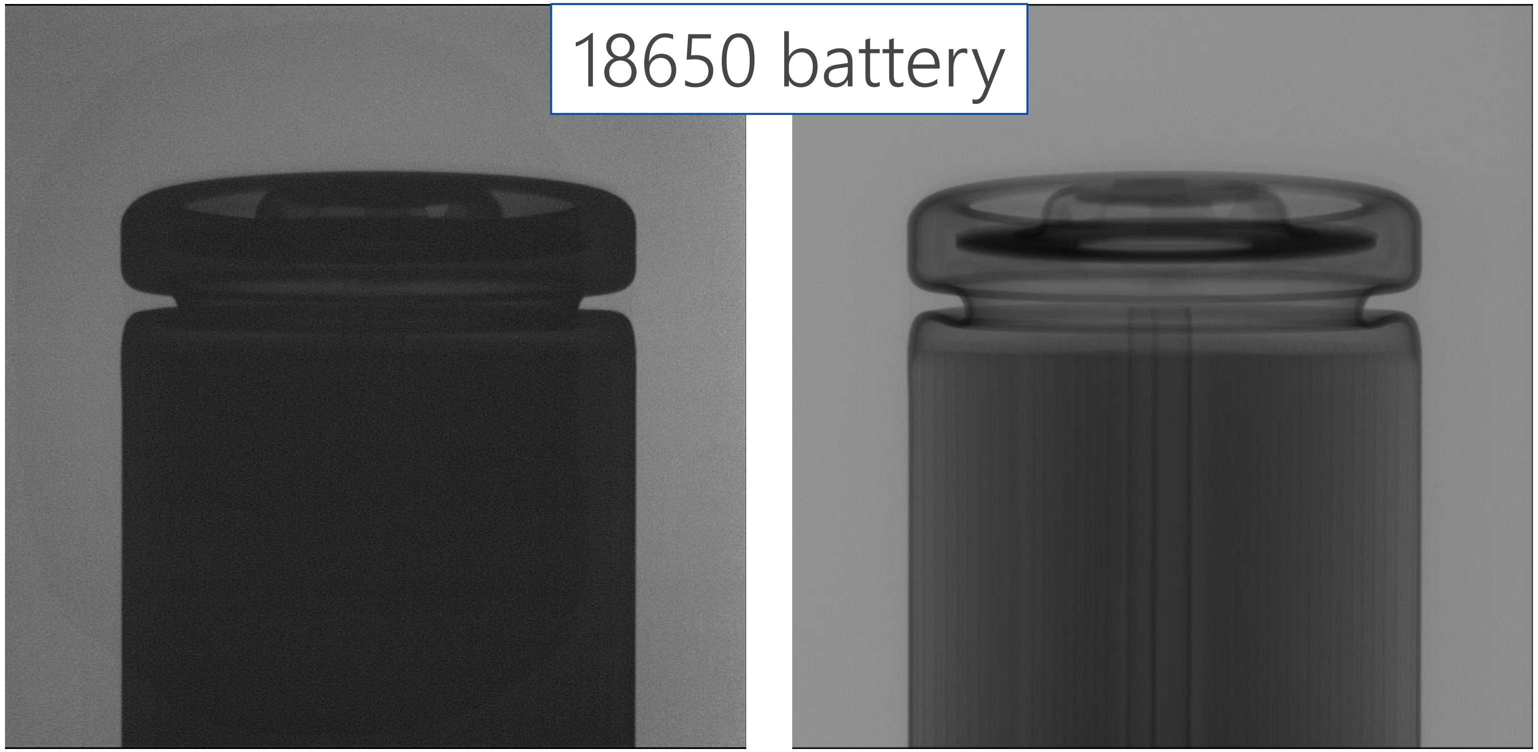


- Bremsstrahlung vs. characteristic radiation



- X-ray energy
  - Bremsstrahlung radiation (change applied voltage, kV)

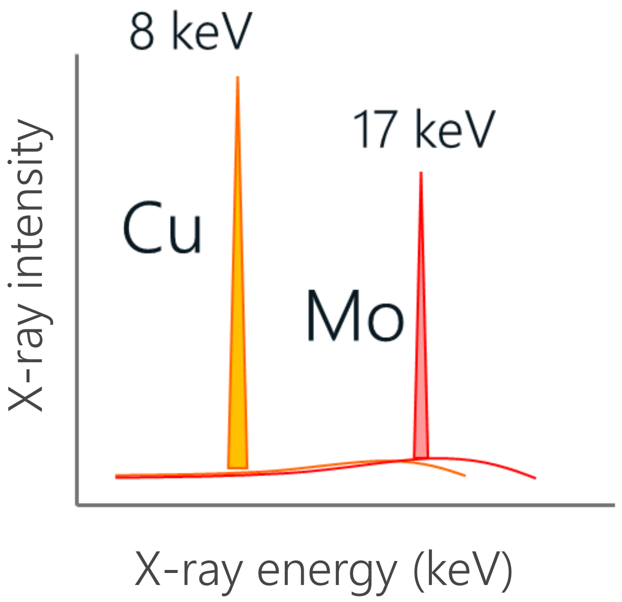


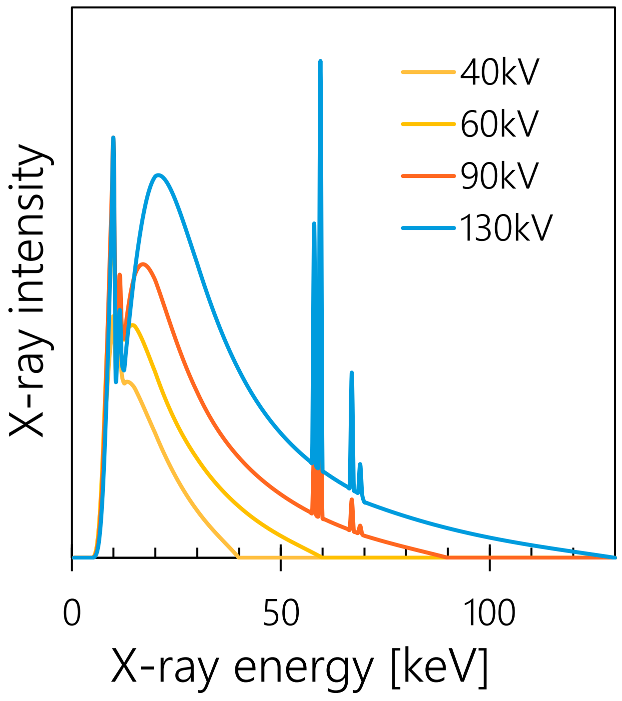


50 kV

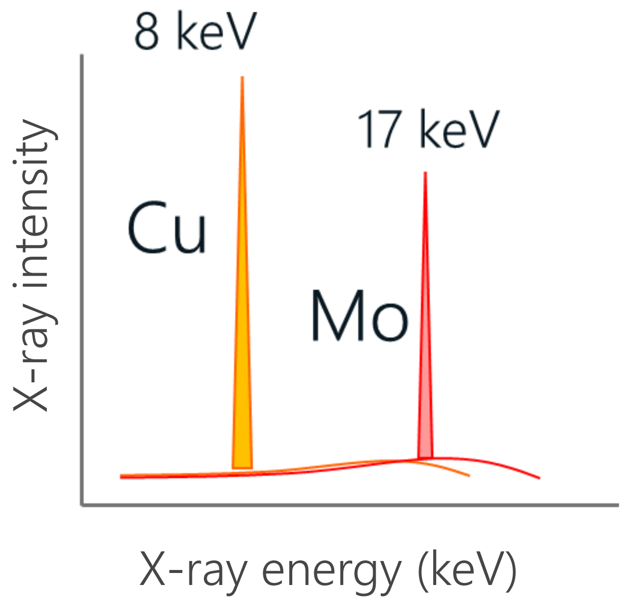
200 kV

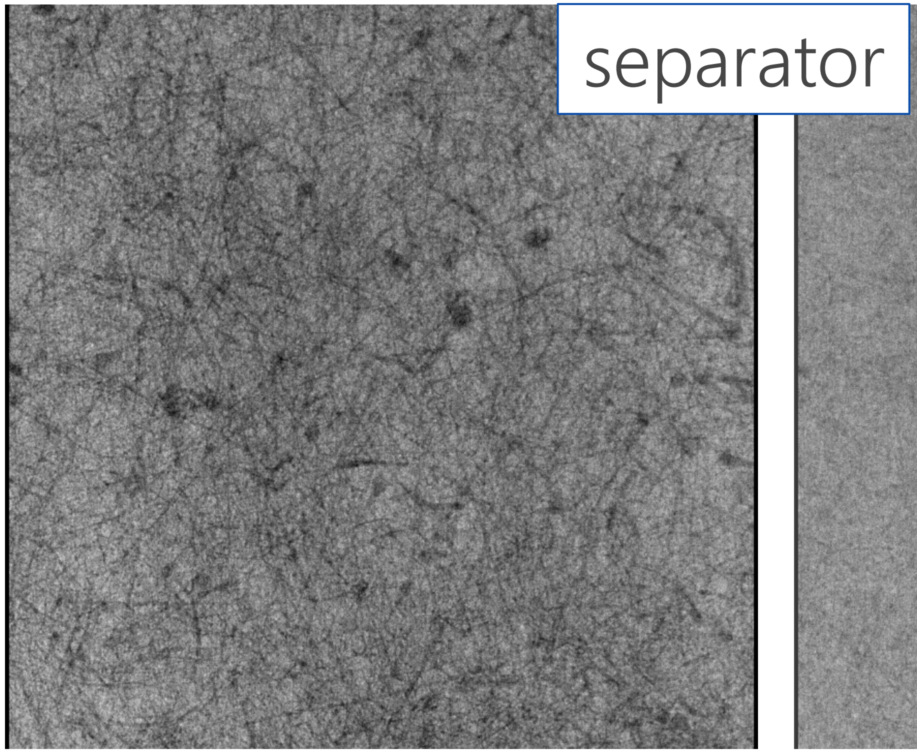
- X-ray energy
  - Characteristic radiation (change target material - Cu, Mo)



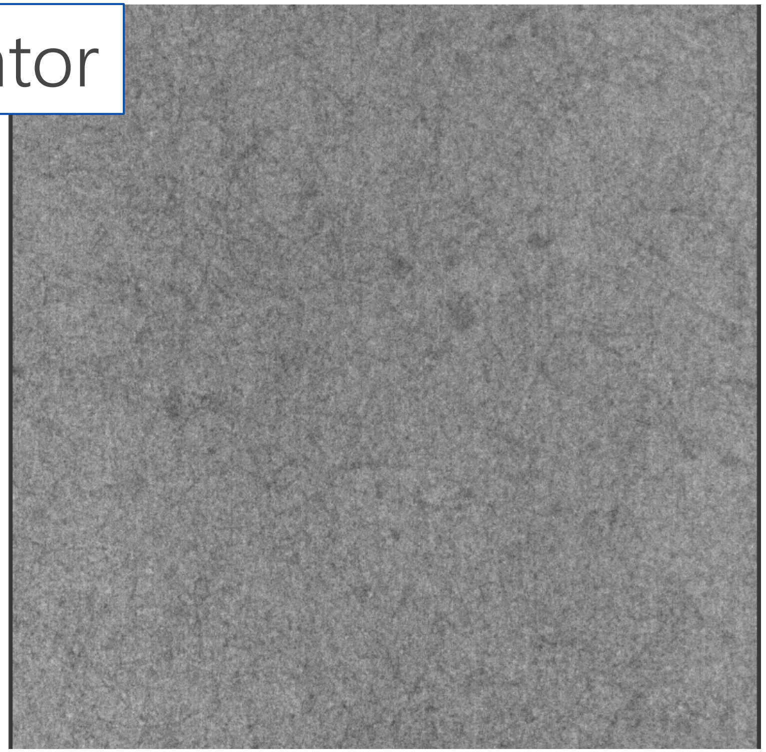


- X-ray energy
  - Characteristic radiation (change target material - Cu, Mo)





Cu (8 keV)

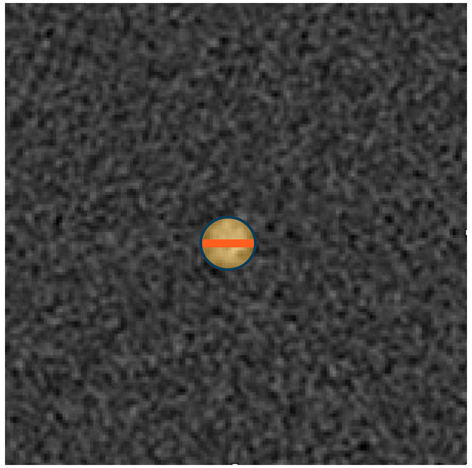


Mo (17 keV)

Spatial resolution & FOV

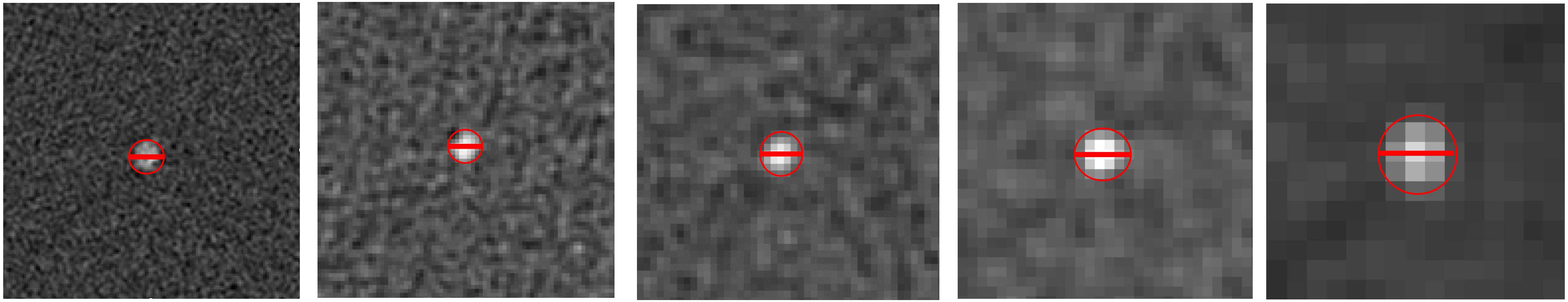
Voxel size

1.3 μm

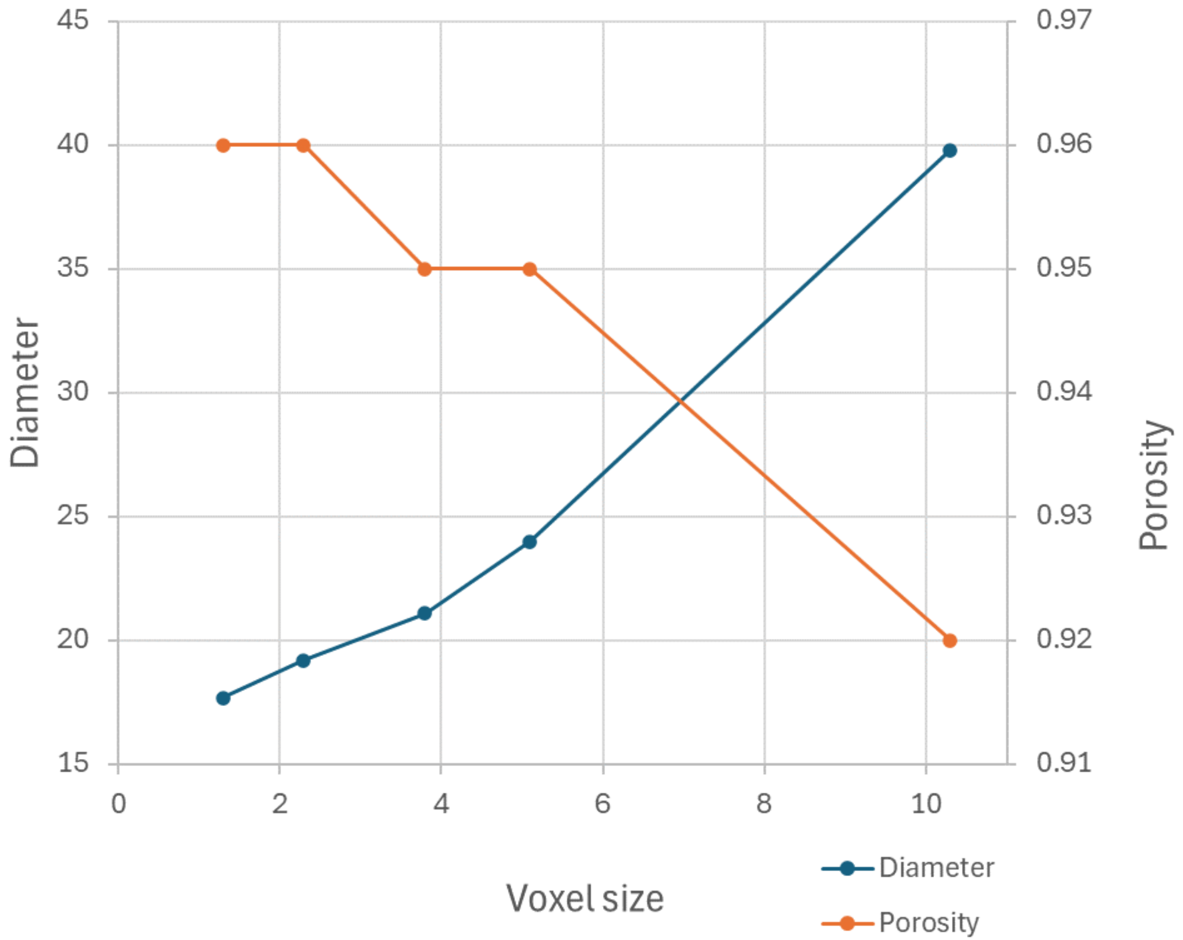


17.7 μm

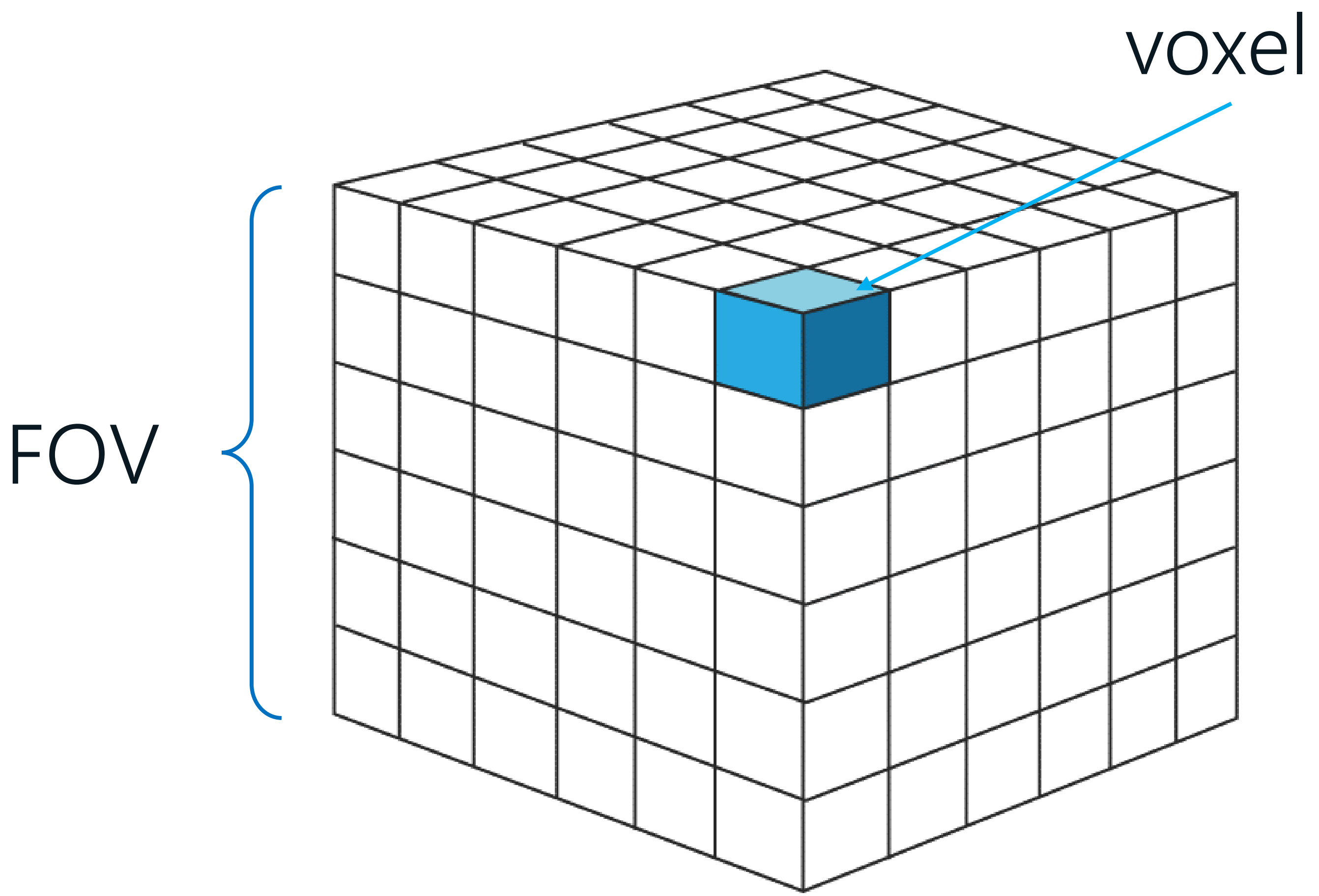
Voxel size				
1.3 µm	2.6 µm	3.8 µm	5.1 µm	10.3 µm



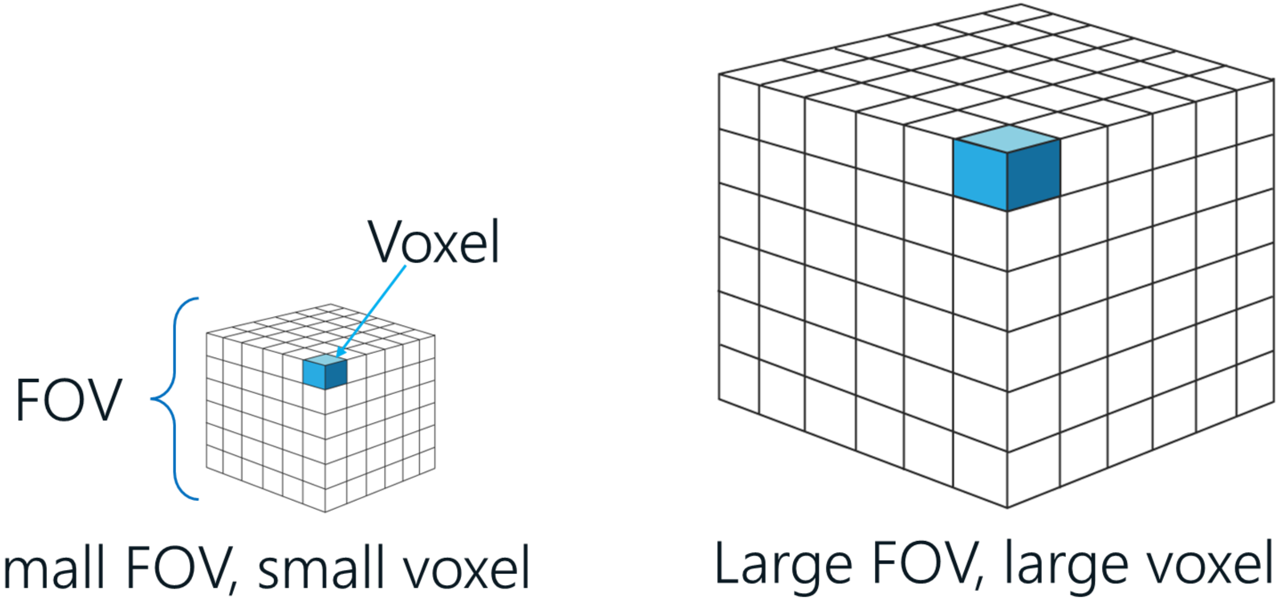
	1.3 µm	2.6 µm	3.8 µm	5.1 µm	10.3 µm
Diameter	17.7 µm	19.2 µm	21.1 µm	24.0 µm	39.8 µm
Porosity	0.96	0.96	0.95	0.95	0.92



Voxel size < ⅕ feature size

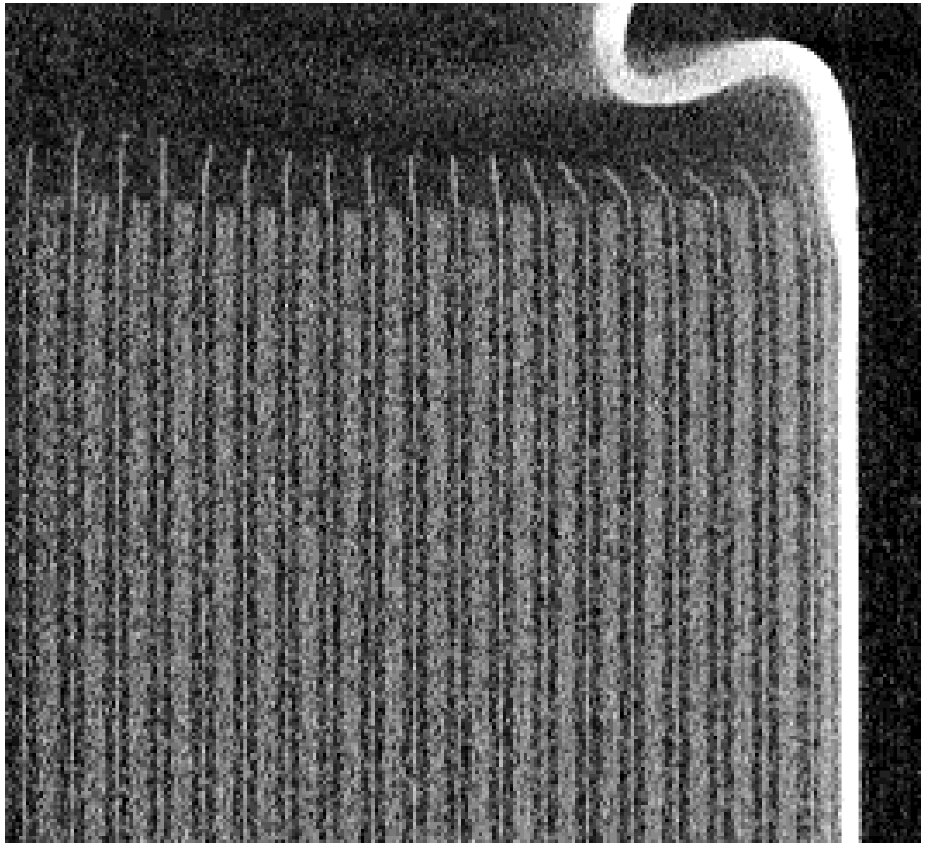

voxel
FOV

FOV [mm]	Voxel [µm]	File size
100 x 100 x 100	1	2000 TB

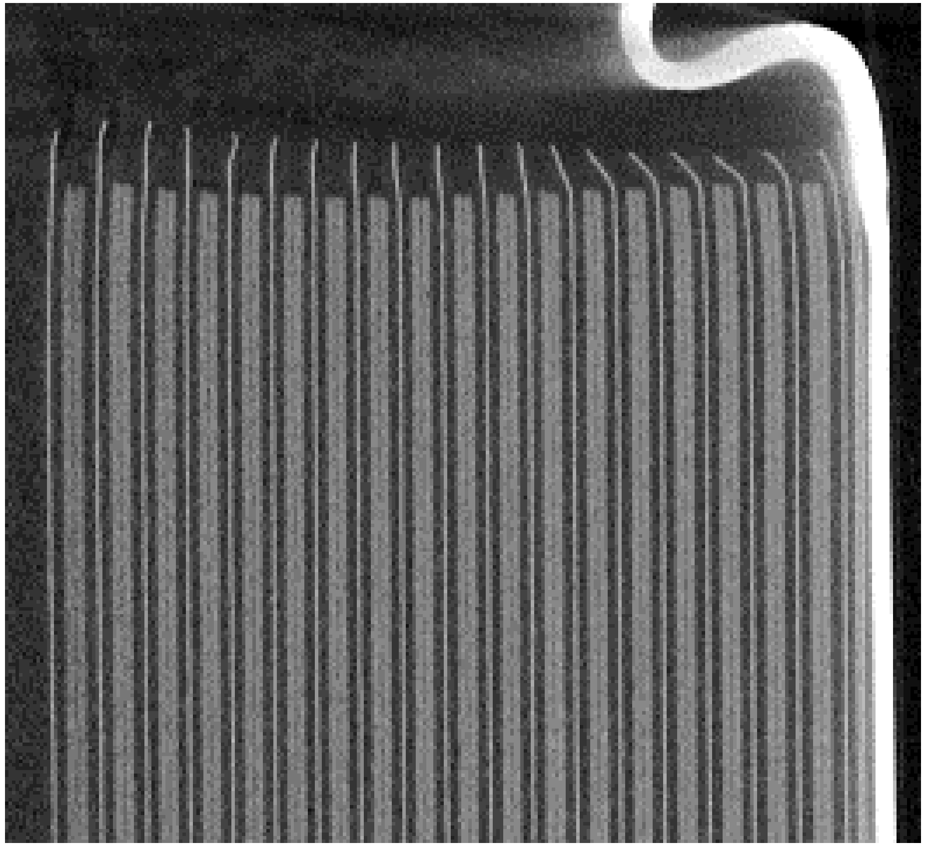


Signal-to-noise

18650 battery



8.5 min

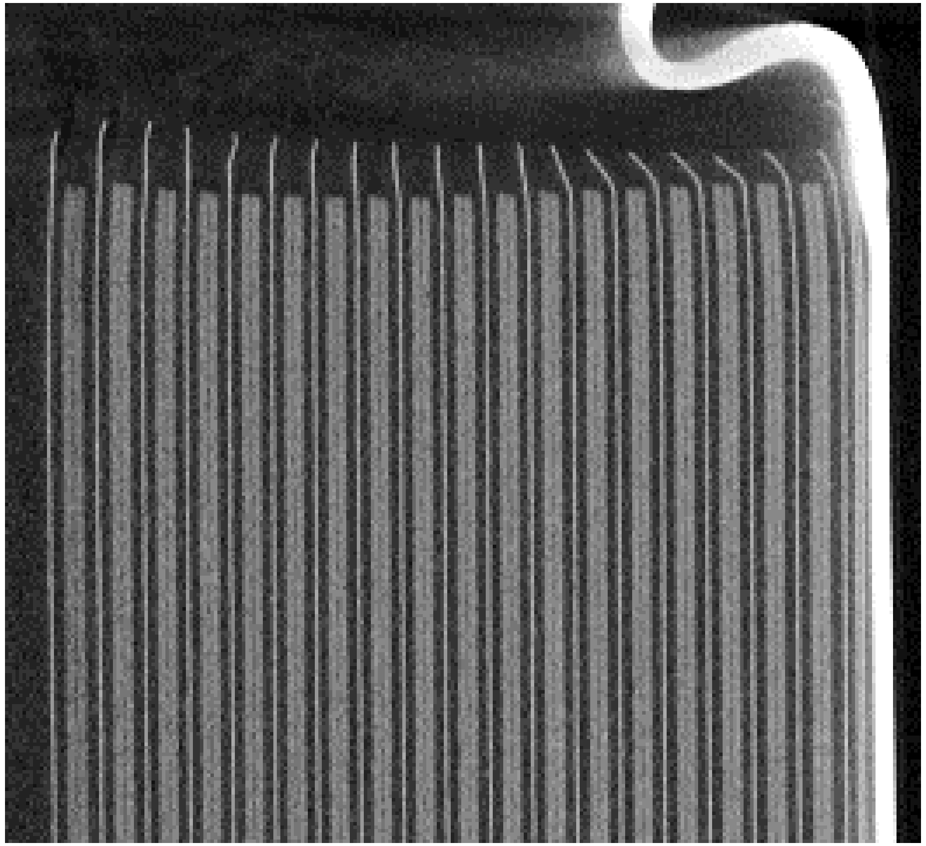


136 min

18650 battery

Improving signal-to-noise

- Increase the scan time
- Shorten the source-to-detector distance
- Bin pixels



136 min

Experiment type

CT experiments with batteries

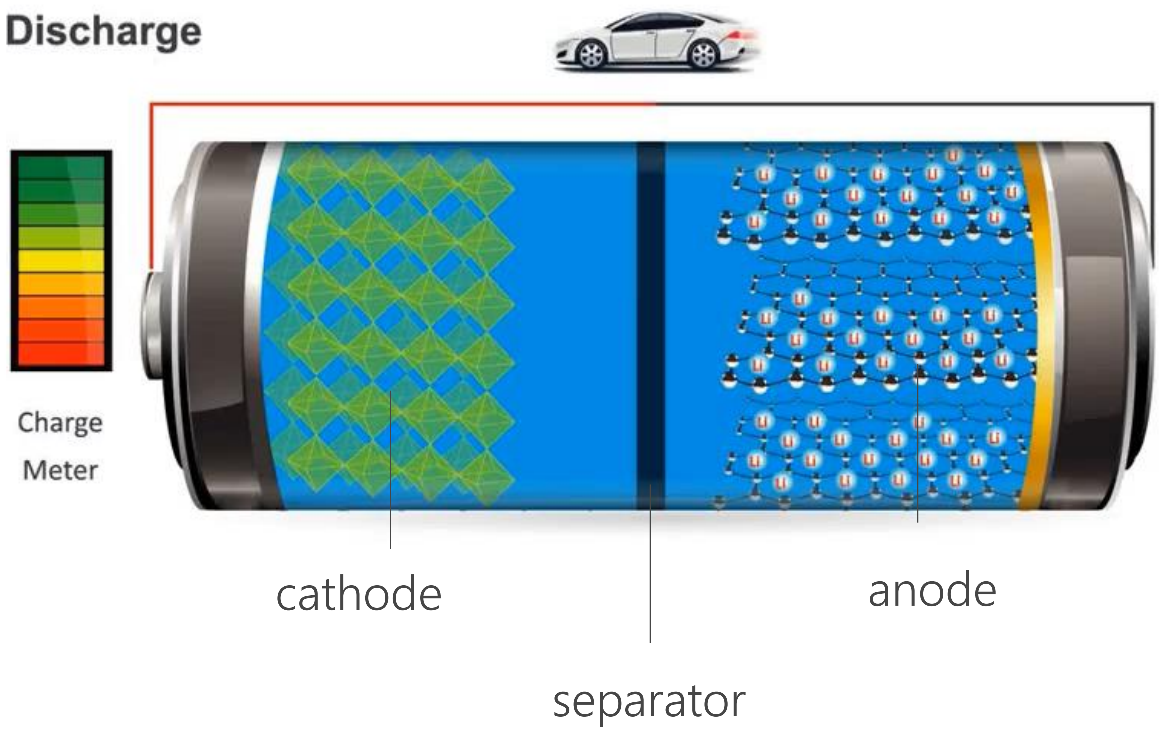
- Battery materials
  - Ex situ – Raw & cycled materials
  - In situ - Cycled materials
- Battery cells
  - In situ
  - Operando

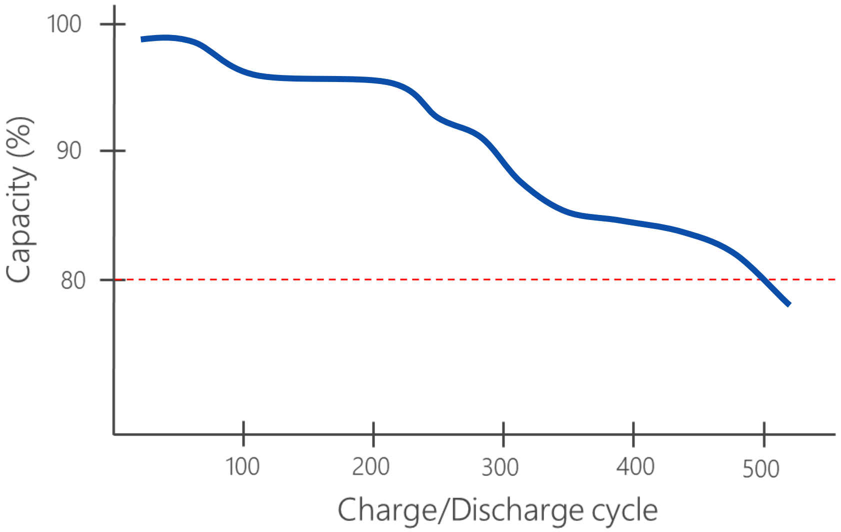
Polling Question #2

Microsoft Stock

What information can we extract from CT data about batteries?

Li-ion batteries

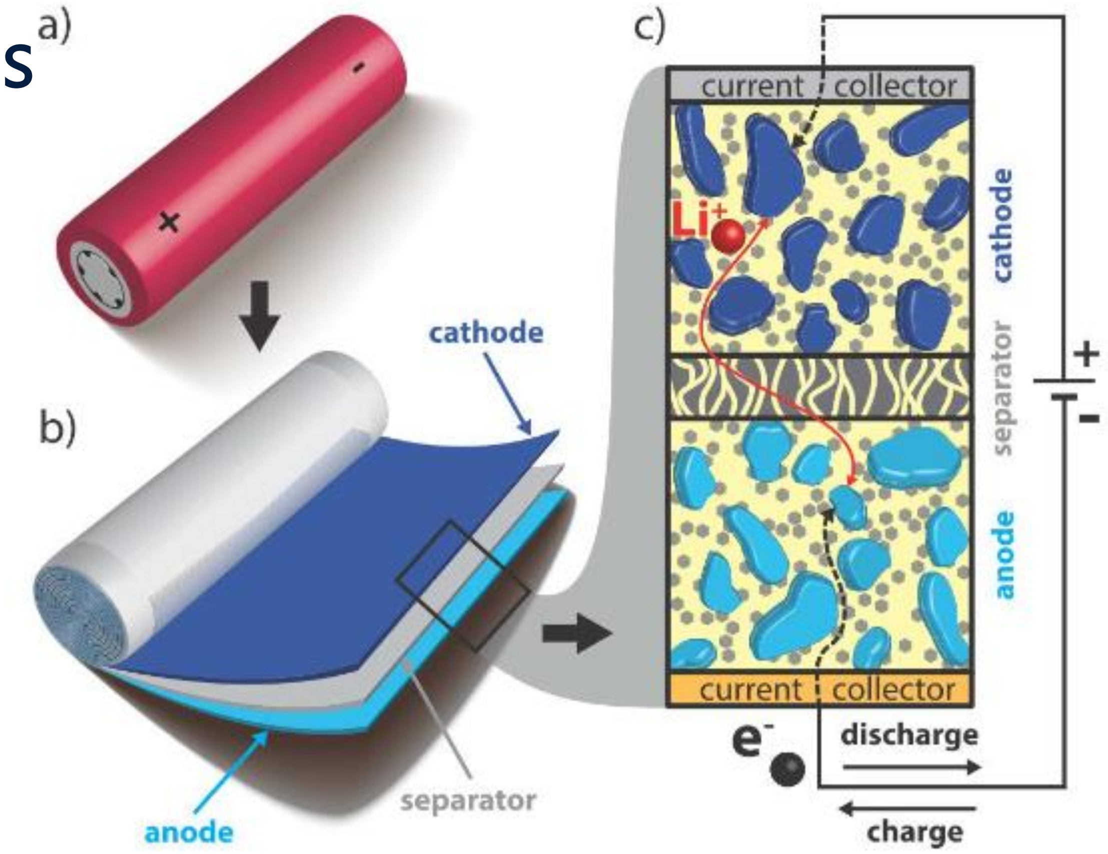




https://www.energy.gov/energysaver/articles/how-lithium-ion-batteries-work

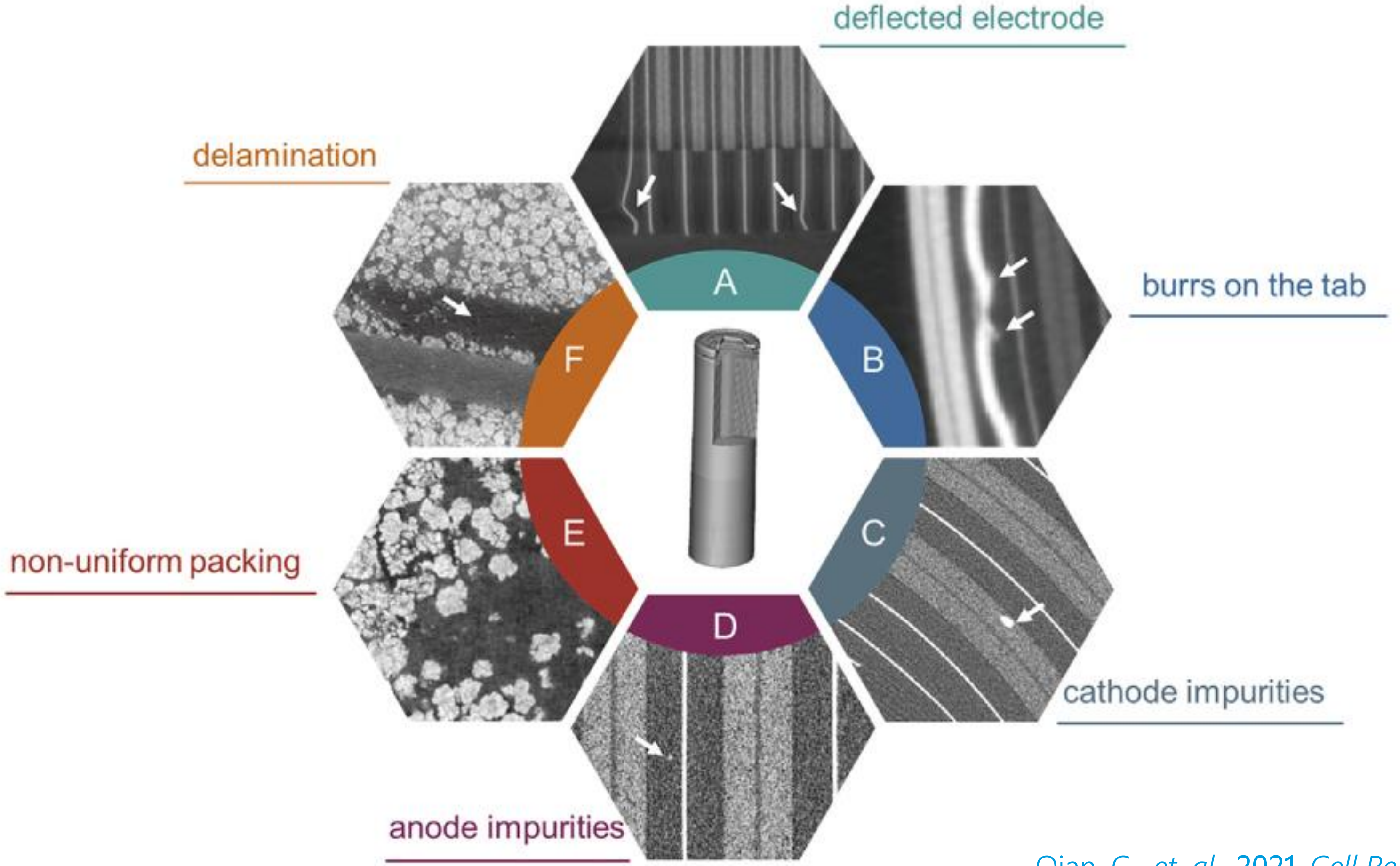
nm-to-μm scale CT features

- Cathode/anode design
- Cathode/anode performance
- Separator design
- Electrolyte distribution
- Battery cell design

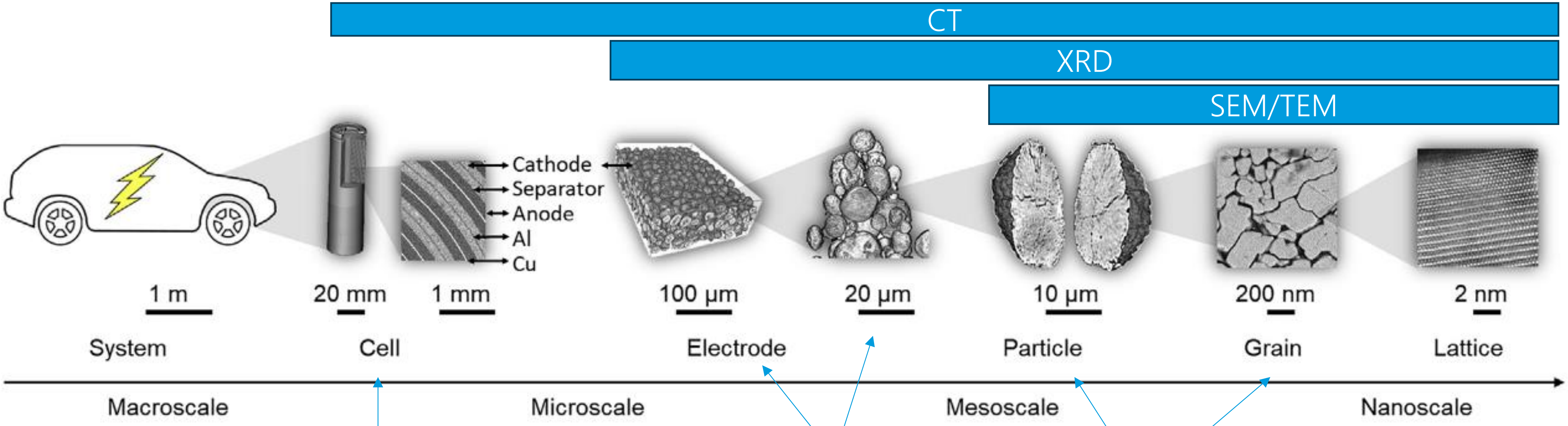


Pietsch, P., Wood, V., 2017. *Annu. Rev. Mater. Res.* 47, 451–479.

µm-to-m scale CT features



Qian, G., *et. al.*, 2021. *Cell Reports Physical Science 2, 100554.*



Quality control of cell structure
Electrode deflection
Electrode thickness
Defect and failure analysis
Delamination
Cathode/anode impurities

Electrode deposition & homogeneity
Particle size
Particle shape
Porosity
Permeability
Tortuosity

Grain size
Grain shape
Porosity
Surface area

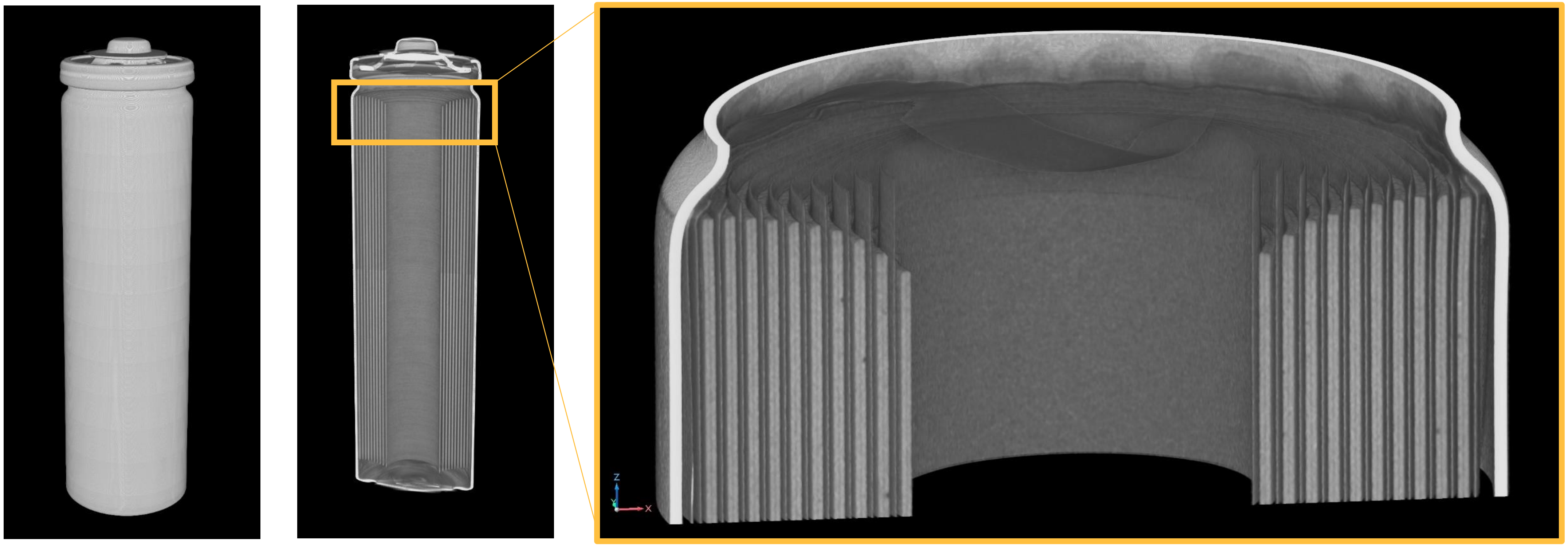
Qian, G., *et. al.*, **2021**. *Cell Reports Physical Science 2*, 100554.

Polling Question #3

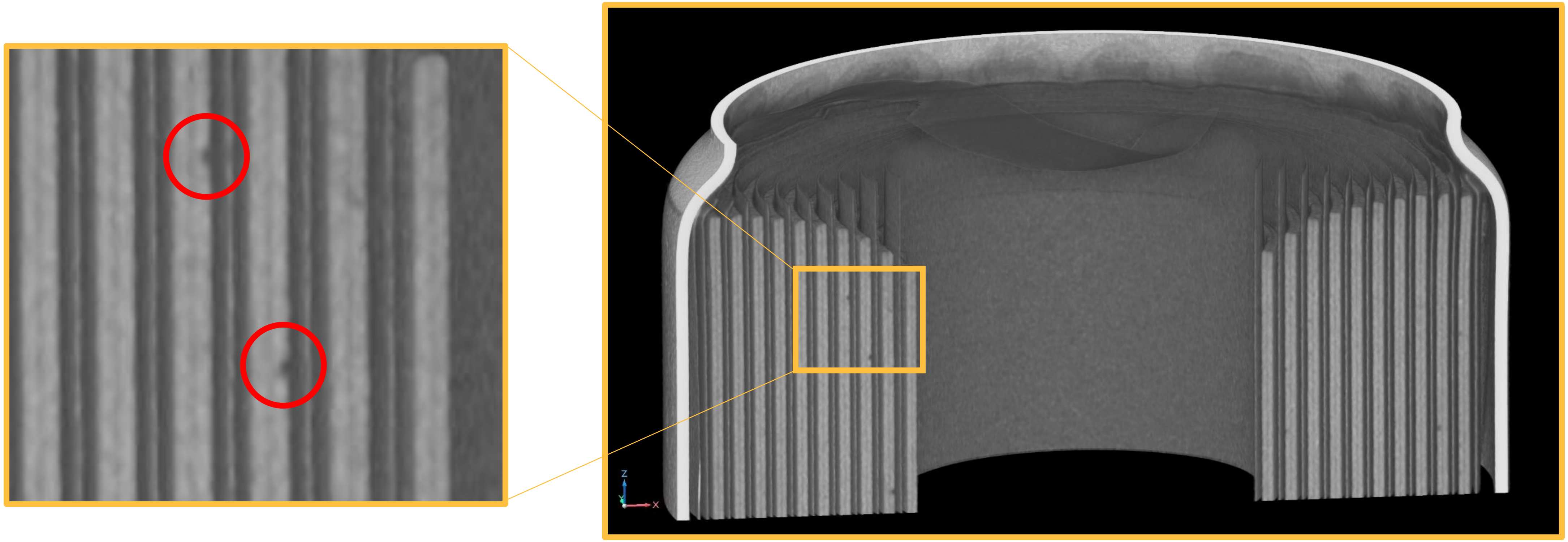
Microsoft Stock

Battery analysis examples

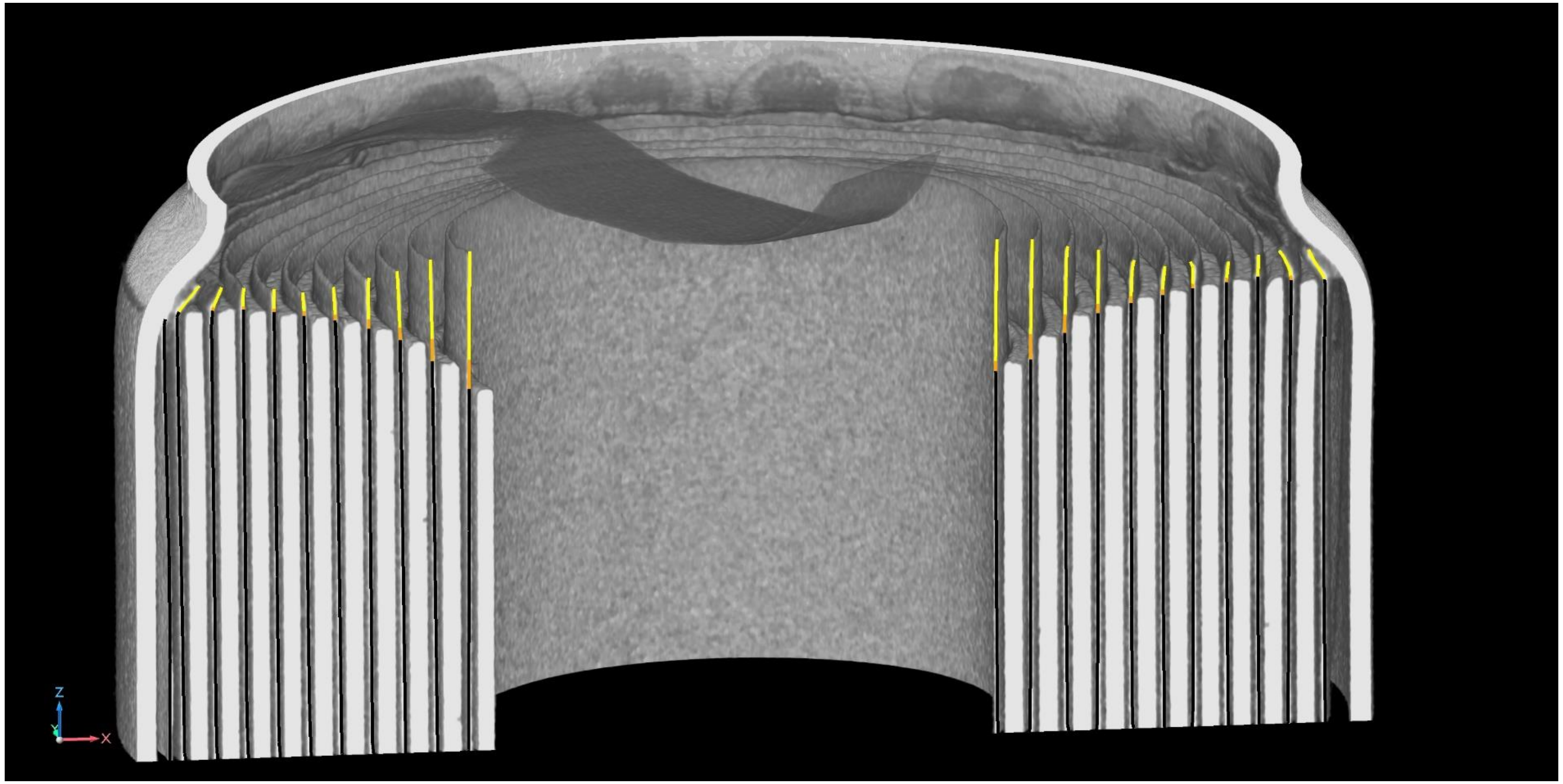
18650 cell analysis



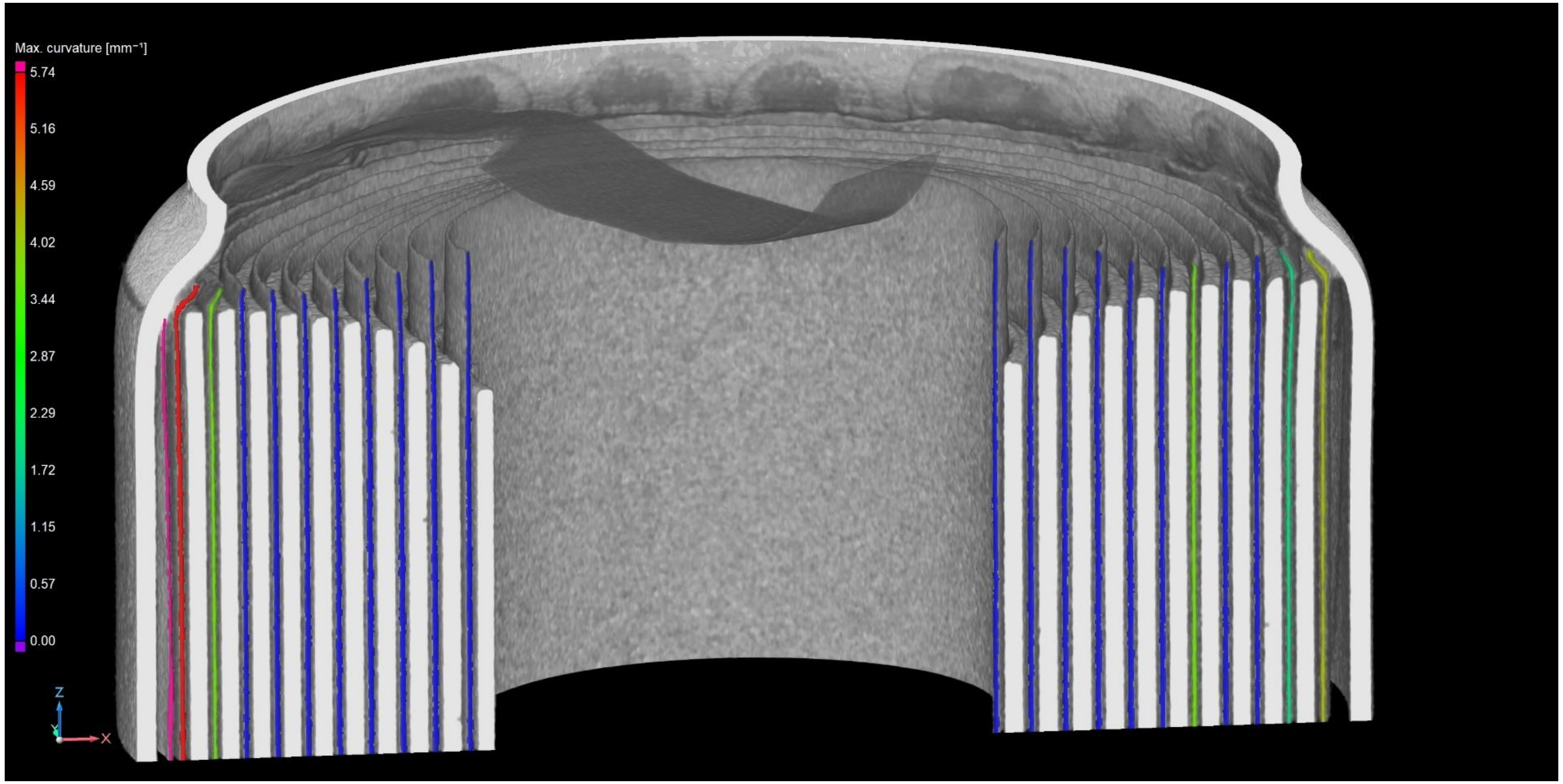
18650 cell analysis



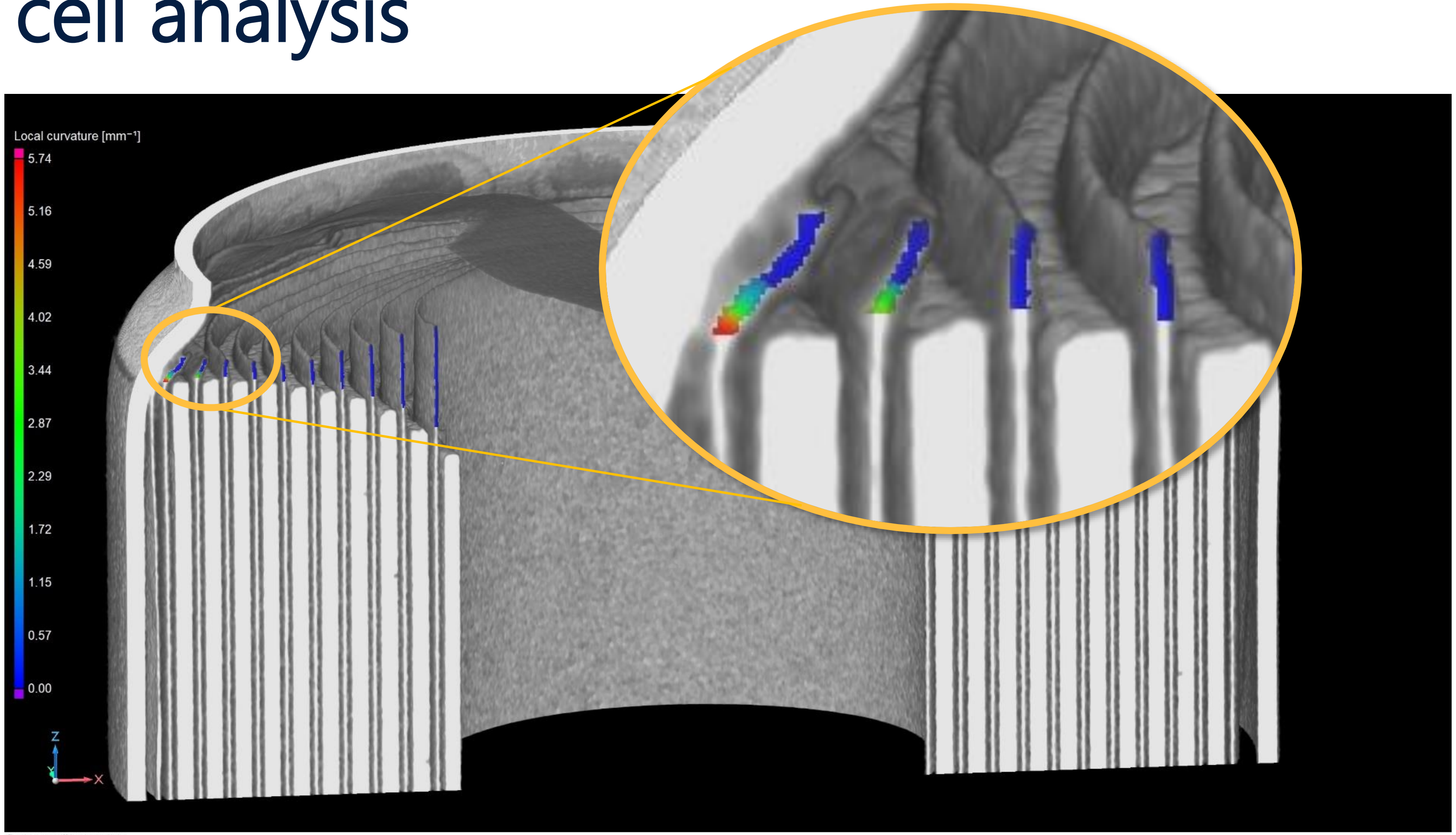
18650 cell analysis



18650 cell analysis



18650 cell analysis



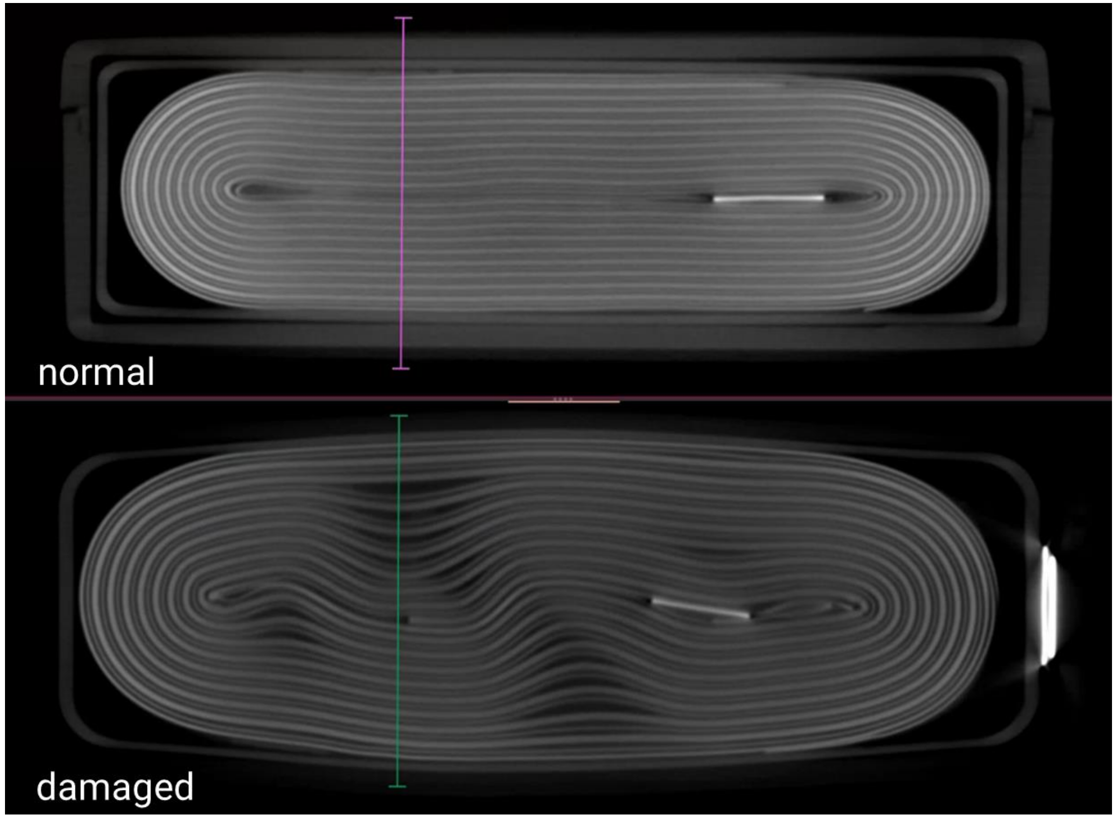
Scene coordinate system
3D

Li-ion battery from digital camera

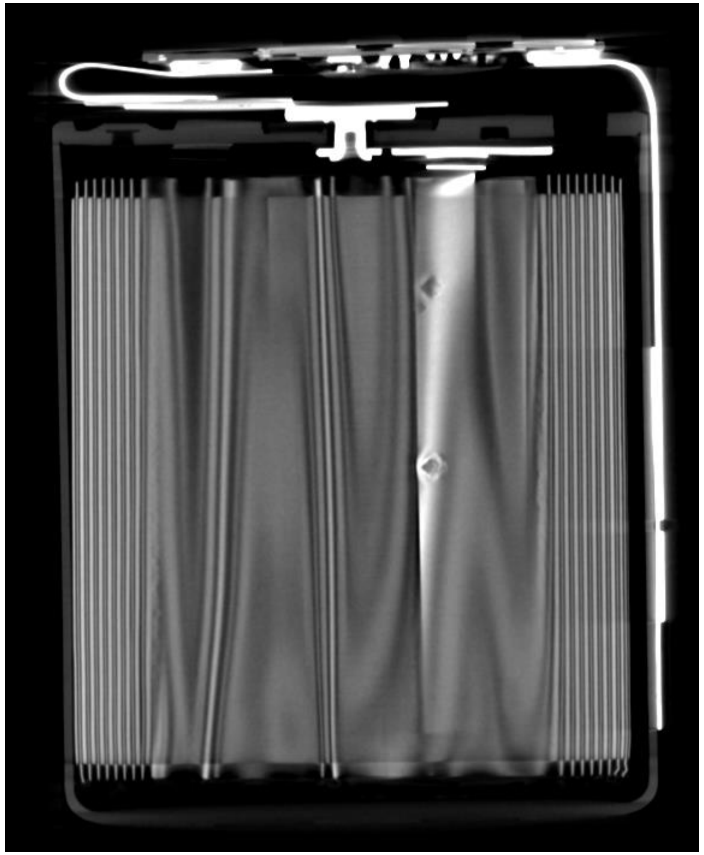
Normal

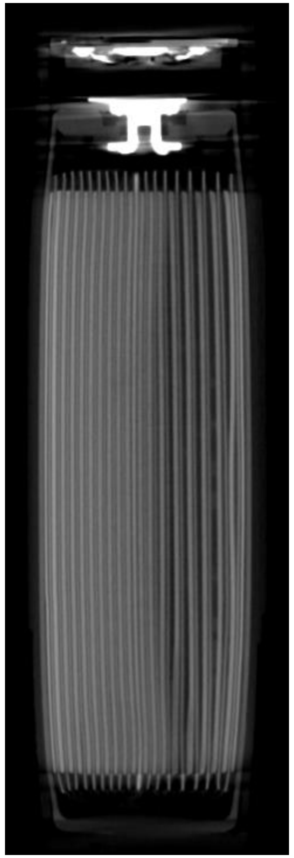
Damaged

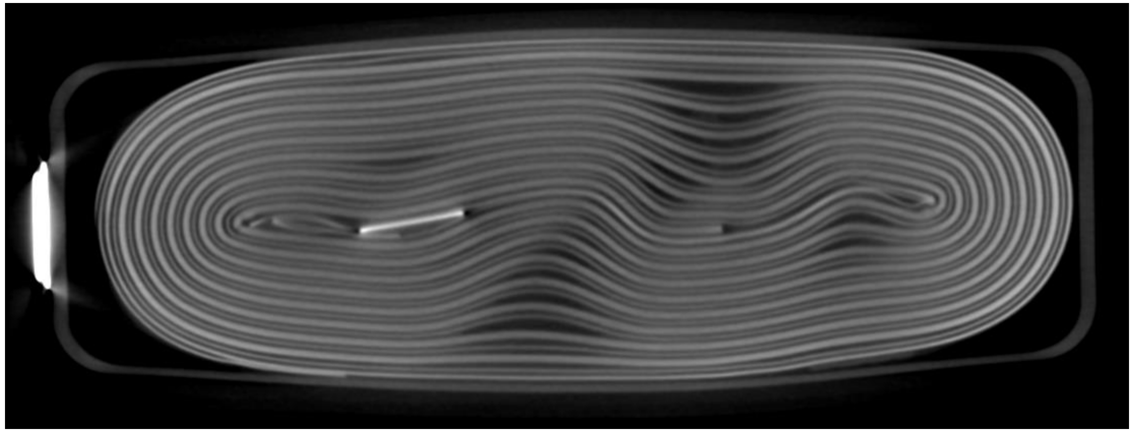
Li-ion battery from digital camera



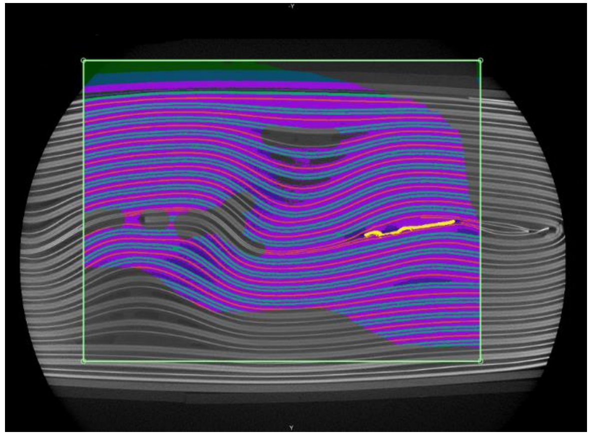
Li-ion battery from digital camera



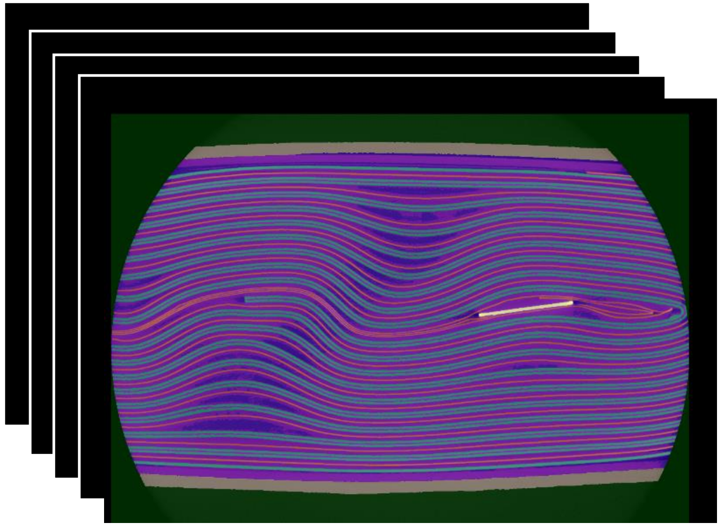




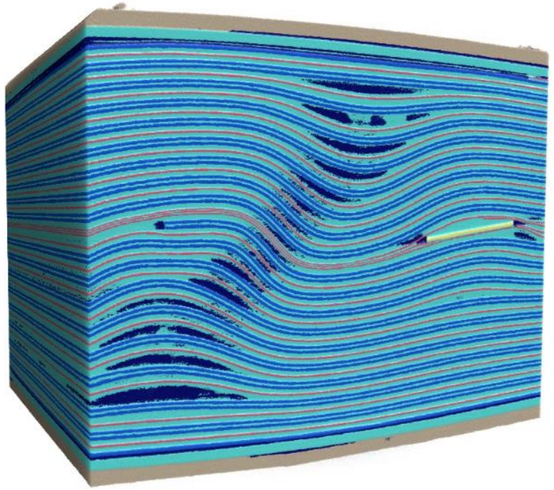
Li-ion battery from digital camera



Label Ground Truth and train deep learning model

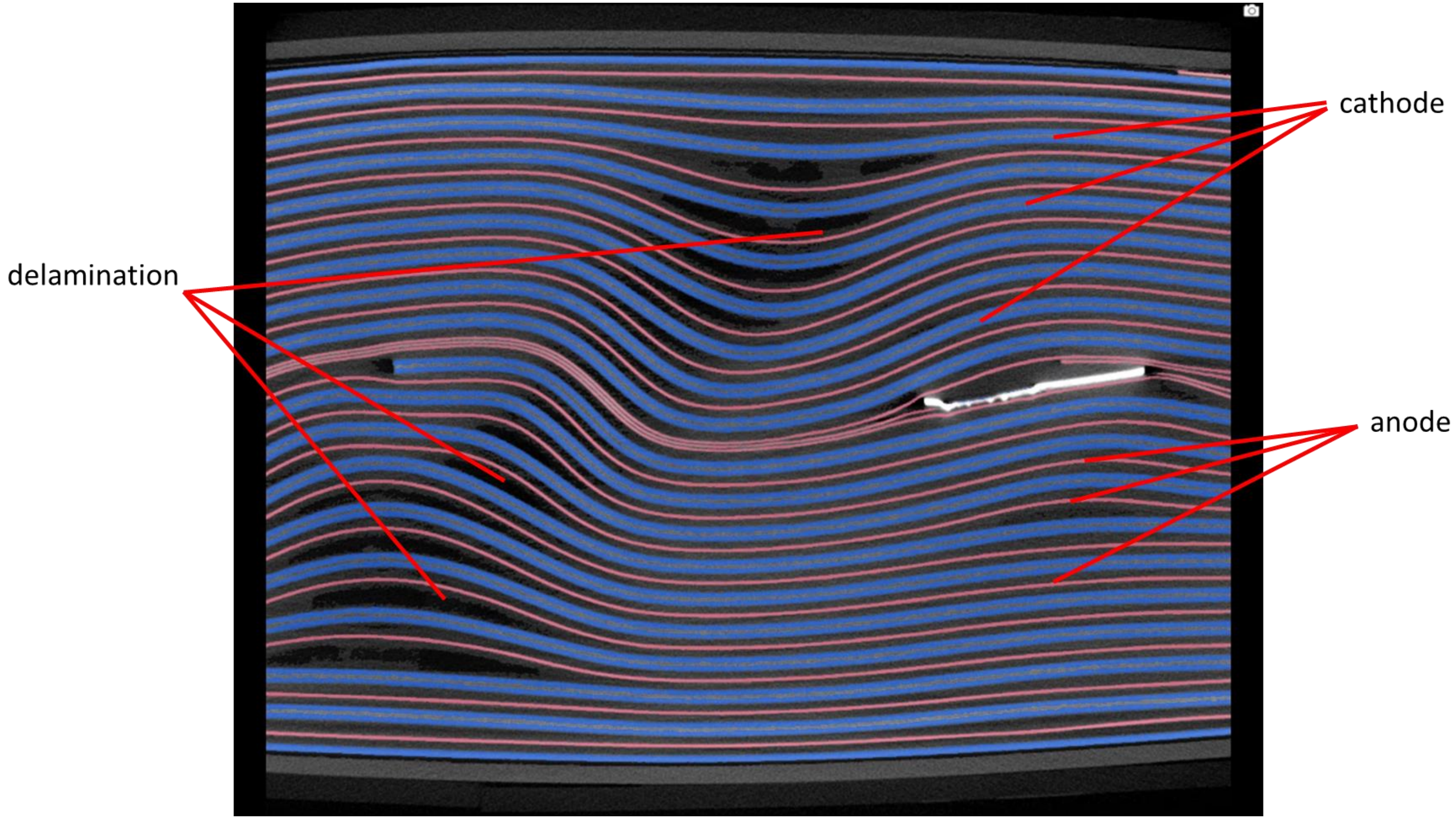


Deep learning segmentation

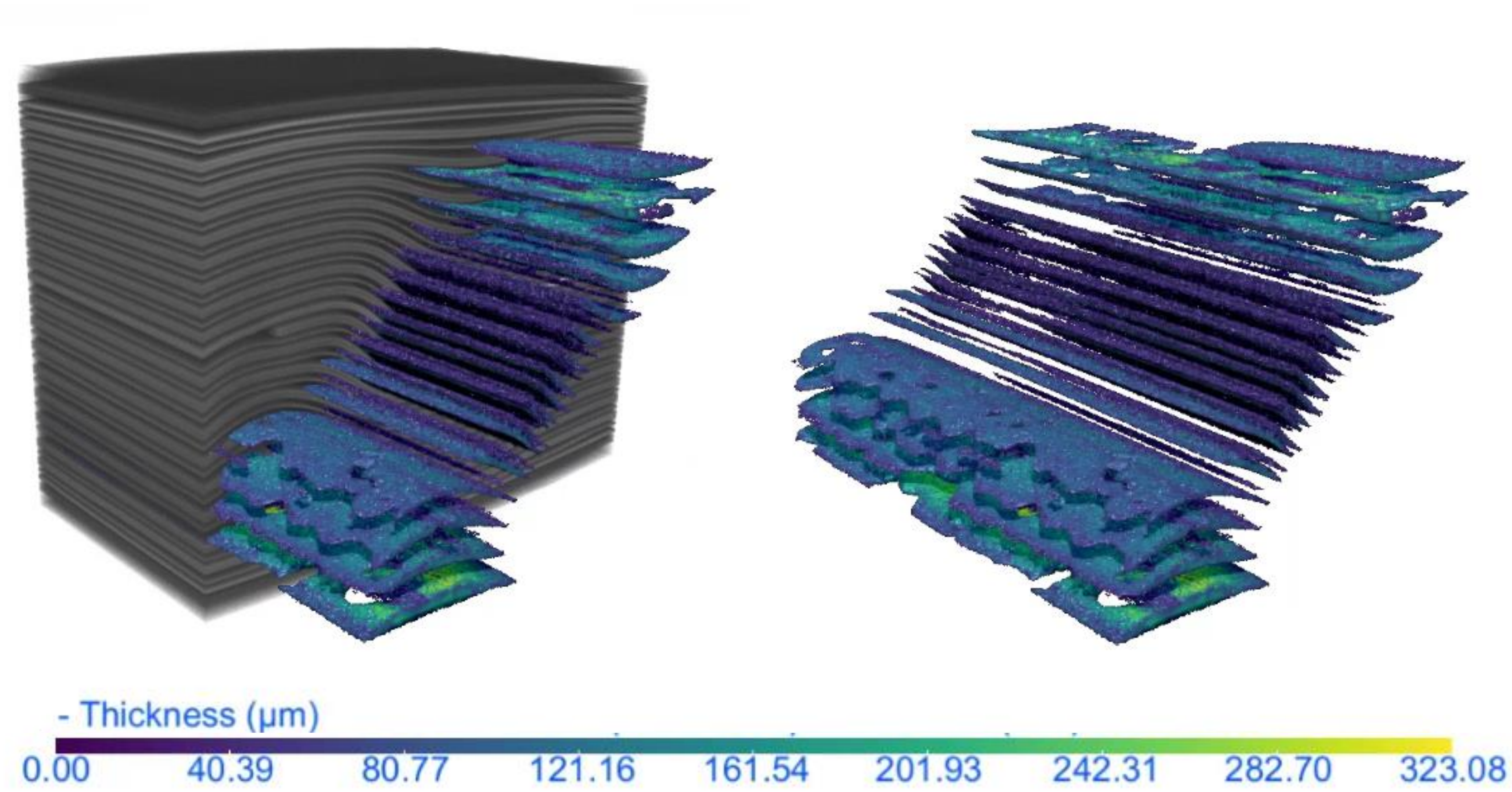


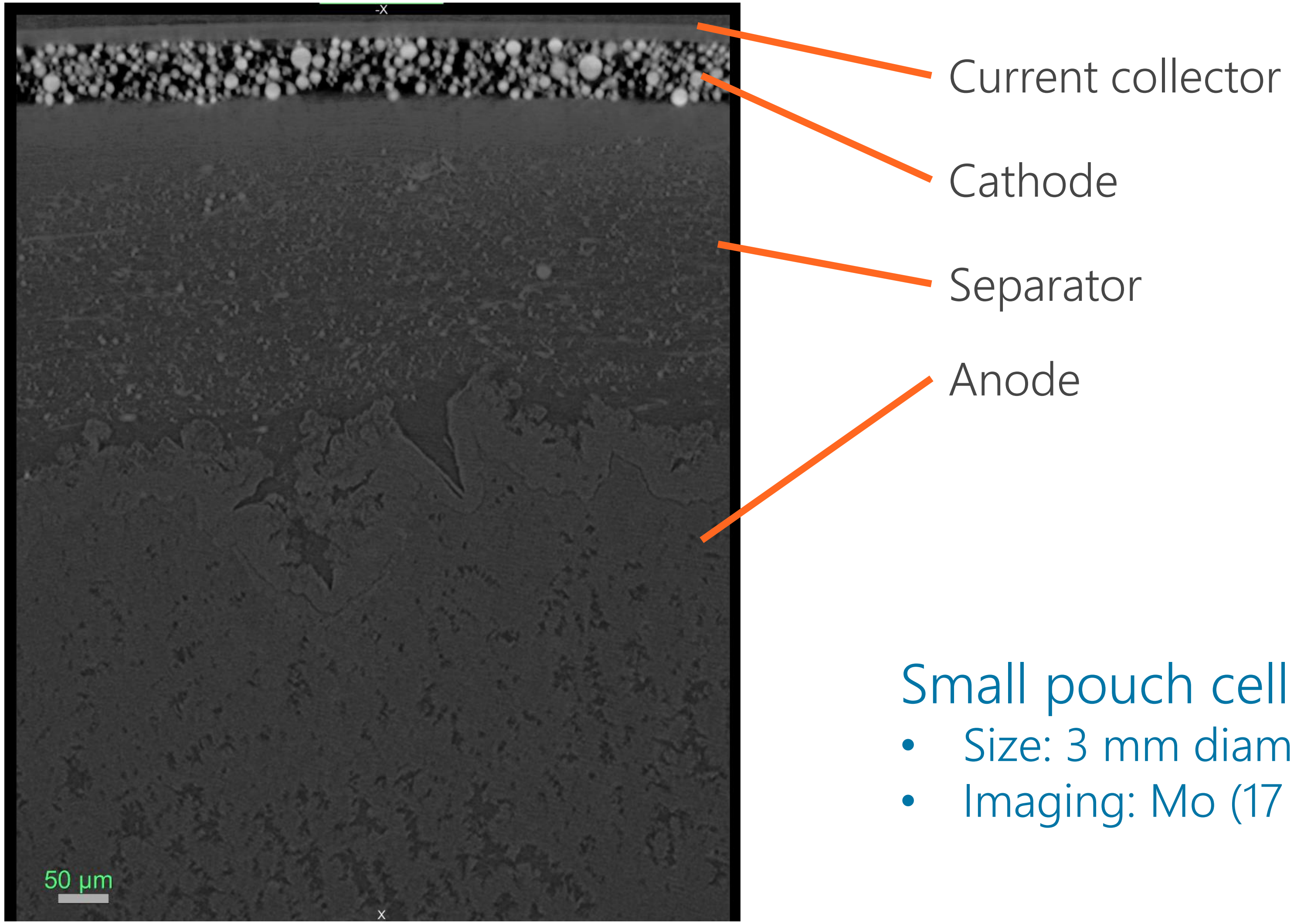
3D segmented data

Li-ion battery from digital camera



Li-ion battery from digital camera





Small pouch cell

- Size: 3 mm diameter
- Imaging: Mo (17 keV), 10X, 0.66 µm voxel

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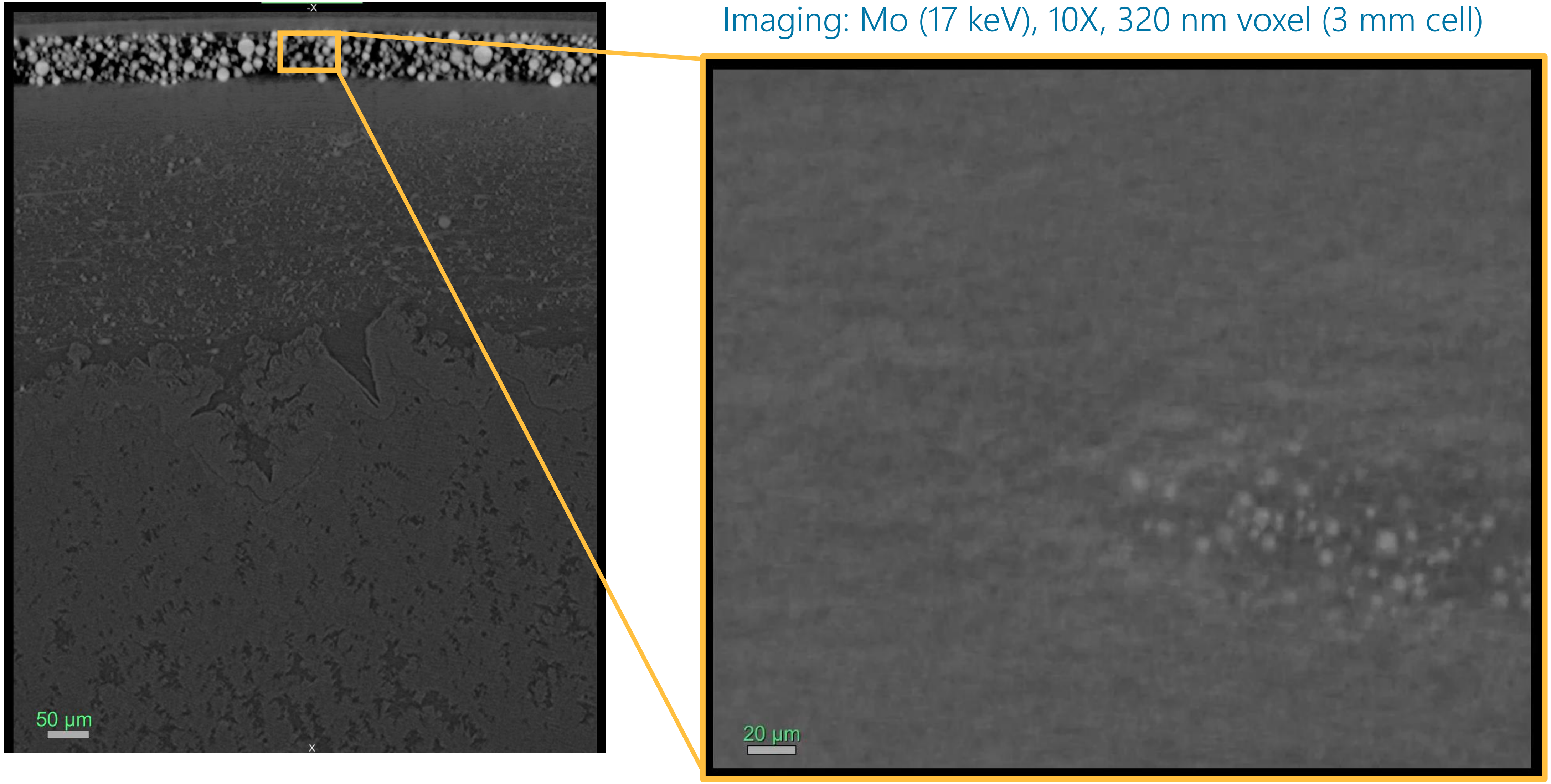
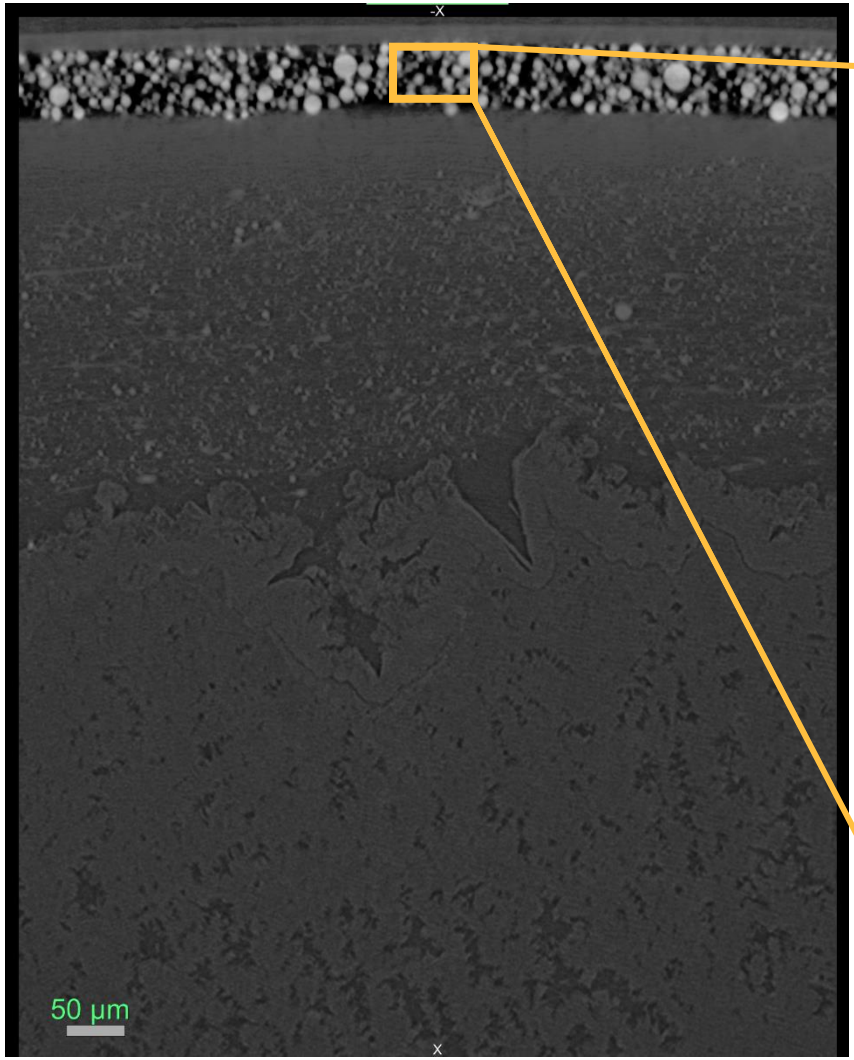
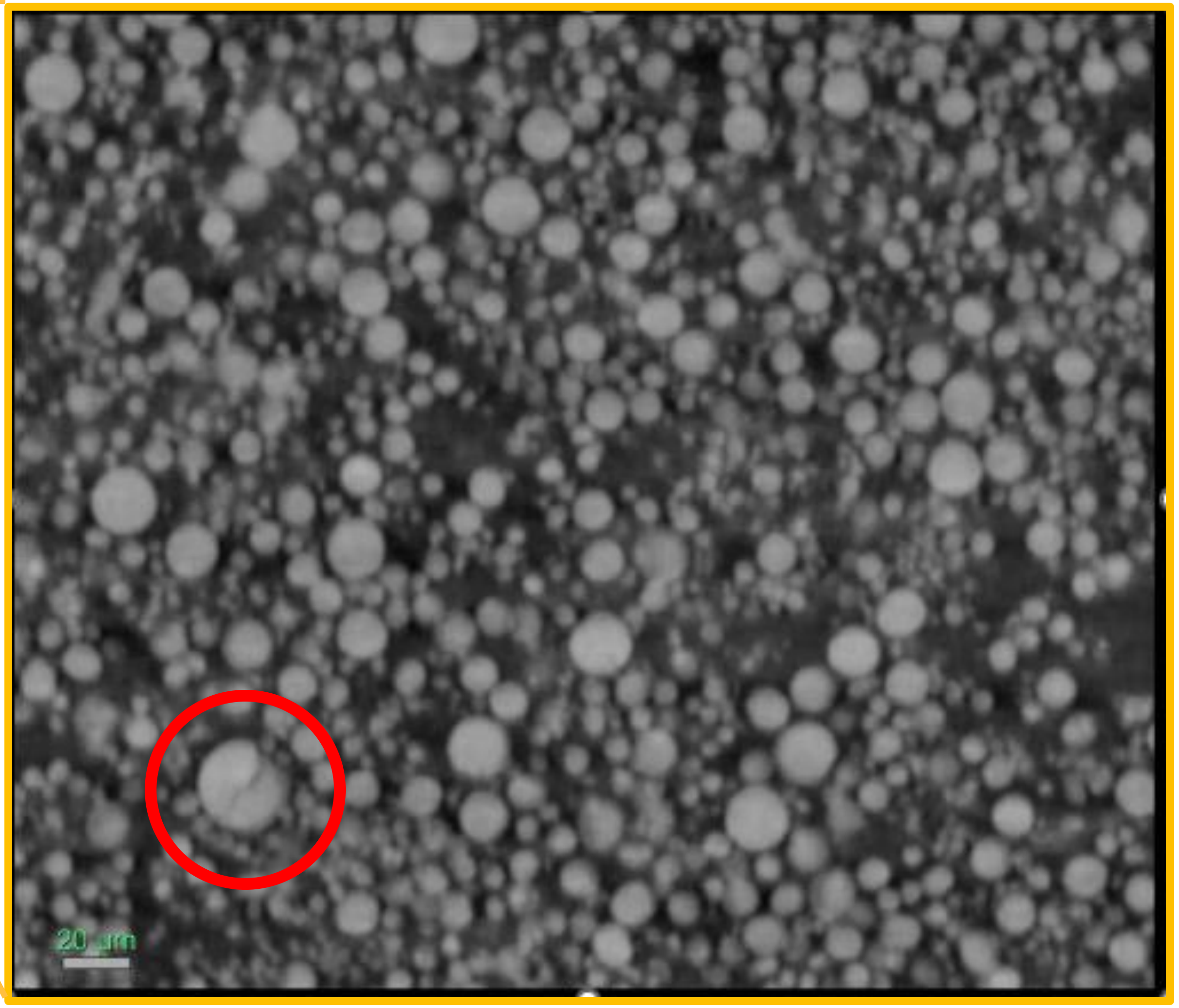
Imaging: Mo (17 keV), 10X, 320 nm voxel (3 mm cell)

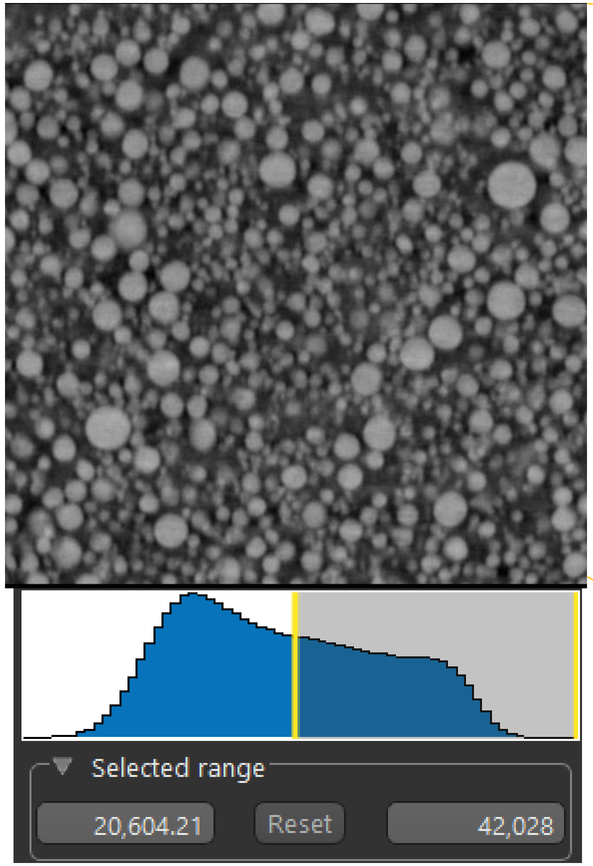


Rigaku

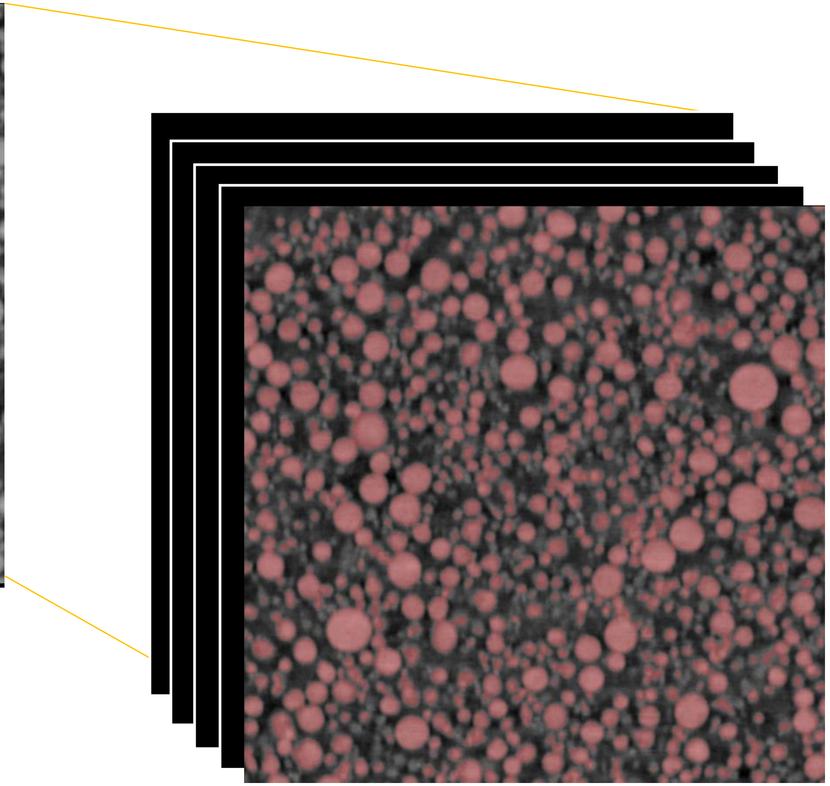
Imaging: Mo (17 keV), 10X, 320 nm voxel (3 mm cell)



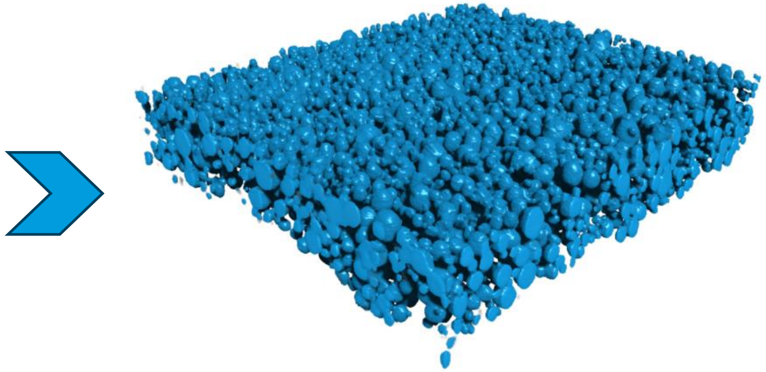




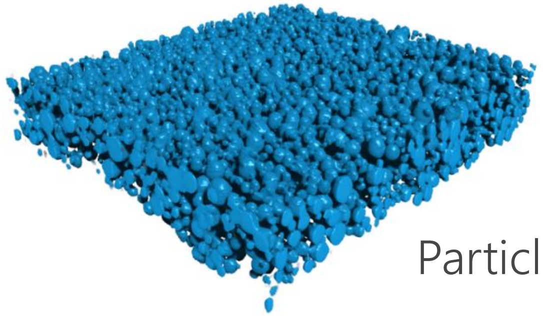
Histogram threshold

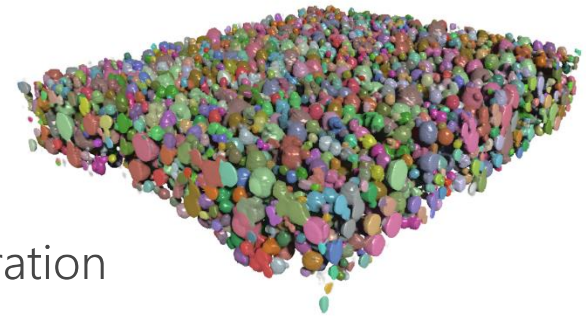


2D cross-sections

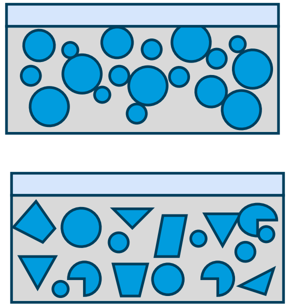


3D segmented Data



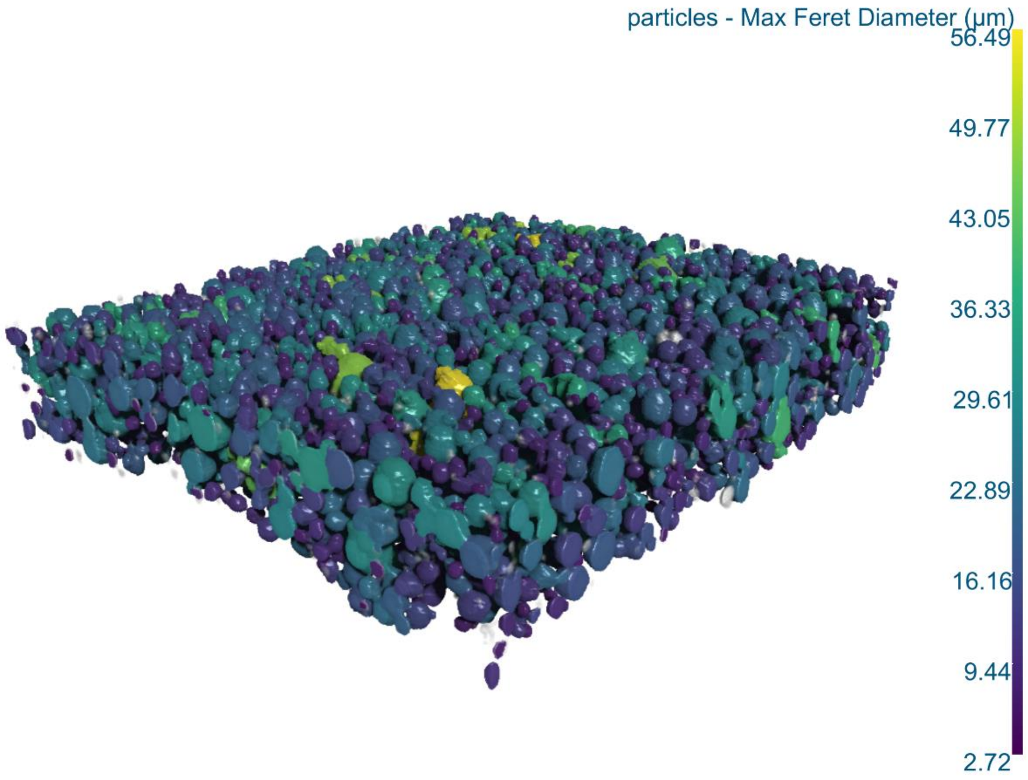


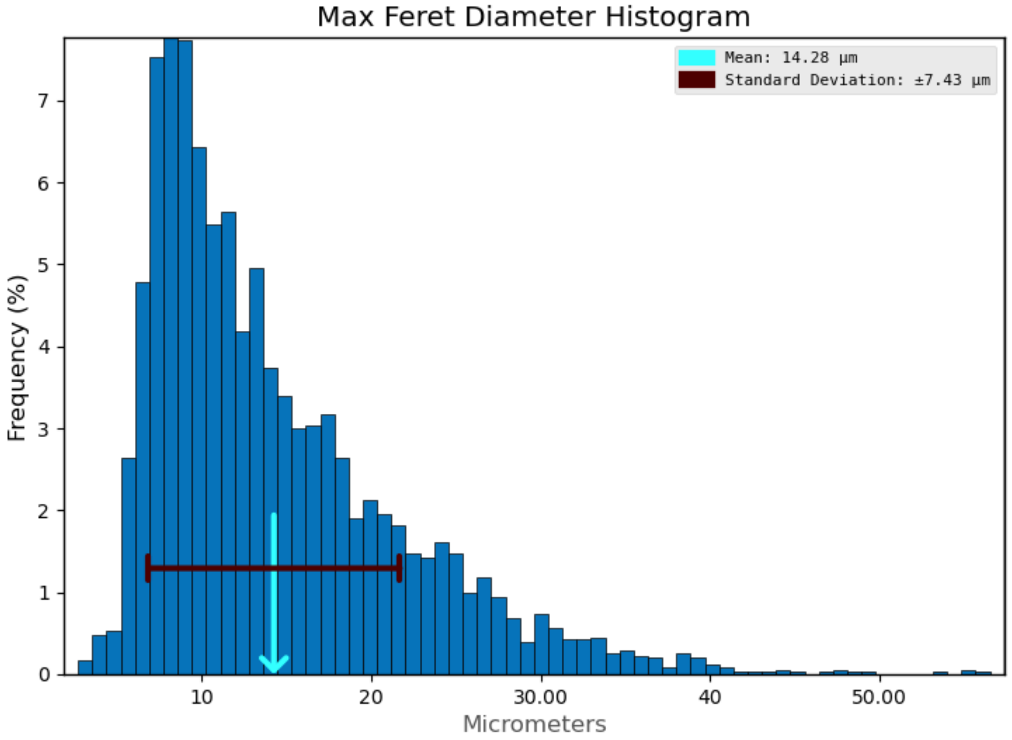
Particle separation
using
watershed transformation

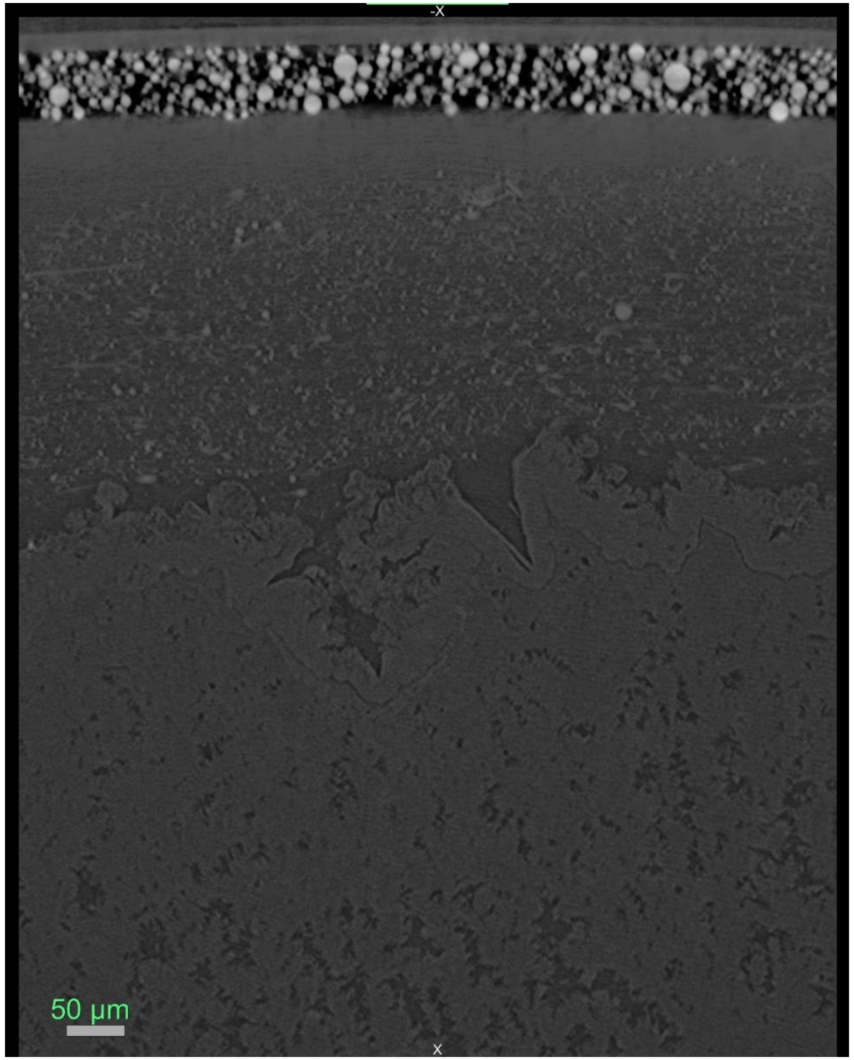


- particle size
- distribution
- porosity
- particle shape
- change over time

Particle size distribution

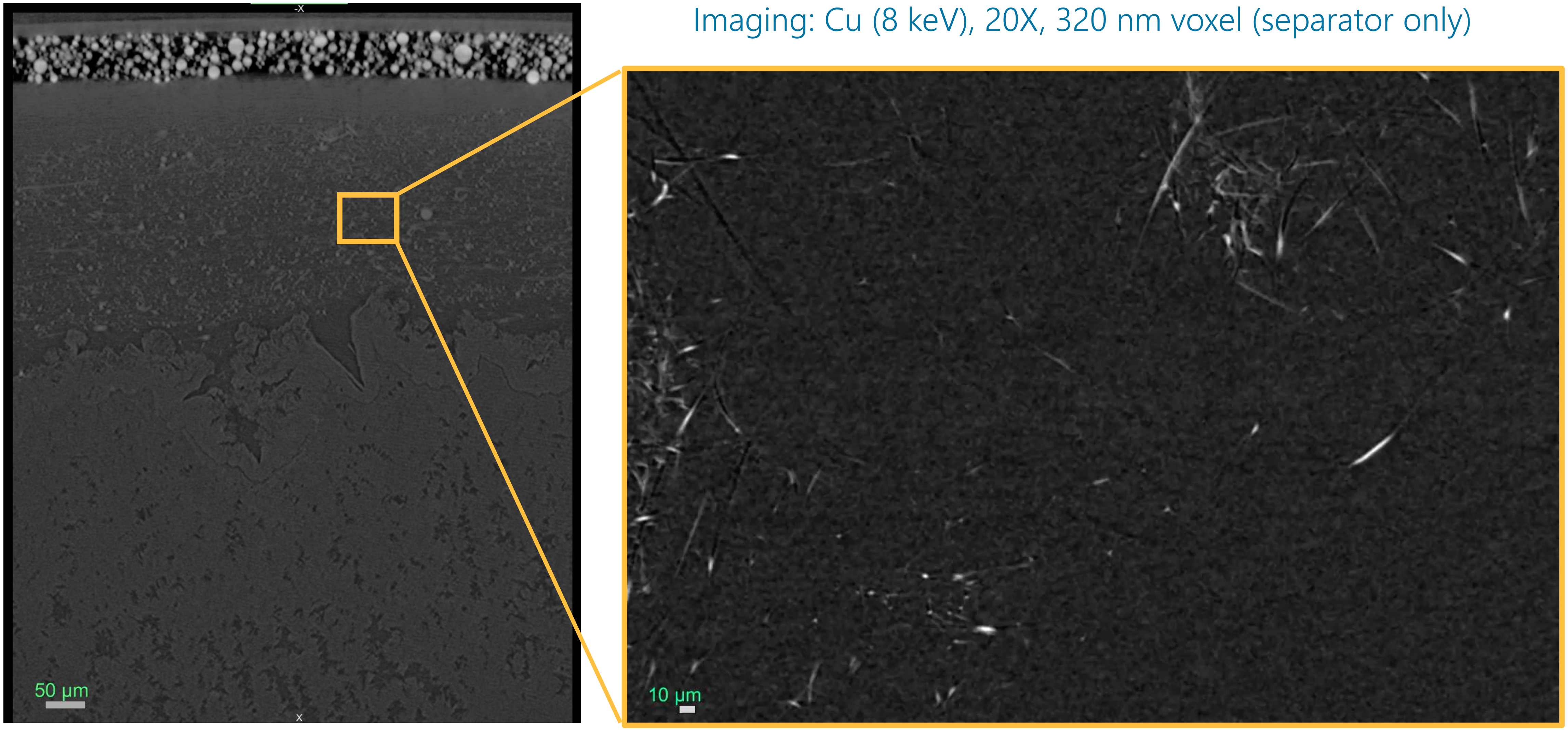


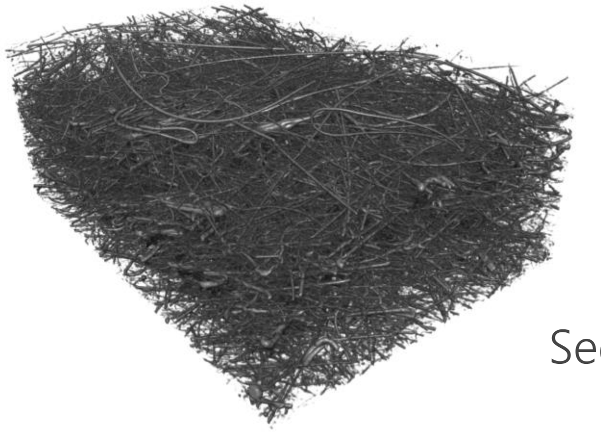



-X
50 µm
X

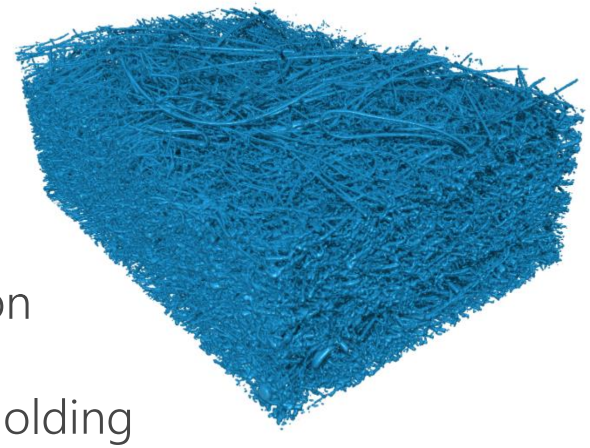
Rigaku

Imaging: Cu (8 keV), 20X, 320 nm voxel (separator only)

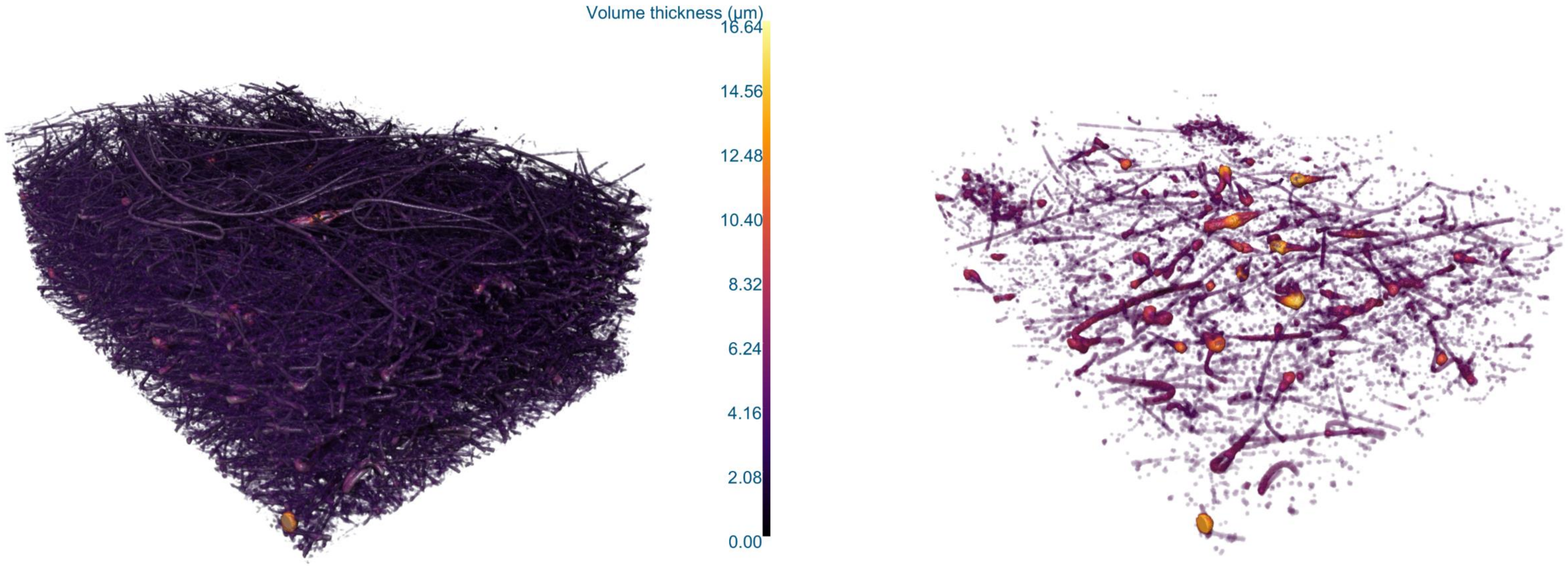


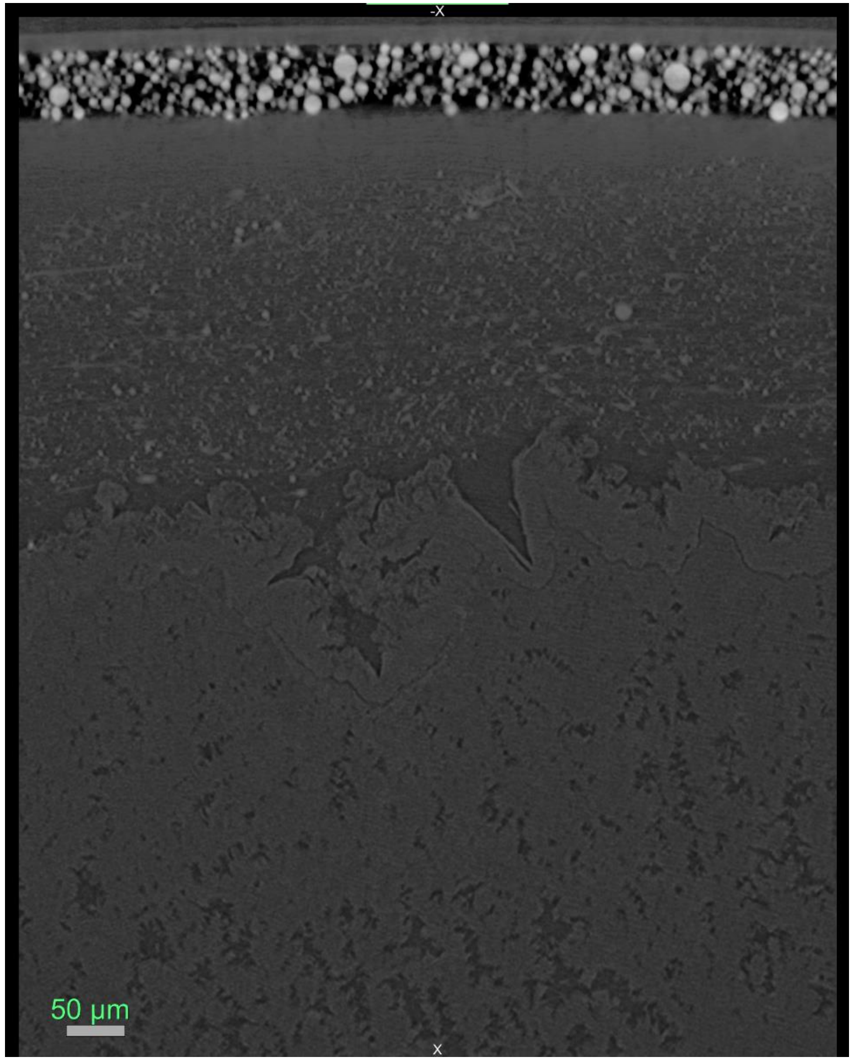


Segmentation
using
histogram thresholding

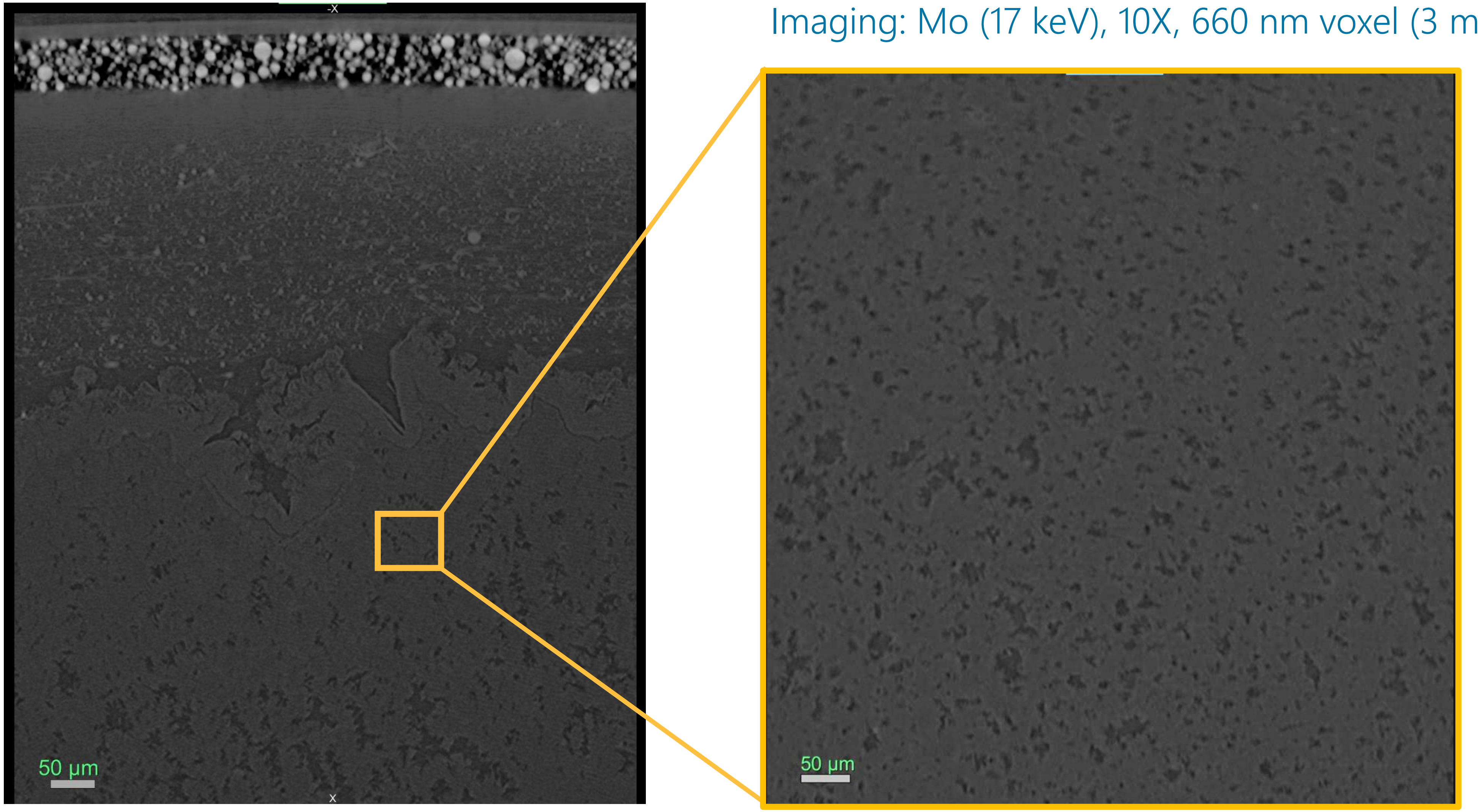


Separator properties:
- Porosity
- Thickness
- Permeability
- Distribution


Volume thickness (µm)
16.64
14.56
12.48
10.40
8.32
6.24
4.16
2.08
0.00


-X
50 µm
X

Imaging: Mo (17 keV), 10X, 660 nm voxel (3 mm cell)



ACS APPLIED MATERIALS & INTERFACES

www.acsami.org Research Article

Visualization and Control of Chemically Induced Crack Formation in All-Solid-State Lithium-Metal Batteries with Sulfide Electrolyte

Misae Otoyama, Motoshi Suyama, Chie Hotehama, Hiroe Kowada, Yoshihiro Takeda, Koichiro Ito, Atsushi Sakuda, Masahiro Tatsumisago, and Akitoshi Hayashi*

Cite This: *ACS Appl. Mater. Interfaces* 2021, 13, 5000−5007

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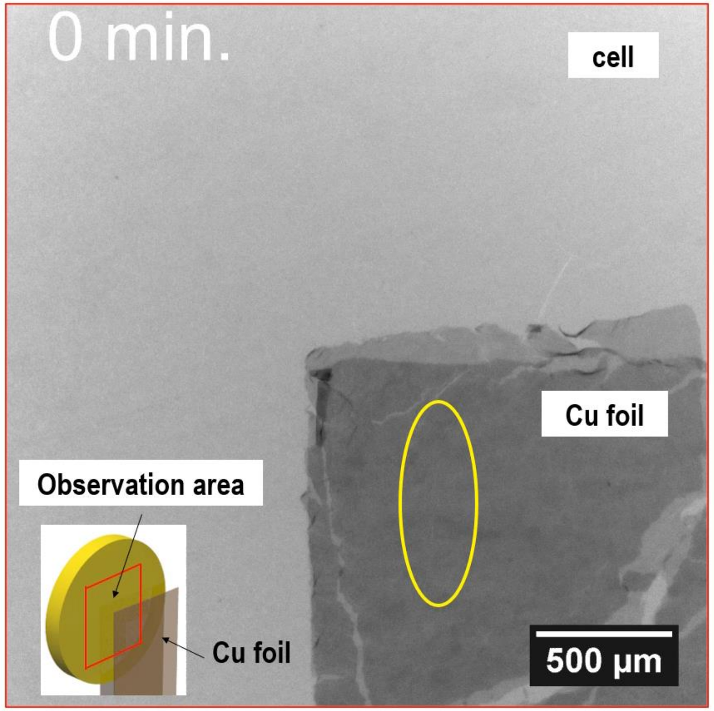
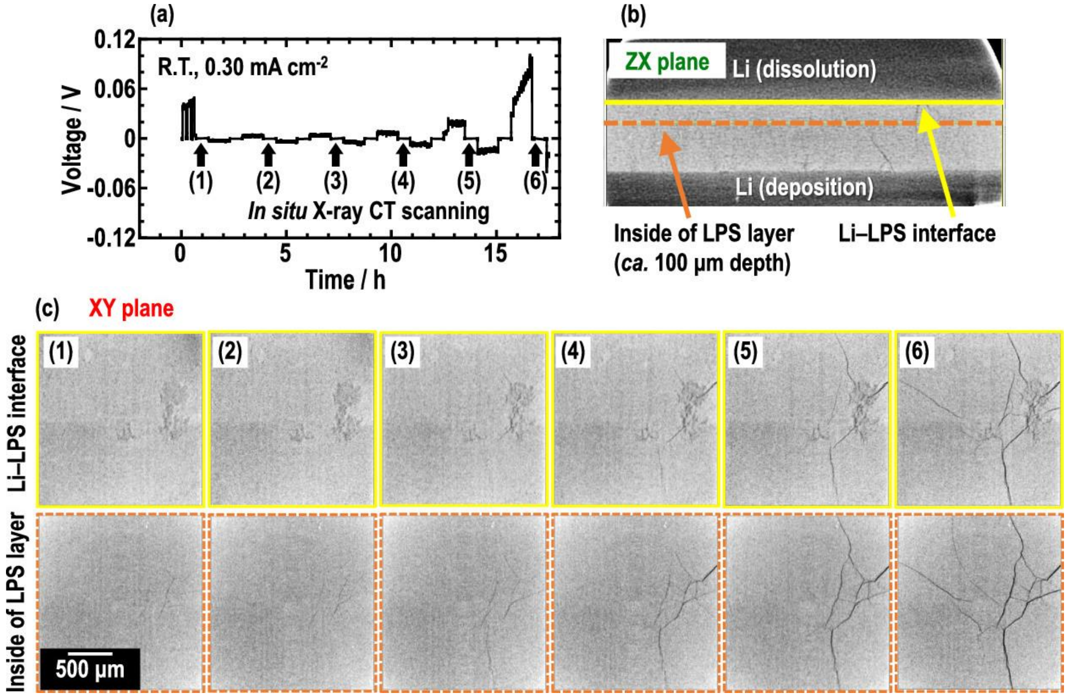
X-ray projection images during the galvanostatic test (without rotating)





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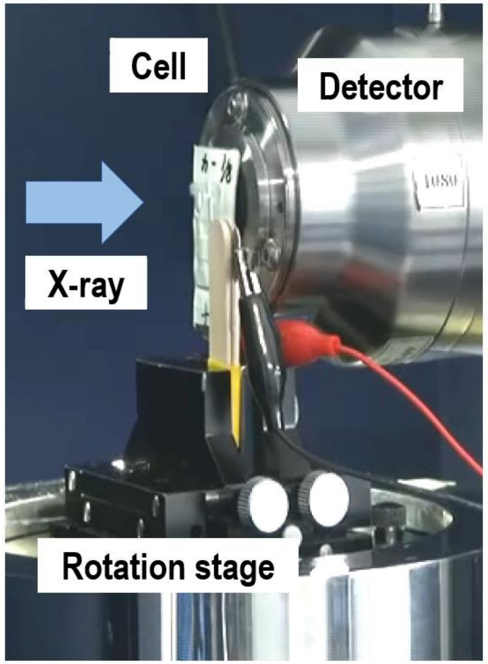
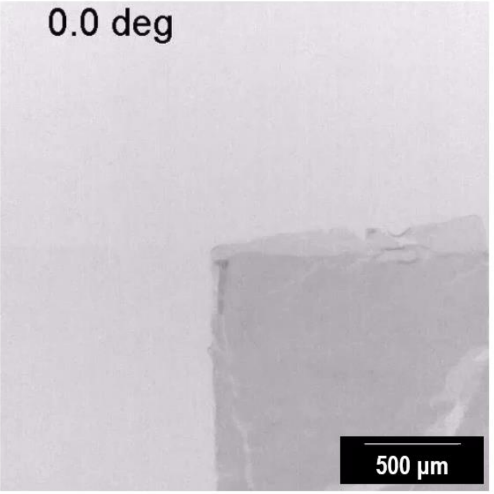
Imaging: Mo (17 keV), 5X, 2.5 μm voxel

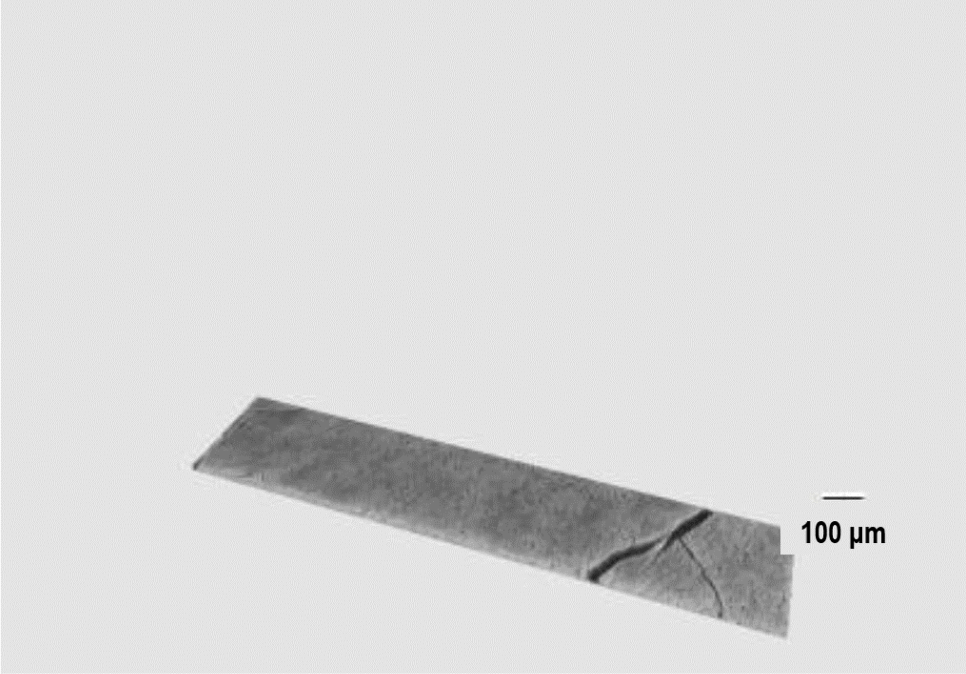
X-ray CT measurement



X-ray projection image



3D rendered movie of the cell after short-circuiting



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We discussed:

- What is X-ray CT?
- What are the considerations when using X-ray CT for battery research?
- What information can we extract from CT data about batteries?
- Battery analysis examples

Questions & Answers

We'll follow up with your questions.

Recording will be available tomorrow.

Register for the next workshop.

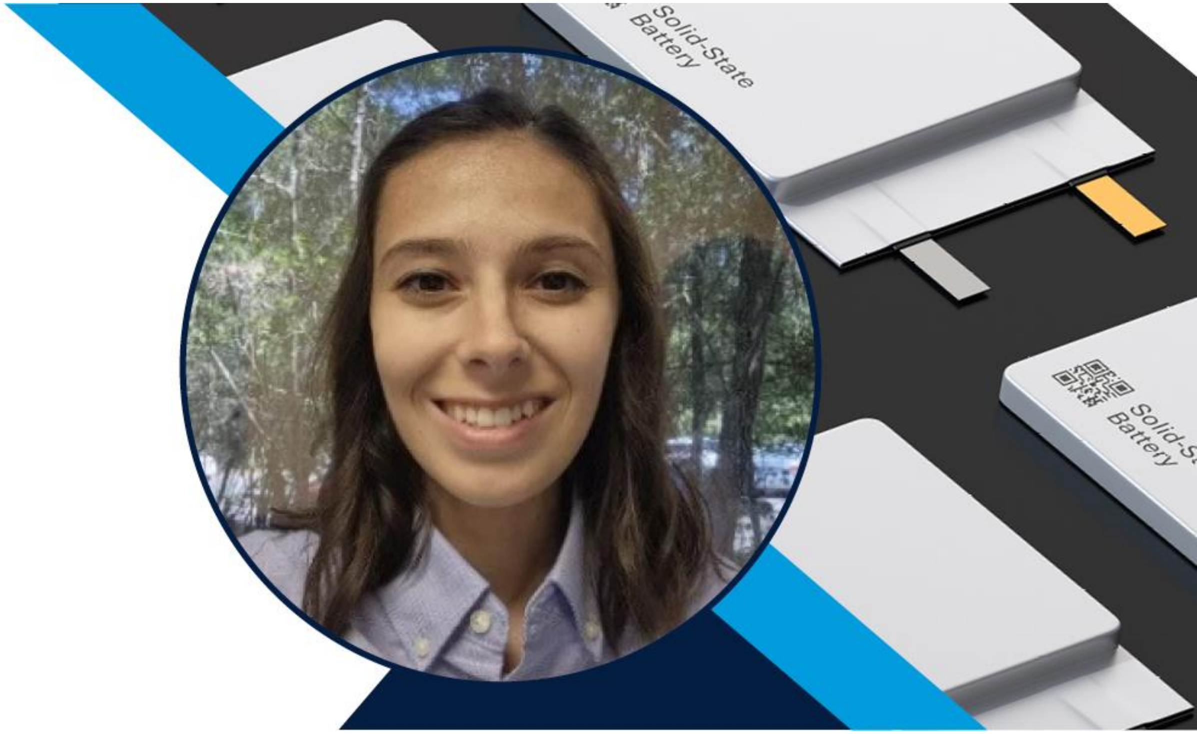
Rigaku

Beneath The Surface: X-Ray Analyses of Battery Materials and Structures

A Battery Webinar Series by Rigaku

Non-destructive Elemental Analysis of Batteries Using XRF

October 16, 2024 at 1:00 PM



Register at rigaku.com

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THANK YOU