The Electrochemical Innovation Lab X-ray Suite: from macro- to nano-CT

Dr. Francesco Iacoviello, Toby Neville
& the Electrochemical Innovation Lab

f.iacoviello@ucl.ac.uk

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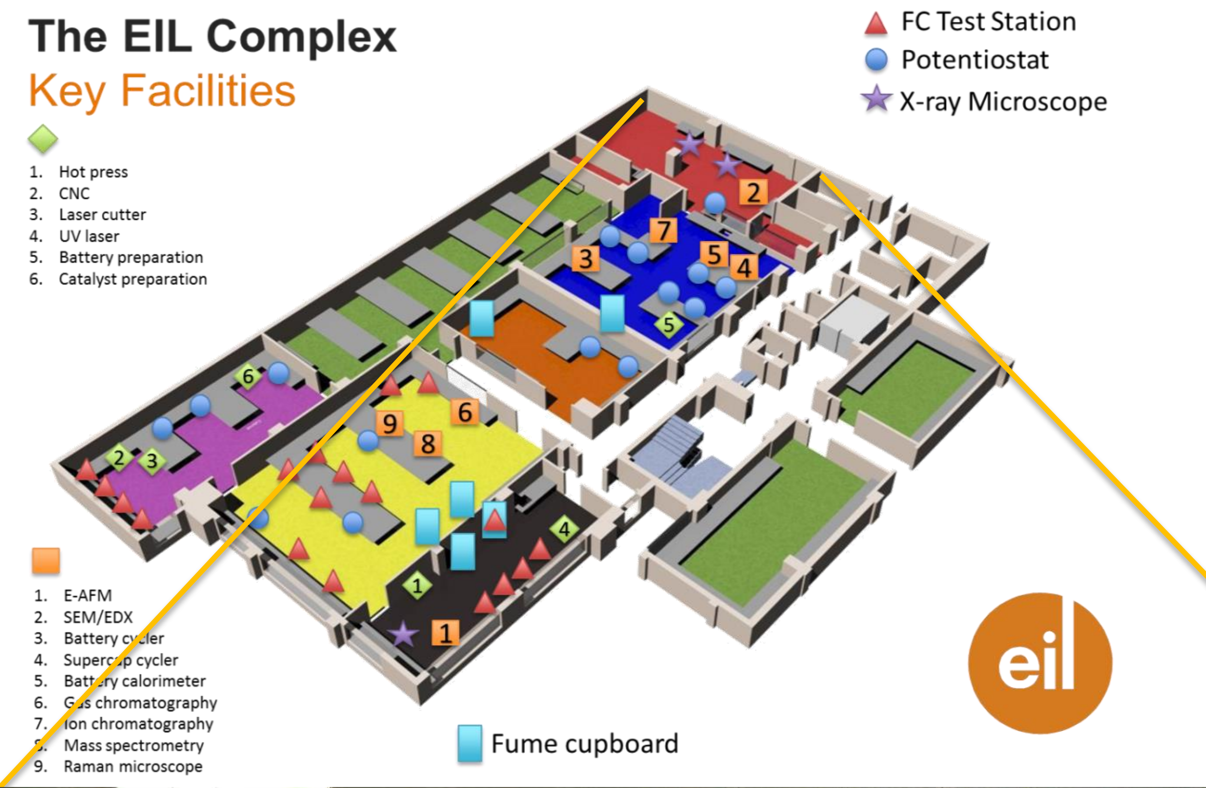
The EIL Complex

Key Facilities



◆
1. Hot press
2. CNC
3. Laser cutter
4. UV laser
5. Battery preparation
6. Catalyst preparation

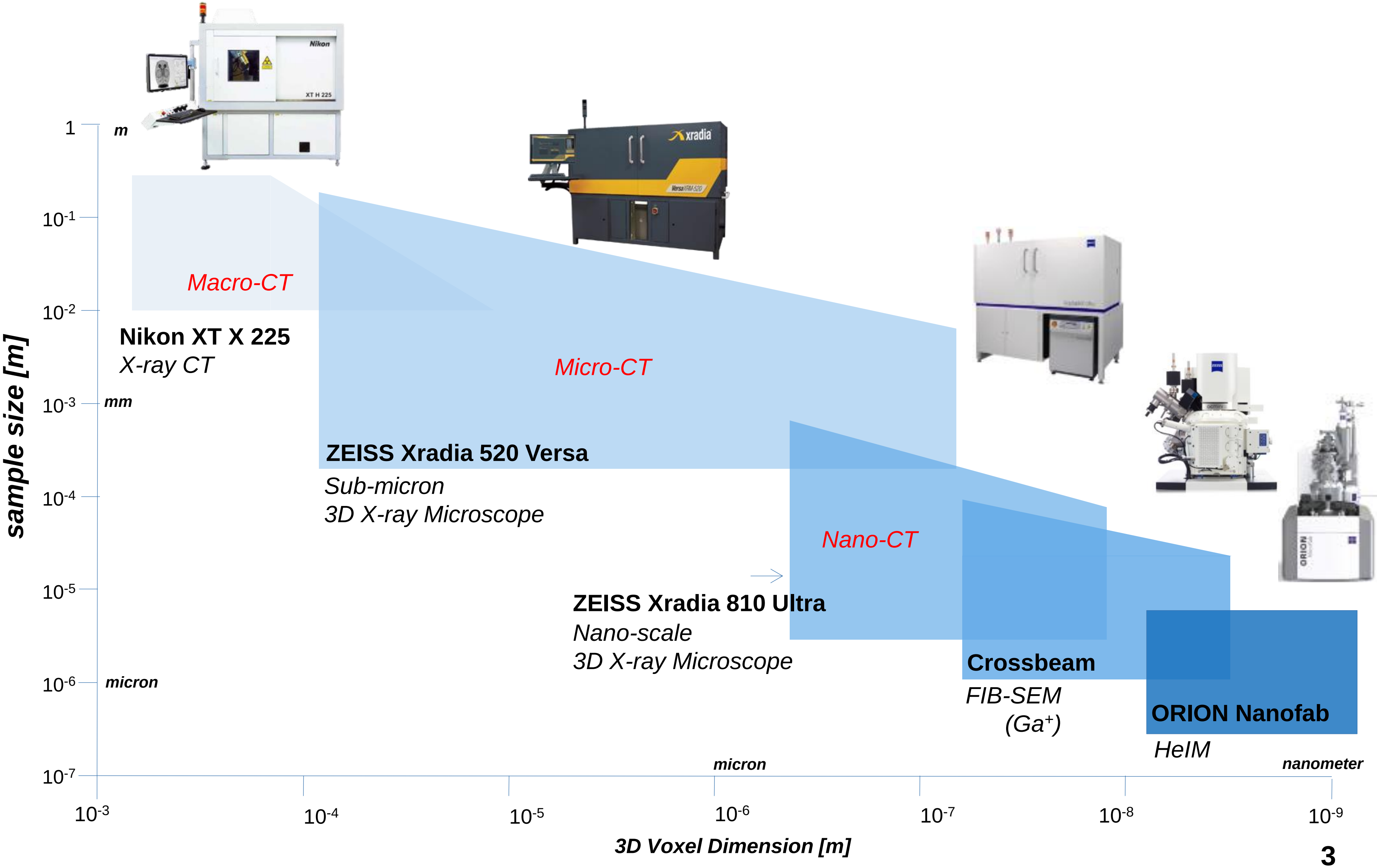
🟧
1. E-AFM
2. SEM/EDX
3. Battery cycler
4. Supercap cycler
5. Battery calorimeter
6. Gas chromatography
7. Ion chromatography
8. Mass spectrometry
9. Raman microscope

Nikon 225 XT
Resolution 3 micron+, 225kV source

ZEISS Xradia 520 Versa
Resolution 130nm - 10s μm

ZEISS Xradia 810 Ultra
Resolution 16 - 260nm

The EIL microscopy Suite



Nikon XT X 225
X-ray CT

ZEISS Xradia 520 Versa
Sub-micron
3D X-ray Microscope

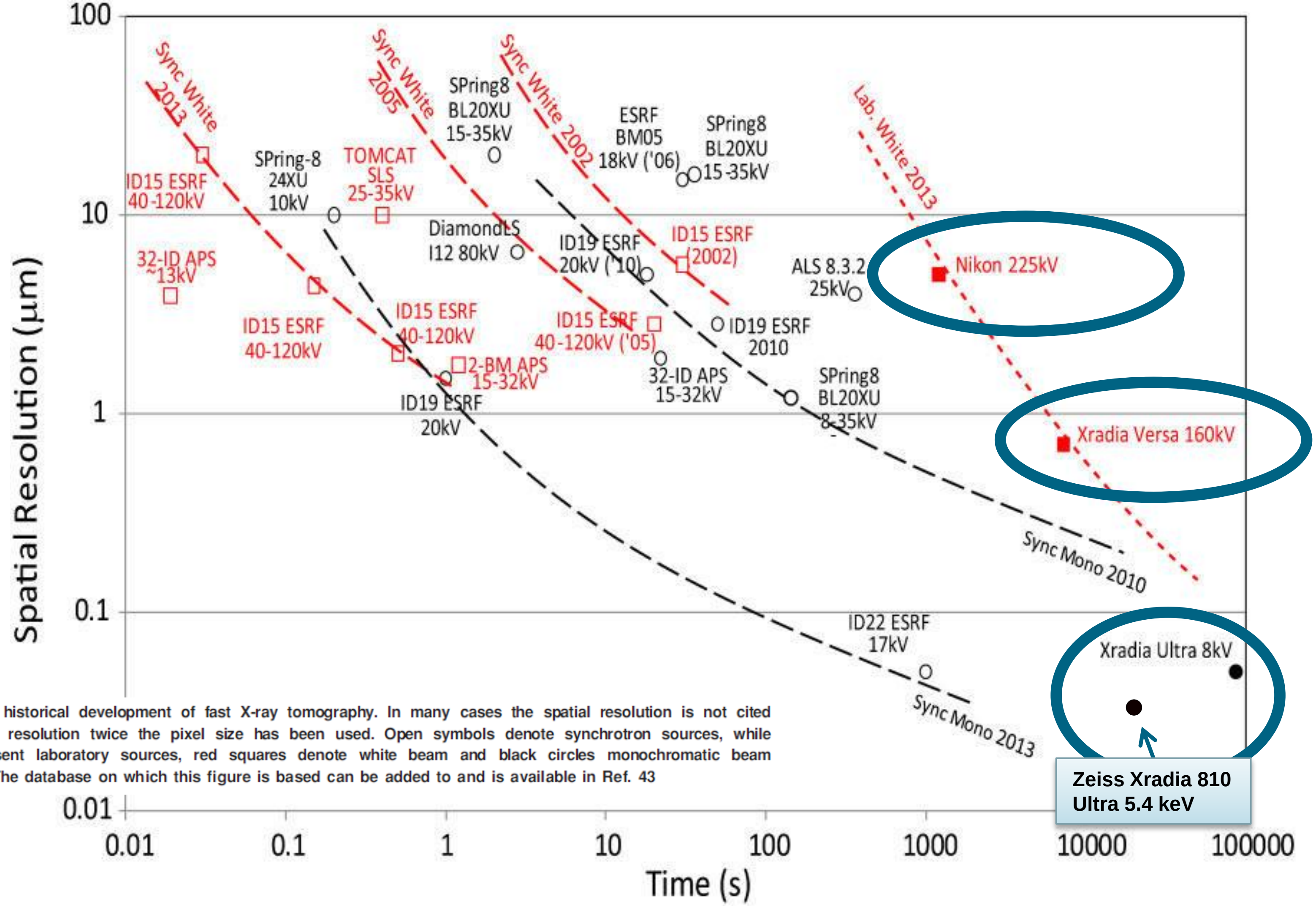
ZEISS Xradia 810 Ultra
Nano-scale
3D X-ray Microscope

Crossbeam
FIB-SEM
(Ga^+)

ORION Nanofab
HeIM

Quantitative X-ray Tomography

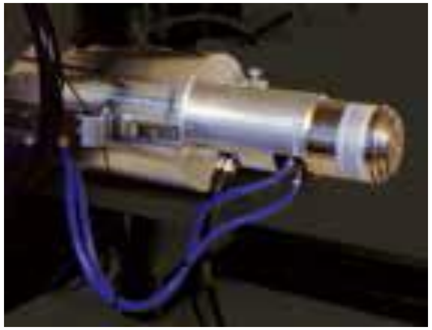
Maire & Withers, *Int. Materials Review* (2014)



1 Plot showing the historical development of fast X-ray tomography. In many cases the spatial resolution is not cited and an estimated resolution twice the pixel size has been used. Open symbols denote synchrotron sources, while filled ones represent laboratory sources, red squares denote white beam and black circles monochromatic beam scanners.[26,31–42] The database on which this figure is based can be added to and is available in Ref. 43

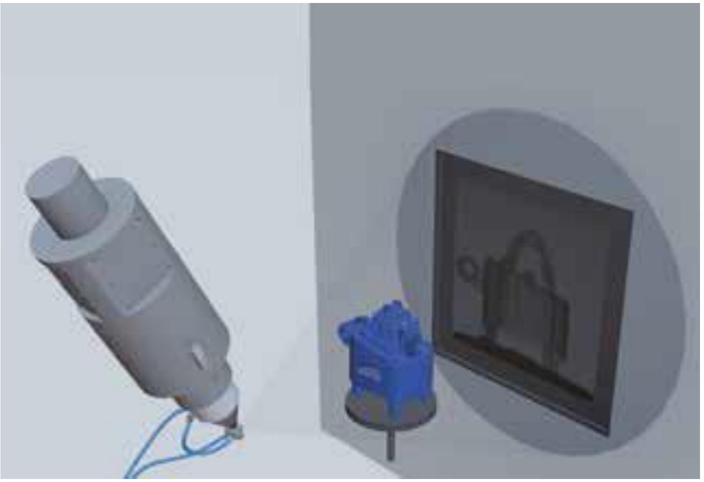
Zeiss Xradia 810 Ultra 5.4 keV

Nikon XT H 225



180 kV transmission target
minimum pixel size of 1 µm

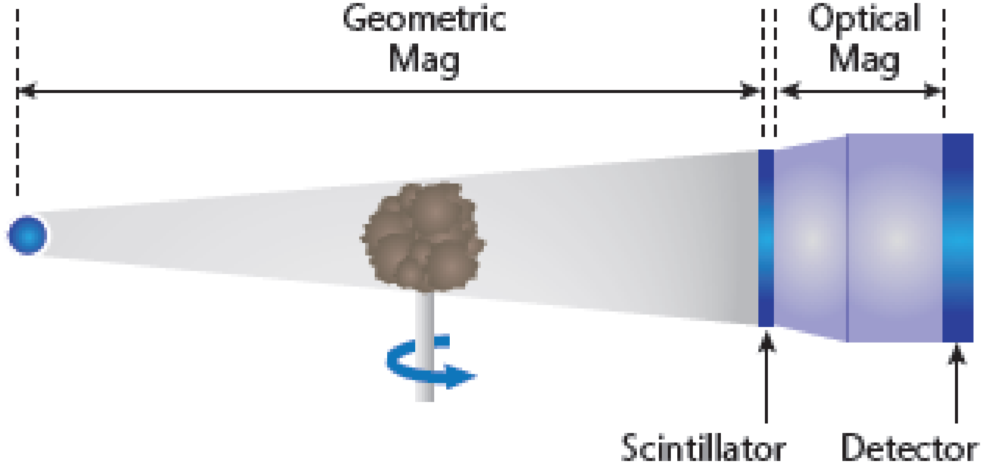
225 kV reflection target
minimum pixel size of 3 µm



PerkinElmer Detector
PerkinElmer 1620

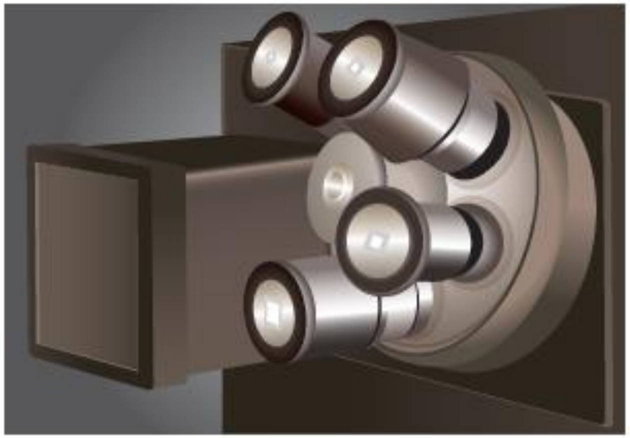
- 16-bit,
- 2000 x 2000 px,
- 200 µm per px
- Frame rate binning 1 3.75 fps

ZEISS Xradia 520 Versa

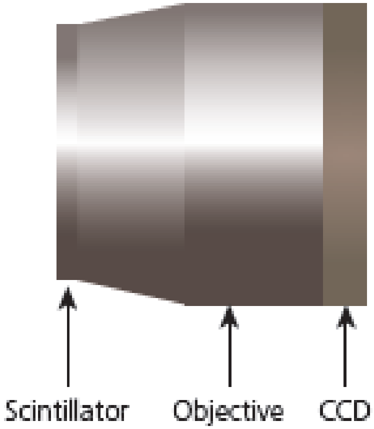


Filters carousel

Objective lenses



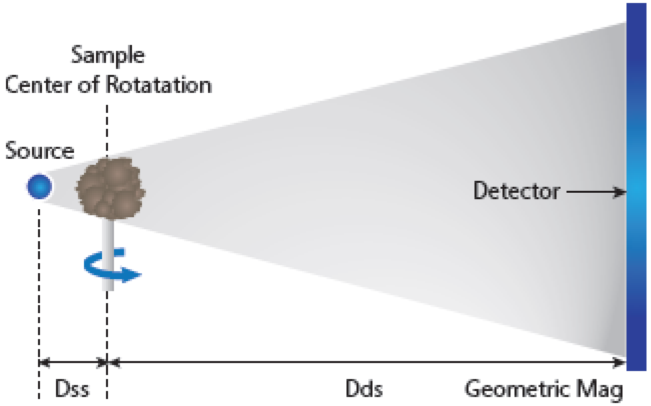
2048 x 2048 px



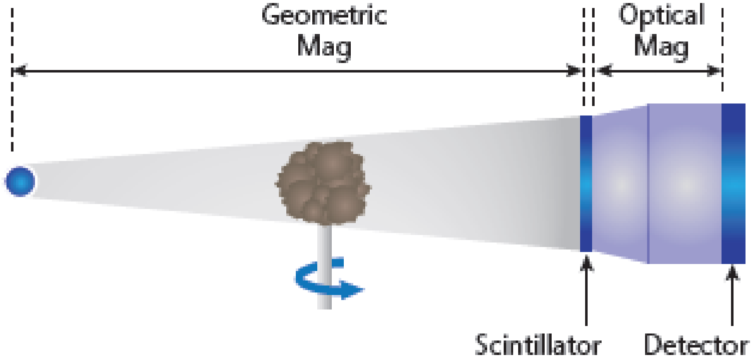
ZEISS Xradia 520 Versa

Source Spot size
2.0 um FWHM @ 30 kV
2.9 um FWHM @ 160 kV

Imaging	
Spatial Resolution	0.7 um
X-ray source tube voltage range	30 -160 kV
Max output	10 W
Objective lenses	0.4X, 10X, 20X, 40X
Stage travel rotation	360°

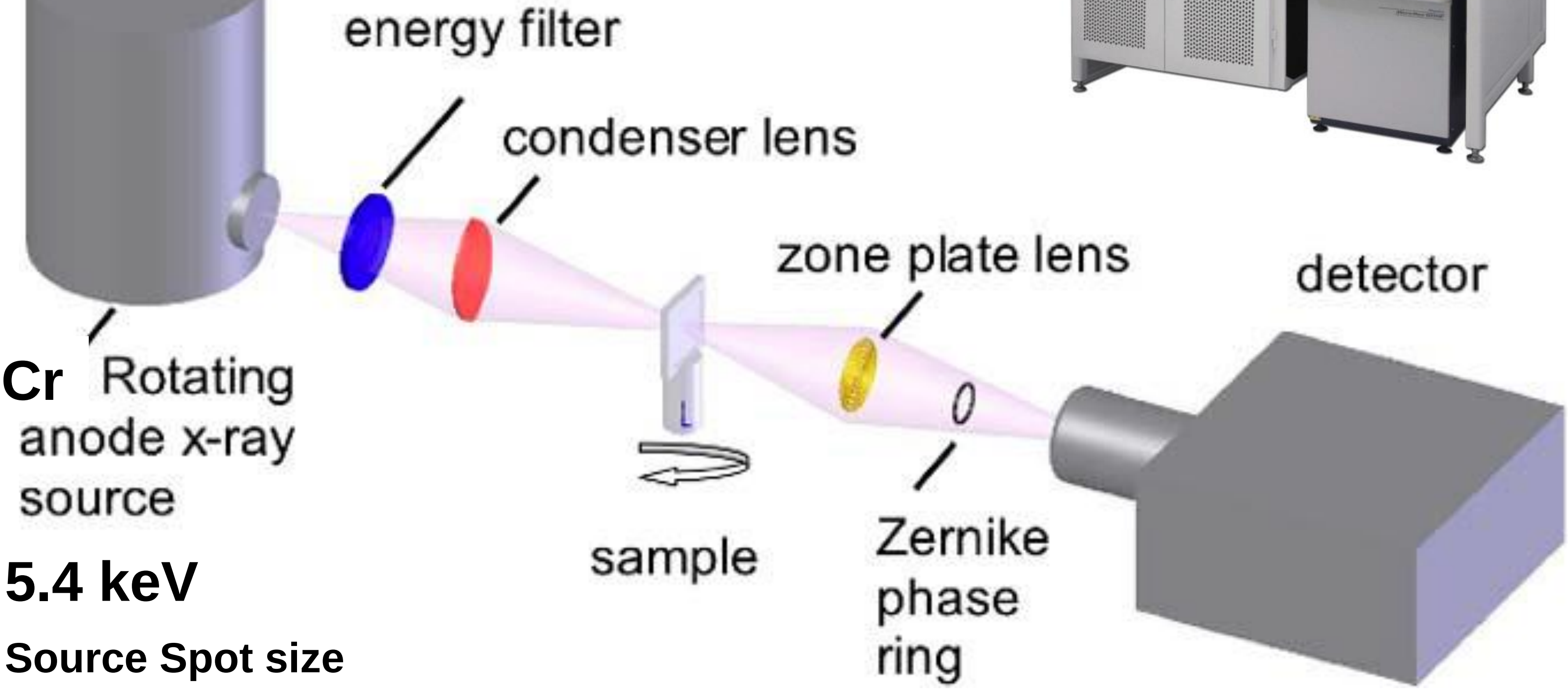


Nikon XT H 225



ZEISS Xradia 520 Versa

ZEISS Xradia 810 Ultra



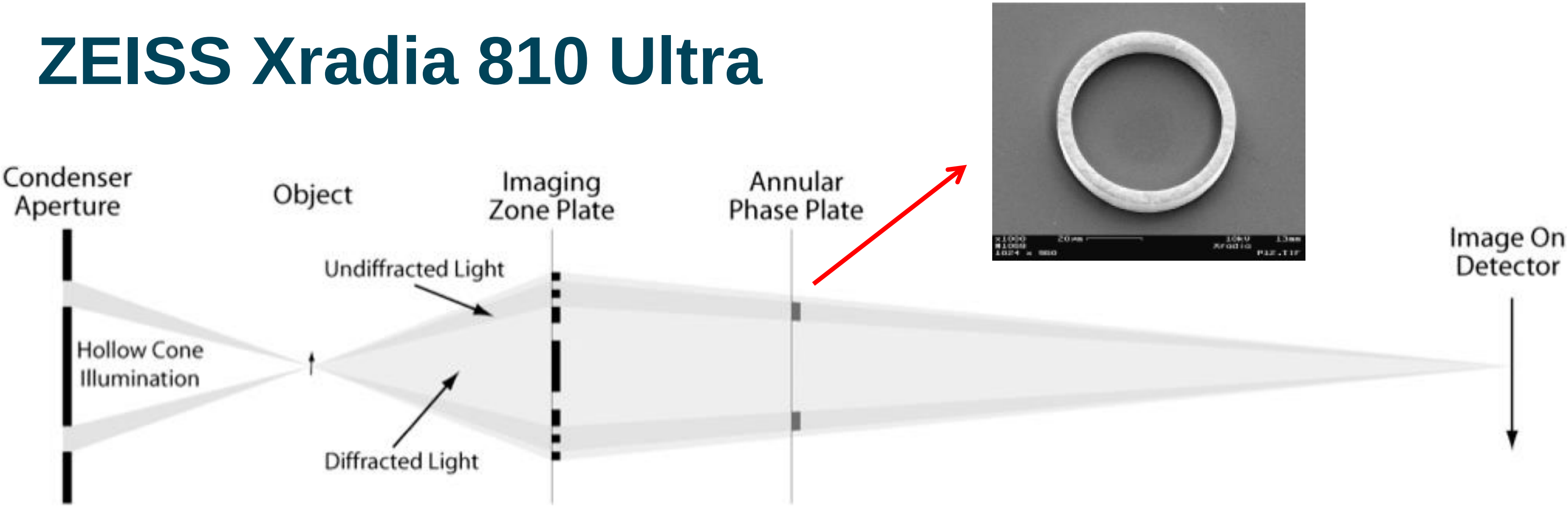
5.4 keV

Source Spot size

75 um ± 15% FWHM @ 35 kV

1024 x 1024 px

ZEISS Xradia 810 Ultra



- The condenser lens is a reflective ellipsoidal capillary with ~90% reflection efficiency, which refocuses ~75 um X-ray source spot from the rotating anode onto the sample.

- A Fresnel lens zone plate objective lens with **35 nm outermost zone** width and 700 nm zone height magnifies the image onto high resolution X-ray detector.

- The Zernike phase ring is positioned near the back focal plane of the zone plate lens to phase shift the X-rays not scattered by the sample by **$3\pi/2$ (negative phase contrast)**.

	1. Large Field of View mode (LFOV)	2. High-Resolution mode (HRES)
Spatial resolution	150 nm	50 nm
Field of View	65 μm	16 μm
Voxel size	64 nm	16 nm
Magnification	200 x	800 x

X-ray Source	
Source type	Rotating Anode
Target Material	Chromium
X-ray Photon Energy	5.4 keV
Voltage	35 keV
Power	0.9 kW

Sample preparation

- Sample to fit in the field of view (65 um for 810 ULTRA LFOV, 16 um for HRES)
- Manual mounting vs Laser preparation
- Sample asymmetry (e.g. High aspect ratio)

Laser Sample preparation

- Robust, cost-effective, faster than FIB-lift out.
- Key to accessing small enough sample thicknesses to give higher transmissions and importantly to give good signal-to-noise ratios → 30-40 µm diameter
- FIB technique: Used only when necessary → 10-30 µm diameter

Examples

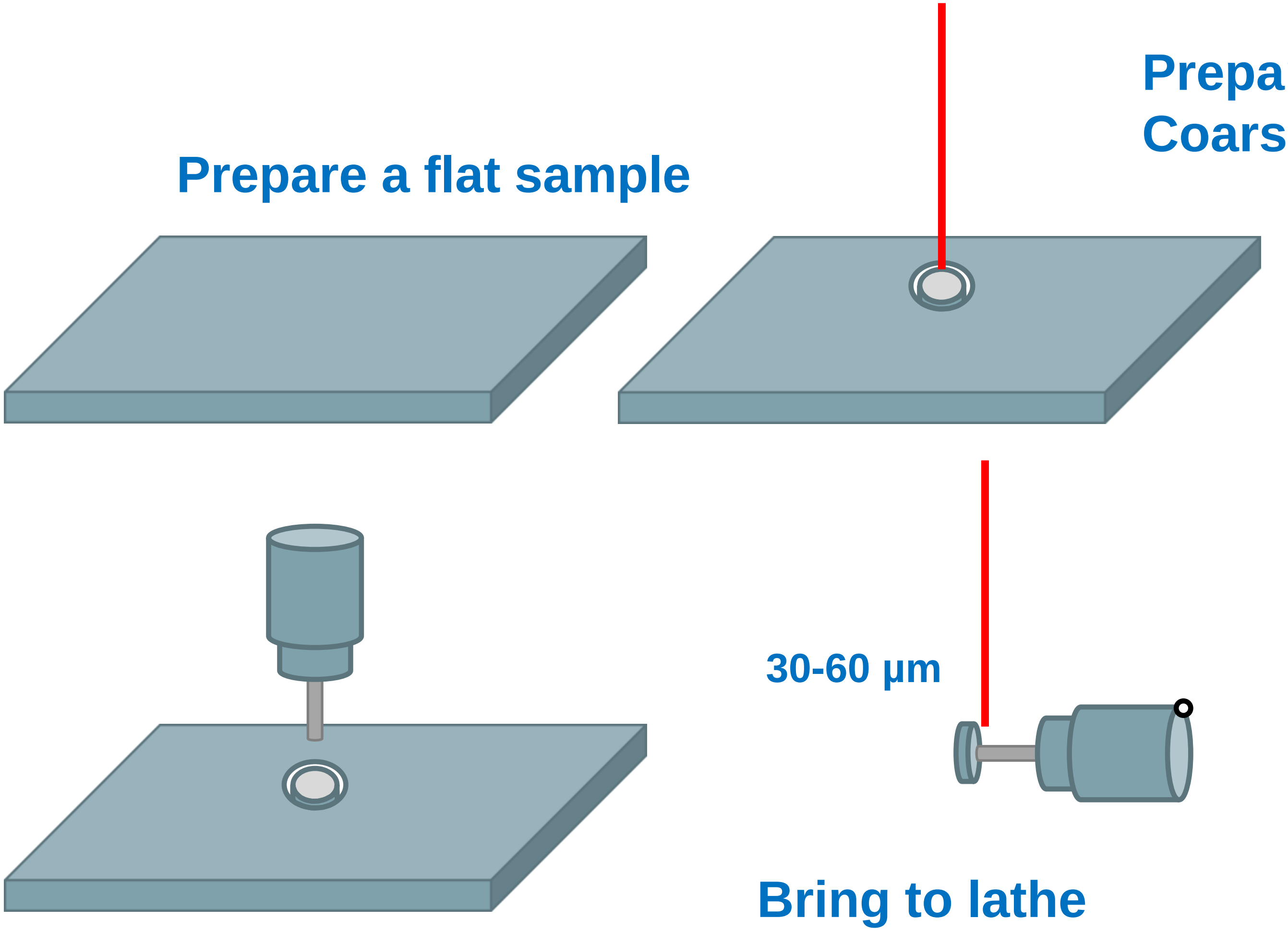
SOFC → maximising FOV we have more chance of being representative for a given microstructure

LIB → First example of pillars of this type → gives access to directional information as full thickness from separator to current collector is captured

Shale → multi-scale approach. Altering sample size allows identification of extra phase. Fractures and bedding structure evident at micro-CT level but clay mineral information becomes observable at nano-CT level
(**Backeberg, Iacoviello et al.** (2017) *Scientific Reports*)

Bailey *et al.* *(2017)* – *Journal of Microscopy*

Sample Laser preparation - Oxford Laser



Prepare a flat sample

Prepare
Coarse pillar

Glue pillar to dowel

30-60 µm

Bring to lathe
Reduce pillar to desired size

Multi-Scale Characterisation

PCCP

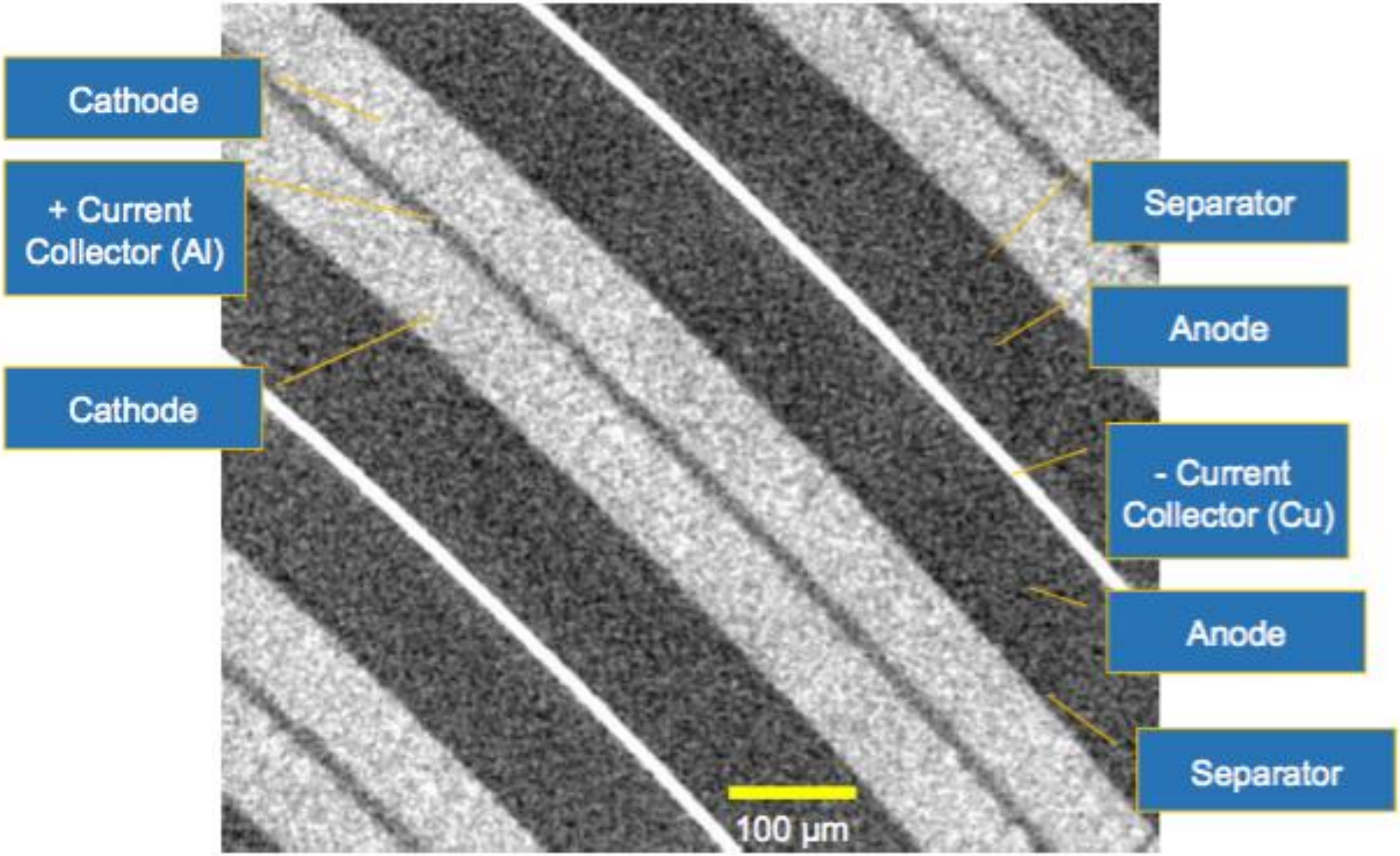
Physical Chemistry Chemical Physics

ROYAL SOCIETY OF CHEMISTRY

PAPER

175 YEARS

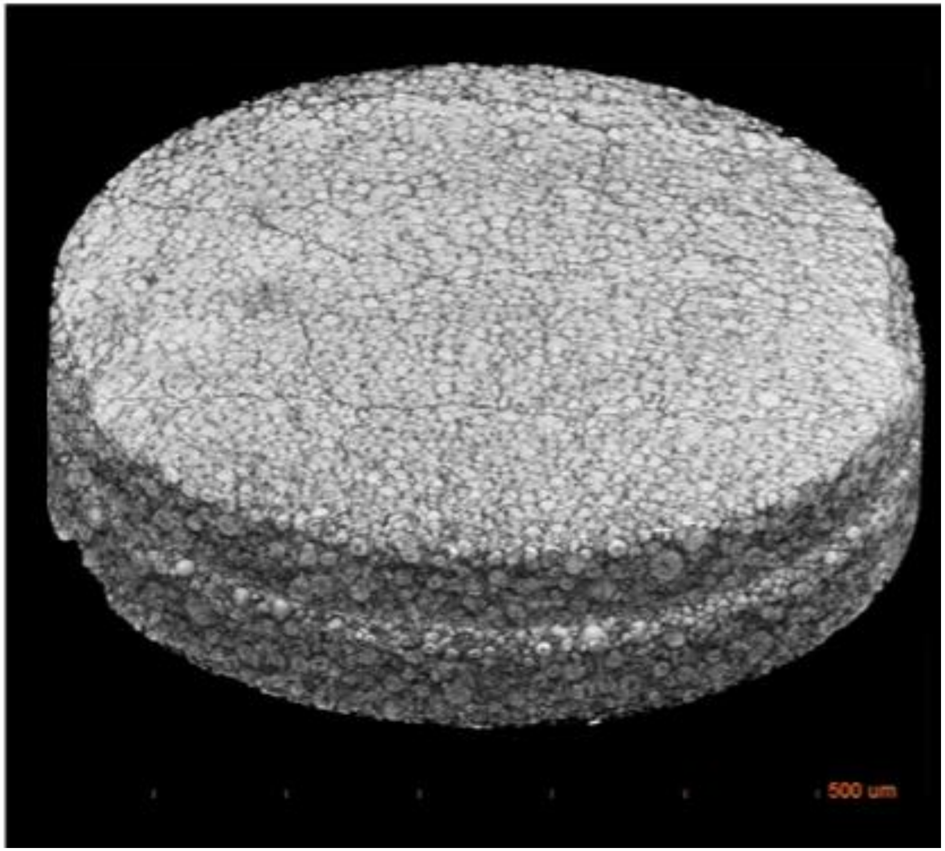
Multi-Scale Imaging of a Panasonic 18650

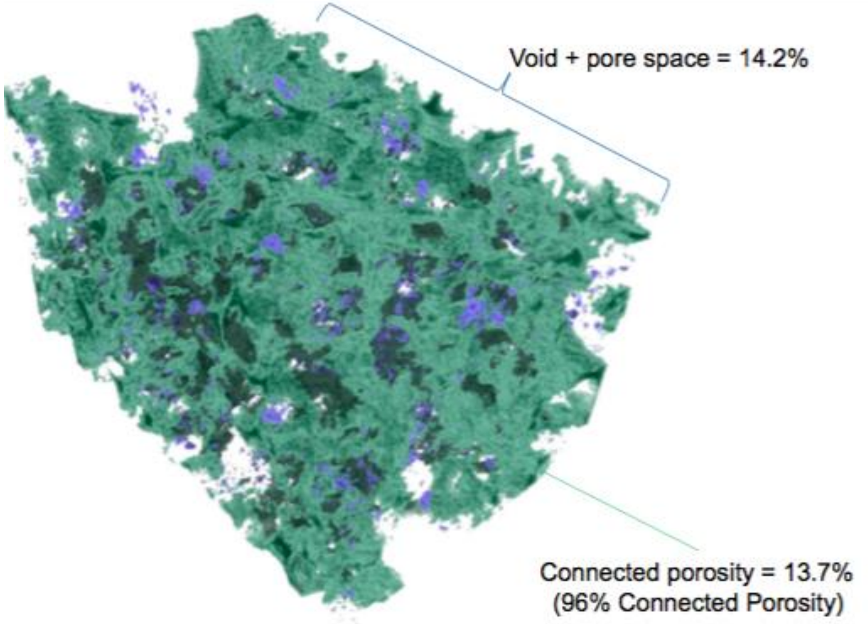


Full Body Scan: Versa 410 22µm voxel

ROI Scan: Versa 410 1.8µm voxel

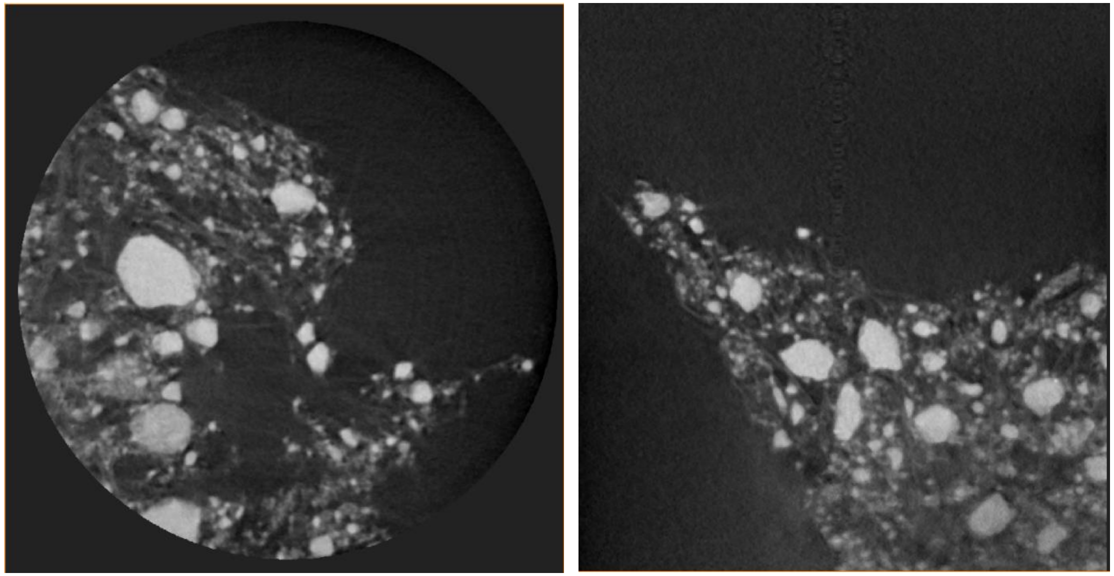
Multi-Scale Imaging of a Panasonic 18650: Depackaged Cathode Material



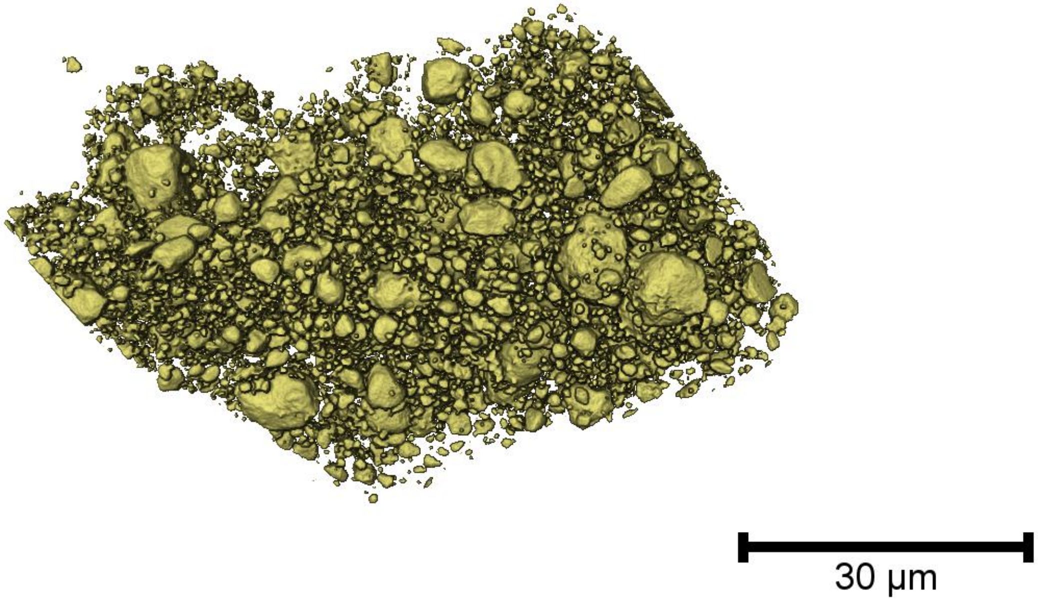


Cathode Scan: 350nm voxel, Versa 520

Cathode ROI Scan:
Ultra 810 130nm voxel



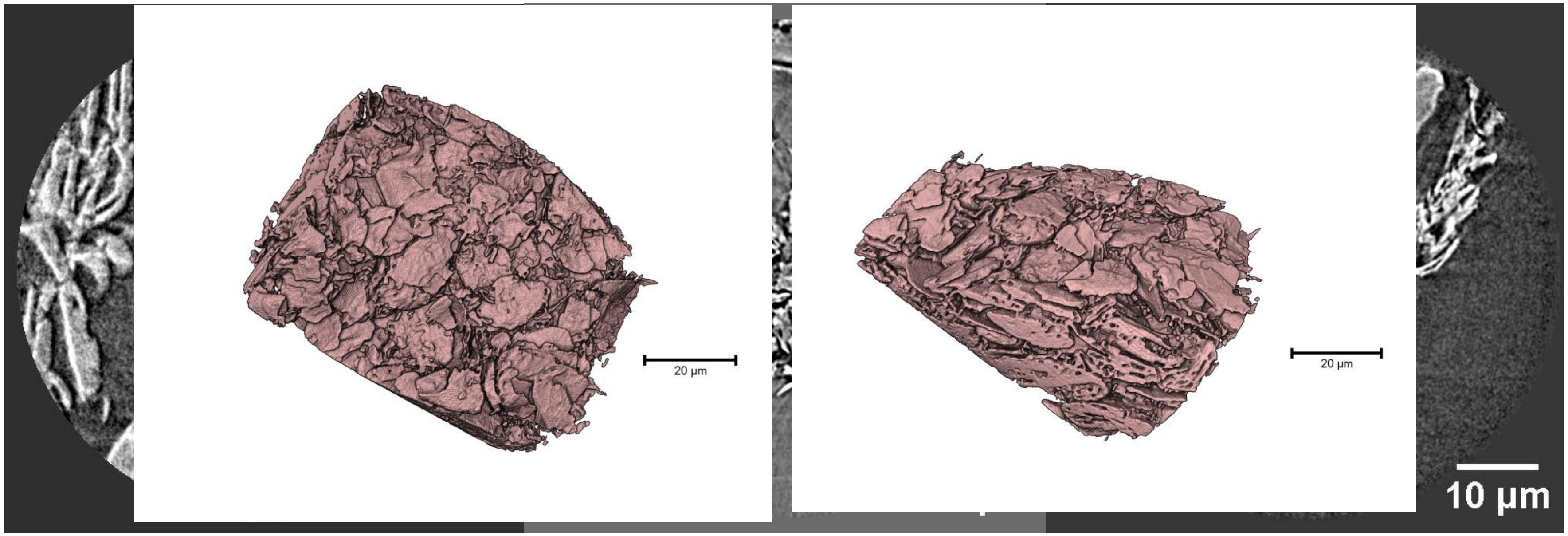
Silicon electrode with nano-sized Si particles (250 nm average particle size)



Ultra 810
23 s exp, 1601 proj.
63.1 nm/voxel

Combining absorption and phase contrast images using the *DSCoVeR* tool

Graphite electrode (87 % TIMCAL SLP-30, 3% Super P carbon, 10% PVDF binder)



Absorption contrast image
(30s exp, 1601 proj, LFOV)
0.126 µm/voxel

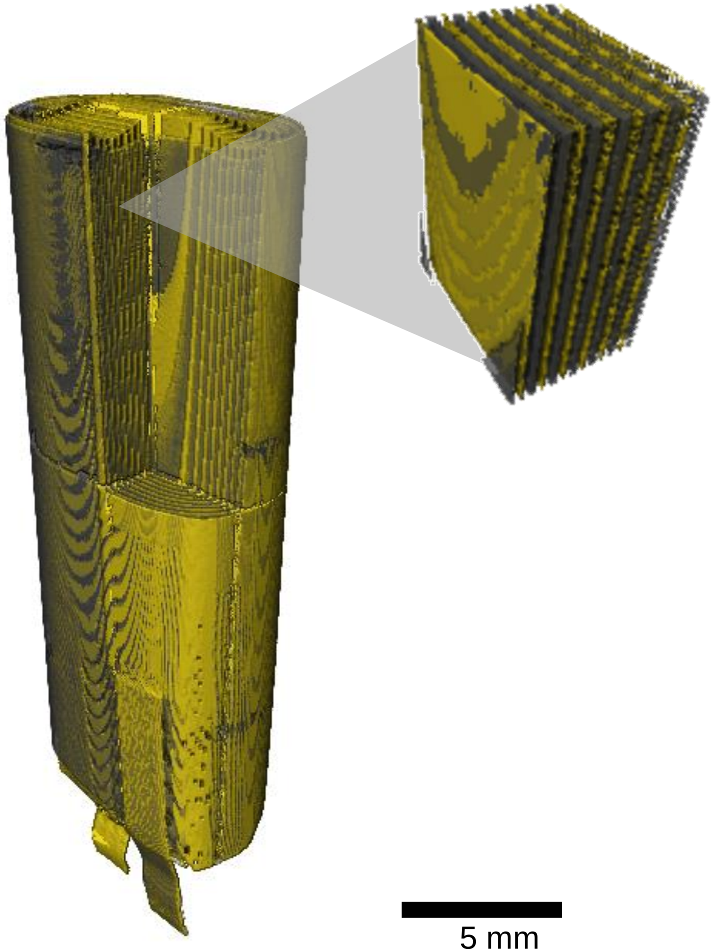
Phase contrast image
(17s exp, 1601 proj, LFOV)
0.126 µm/voxel

Combination of both
images in DSCoVeR

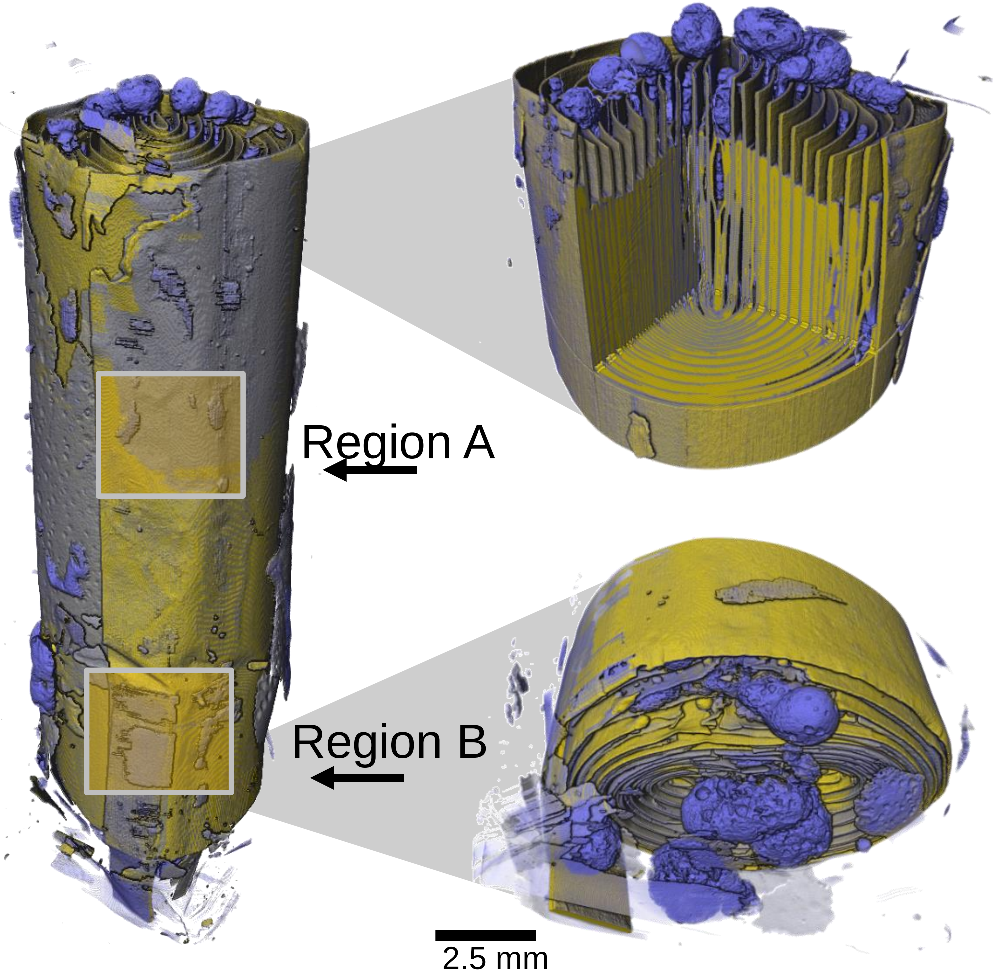
Li- Polymer Cell: High Voltage Failure

Versa 520 wt. Vertical Stitching, Voxel Size 9.9um

Before Failure



After Failure

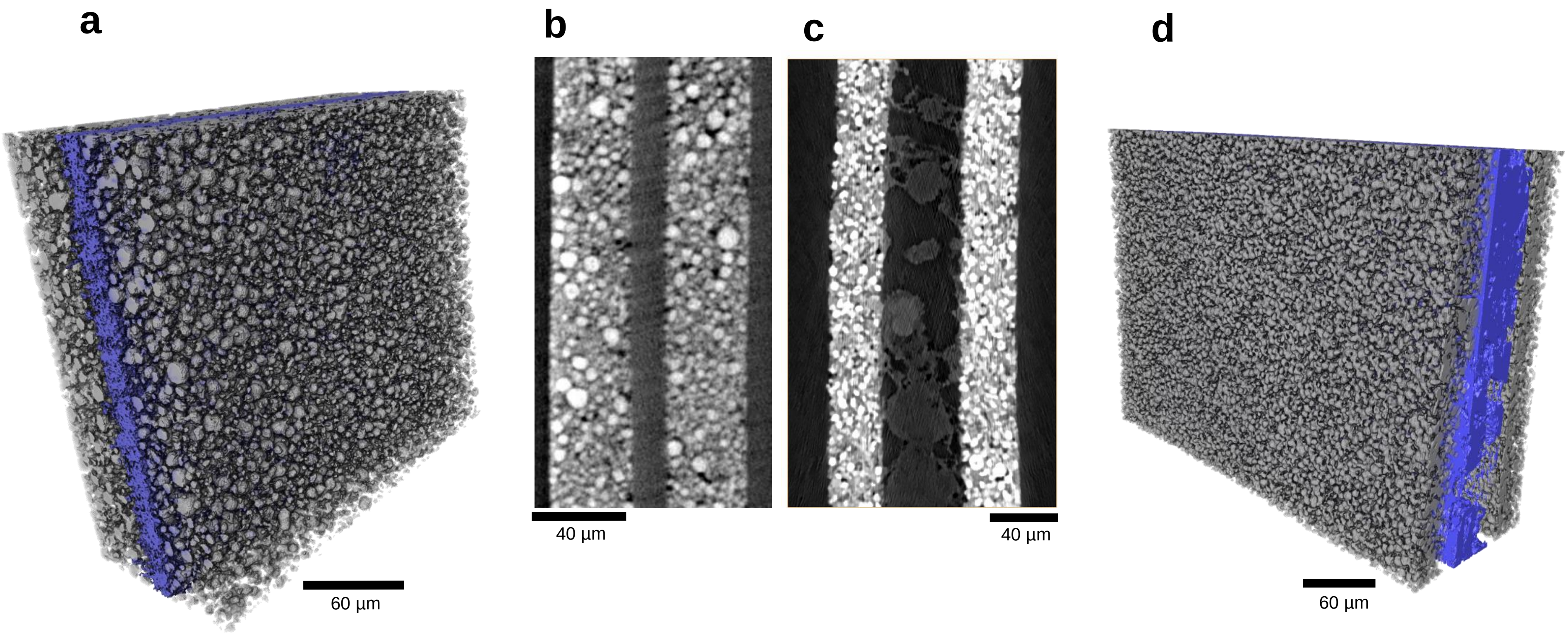


Li- Polymer Cell: High Voltage Failure

Versa 520:Voxel Size 420 nm

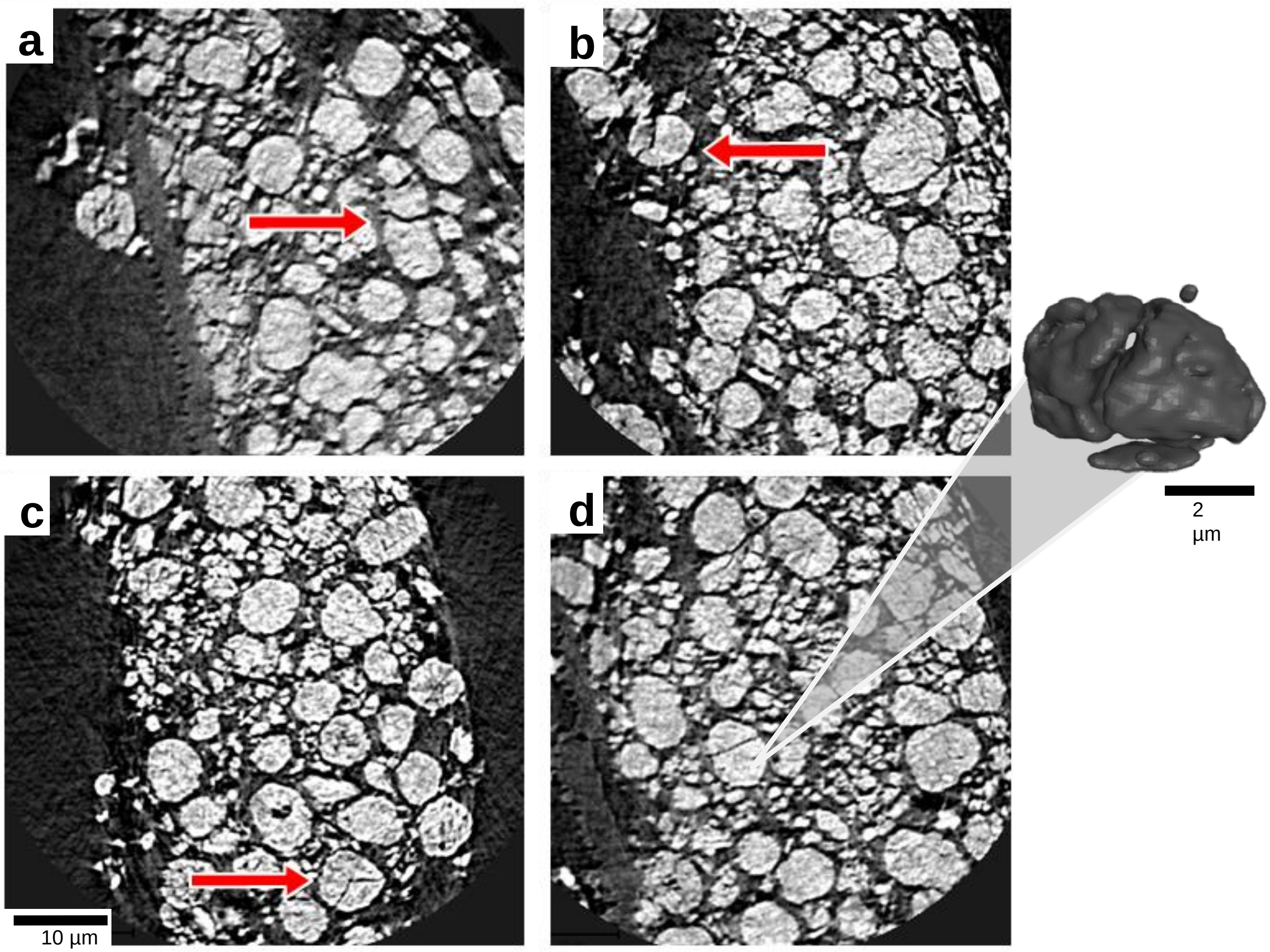
Before thermal runaway

After thermal runaway



Post Mortem Tomography:

Extensive particle cracking effects, Versa 520



Electrochemical Testing

- 32 Potentiostat Channels
- 80 Battery Cycling Channels
- kW scale Electronic Loads
- Battery Failure Testing Facilities

Thank You

Questions?

Dr. Francesco Iacoviello, Toby Neville
& the Electrochemical Innovation Lab

f.iacoviello@ucl.ac.uk

www.ucl.ac.uk/eil
www.ucl.ac.uk/eil/xray

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