X-ray Microscopy for Material and Life sciences research

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XRM Product and Applications specialist
Paris, 2019-12-02

Four ZEISS Future-Shaping Segments
2017/18

Semiconductor Manufacturing Technology

1.531 billion euros revenue

3,438 employees

Industrial Quality & Research

1.549 billion euros revenue

6,773 employees

Medical Technology*

1.546 billion euros revenue

4,889 employees

Consumer Markets

1.106 billion euros revenue

10,544 employees

As of 30 September 2018 *The values deviate from the published figures of Carl Zeiss Meditec AG as a result of different consolidation models.

Segment
Industrial Quality & Research

Industrial Quality Solutions Portfolio:

- Contact and optical measuring systems
- Computed tomography
- Measuring, analysis and management software
- Services

Research Microscopy Solutions Portfolio:

- Light microscopy
- Laser scanning microscopy
- Electron and ion microscopy
- X-ray microscopy

Strategic Business Unit
ZEISS Research Microscopy Solutions

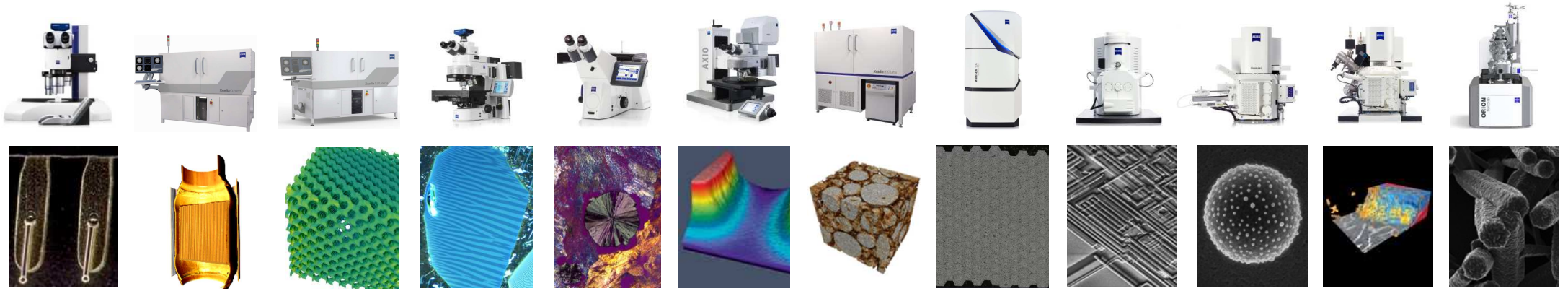
- Biggest microscopy player in the market

- The only company with a unique offering of light, electron and X-ray microscopes

- Dedicated research solutions for life sciences and materials microscopy in academia and industry

ZEISS Microscopy Portfolio
Multi-Scale Characterization for Multi-Scale Research

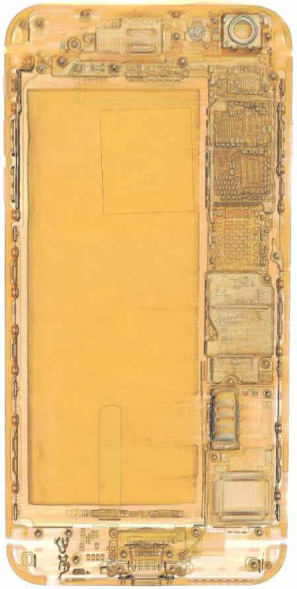
A complete microscopy portfolio…



…to address multi-scale research challenges.

Why Do We Use X-ray Microscopy?
Materials characterization in 3D

→ **Visualize, characterize, and quantify internal three dimensional structures of objects without physical cutting**



3D X-ray Imaging for Research Applications

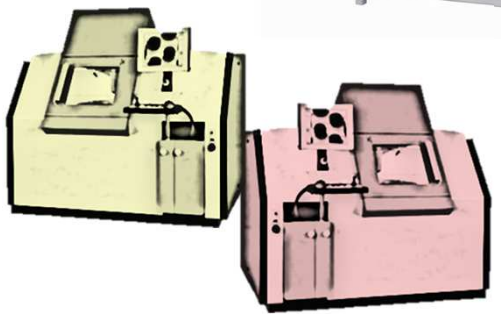
X-ray microCT

Projection-based geometric magnification architecture

Xradia Context

0.95 µm spatial resolution

Other Commercial Systems



X-ray Microscopy

Synchrotron technology extended to the lab

Two-stage magnification with scintillator-coupled optical objectives

Xradia Versa Family

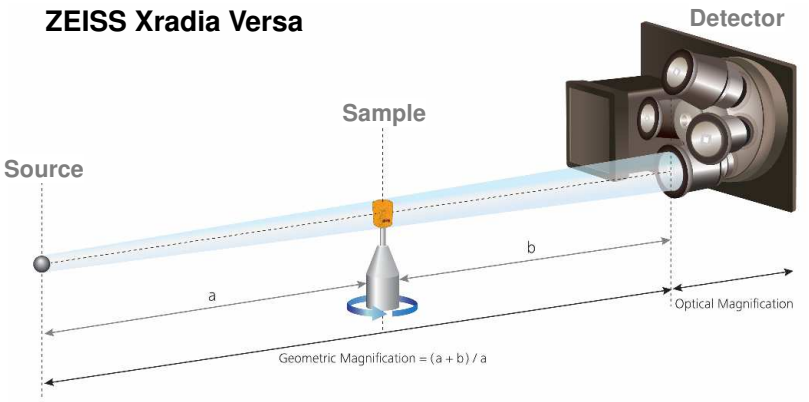
0.5 µm spatial resolution, RaaD

Transmission XRM architecture with X-ray focusing optics (condenser, zone plate)

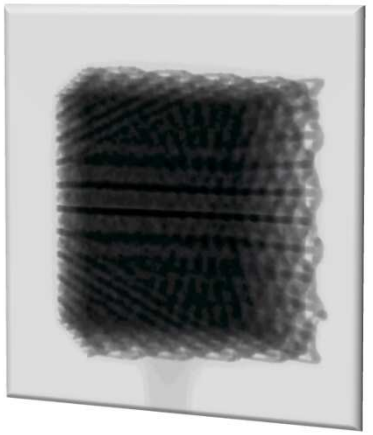
Xradia Ultra Family

50 nm spatial resolution

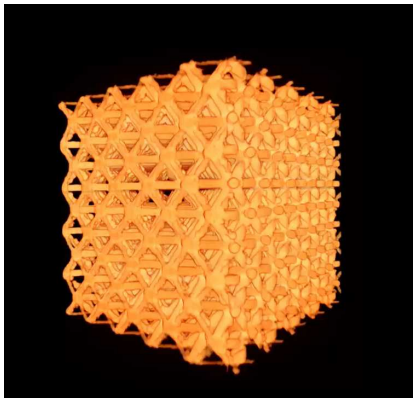
Tomography in 3D X-ray Microscopy
How it works



Projections

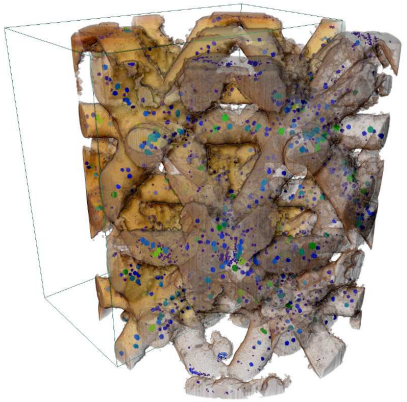


3D Reconstruction

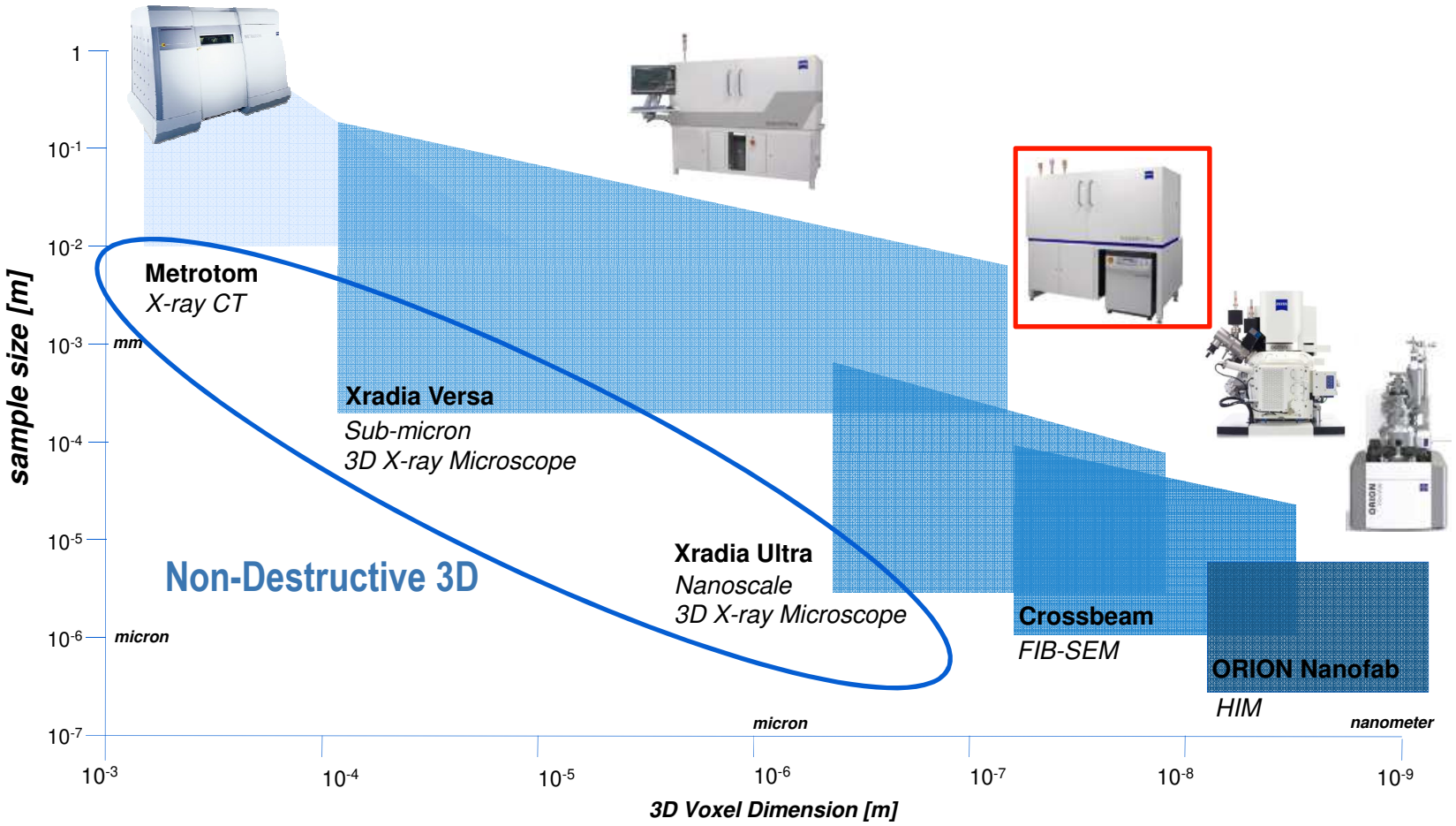


Virtual slices

Quantitative Analysis



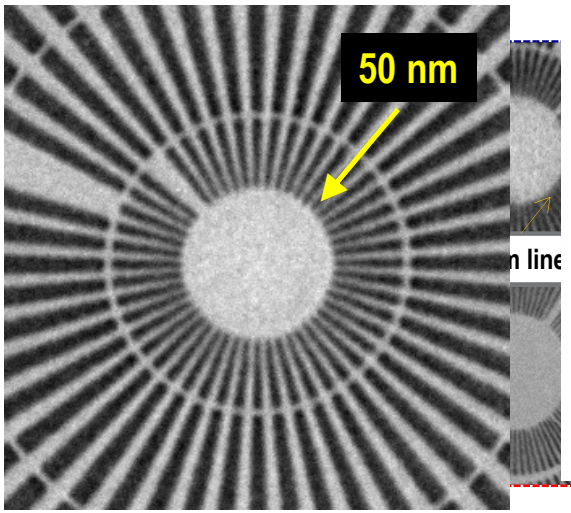
ZEISS
A complete 3D-Microscopy portfolio



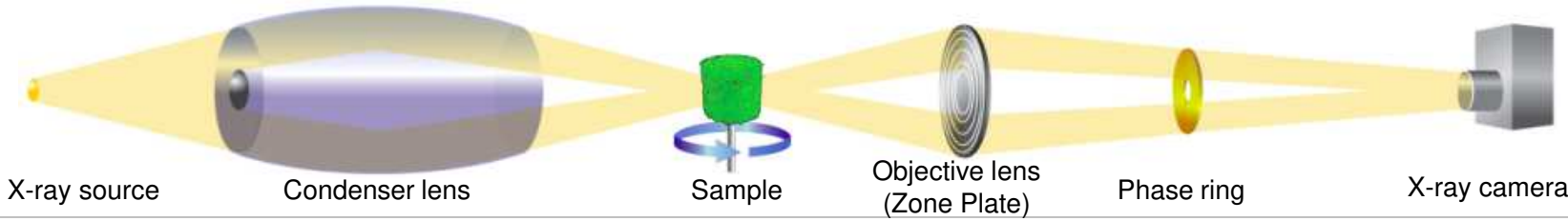
ZEISS Xradia Ultra
3D X-ray Nanotomography down to 50 nm Resolution

50 nm spatial (16 nm voxel) resolution

- High brightness X-ray source
  - **Xradia 810 Ultra: 5.4 keV**
  - **Xradia 800 Ultra: 8.0 keV**
- Advanced X-ray optics
- Absorption and Zernike phase contrast

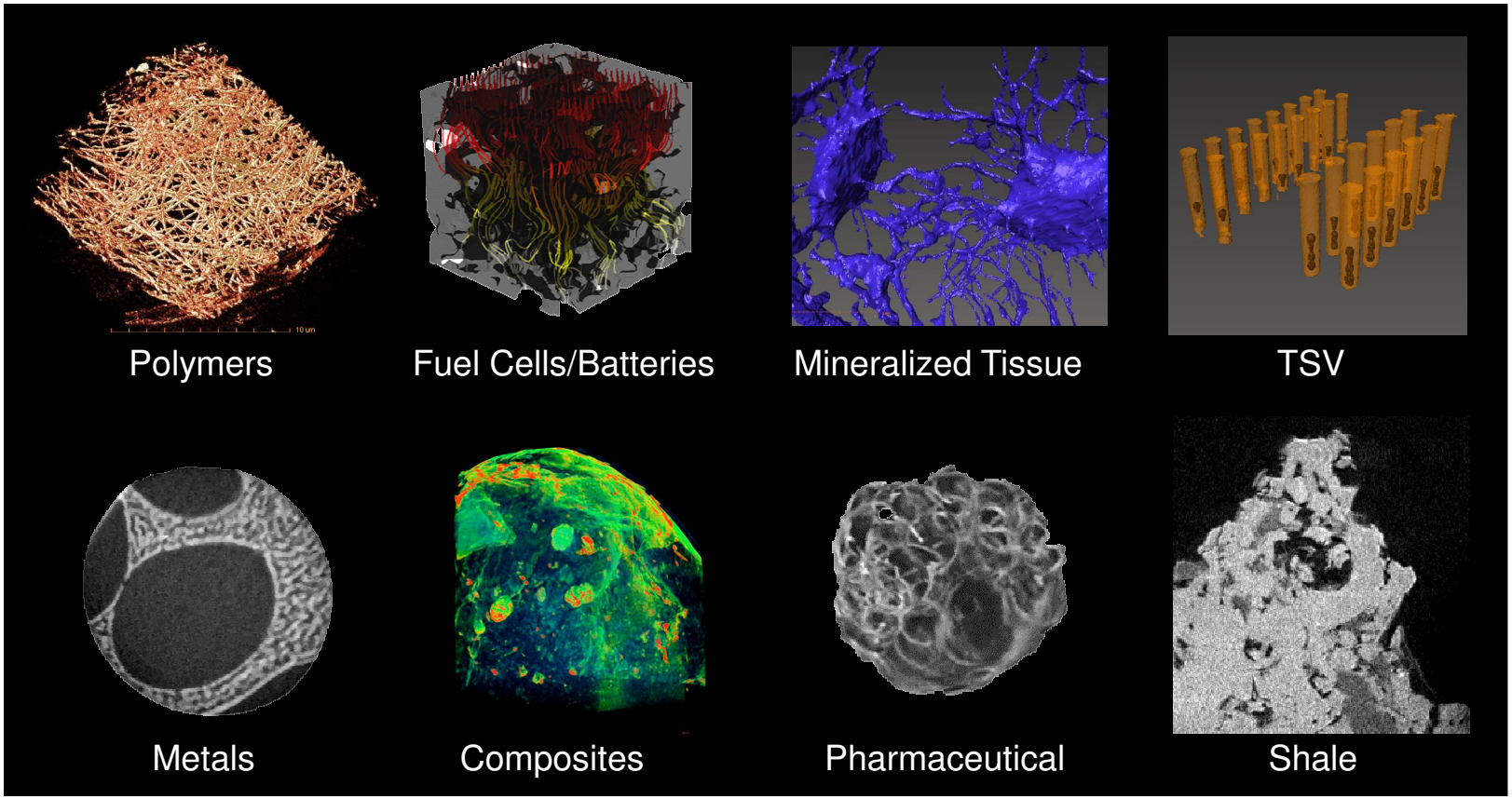


Mode	Mag	2D Res	Voxel	Field of View
Large Field of View	200X	150 nm	64 nm	65 µm x 65 µm
High Resolution	800X	50 nm	16 nm	16 µm x 16 µm

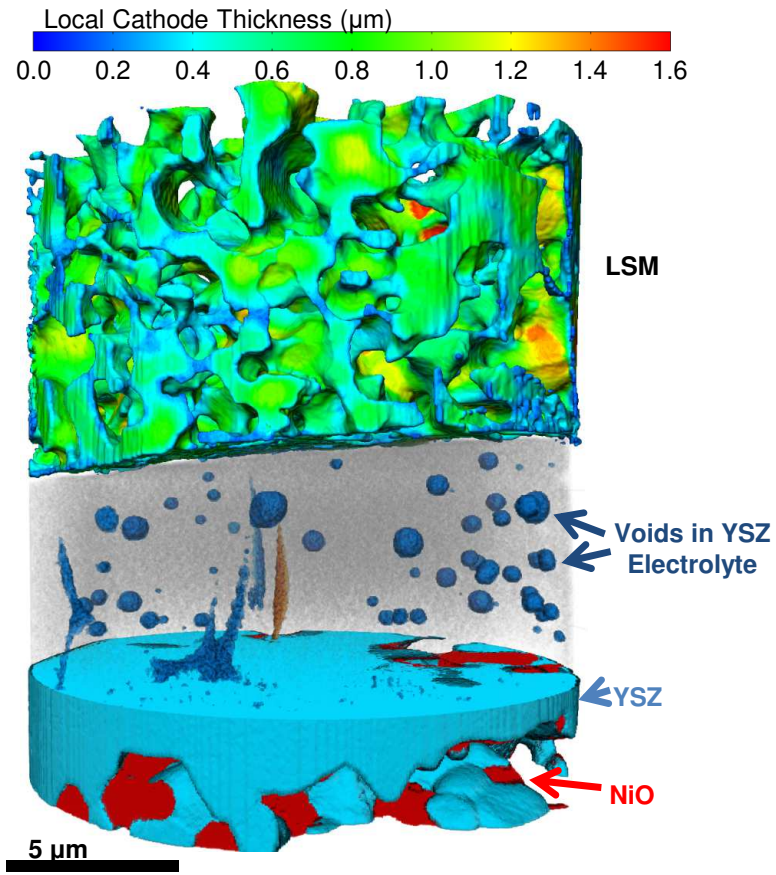


Xradia Ultra
Applications for Research

Diverse interests and applications…



Analysis and Measurements

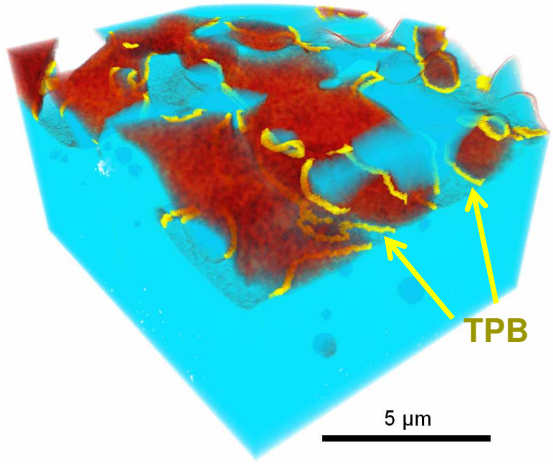


Local Size Measurements

ORS Dragonfly Pro used to measure the local variation in feature sizes of the LSM network

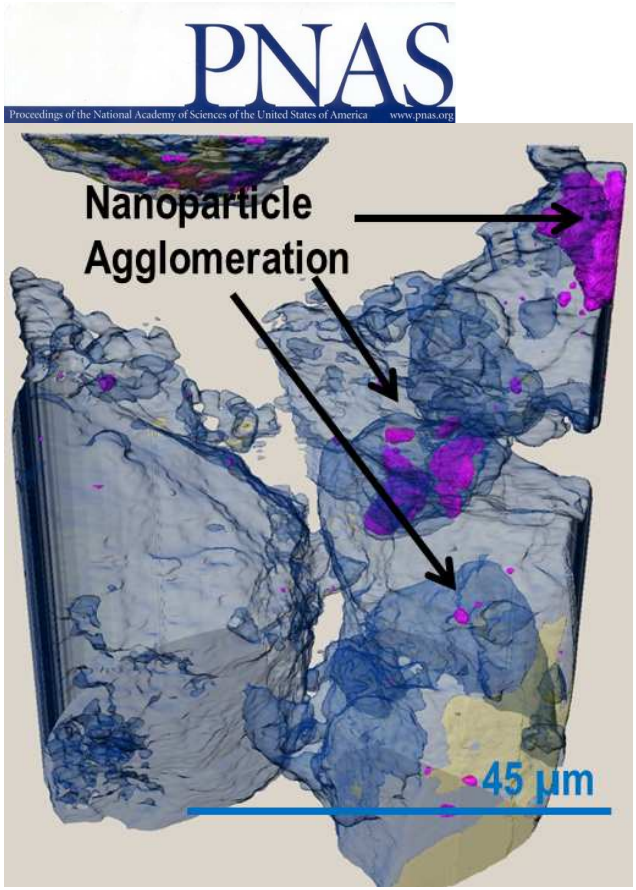
Anode Triple Phase Boundary Visualization

Generated by a series of dilations on the NiO, YSZ, and pore phases, shows the locations of electrochemical reaction sites



ZEISS Xradia Ultra:
Soybean susceptibility to manufactured nanomaterials

J. Priester, et. al., *PNAS, 109, 37, E2451-E2456* (2012)

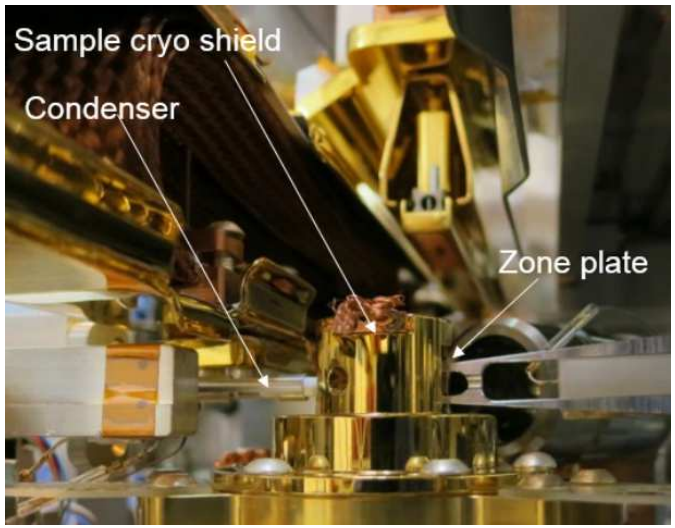


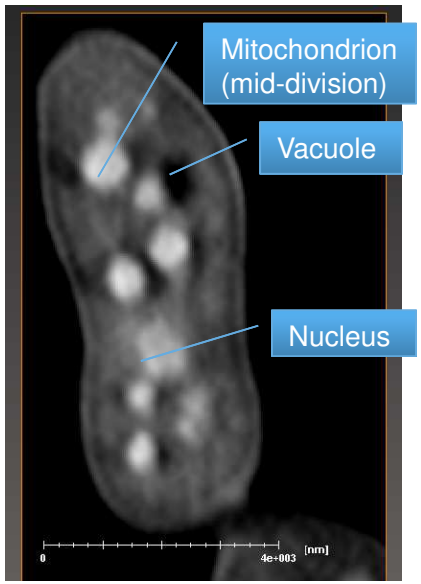
- Effect of the uptake of manufactured nanomaterials in soybean root structures observed by nanoscale XRM
- Zernike phase contrast utilized to image CeO_2 particles and root matrix materials
- Left: Ultra XRM (Zernike) 3D image of Ce accumulations in root nodules. Arrows indicate higher density, as correlated to SEM information
- XRM provides straight-forward approach to quantifying CeO_2 accumulation (size, distribution) without the need for physical serial sections.

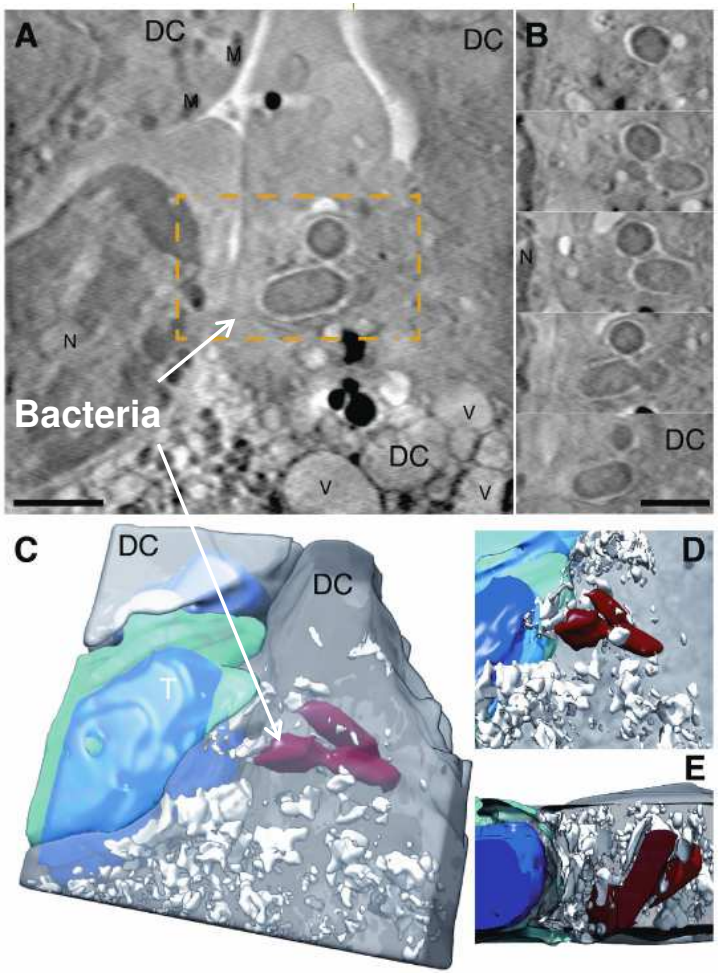
DOI: 10.1073/pnas.1205431109

Xradia 825 Ultra
Lab Soft X-ray Cryo Microscope for Cellular Imaging

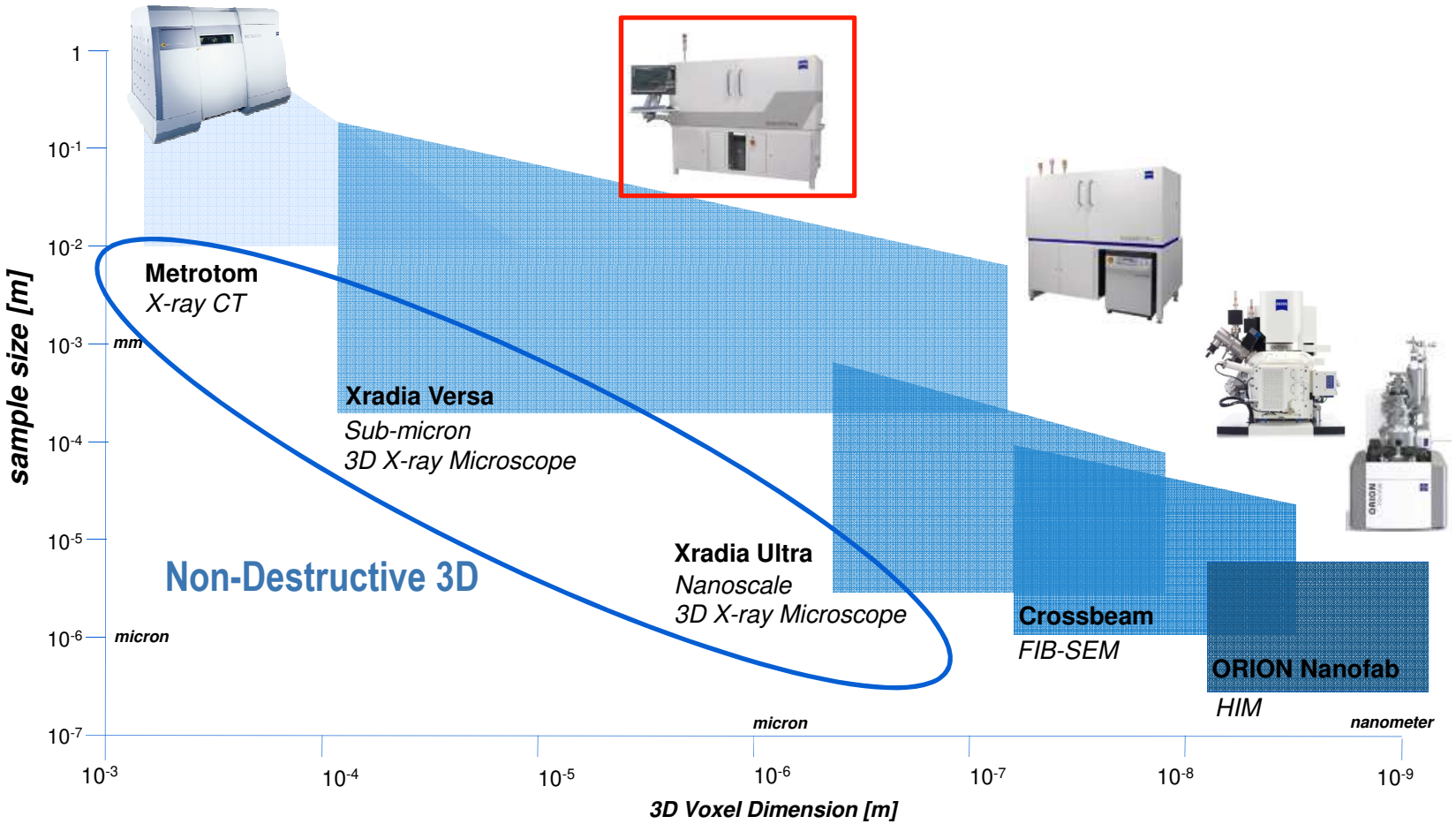
- Cryo TXM coupled to laboratory based nitrogen plasma soft x-ray source (430 eV)
- Imaging of frozen hydrated yeast cell
- Initial demonstration of correlative imaging: Optical fluorescence (function) and x-ray (structure) imaging







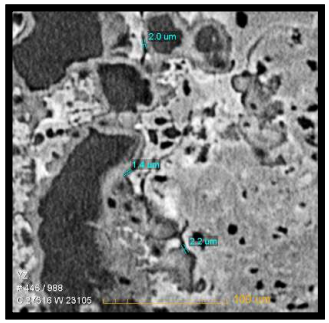
ZEISS
A complete 3D-Microscopy portfolio



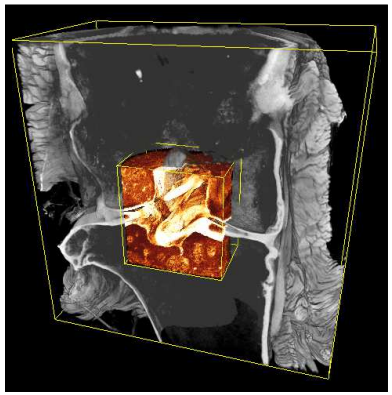
Diversity of Applications in Academia
Both the appeal and the challenge

8” concrete

Indented rat ulna

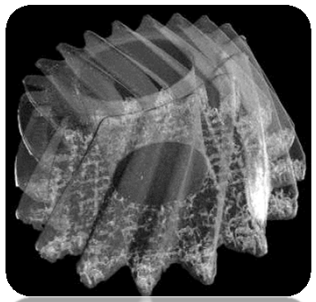


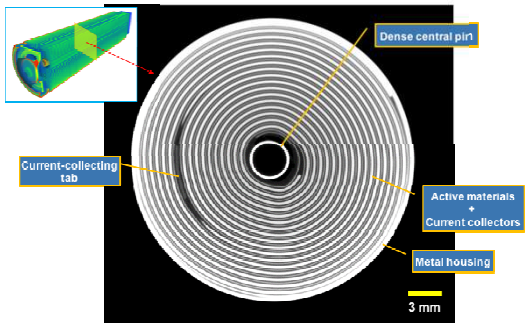
Mouse knee



Plant root

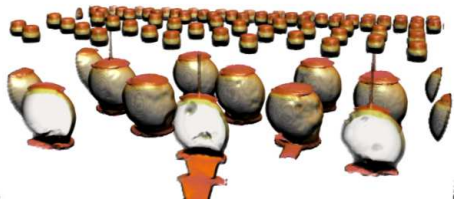
3D printed Al

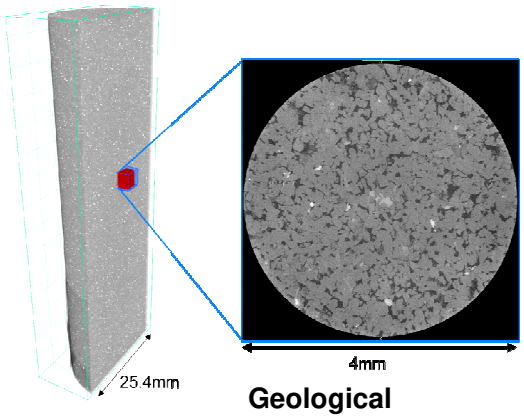




18650 Li-ion battery

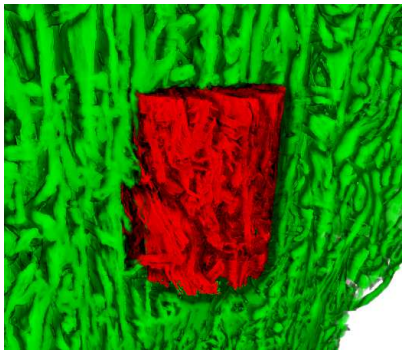
Solderball interconnects



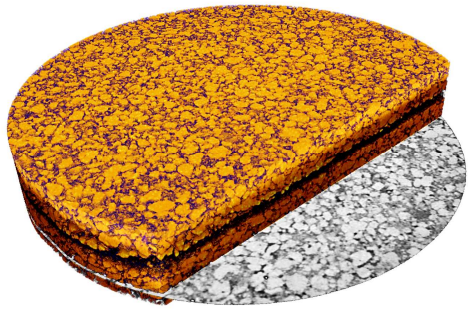


Geological core sample

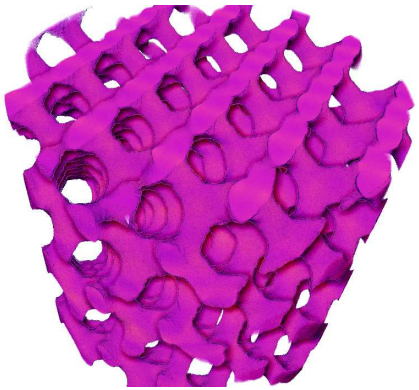
Materials Science
Applications for X-ray microscopy



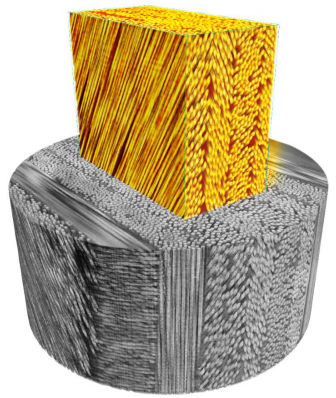
Polymers & Biomaterials



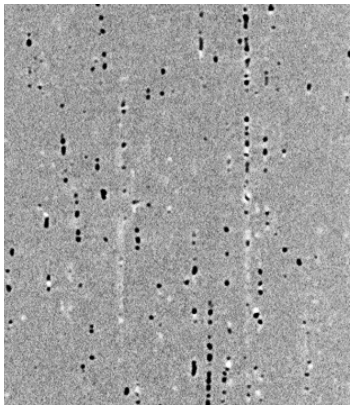
Energy Materials



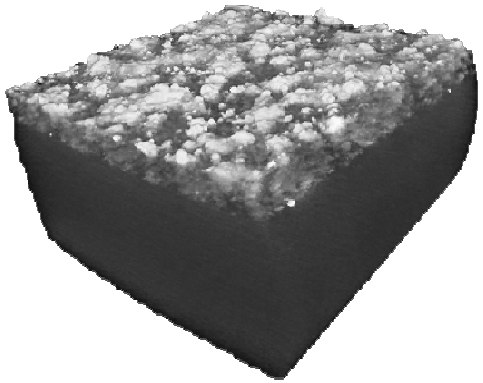
Ceramics



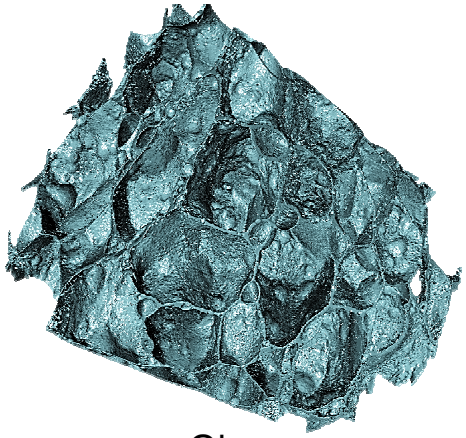
Composites



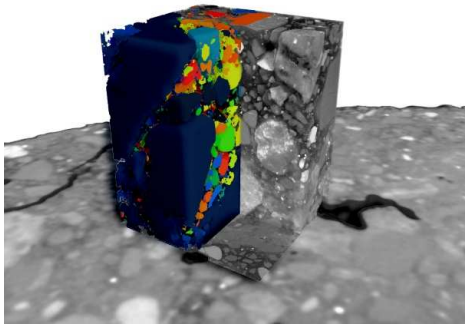
Metals



Coatings



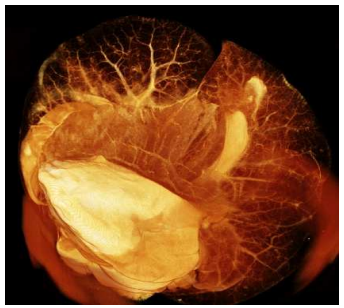
Glass



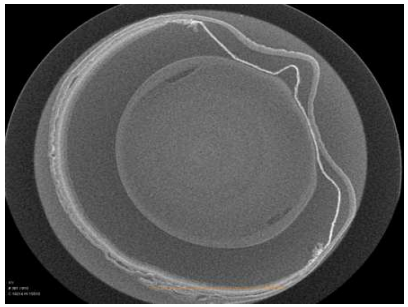
Concrete

Soft Tissue XRM Imaging
High Resolution and Contrast Offers Great Benefit to Imaging

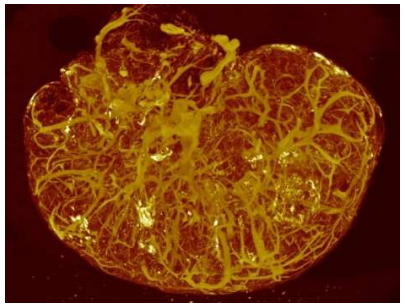
Lung



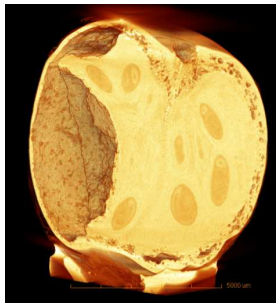
Eye



Ovary

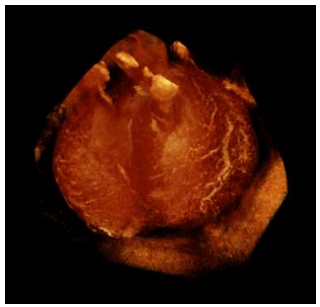


Plant Tissue

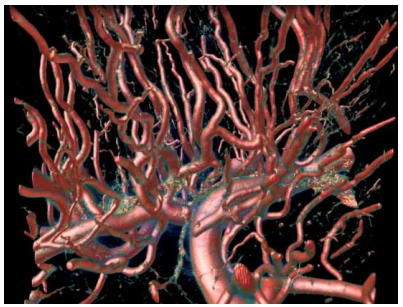


Brain

Heart

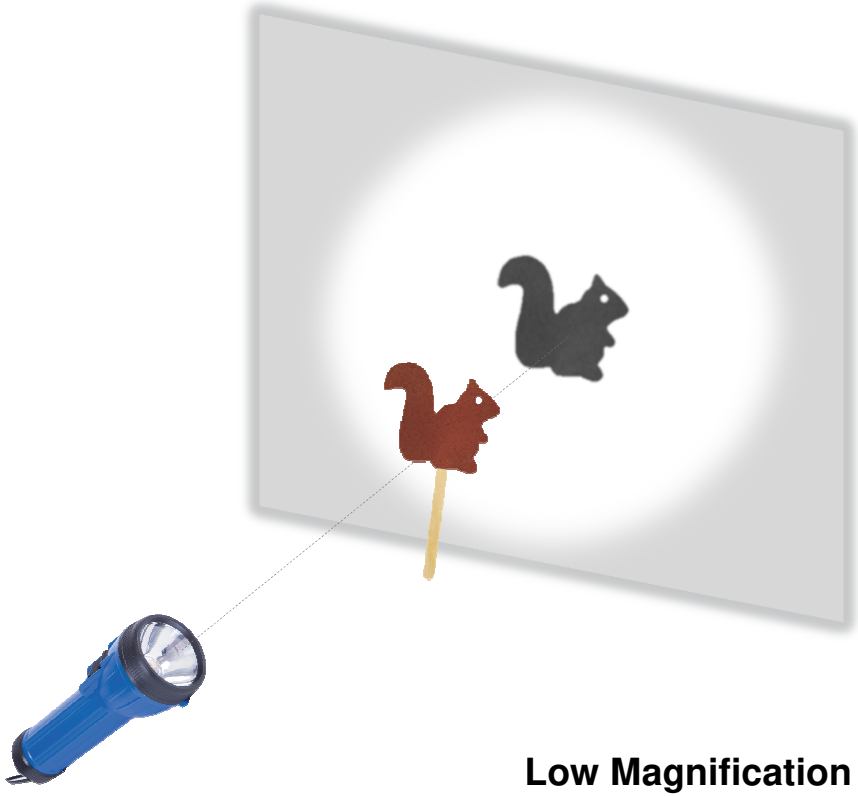


Vasculature

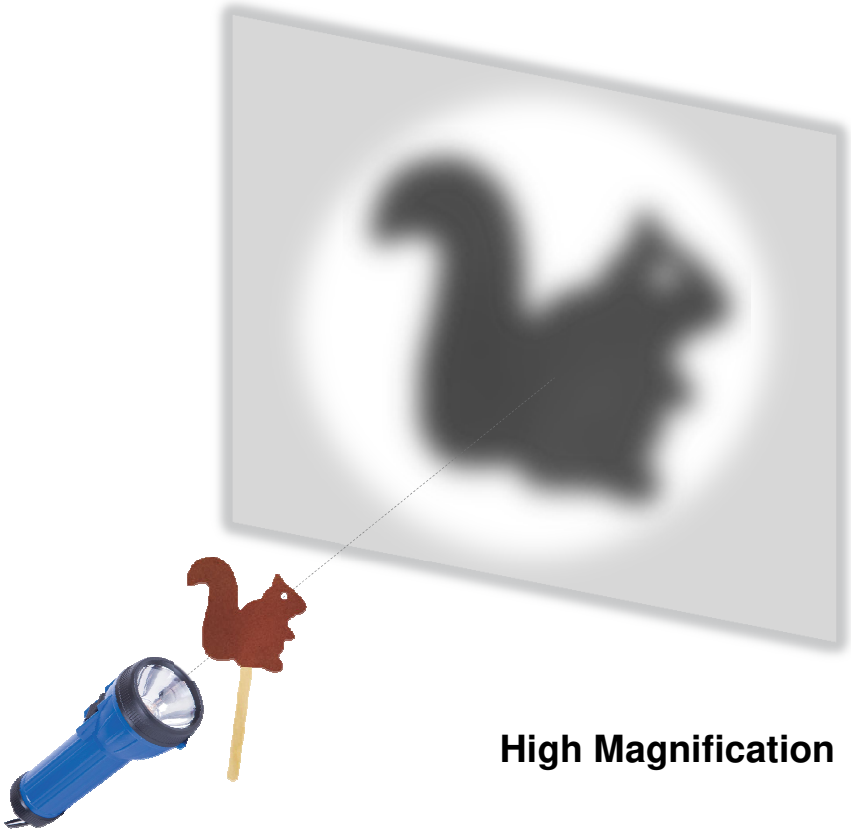


Liver

Defining Geometric Magnification
Shadow projections



Low Magnification

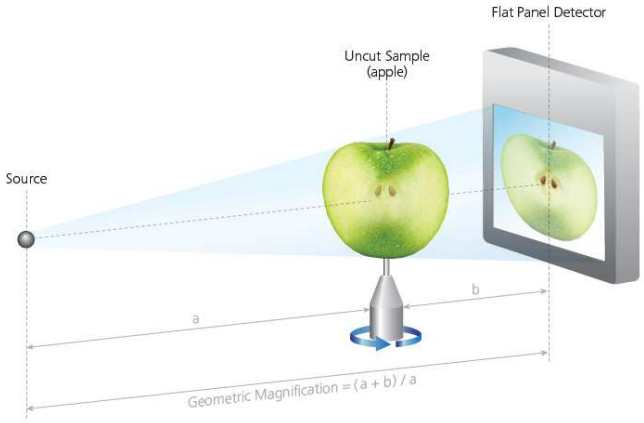


High Magnification

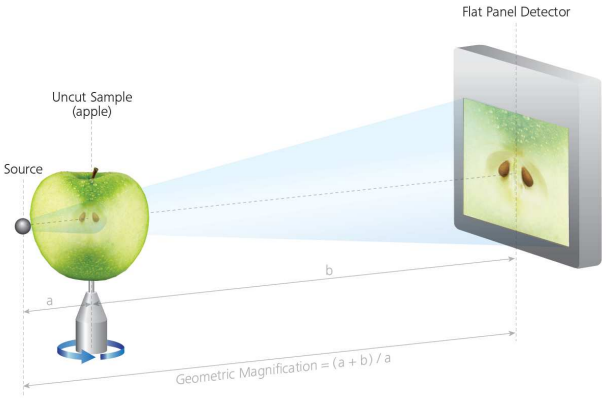
Limitations of microCT Geometric Magnification

"You can only get so close"

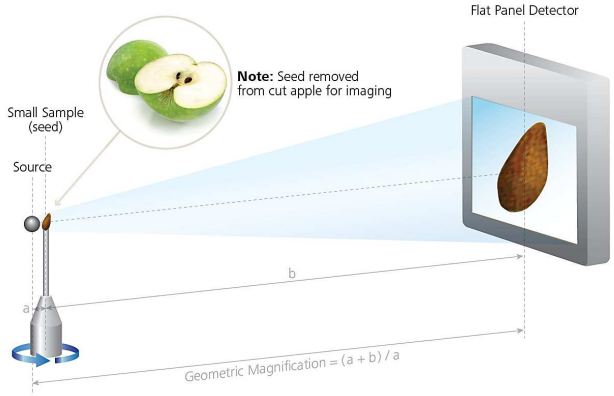
With microCT architecture…



…you can image the whole object…

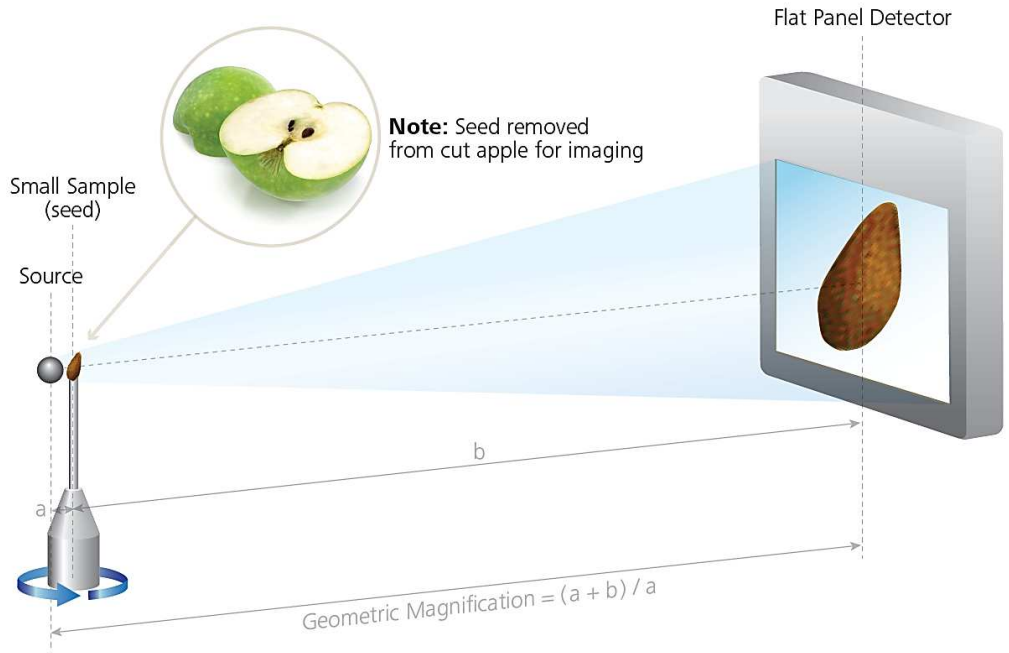


…and then you can zoom in *a little.*



But if you want to see the small things (seed), you need to cut it open

Chopping Up Samples for Higher Resolution
When all you have is microCT geometric magnification



Cutting an apple might be OK, but what if…

…it is a precious sample you can't destroy?

…it is an intact device (battery, electronics component)?

…cutting your sample risks damaging the structure?

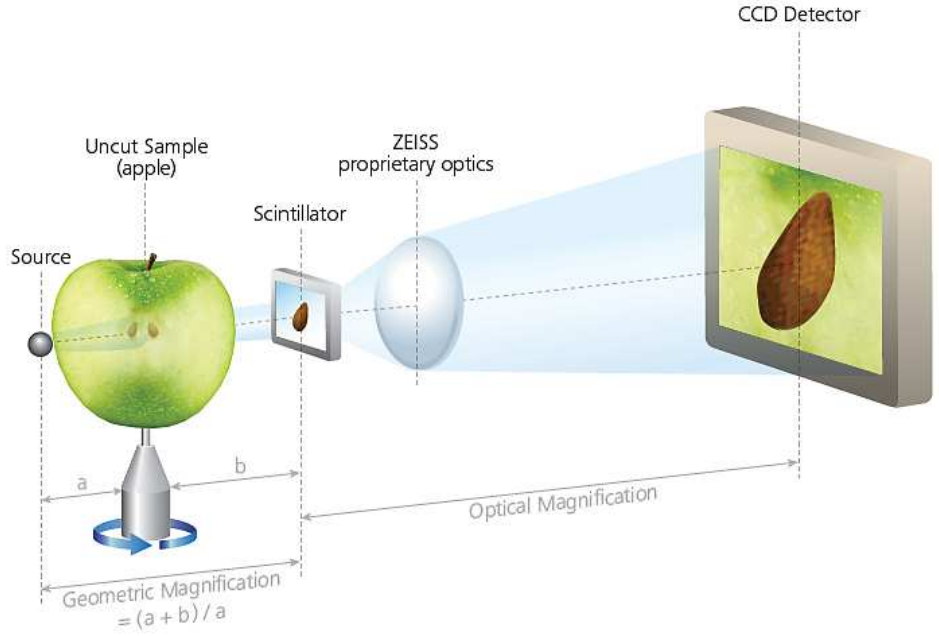
…you need to preserve your sample for future studies?

…you have sparse features and don't know where to cut?

…you are working inside an *in situ* chamber or rig?

→ There are frequent cases where working with larger or intact samples is beneficial

X-ray Microscopy with Two-Stage Magnification
Geometric + optical magnification

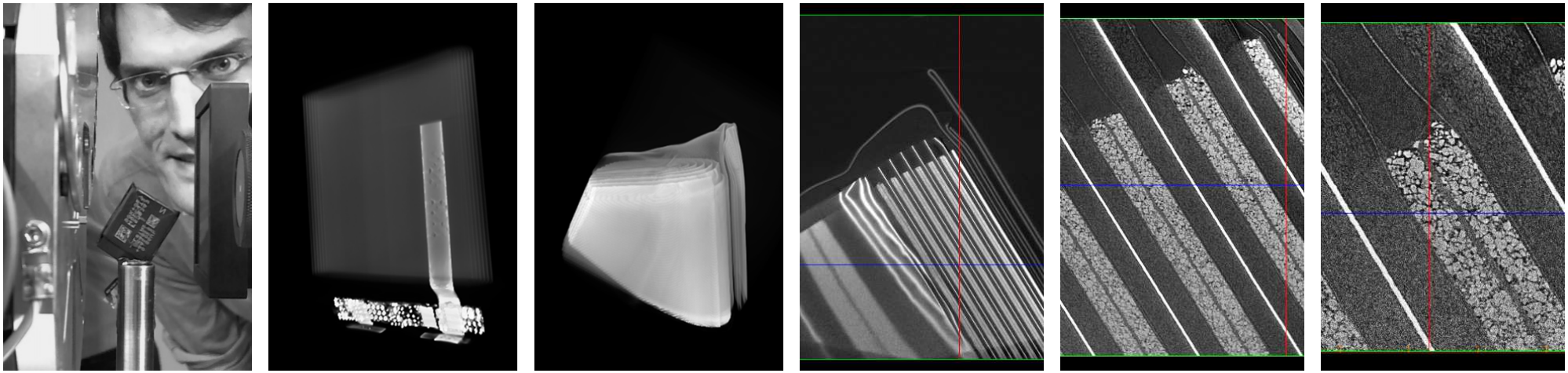


Only an **X-ray microscope** can scan an apple seed at high resolution ***without cutting*** the apple open (RaaD = Resolution at a Distance)

ZEISS Xradia Versa -
Multiple scintillator-coupled optics for different magnification

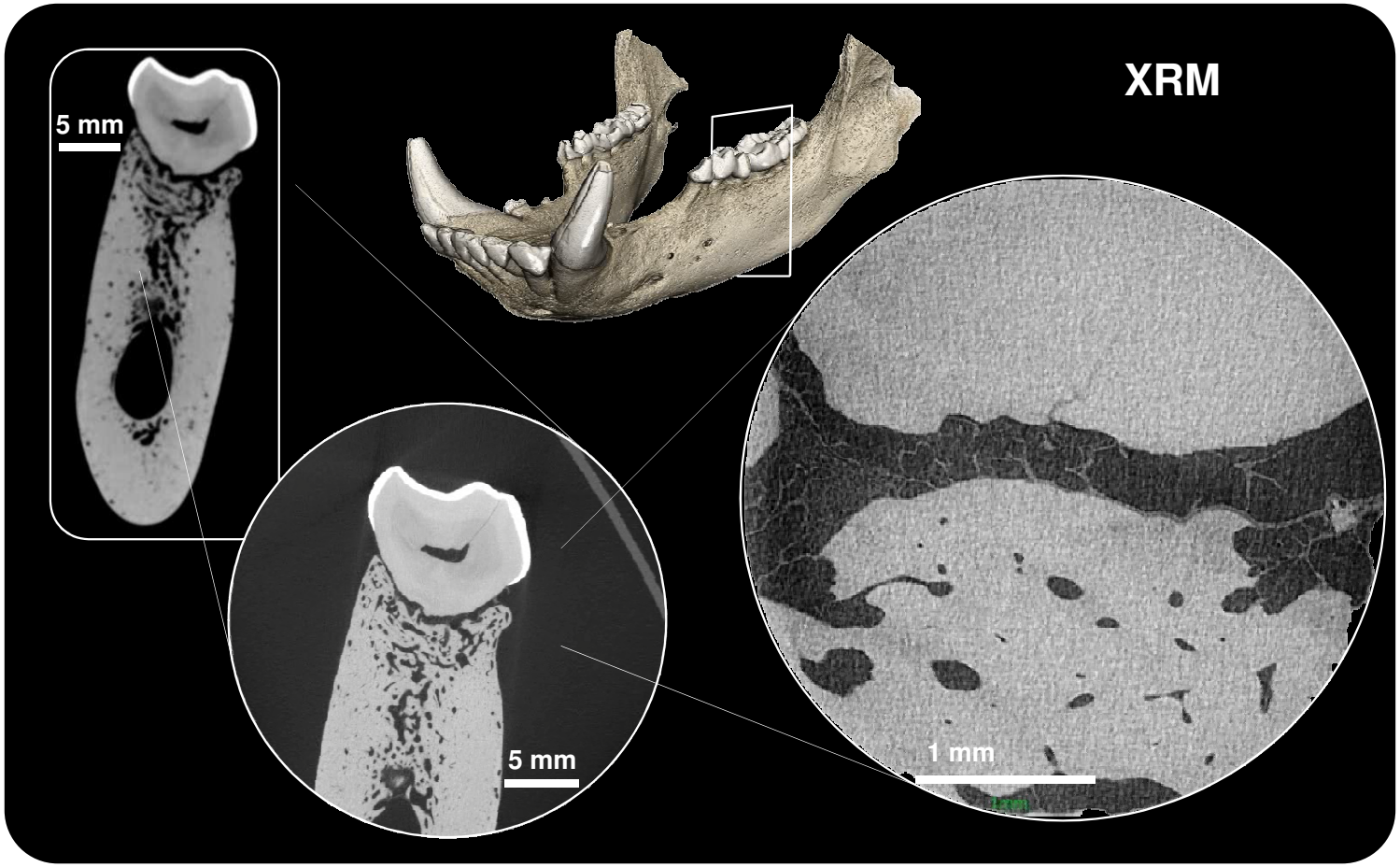
What Can We Do with RaaD?
Not just for apples

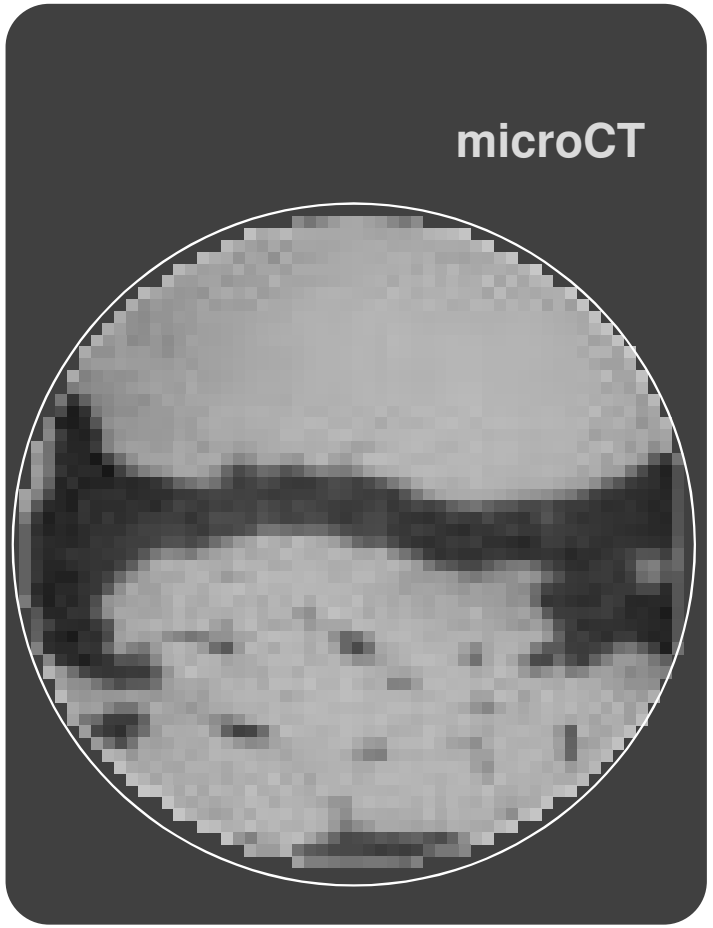
Analysis of lithium ion batteries is challenging – many critical quality and safety effects only become apparent with aging.



- **X-ray microscopes (XRM)** scan the *intact* battery to identify areas of interest and zoom-in for high resolution imaging
- With **traditional X-ray microCT** to scan at this resolution requires *complete disassembly of the battery - requiring glovebox and solvents, skill and time*

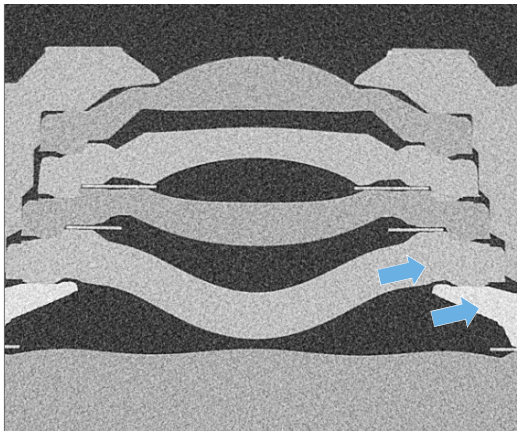
High Resolution
For precious samples or artifacts





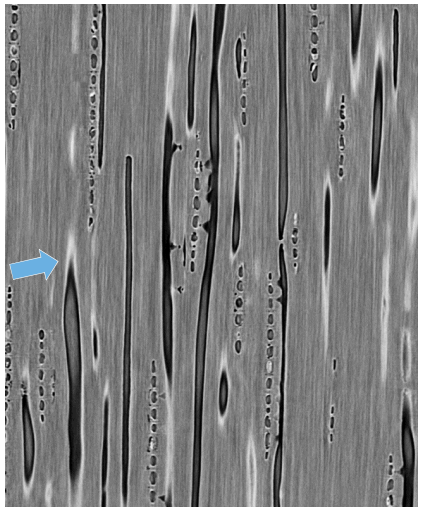
Beyond Absorption (Density) Contrast

Advanced absorption with optimized scintillator optics



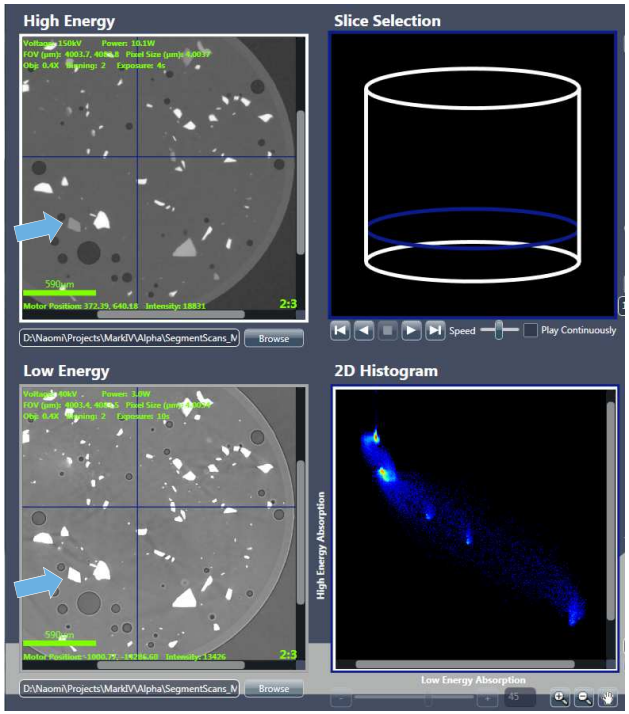
Mobile phone camera lens assembly

Propagation phase contrast for edge enhancement & low density phases



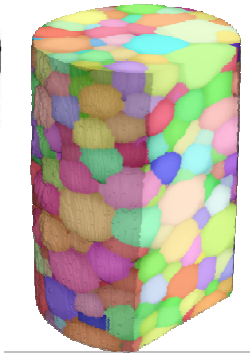
Vasculature in wood

Dual scan contrast visualizer (DSCoVer) for differentiating similar-Z phases



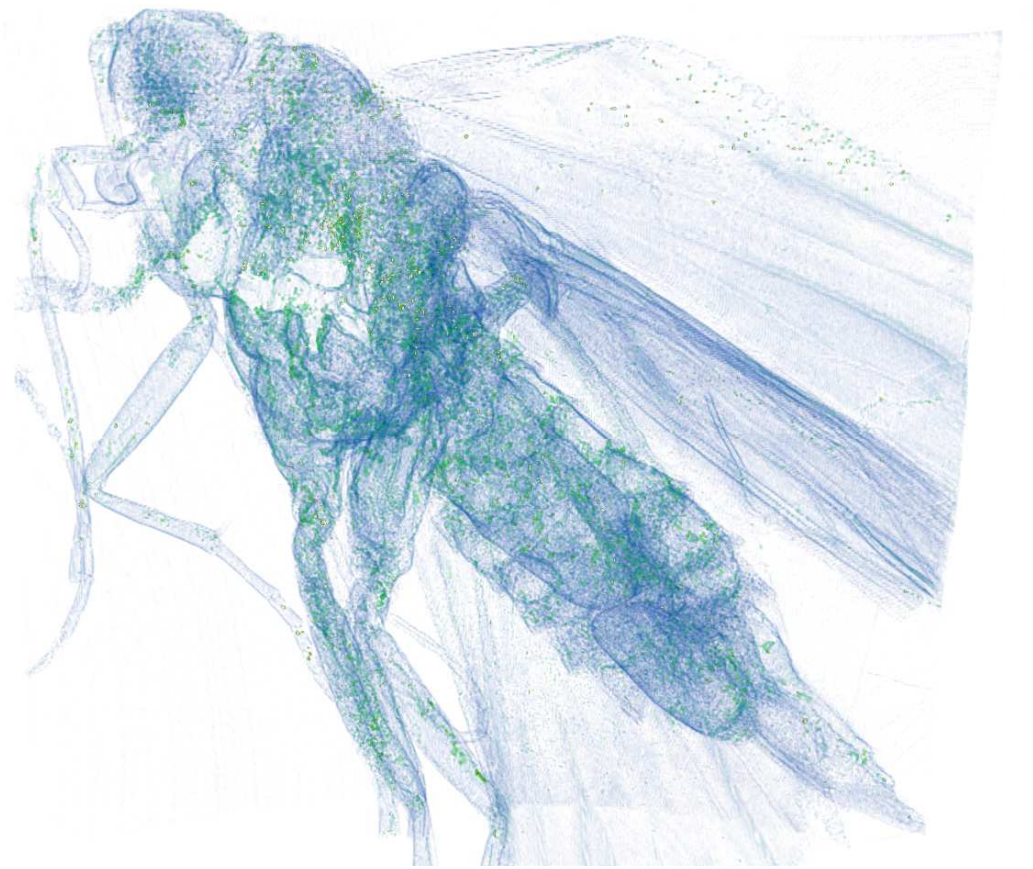
Al-Si composite

Diffraction contrast tomography (LabDCT) to map polycrystalline materials



Ti alloy

Phase contrast – insect in amber & DSCOVER

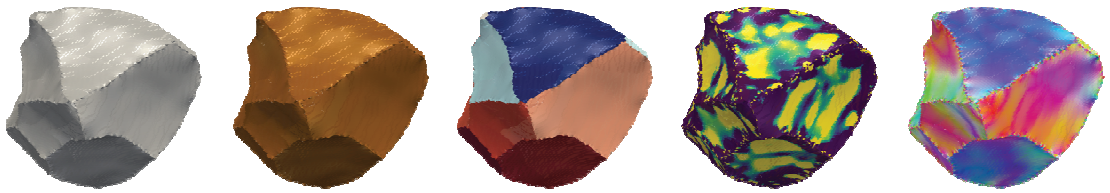


Absorption contrast removed

LabDCT with **GrainMapper3D** provides Comprehensive Information on Grain Structure

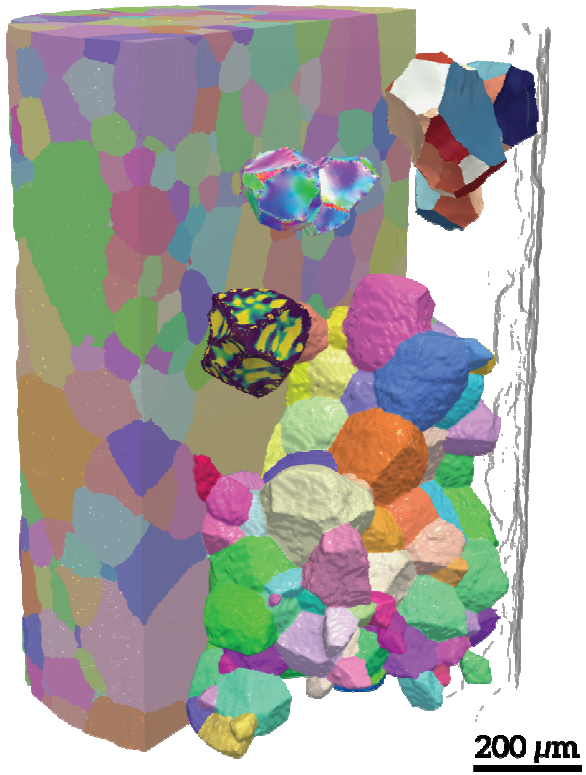
GrainMapper3D offers:

- ✓ Grain Centroid Position
- ✓ Grain Size
- ✓ Grain Orientation
- ✓ Grain Shape
- ✓ Grain Boundary Information



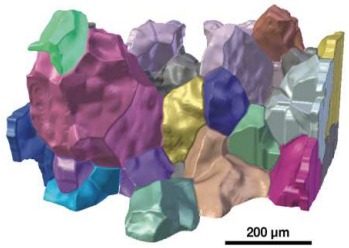
Left-right:
Faces of a selected grain color coded in random color, by IPF color, misorientation to neighboring grains, grain boundary curvature and grain boundary normal direction in crystal reference system.

GB processed with Dream3D

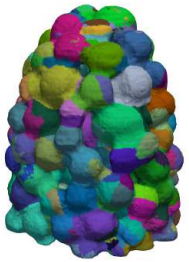


3D grain map of an Armco iron sample.
Half the sample volume is removed to reveal inner grain (clusters). Courtesy of Prof. Burton R. Patterson, University of Florida, United States.

LabDCT
Non-destructive 3D grain imaging



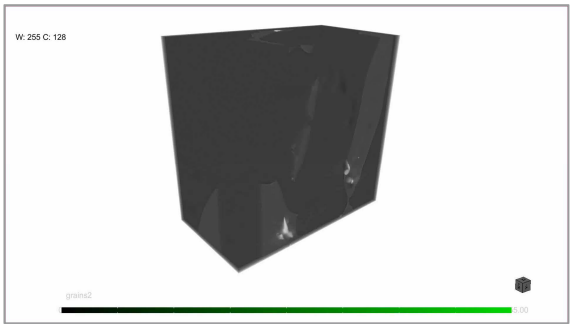
Al alloys



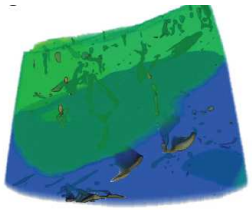
Sintered Cu Spheres

Al-Cu

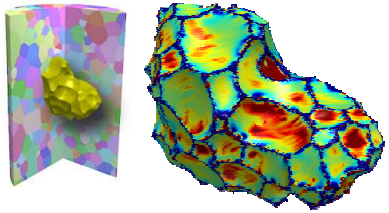
200 µm



Olivine

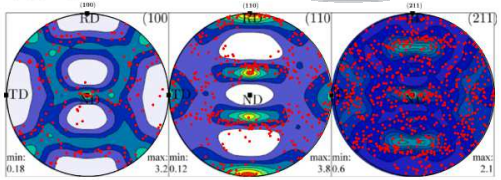


Polysilicon

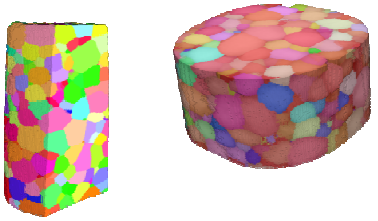


Iron

700 °C



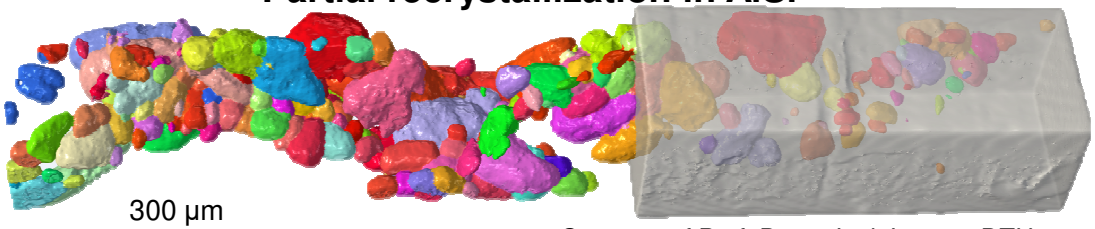
Recrystallization in rolled steel



Titanium alloys

Others:
Magnesium, Silicon-Carbide, Diamond

Partial recrystallization in AlSi



Courtesy of Prof. Dorte Juul Jensen, DTU

Materials Science Publications Using ZEISS X-ray Microscopes

#BuildTheFuture

Since January 2016…

603
XRM materials science publications

42
Countries publishing XRM materials science papers

3.8
On average, new ZEISS XRM materials science publications *every week*

6
Continents with co-authors

500+
Unique institutions listed as authors or co-authors

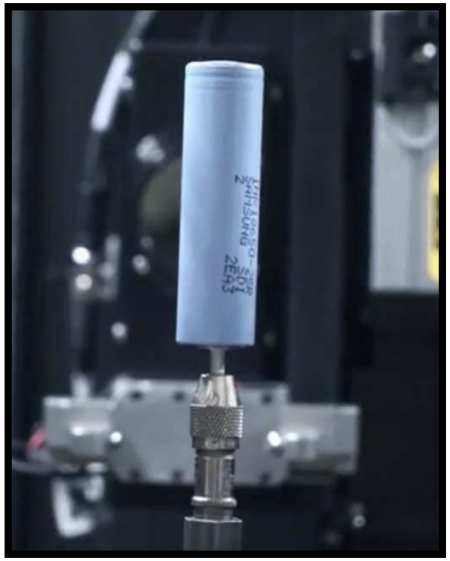
ZEISS Xradia Versa
ZEISS Xradia Ultra
ZEISS Xradia Context microCT

XRM Applications in Materials Science

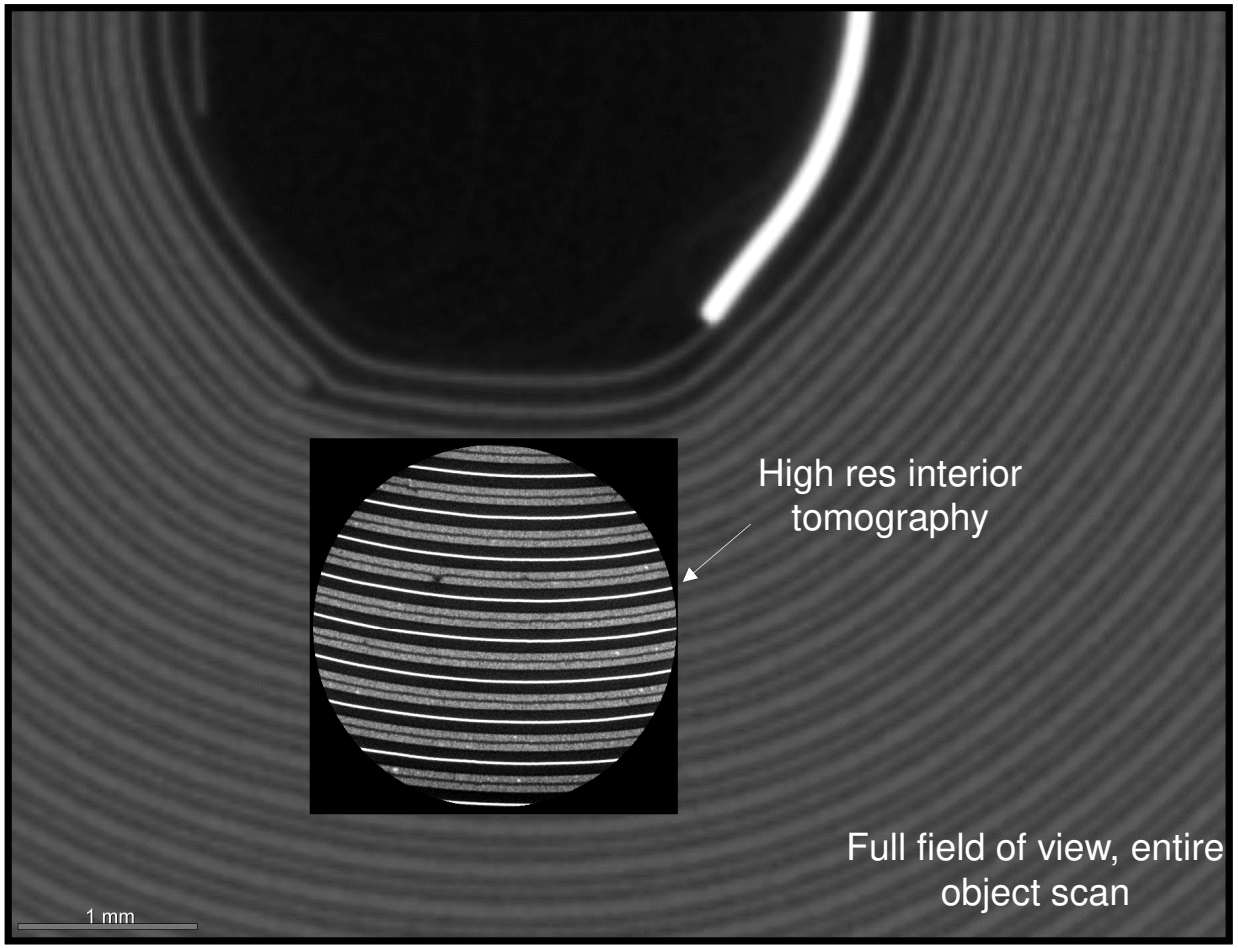
- Engineering Materials (metals, composites, etc)
- Soft/Environmental/Biomaterials
- Energy Materials
- Nano and Functional Materials

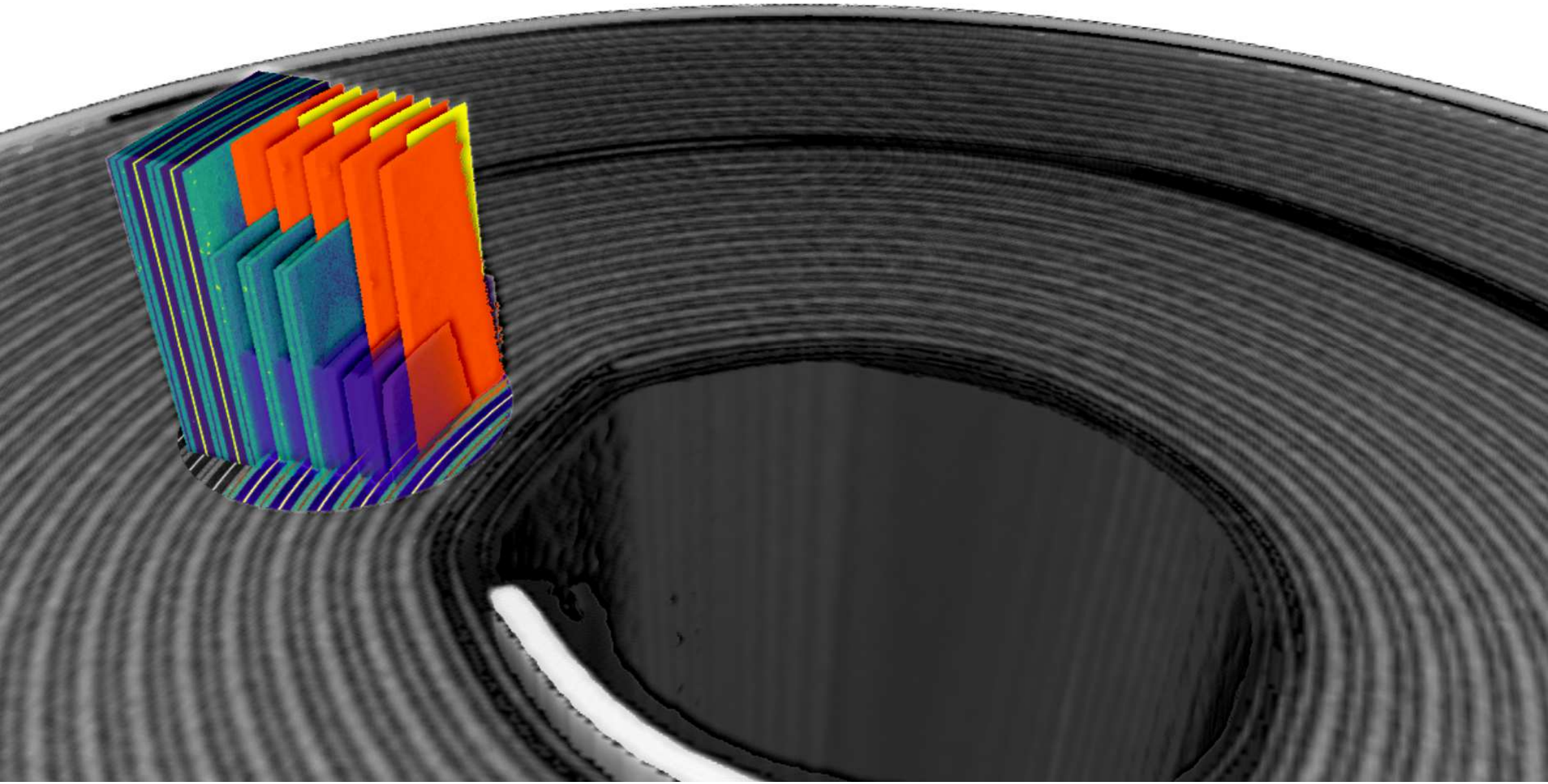
*as of January 2019

18650 Li-ion Battery
High resolution interior tomography

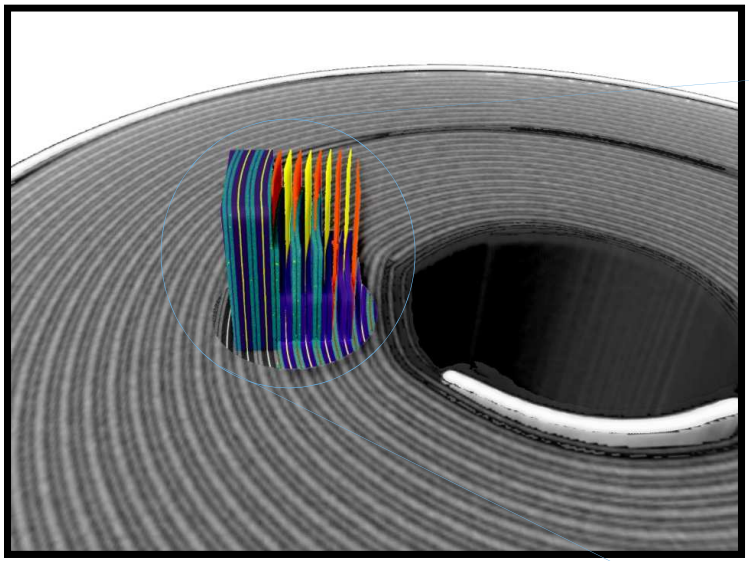


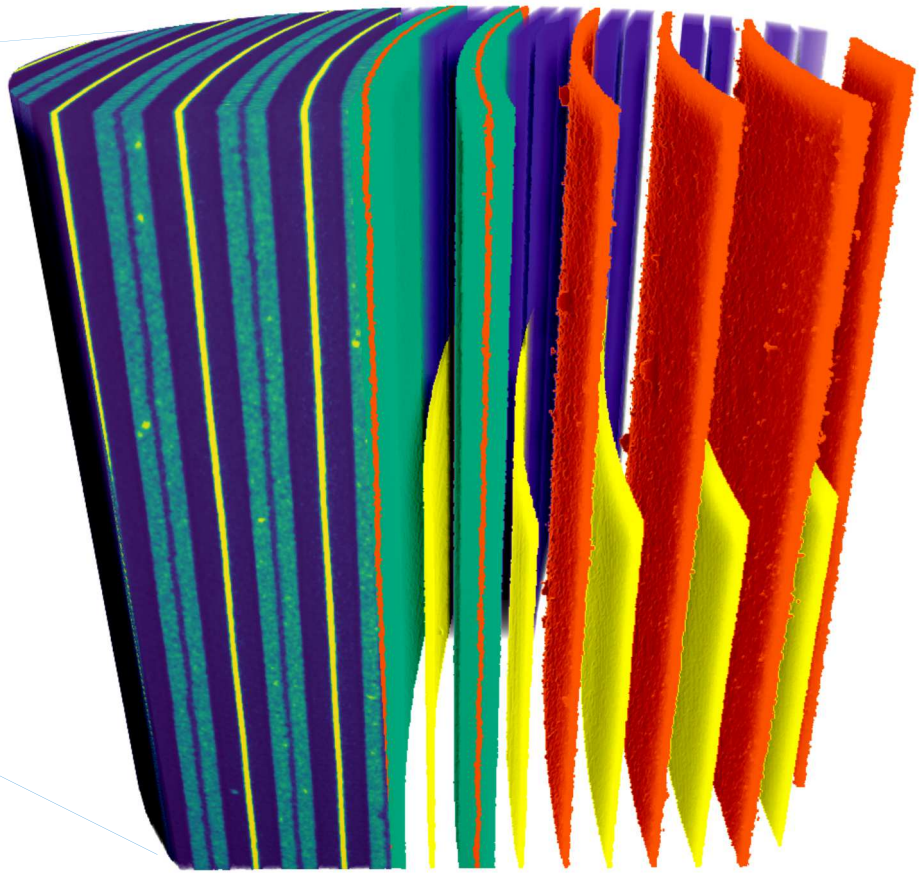
- Intact 18650 Li ion battery





18650 Li-ion Battery
High resolution interior tomography

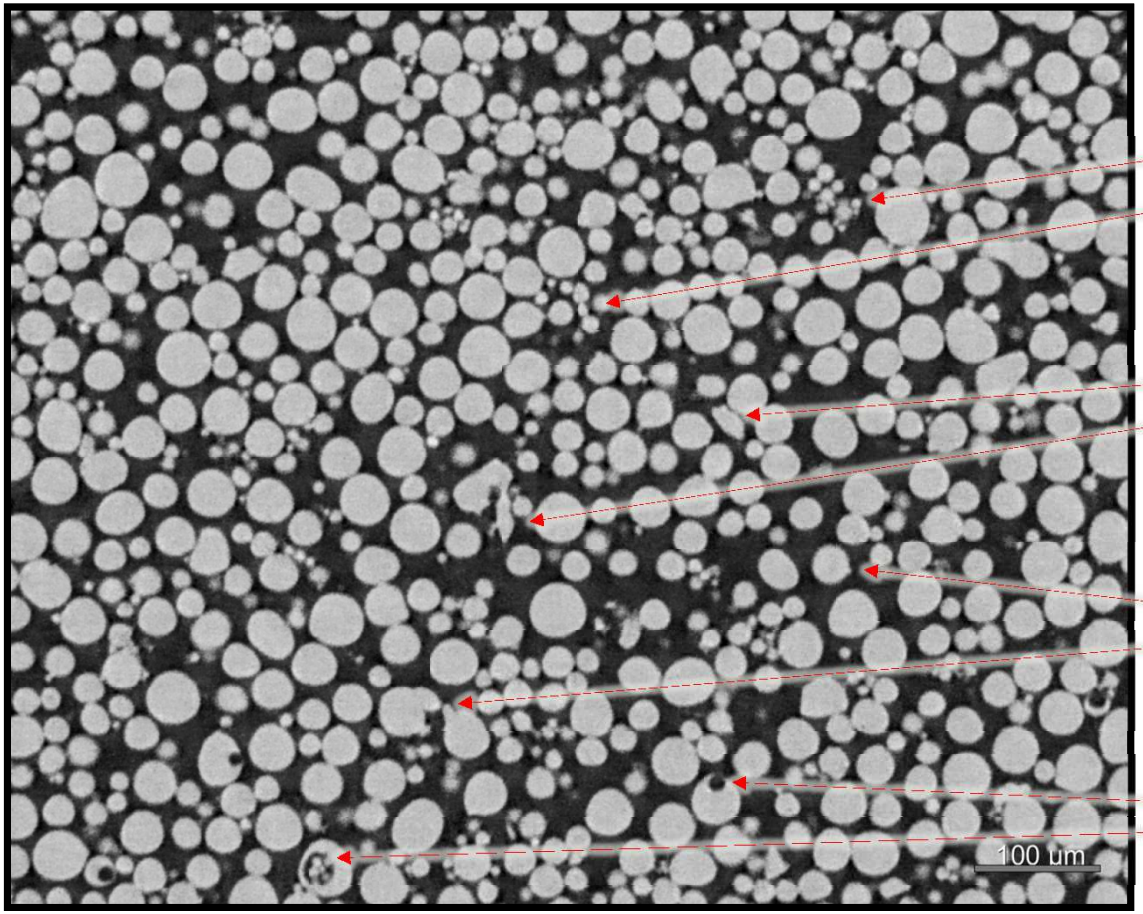




Legend

Cathode
Anode
Al current collector
Cu current collector

Additive Manufacturing
Characterization of feedstock powder

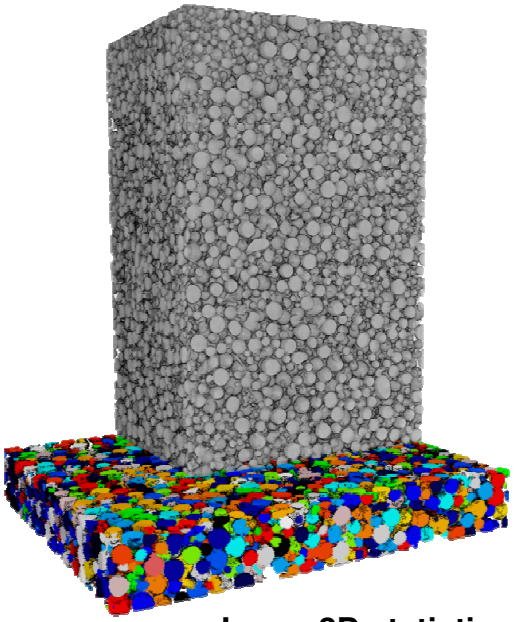


Broad range of particle size

Non-spherical morphology

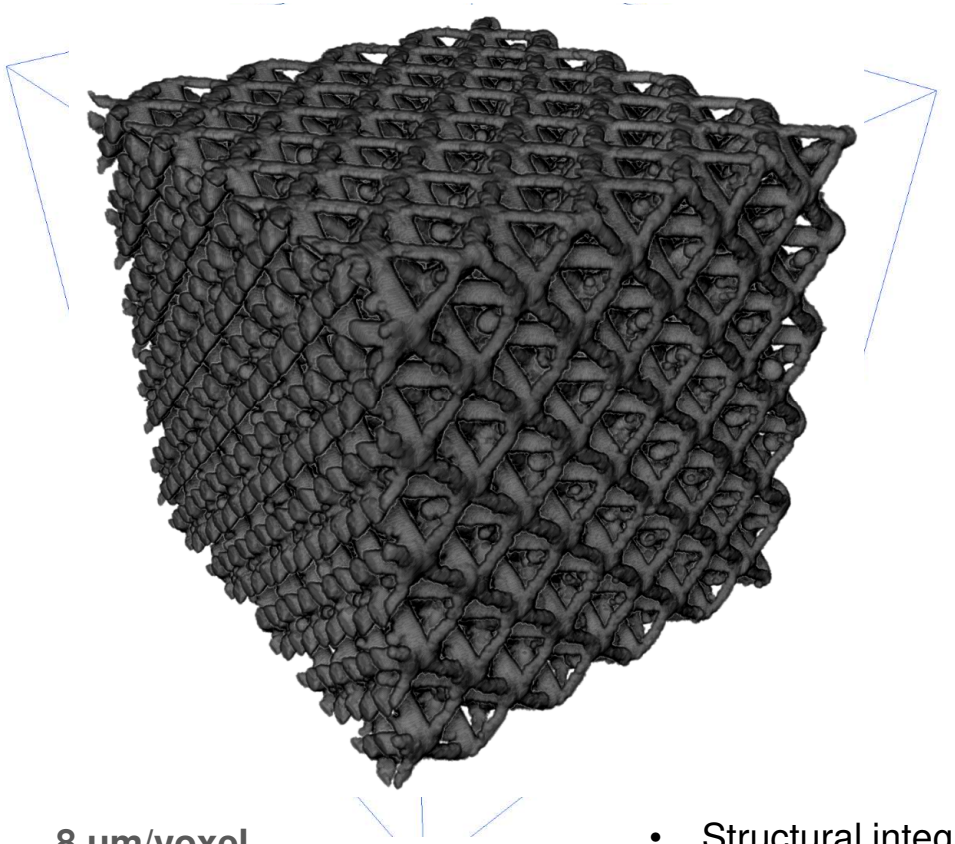
Likely satellite particles

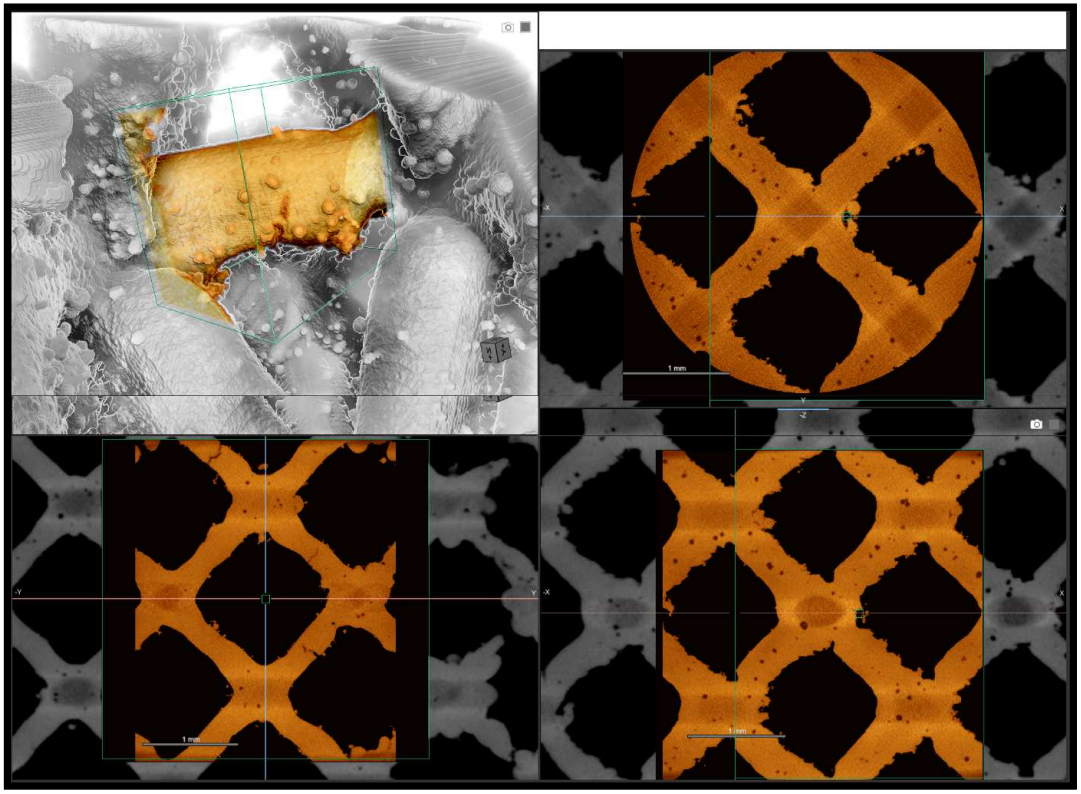
Internal porosity



Large 3D statistics using XRM

Additive Manufacturing
Inconel 3D printed lattice structure





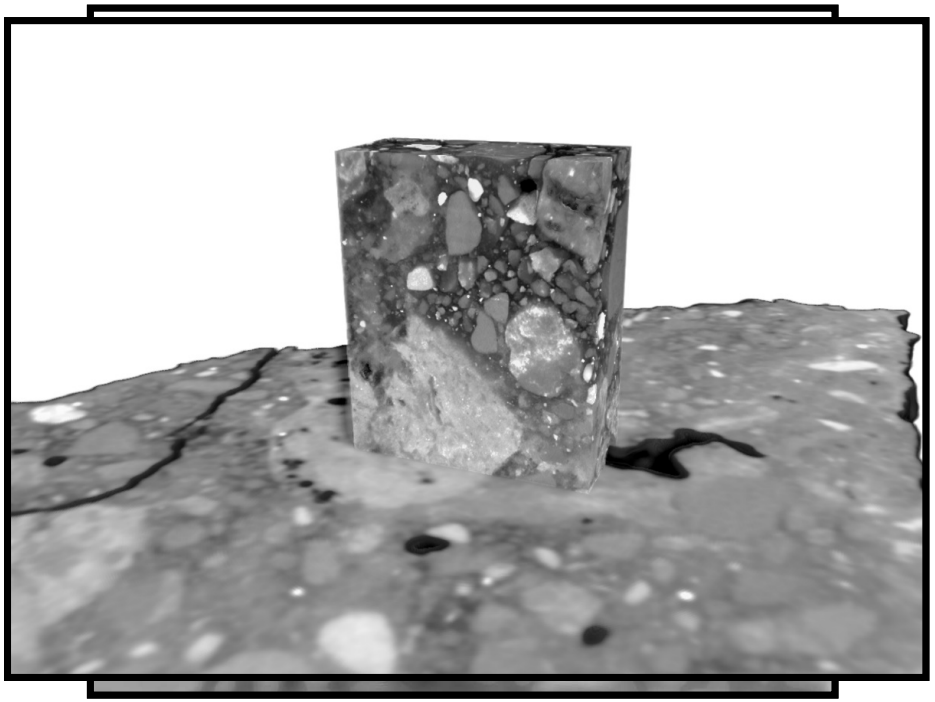
8 um/voxel
3.5 um/voxel

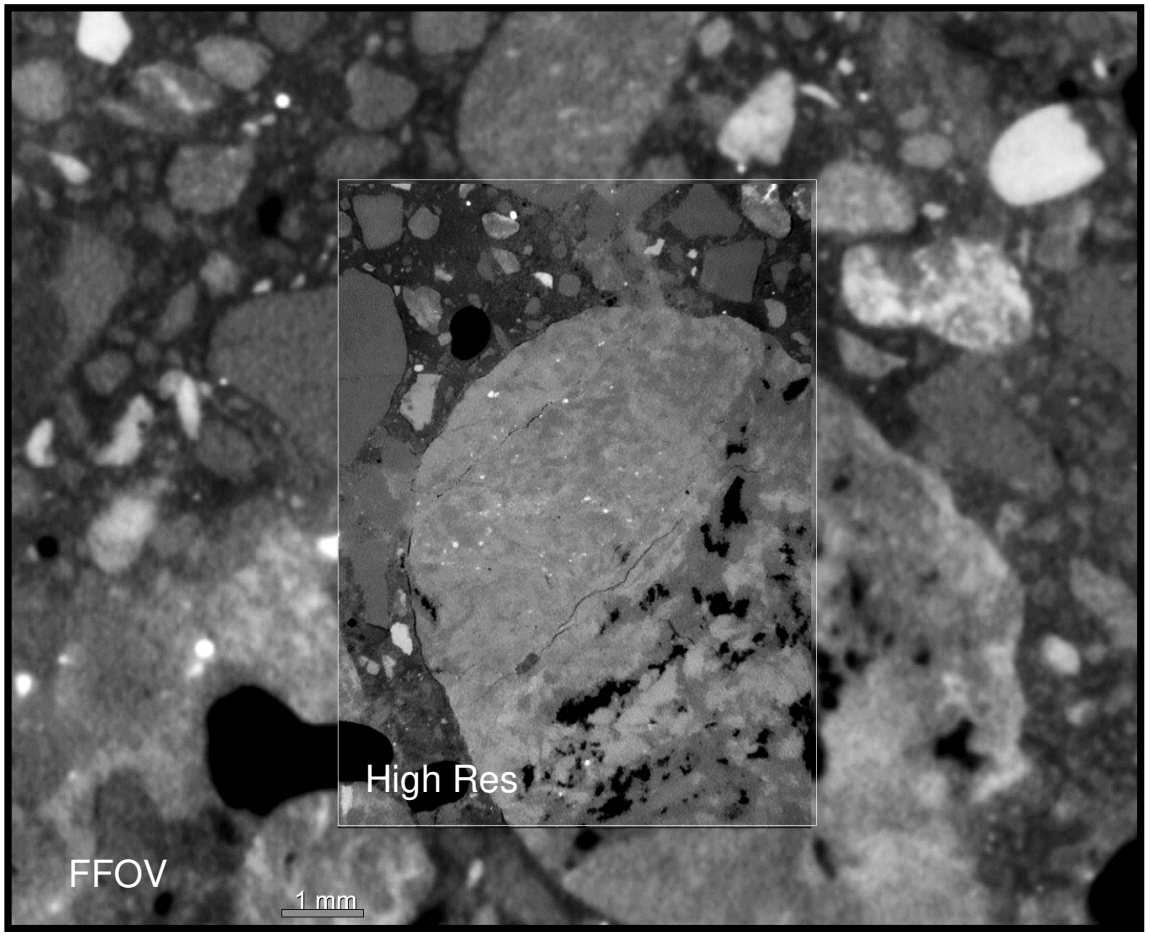
- Structural integrity
- Internal defects (porosity, impurities)
- Surface roughness

Sample courtesy of Kavan Hazeli, Mechanical and Aerospace Engineering, The University of Alabama, Huntsville

Building Materials
Analysis of phases in concrete

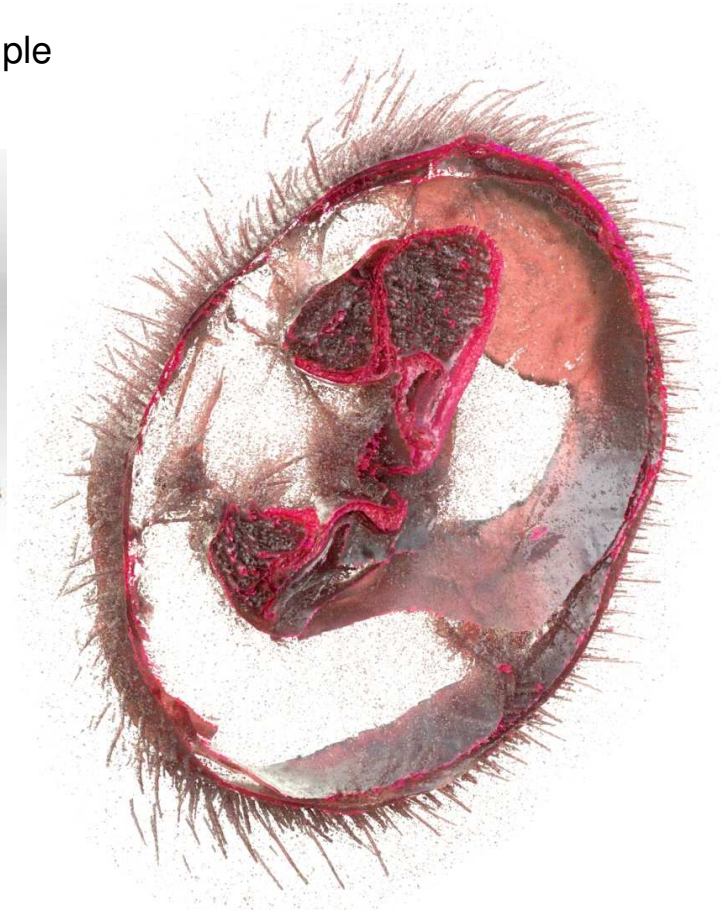
- Interior tomography to target large particle
- Strong absorption contrast reveals numerous solid phases
- Aggregate particles segmented and can be quantified by size, shape, etc.





Virtual Dissection of Precious Samples
High Resolution 3D Image enables Dissection without damage

A digital “mesh” is created from the XRM dataset
Animation or virtual dissection damaging the sample



Ant Sample Courtesy of Brian Fisher, CAS
Image by Graeaex, Inc. (Dave McMahon)
Graeaex@gmail.com

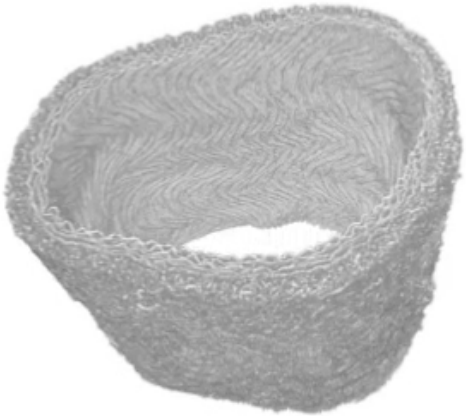
Non-Destructive Species Determination
Classification and Digital Documentation of Specimens

Courtesy of Arjen Speksnijder, Naturalis, Netherlands

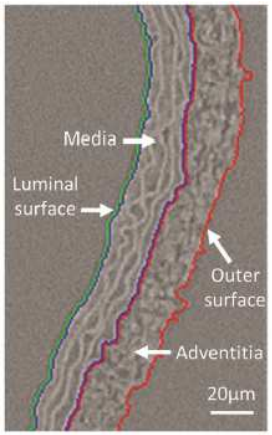
Imaging with XRM Without the Need to Stain

Propagation Phase Contrast Resolves Membranes in Unstained Tissue

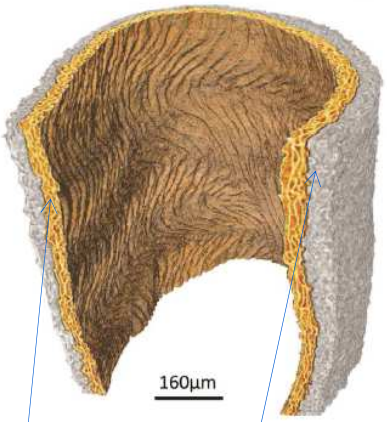
Rat common carotid artery:



Segmenting the layers



Segmentation Result



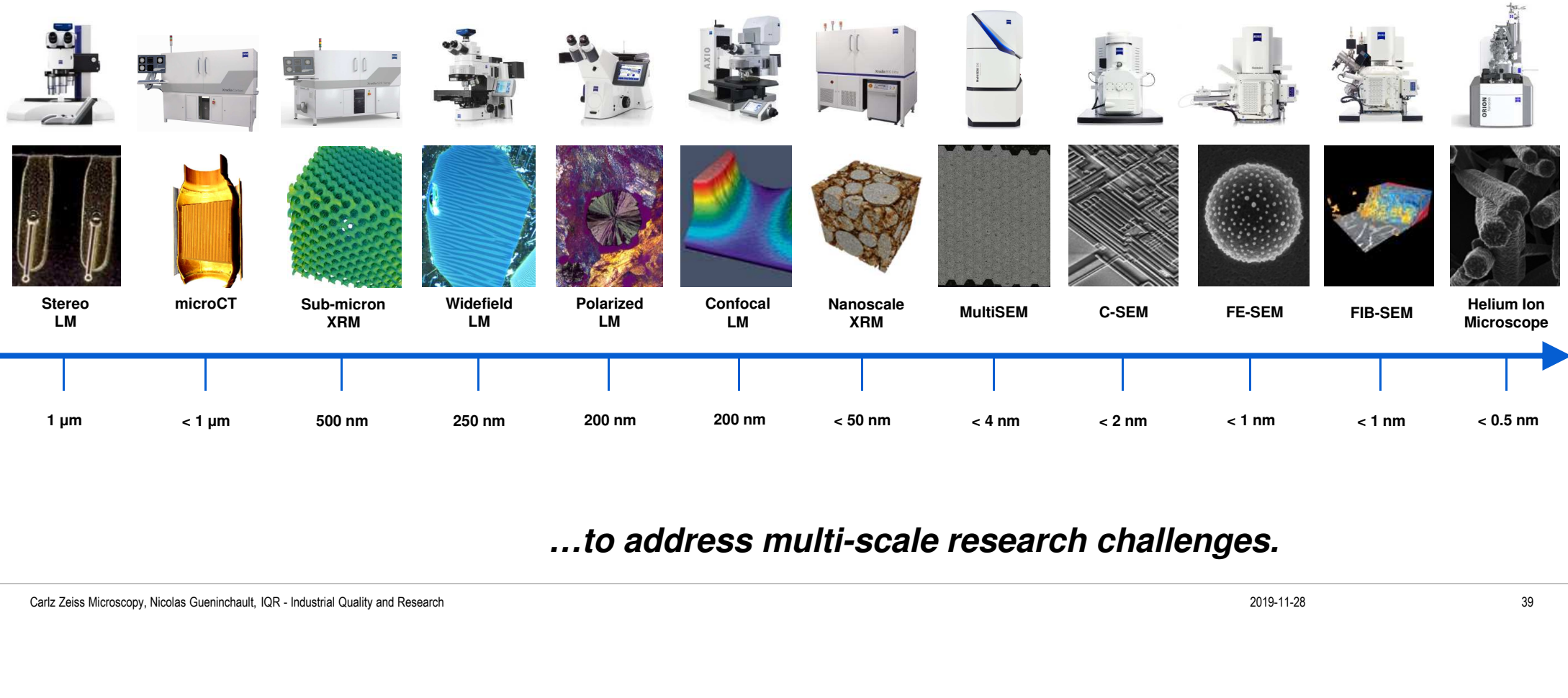
Outer collagen-rich adventitial layer

Inner medial elastic lamellae

Walton, L. et al., Scientific Reports 5:10074 (2015)

ZEISS Microscopy Portfolio
Multi-Scale Characterization for Multi-Scale Research

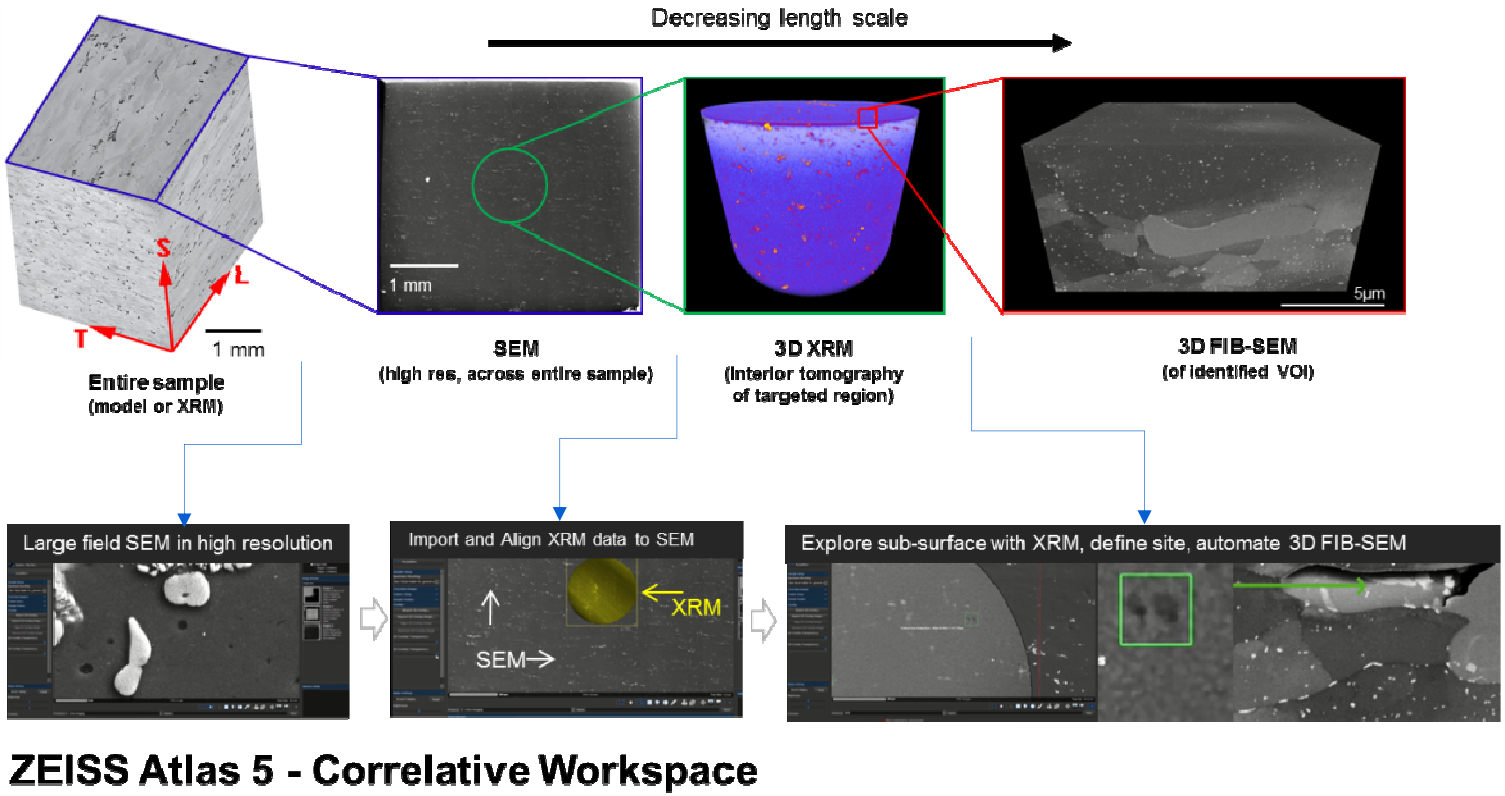
A complete microscopy portfolio…



…to address multi-scale research challenges.

Aluminum 7075 Multi-scale Study
Atlas 5 Correlative Workspace Example

First appeared as Merkle, et al., *Microscopy & Analysis* 134 (2014) S10-S13

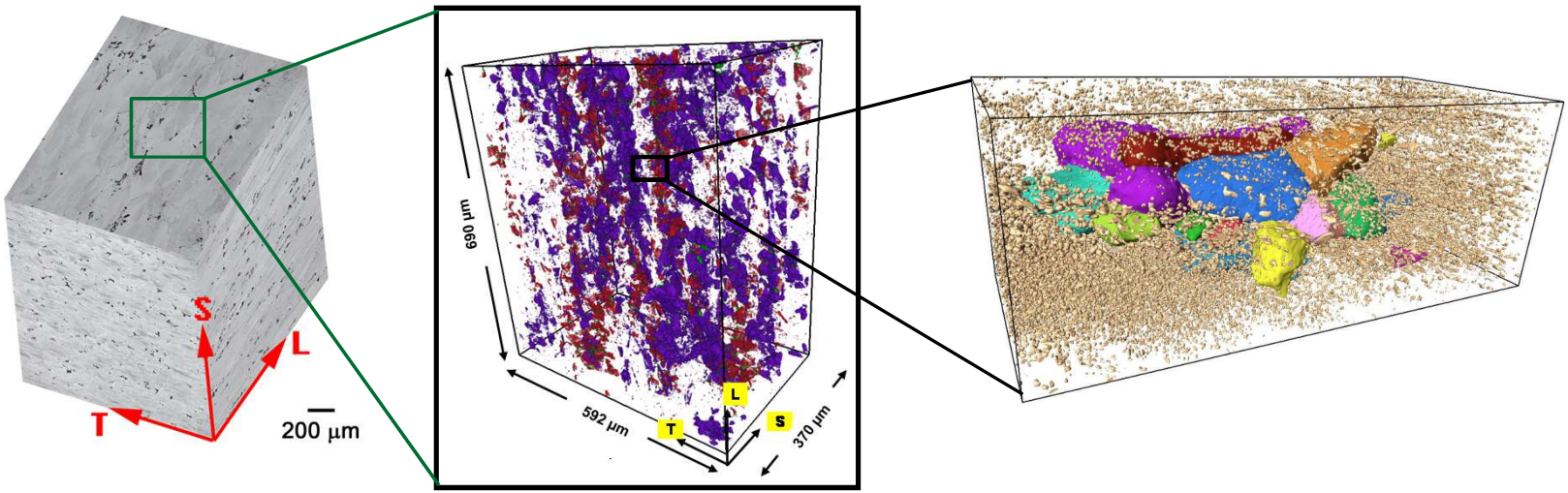


ZEISS Atlas 5 - Correlative Workspace

AA7075 – Linking XRM with FIB-SEM

XRM Data used to identify a representative volume element (RVE)

Decreasing length scale



X-ray microscopy

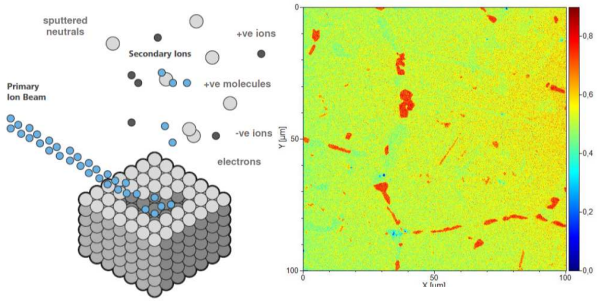
- Grain size & shape
- Inclusion distribution
- ROI / RVE identification

FIB tomography

- High-resolution localization
- Nano-scale precipitates
- Grain contrast

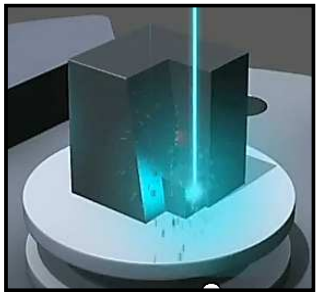
*S. Singh et al., Materials Characterization **118** (2016).*

Adding Imaging modalities



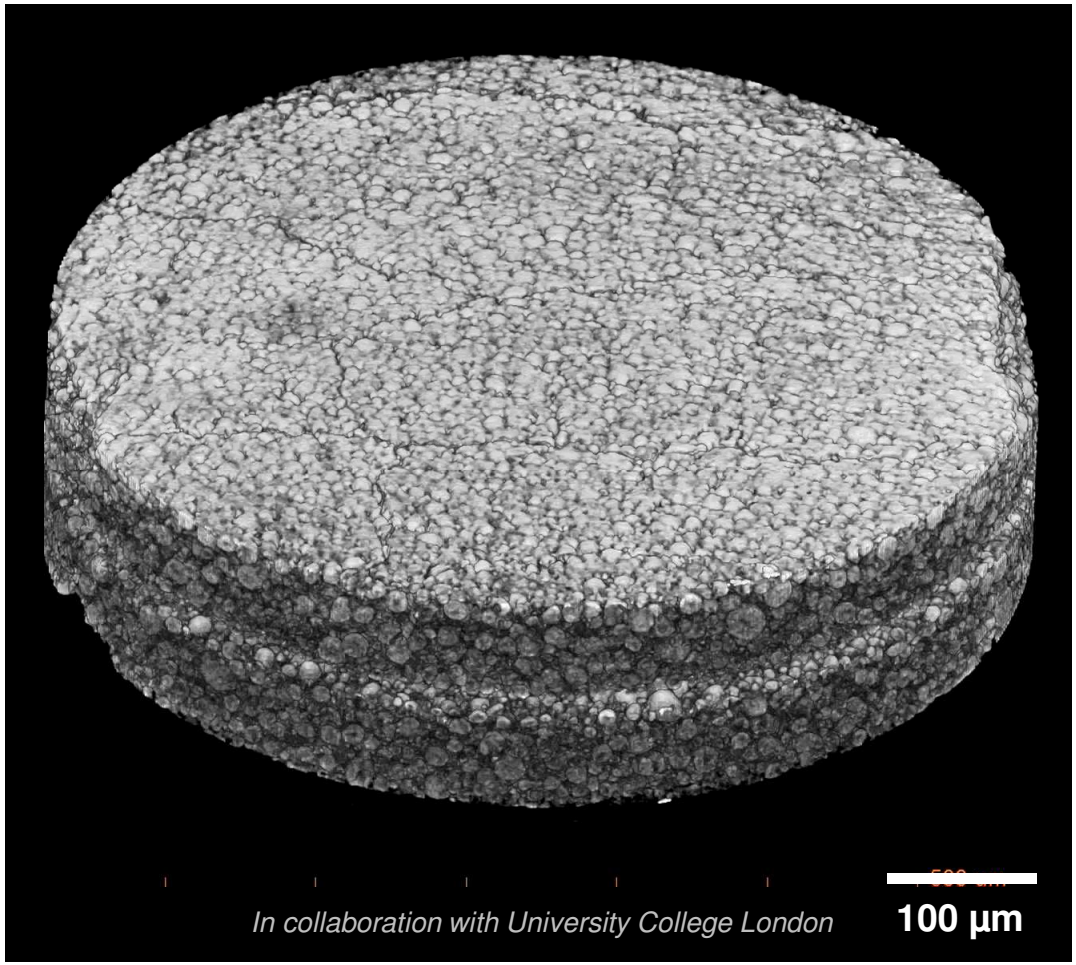
TOF-SIMS

Digging Deeper



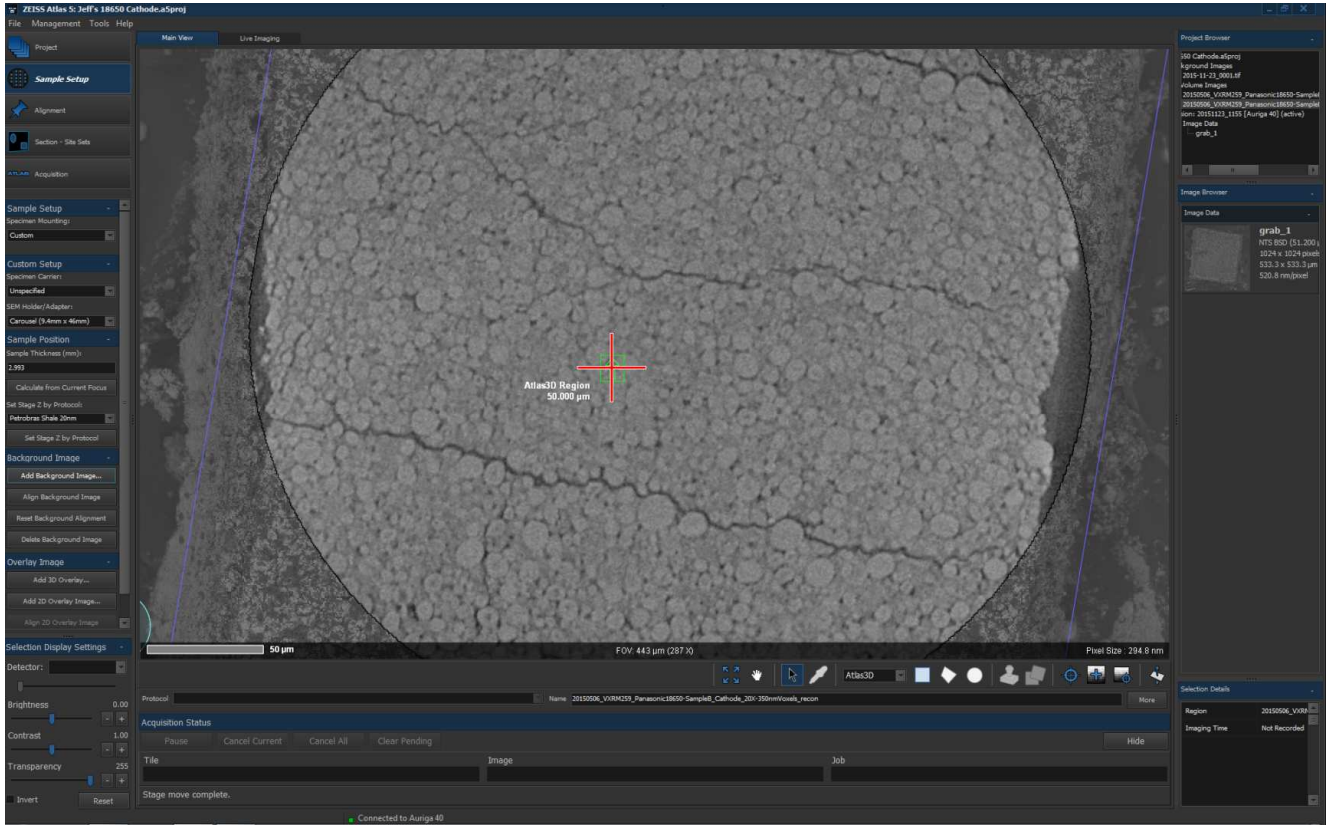
Femtosecond Laser

ZEISS Xradia 520 Versa Imaging: 3D Survey
Depackaged NCA Cathode



Correlative FIB-SEM Analysis
Using Atlas 5

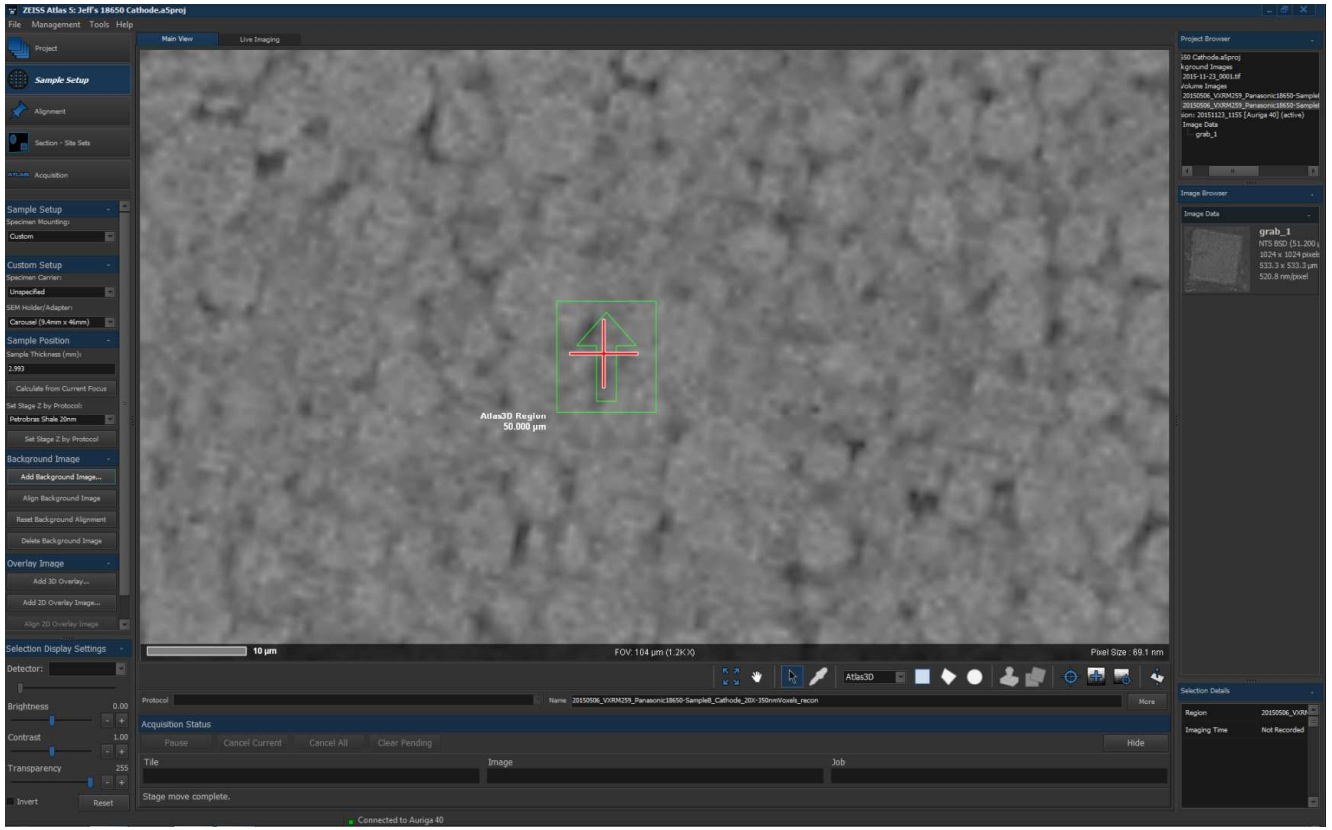
XRM data was used to identify a representative region within the cathode specimen:



In collaboration with University College London

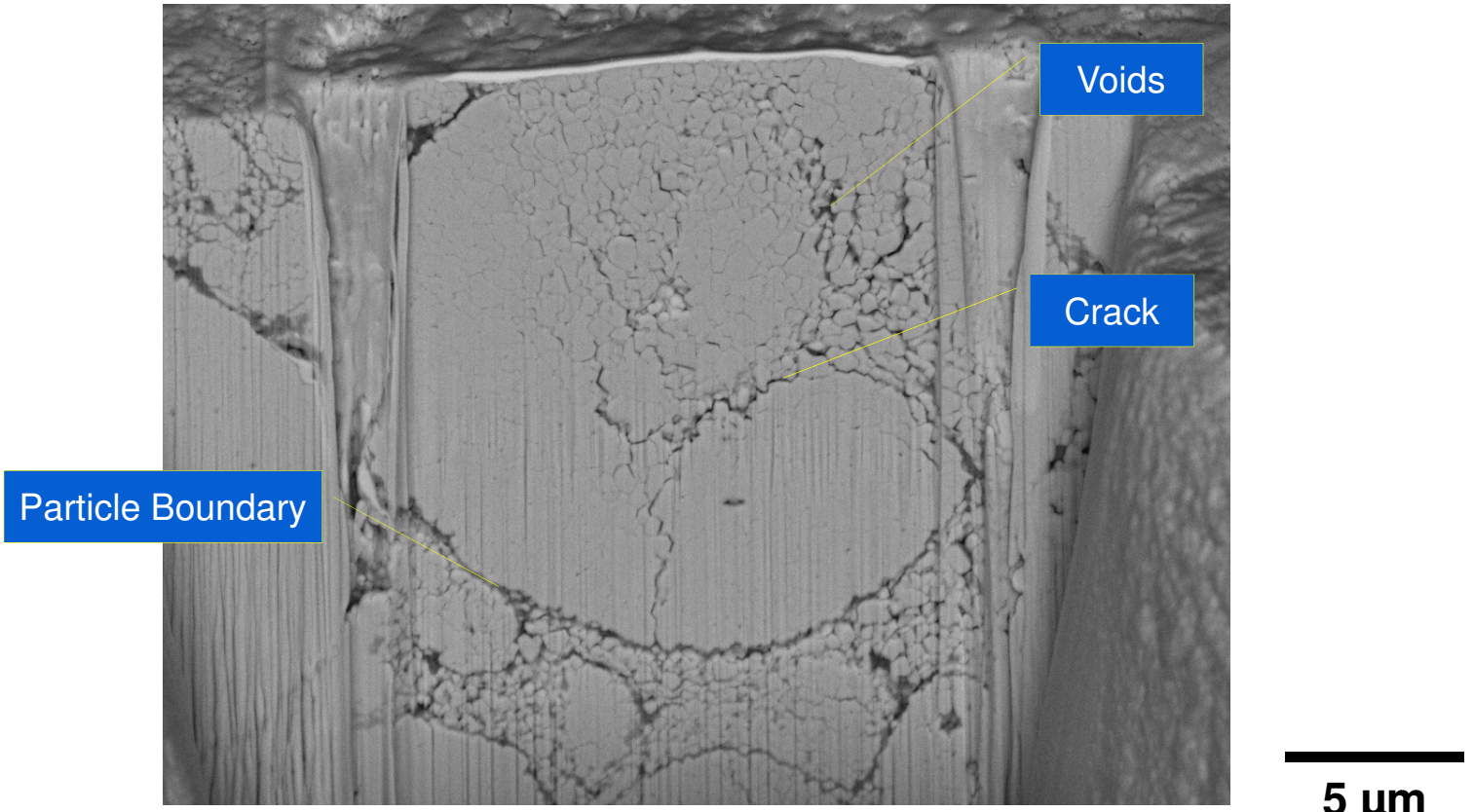
Correlative FIB-SEM Analysis
Using Atlas 5

Identified cathode particle that indicated signs of an internal crack below the surface:



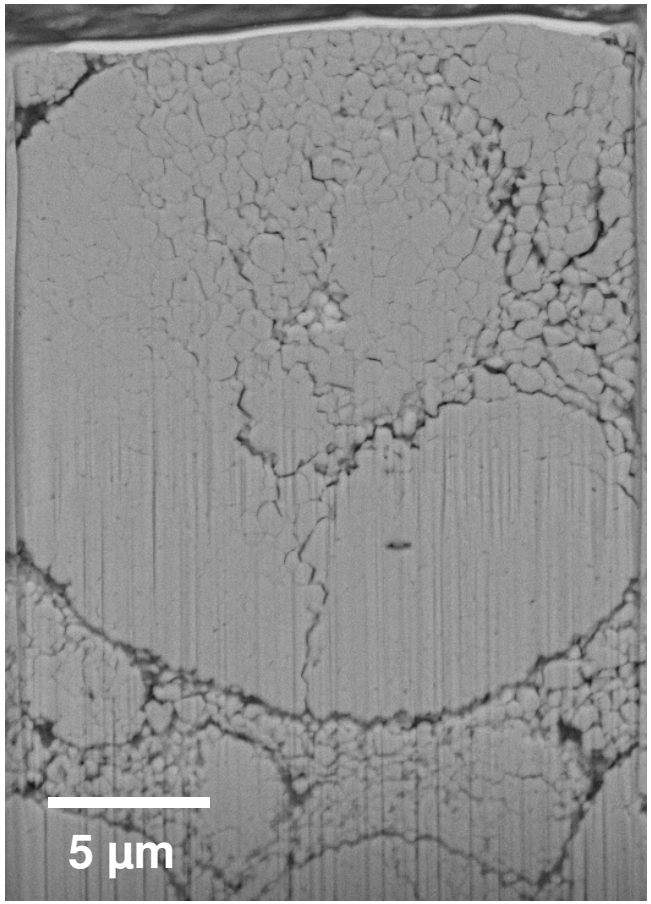
In collaboration with University College London

FIB-SEM Cross-Sectioning
Targeted ROI from XRM Data

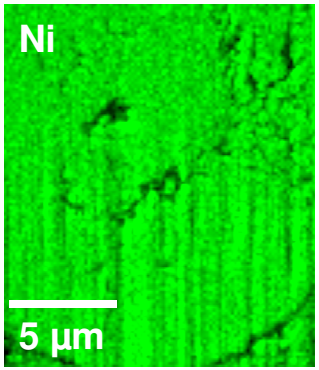


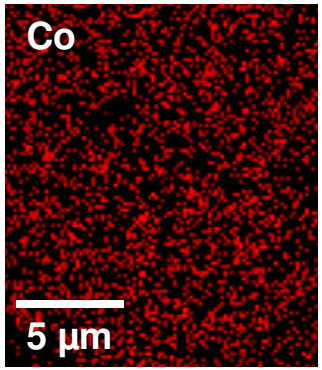
In collaboration with University College London

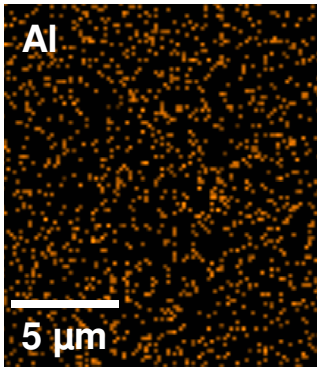
Correlative Chemical Analysis
Targeted ROI from XRM Data

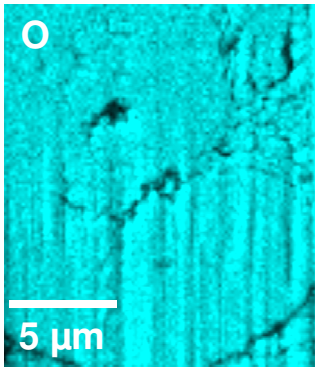


- Hayashi et. al. *JECS* (2014): $LiNi_{0.82}Co_{0.15}Al_{0.03}O_2$
- Mapped composition with EDS at 20 kV, fit to Hayashi data









Element	Wt%
Ni	30%
Co	7%
Al	1%
O	61%

In collaboration with University College London

Aluminum 7075 Multi-scale Study

XRM→ Atlas 5 → XB Correlative Tomography Results

Visual SI Advanced used for Visualization & 3D Rendering

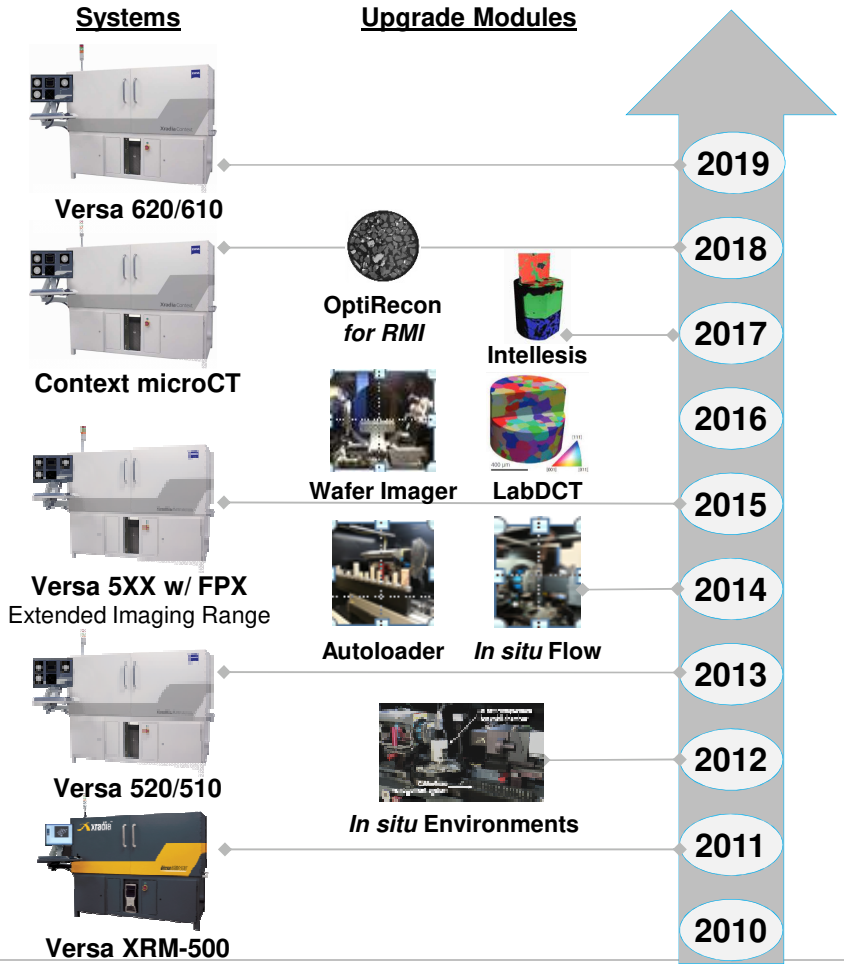
Results


ZEISS

Protect Your Investment
Unprecedented extendibility, unrelenting support

All X-ray systems (since 2011) are field upgradable

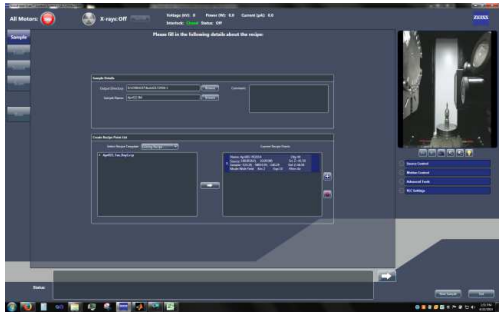
- Upgrade and conversion options keep your investment on the leading edge
- New SW features for continuously enhancing system capabilities:
  - Python Scripting API
  - Scout-and-Zoom enhanced workflow
  - LabDCT v2.0
  - Autoloader Speed-up
  - Adaptive Motion Compensation
  - Vertical Stitching v2.0
  - DSCoVer v1.5
  - Scout-and-Scan™ Control System



Scout-and-Scan™ workflow-based GUI
ZEISS Xradia control system

Intuitive, easy-to-use workflow interface

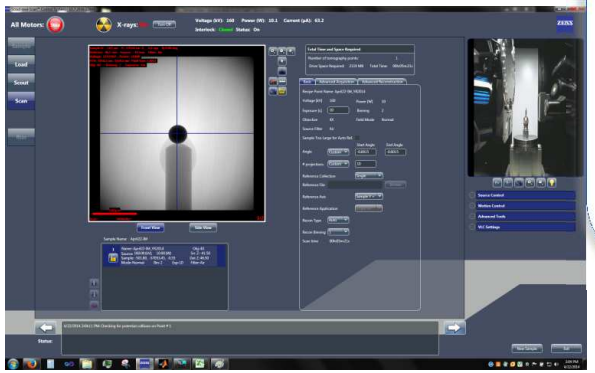
1) Enter Sample Details

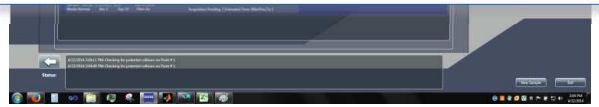


2) Load and Center Sample

3) Position Region of Interest

4) Set-up Exposure Parameters





Versa now with Python API

For scripting ‘unusual’ acquisition, unique scanning strategies, accessing metadata and instrument states, advanced and automated *in situ* experiments

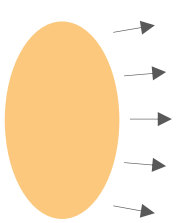
Time for More Photons
Turning up the power

With microCT: **High GeoMag makes you very sensitive to spot size and power**

Only an **X-ray microscope** can

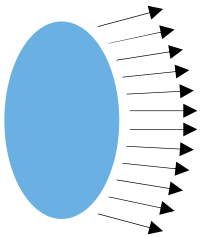
- Achieve high resolution at low GeoMag & larger working distances using RaaD
- Remove sensitivity to X-ray source spot
- Provide stable performance at high resolution for extended time
- Increase power without compromising resolution
- It's not about spot size anymore!

Versa 5XX series
10 W



Increased Power
No Compromises

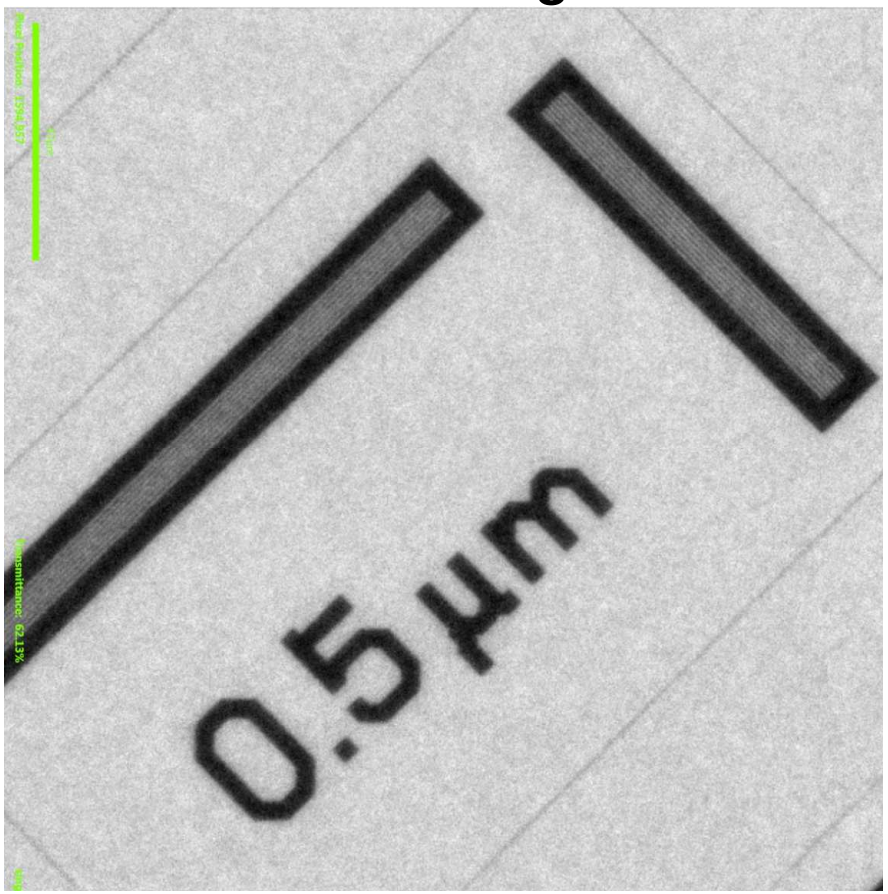
Versa 6XX series
25 W



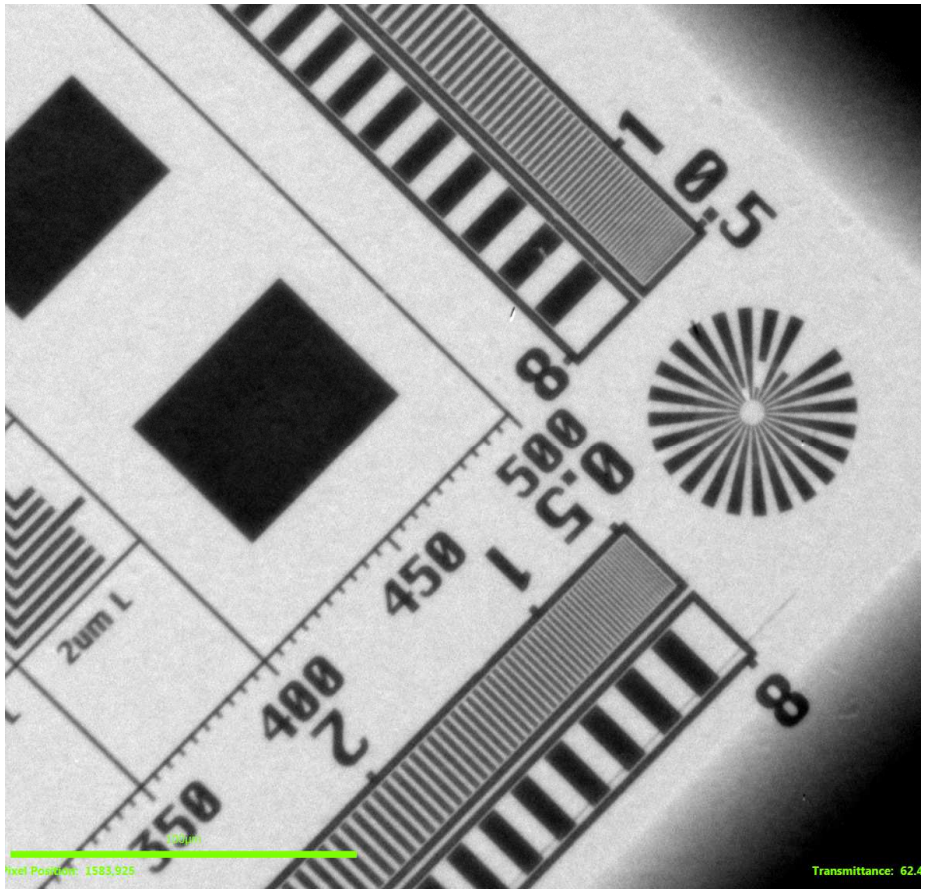
ZEISS Xradia 600-series Versa: Spatial Resolution

Spatial resolution: 500 nm
Minimum voxel size: 40 nm

JIMA Target



X500 Target



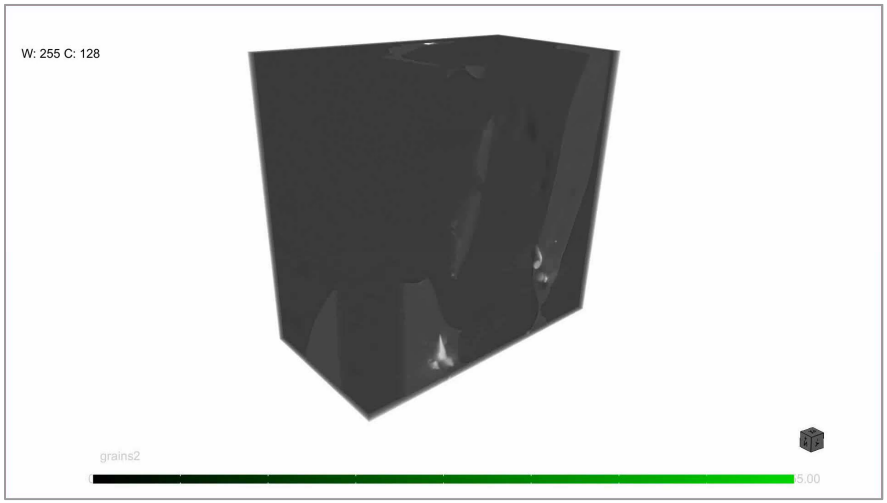
3D Characterization of Crystalline Material Microstructures
Absorption tomography + Diffraction Contrast Tomography (LabDCT)

Al-%4Cu alloy – inclusions and grain microstructure

Courtesy of Prof. Masakazu Kobayashi, Toyohashi University of Technology

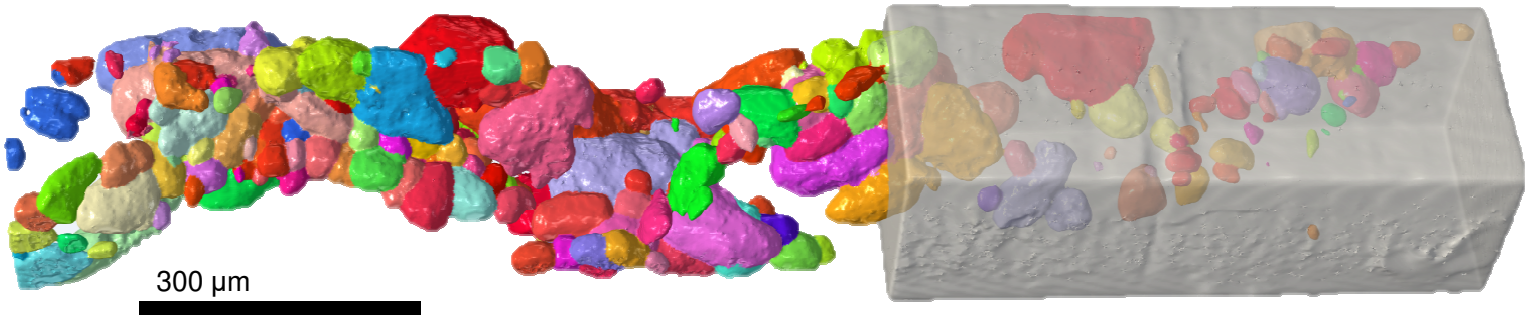
Grain structure in geological materials

Pankhurst *et al.* In Prep

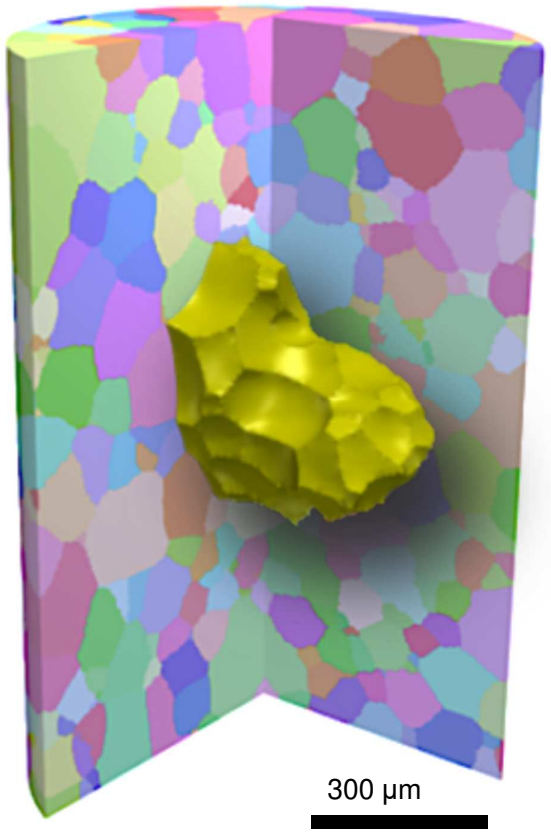


Partial recrystallization in AlSi

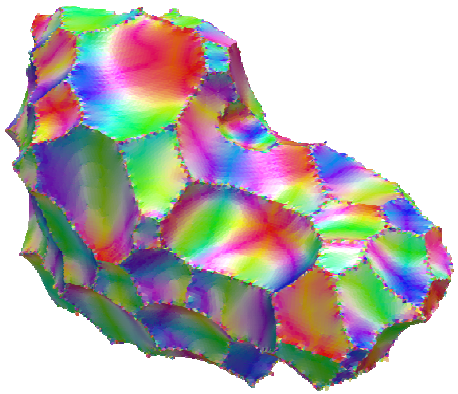
Courtesy of Prof. Dorte Juul Jensen, DTU



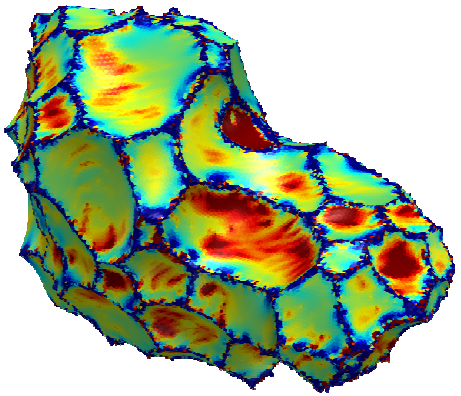
Annealing and Grain Growth
Grain boundary analysis in Armco Iron



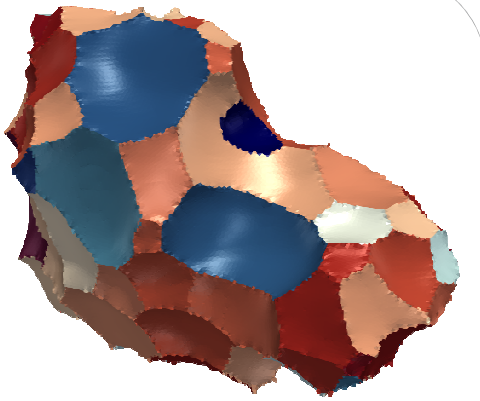
- Abnormal grain growth can lead to anomalous properties
- Diffraction contrast tomography (LabDCT) measures morphology and crystallography of grain structures in 3D
- Grain boundary characterization (inclination, orientation, curvature) can be measured to understand grain growth behavior
- 4D analysis (3D + time) to observe evolution of grain structure through treatment



Grain boundary normal



Grain boundary curvature



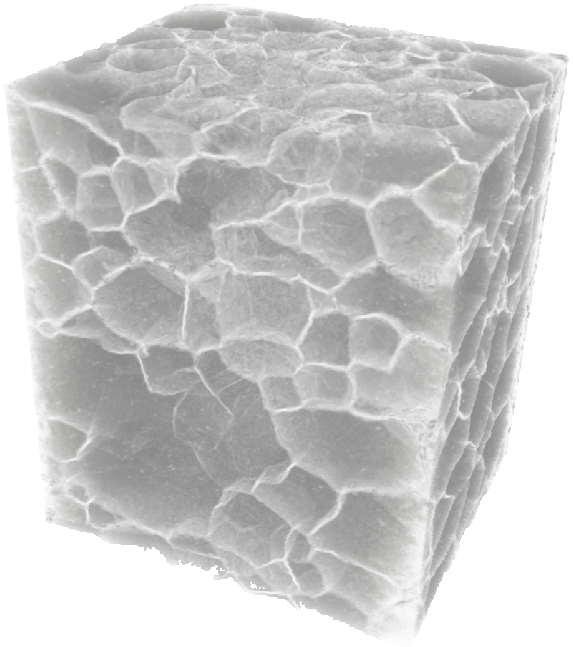
Grain boundary misorientation

Prof. Burton R. Patterson, University of Florida

LabDCT User Story:
Grain boundary wetting correlated to grain boundary energy

Preferential penetration of liquid Ga in the grain boundaries of aluminum

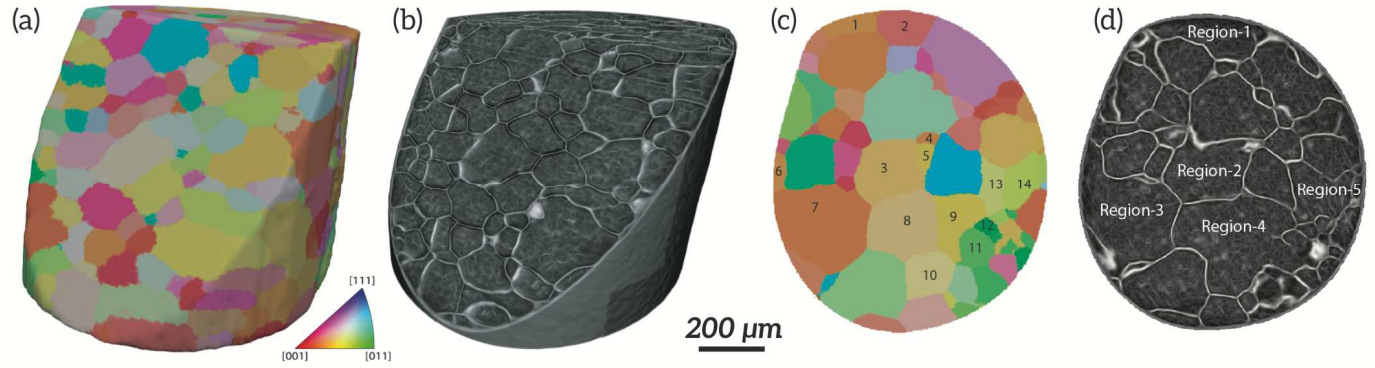
Scripta Materialia, Vol 163, 2019, pp 77-81



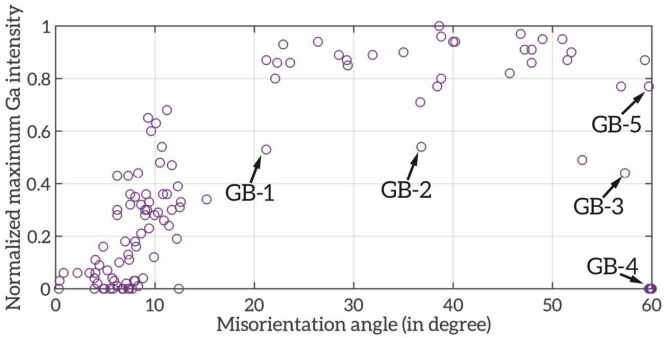
Ga-wetted grain boundary network in the Al matrix

Courtesy of Dr. Yubin Zhang, DTU, Denmark.

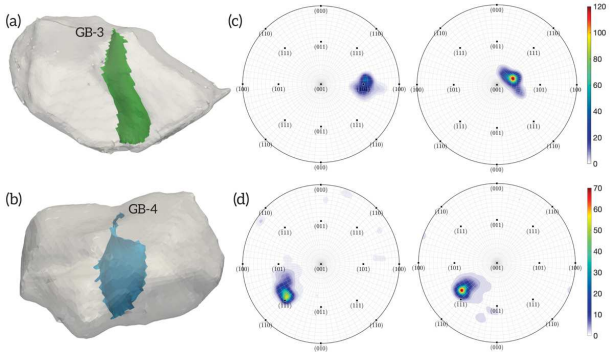
Comparison between Diffraction and Absorption Contrast Tomography



Ga intensity vs. misorientation angle

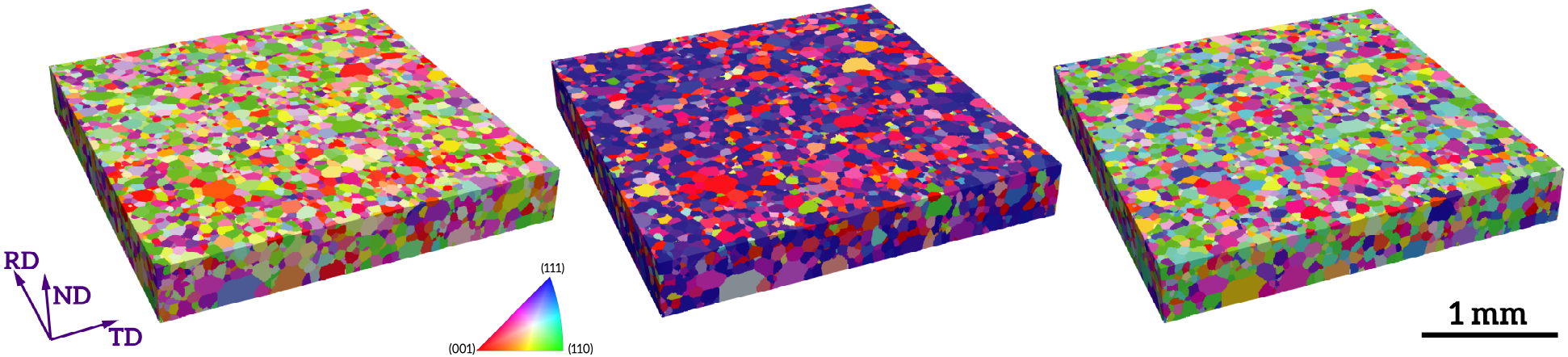


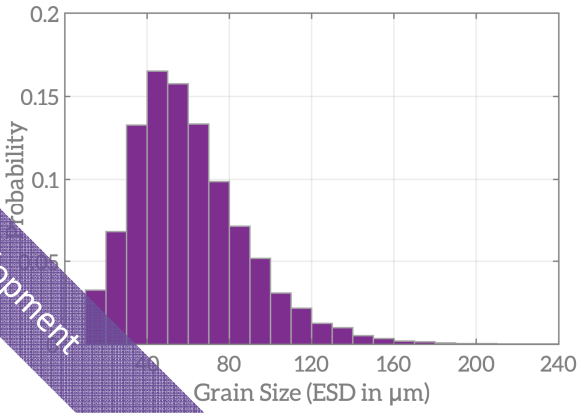
GB plane normal distribution

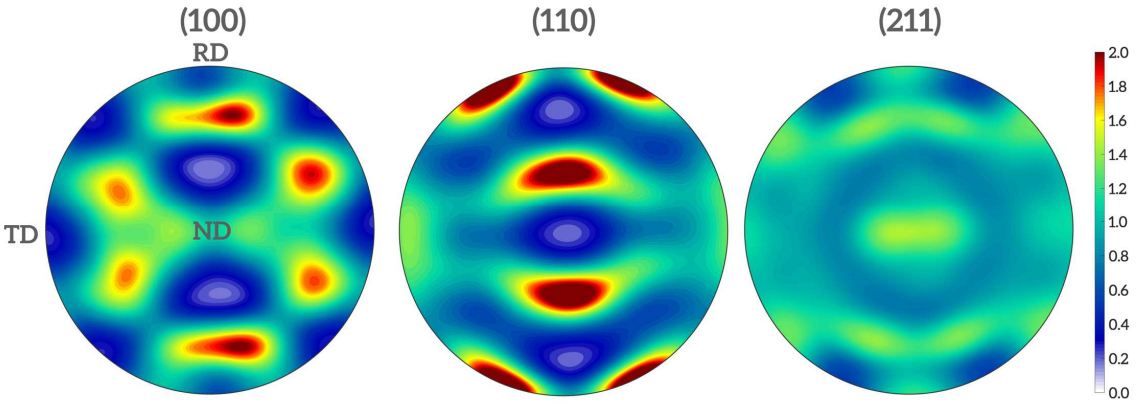


LabDCT User Story:
Large grain statistics in non-oriented electrical steels

3D grain maps of a steel sample, containing over 17,000 grain. *(Left to right: IPF color coded based on RD, ND and TD).*







Ongoing development

Courtesy of Dr. Ivan Petryshynets, Slovak Academy of Science.