Opportunities in 3D and 4D Imaging with X-ray Microscopy
In the materials research laboratory

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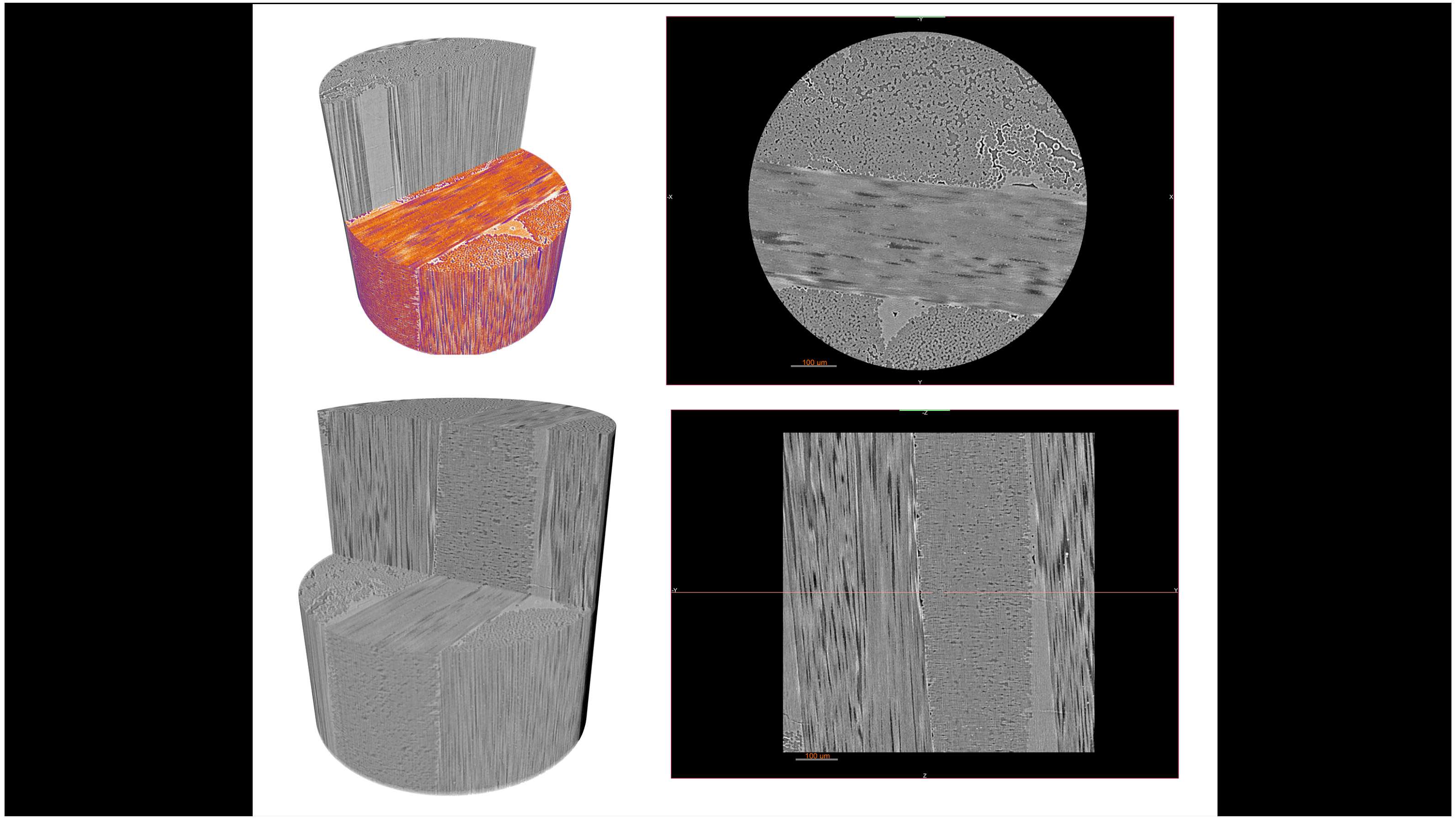
Electron, Ion & X-ray Microscopy Specialist – Materials

X-ray Microscopy Workshop
Purdue University
09 July 2019

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

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



XRM - Carbon Composite (video)

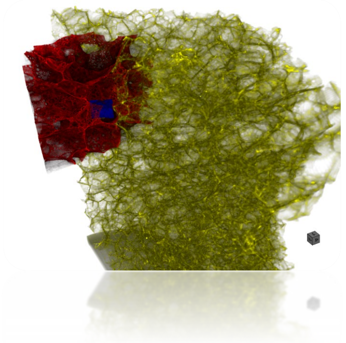
XRM - Li-ion batter (video)

Ceramic matrix composite, imaged at *in situ* working distance with RaaD

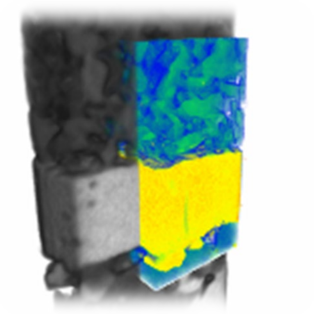
Sample courtesy of Dr. David Marshall, University of Colorado, Boulder

Agenda

Overview of ZEISS X-ray Microscopy in Materials Science



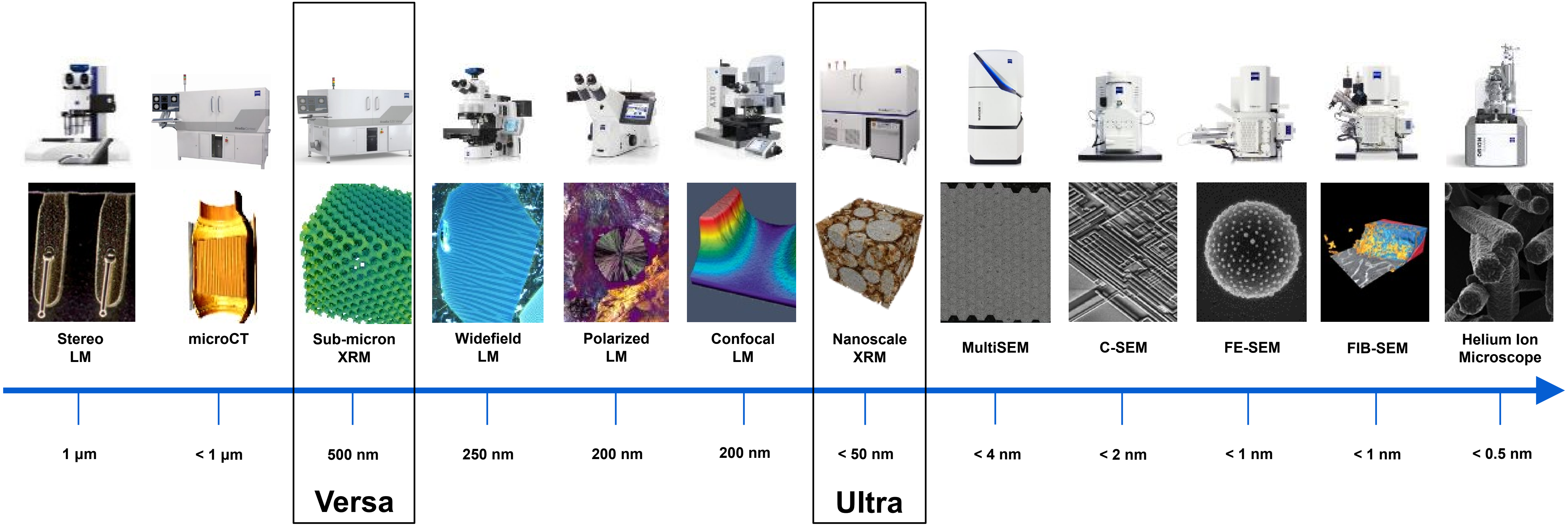
ZEISS Xradia Versa – Technology and Applications



ZEISS Xradia Ultra – Technology and Applications

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

A complete microscopy portfolio…



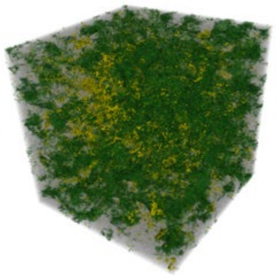
…to address multi-scale research challenges.

ZEISS X-ray Microscopy
Background

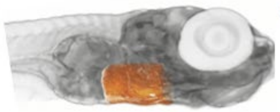
- Xradia was founded in 2000 and acquired by ZEISS in July 2013
- Pioneer in ultra-high resolution 3D X-ray microscopy for Synchrotron
- **Uniquely brought synchrotron 3D imaging performance to lab based instruments**
- Used by premier scientists and industrial researchers worldwide

Materials Science

Natural Resources

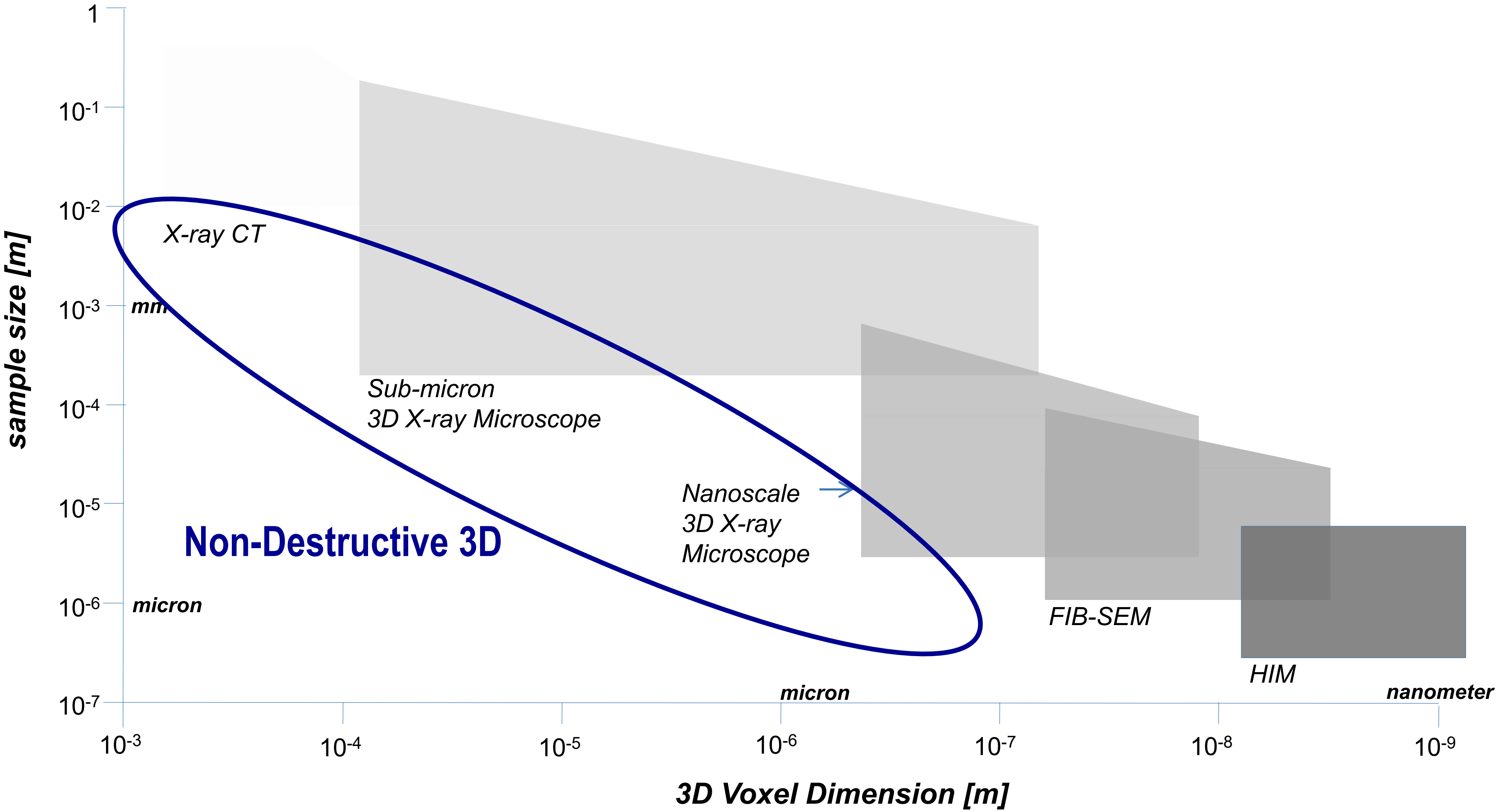


Life Sciences



Electronics

3D Imaging Technologies



ZEISS X-ray Solutions Portfolio
Solutions for every size and application

General research

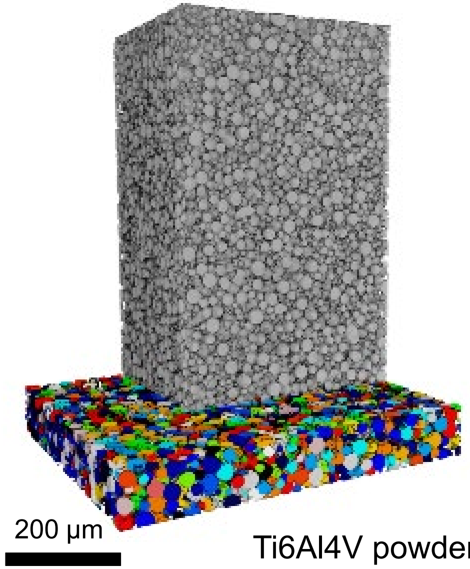
Product-and-process development

Parts manufacturing, Assembly in volume production

	ZEISS Xradia Versa/Ultra X-ray microscopes (XRM)	**ZEISS Xradia Context** microCT	**ZEISS METROTOM** Computed tomography	**SRE MAX from Bosello** a company of the ZEISS group	**ZEISS VoluMax** Computed tomography
Application	High-resolution detail analysis, 3D	Large field of view, full context sub-micron imaging, 3D	Measuring and evaluating entire components	Inspection with high throughput, 2D	Inspection with high throughput
Particular strengths	Resolution in the submicron and nano range	Projection-based geometric mag. High resolution for small samples Field convertible to Versa XRM	Standard-compliant and traceable precision Testing as per VDI/VDE 2630, Evaluation with ZEISS CALYPSO	Customized product design, 2D Radioscope with easy handling Comprehensive project experience	Customized product design, Inline & Atline Computer tomograph Comprehensive project experience
Place of use	Lab	Lab	Measuring lab	In production and near production	In production and near production
Resolution	500 nm (Versa), 50 nm (Ultra)	0.95 µm	3.5 – 6 µm	100– 400µm	3.5– 400µm
Speed	Hours	Hours-Minutes	Minutes	Seconds	Seconds

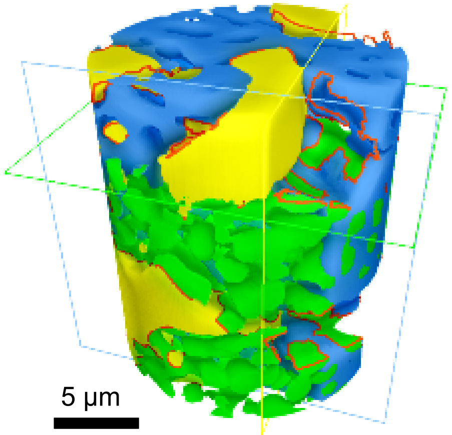
ZEISS X-ray Microscopy for Materials Science
Overview

- **Non-destructive, state of the art 3D imaging**
  - *Highest resolution*
    - Study structures with down to 50 nm resolution
    - Maintain resolution across a wide range of sample sizes
  - *Highest contrast*
    - Synchrotron technology adopted for a lab-source
    - Available 24/7
- **Wide range of sample volumes for multi-scale, hierarchical materials ($cm^3 \rightarrow \mu m^3$)**
- **Unique modalities**
  - 4D material evolution studies, 'time-lapse'
  - High resolution studies within flexible array of *in situ* environments
  - Navigate & correlate:
    - Precursor to complementary and efficient physical sectioning with Crossbeam (3D FIB-SEM)



Ti6Al4V powder for AM

Xradia 620 Versa
Sub-micron 3D X-ray Microscope

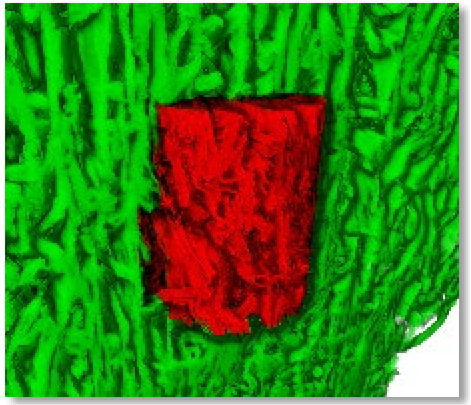


Fuel cell electrode

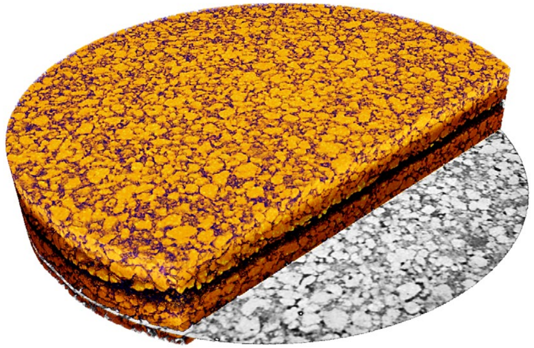
Xradia 810 Ultra
Nanoscale 3D X-ray Microscope

Materials Science

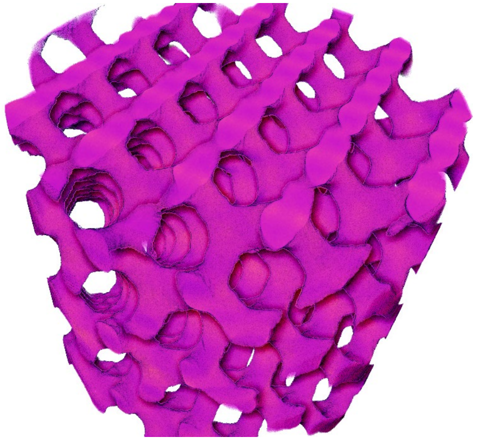
Applications for X-ray microscopy



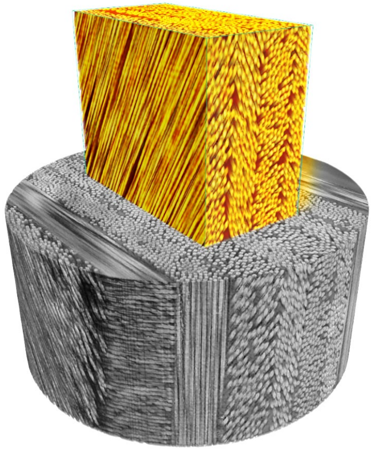
Polymers & Biomaterials



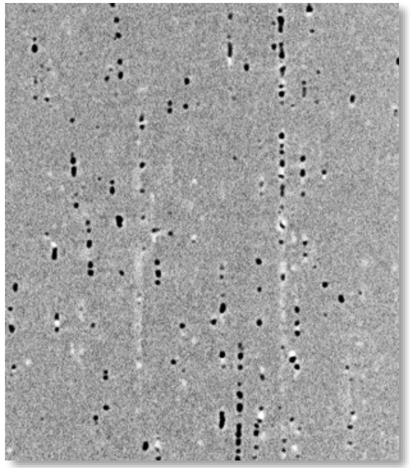
Energy Materials



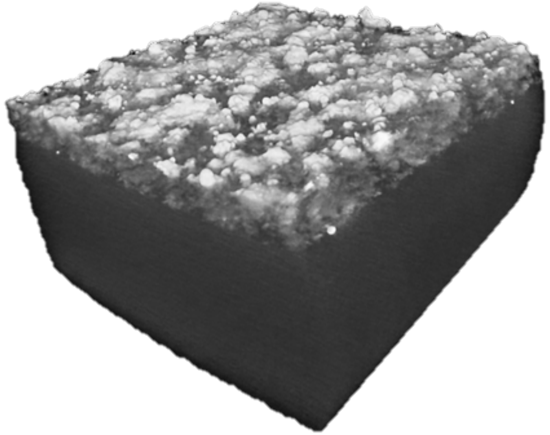
Ceramics



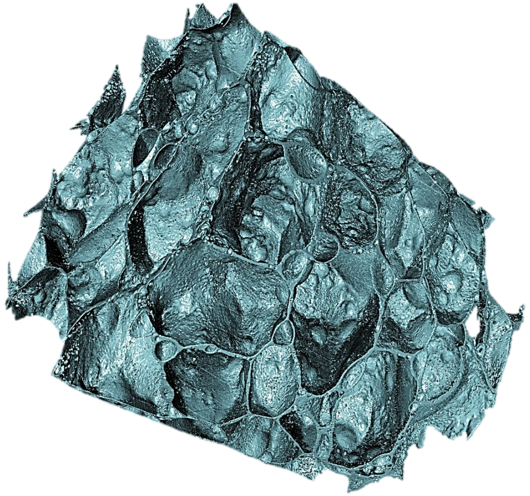
Composites



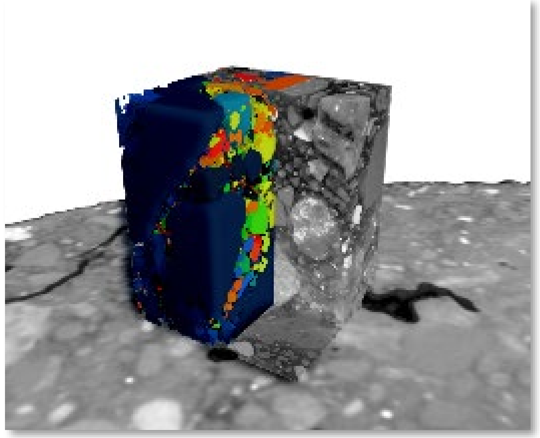
Metals



Coatings



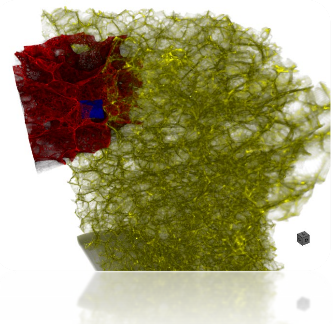
Glass



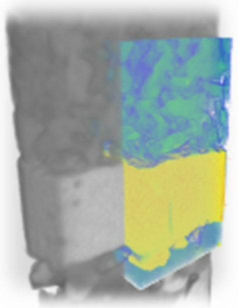
Concrete

Agenda

Overview of ZEISS X-ray Microscopy in Materials Science



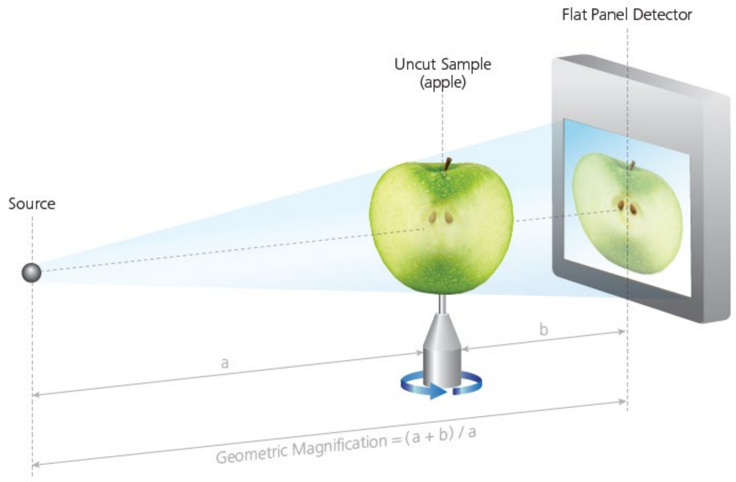
ZEISS Xradia Versa – Technology and Applications



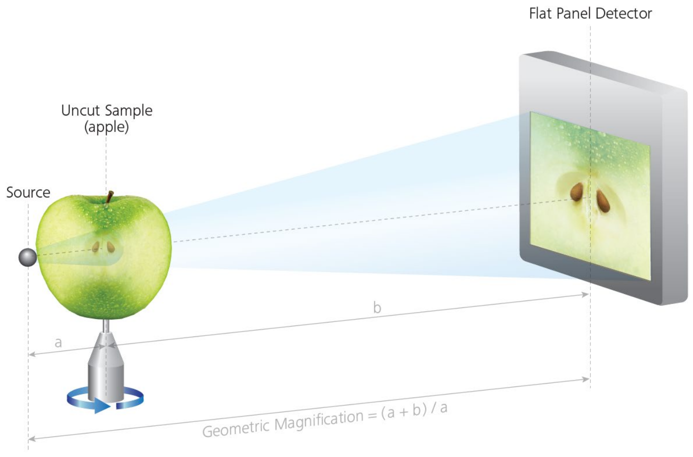
ZEISS Xradia Ultra – Technology and Applications

Limitations of microCT Geometric Magnification
"You can only get so close"

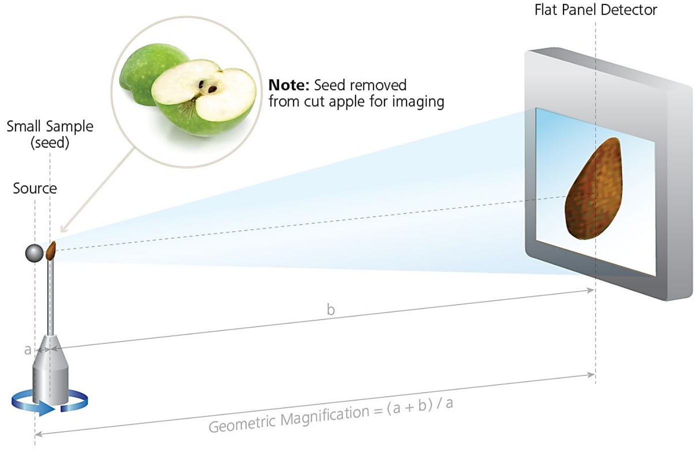
With microCT architecture…



…you can image the whole object…

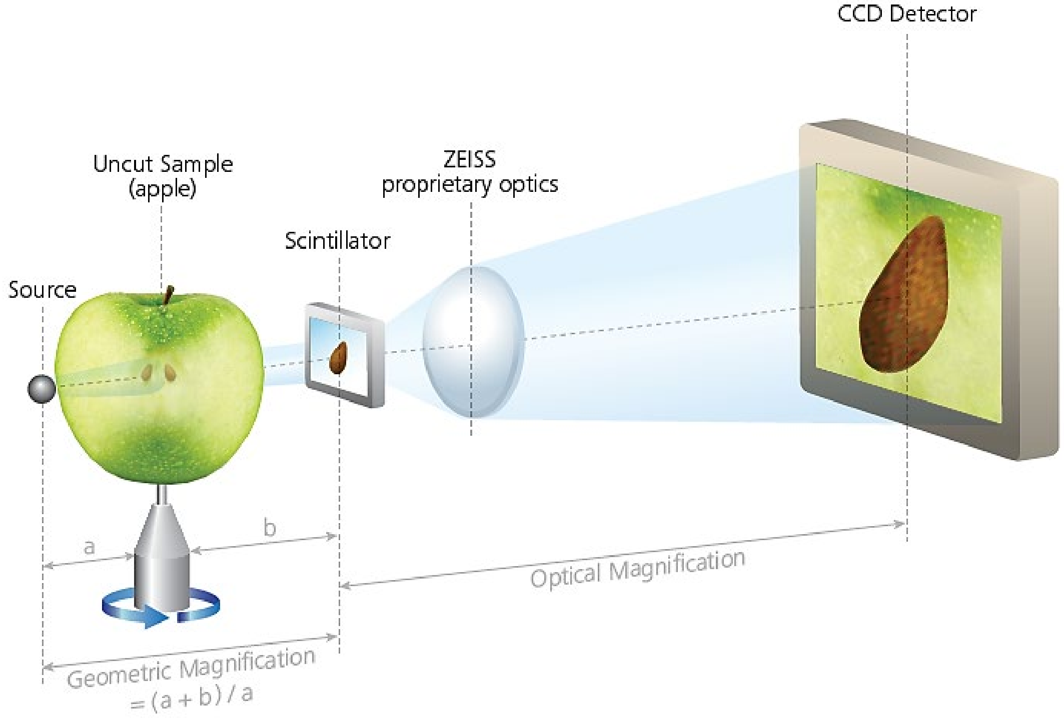


…and then you can zoom in *a little.*



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

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

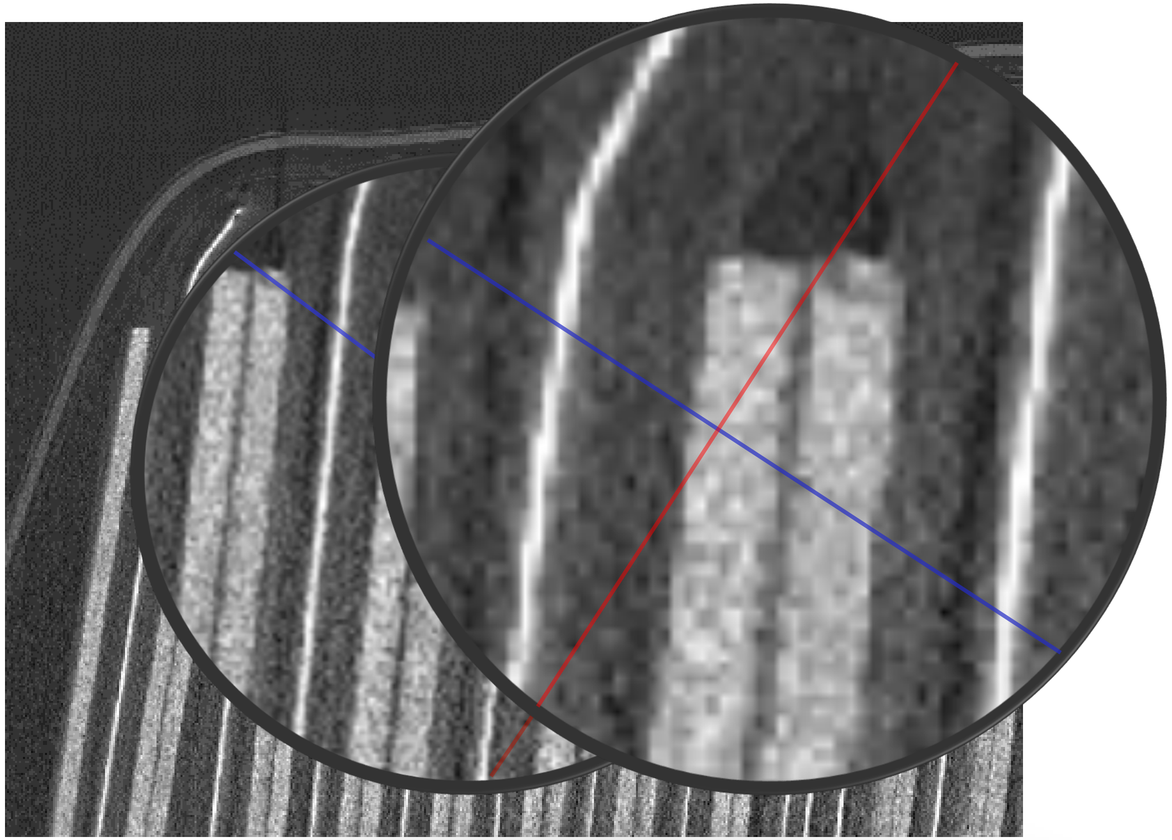


Only an **X-ray microscope** can scan an apple seed at high resolution ***without cutting*** the apple open (RaaD = Resolution at a Distance)

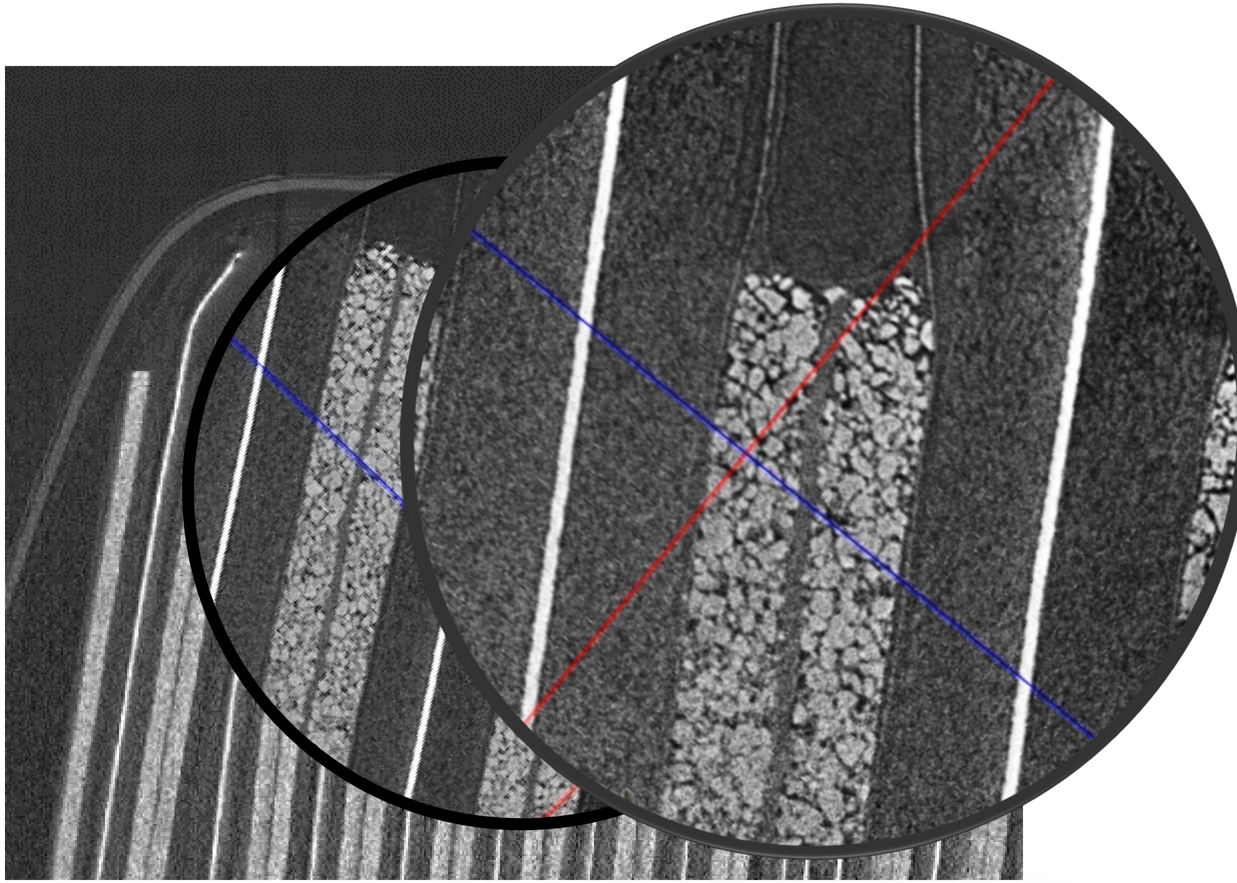
ZEISS Xradia Versa - Multiple scintillator-coupled optics for different magnification

X-ray Microscopy with RaaD
Advantage over microCT – demonstrated on intact Li-ion battery

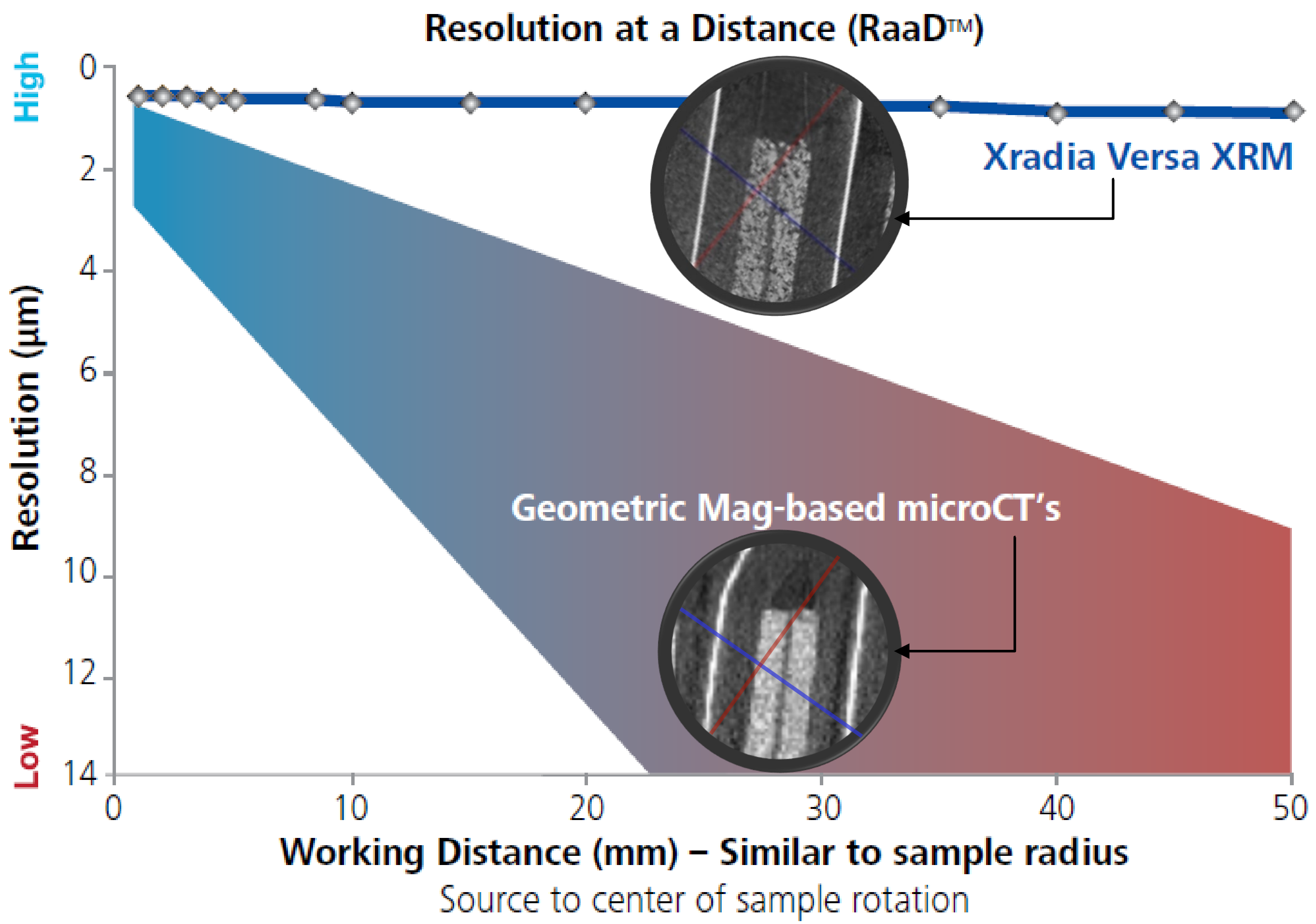
Traditional X-ray microCT



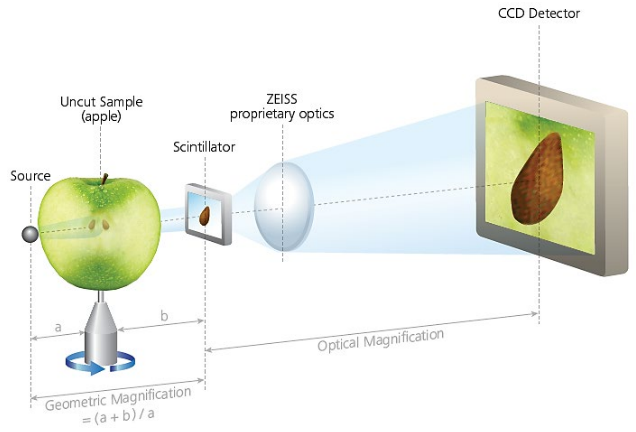
X-ray microscope



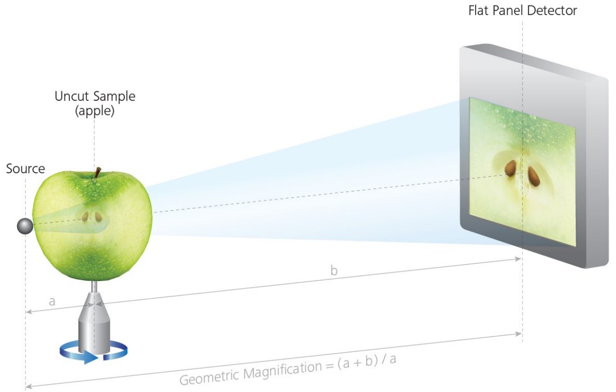
XRM Maintains High Resolution at Large Working Distances



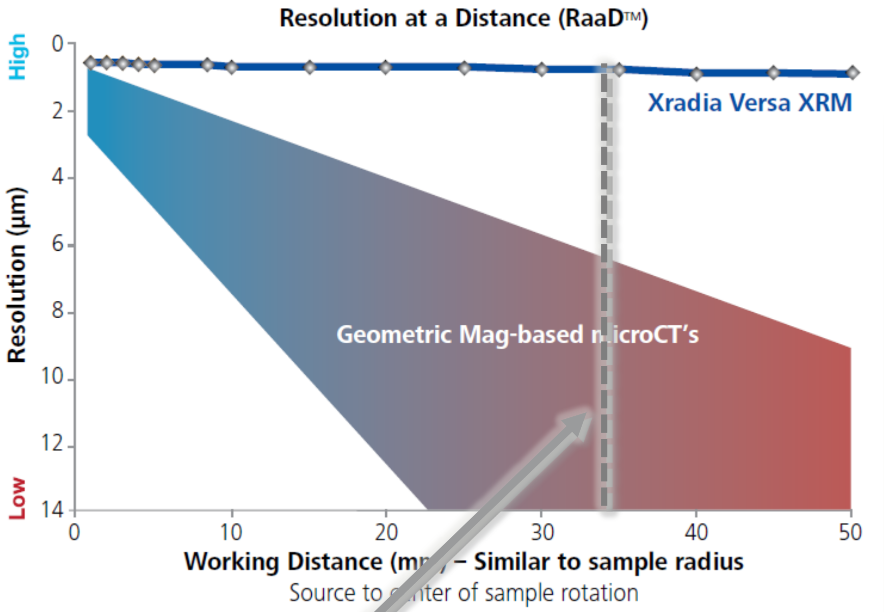
XRM 2-stage magnification architecture



Traditional microCT architecture

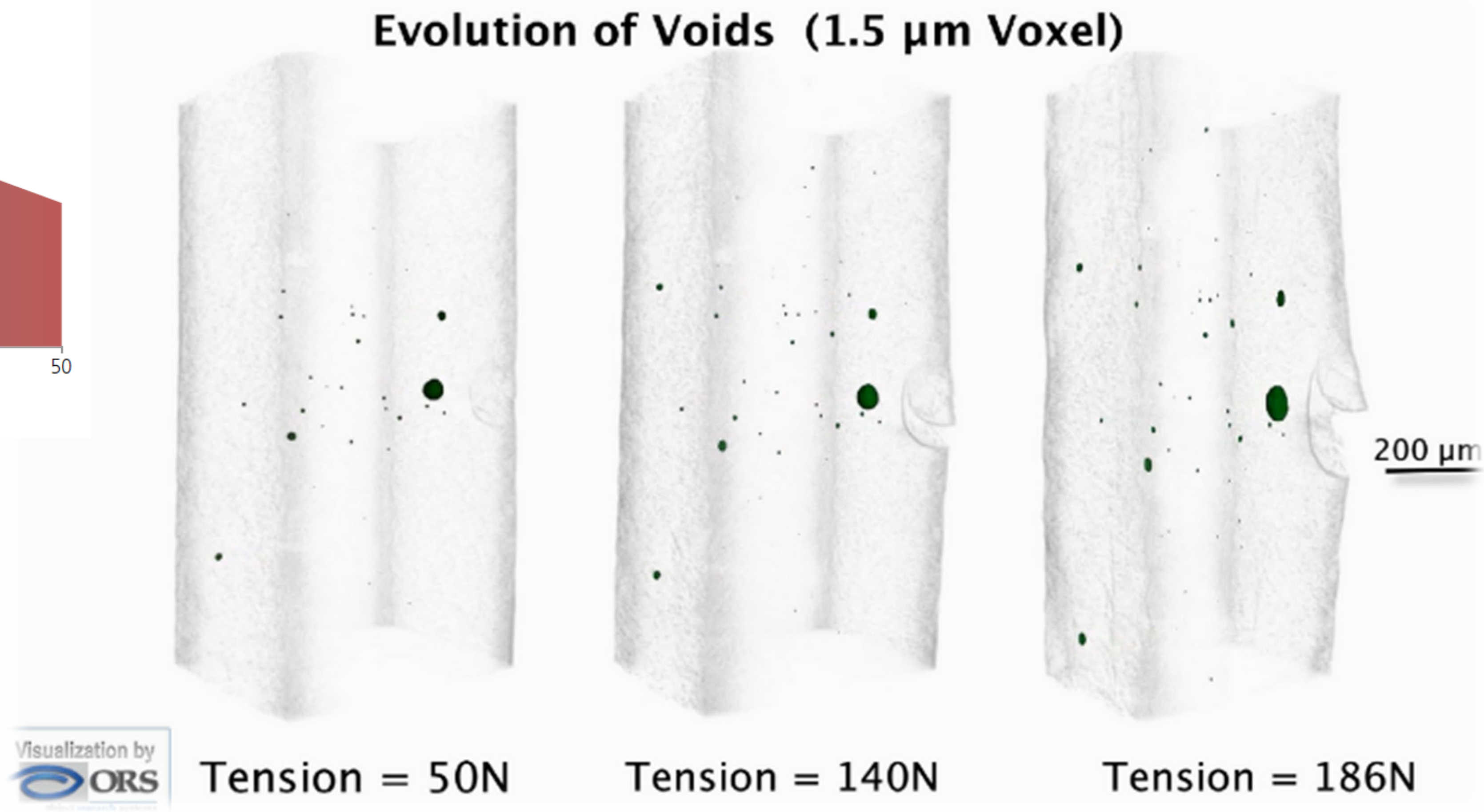


Resolution at a distance (RaaD™)
Provides best results for imaging *in situ* experiments



Deben CT5000-TEC
Load stage

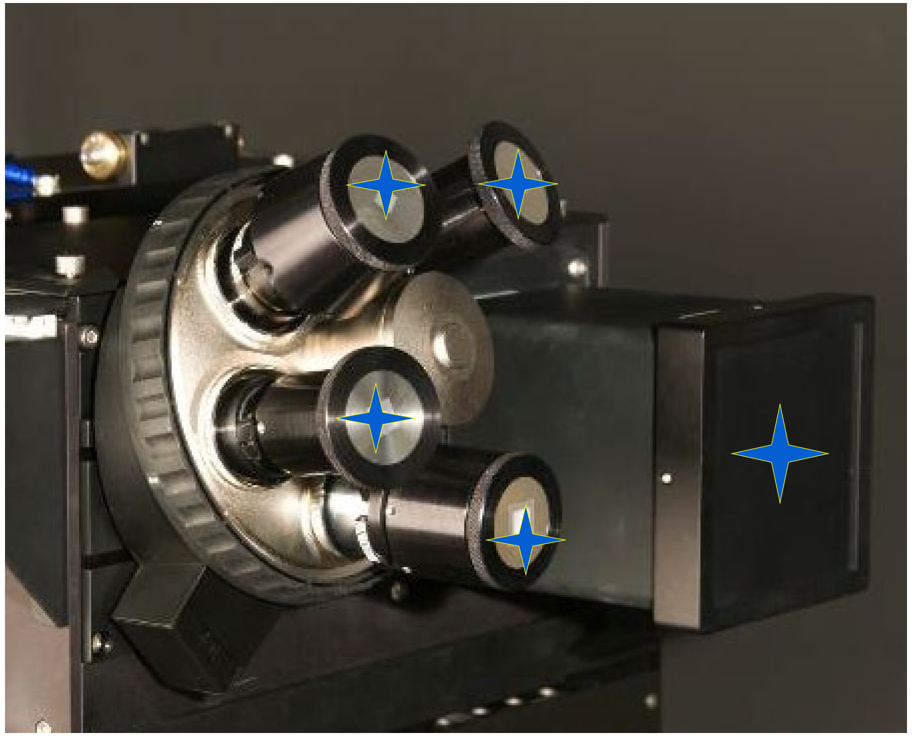
Laser Welds in Steel
in situ tension testing and evolution experiment

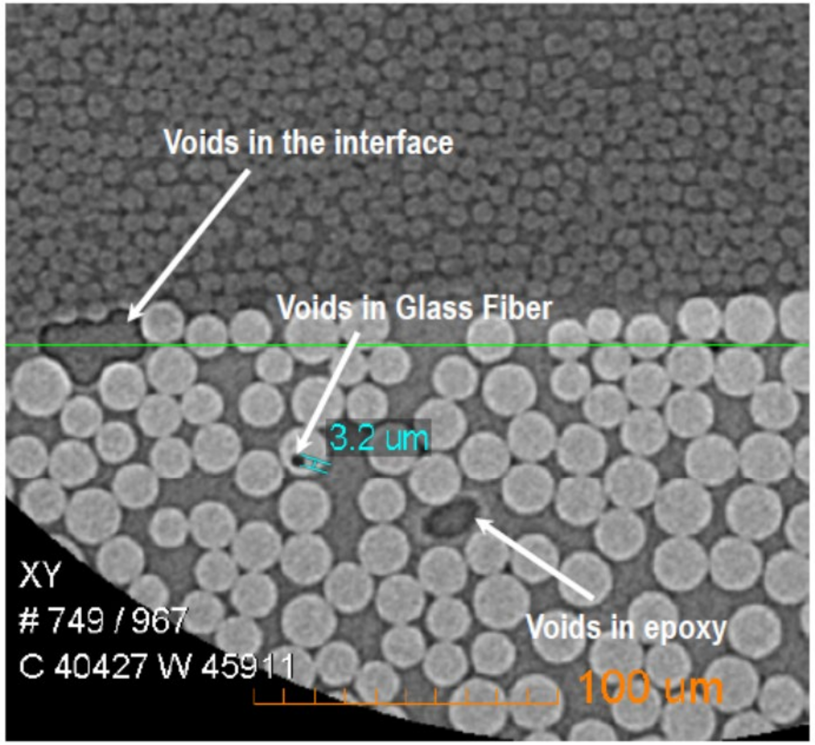


Sample courtesy of Sandia National Lab

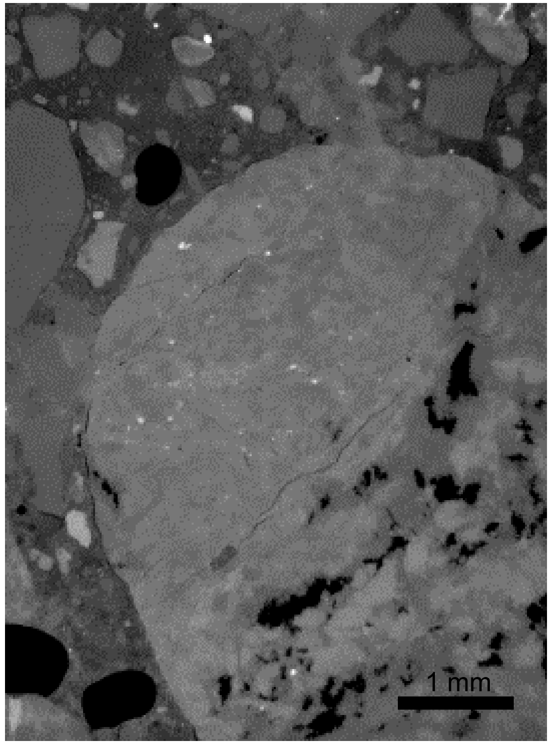
Xradia Versa: Unique Contrast Advantages

High Absorption Contrast





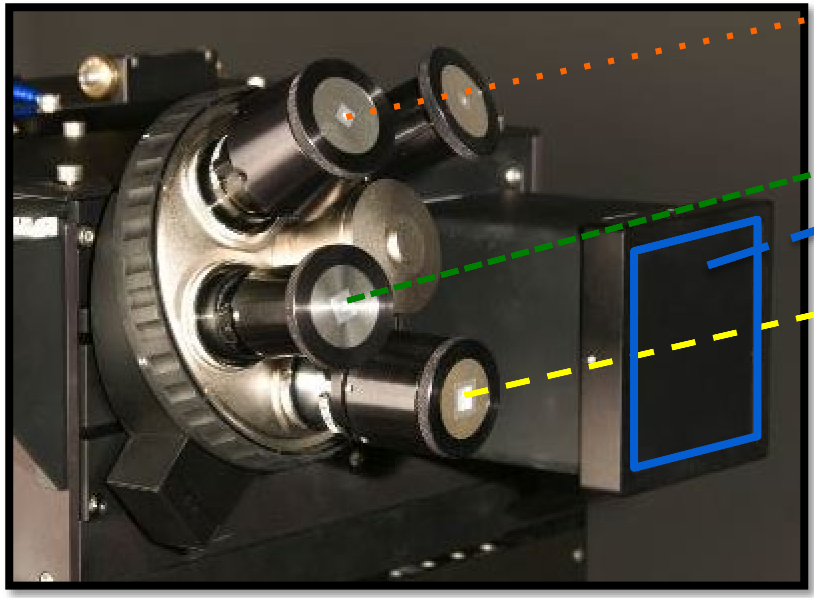
Carbon and glass fiber-reinforced polymer

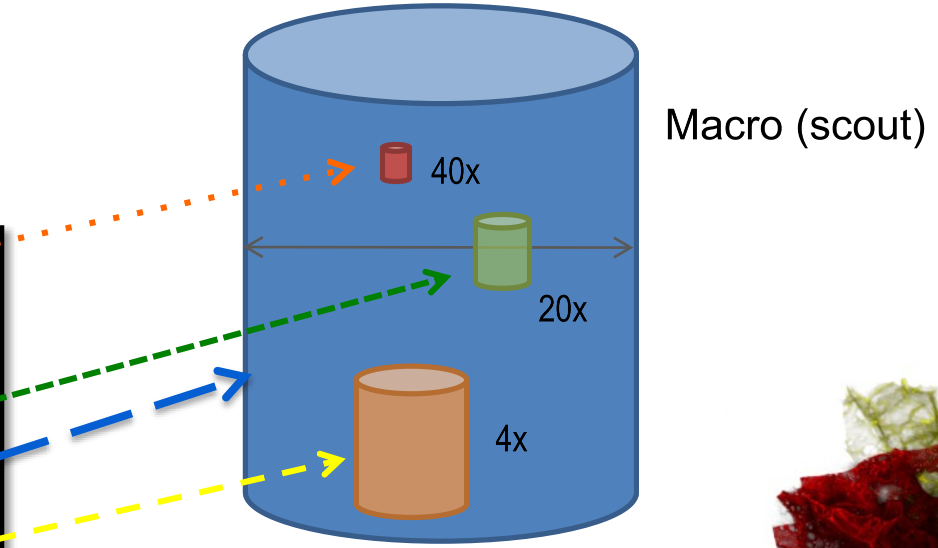


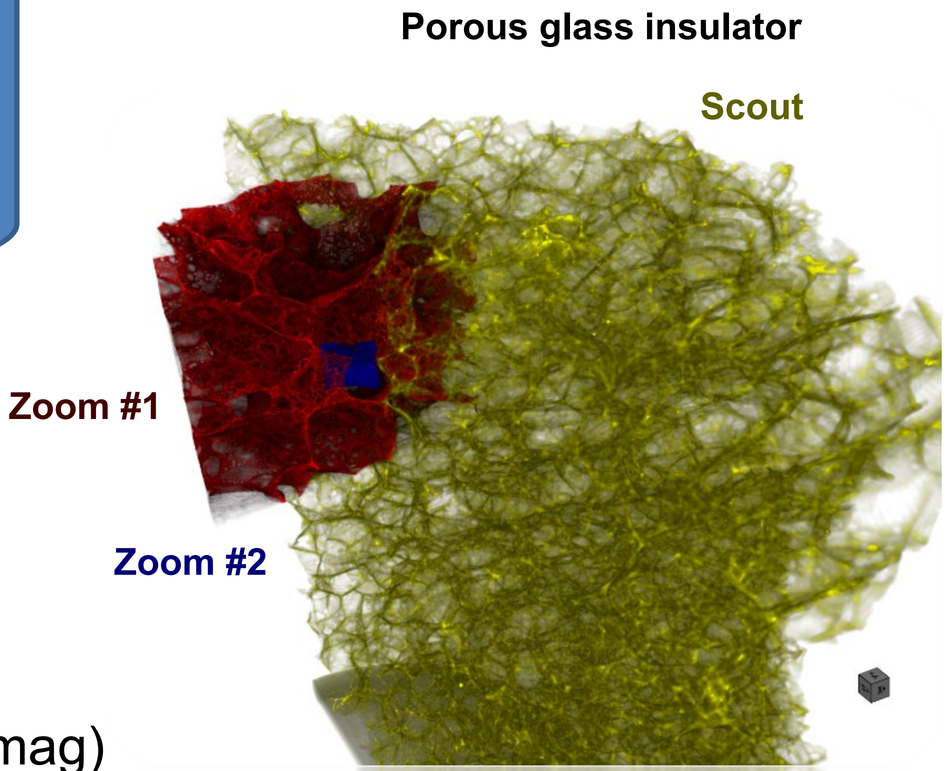
Concrete, interior tomography

Proprietary scintillators optimized by detector

Interior Tomography
Enabled by RaaD





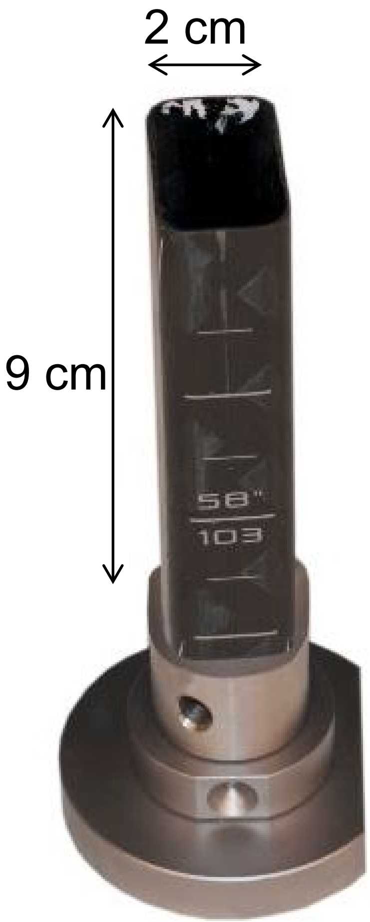


Scout for features of interest (lower mag)

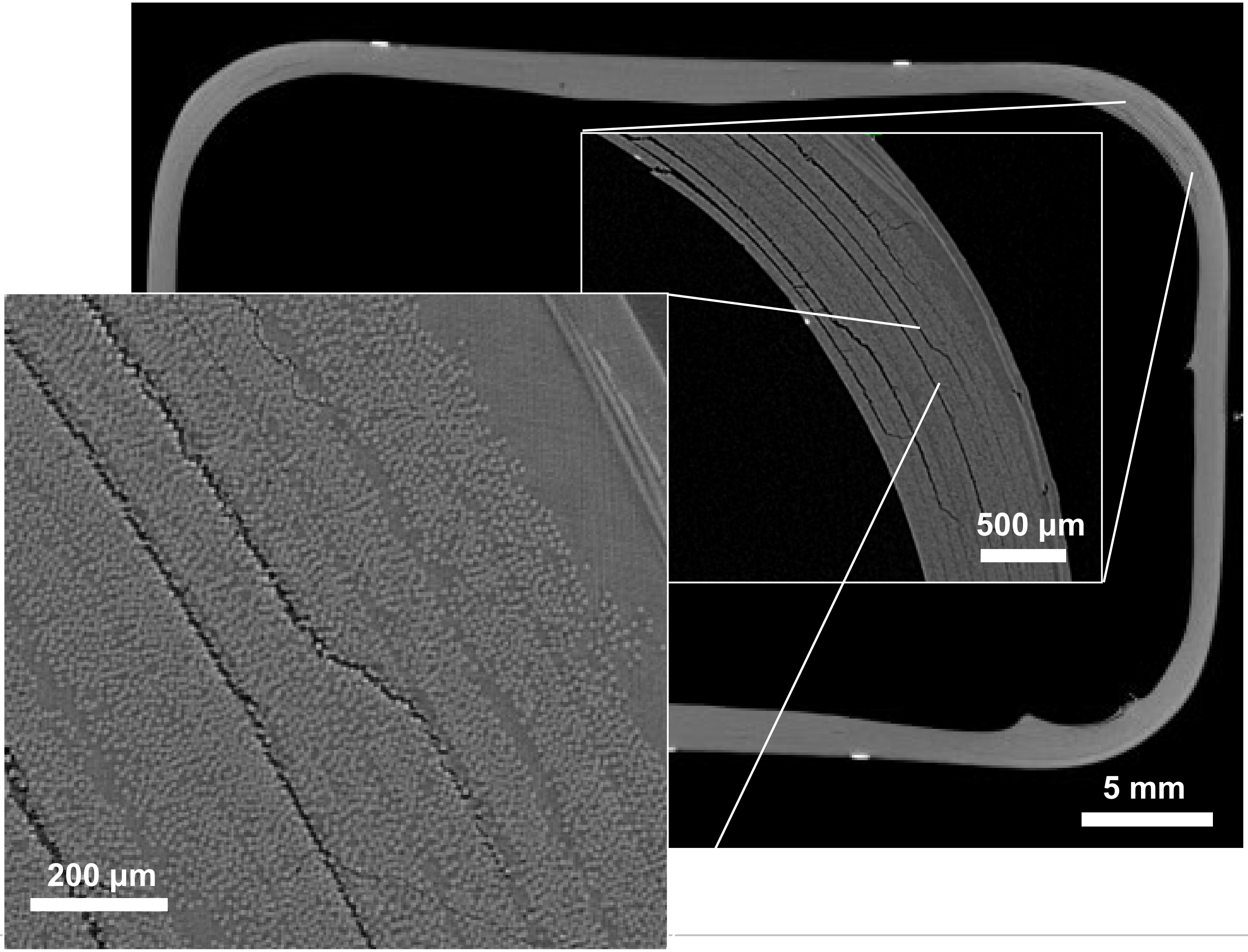
→ **Zoom** in (higher mag)

Sample courtesy of Martin Bonderup Østergaard, Dr. Rasmus R. Petersen and Prof. Yuanzheng Yue from Aalborg University, and Dr. Jakob König from Jozef Stefan Institute

Hockey stick - Fiber Reinforced Composite
“Scout-and-Zoom” workflow



Carbon Fiber Composite

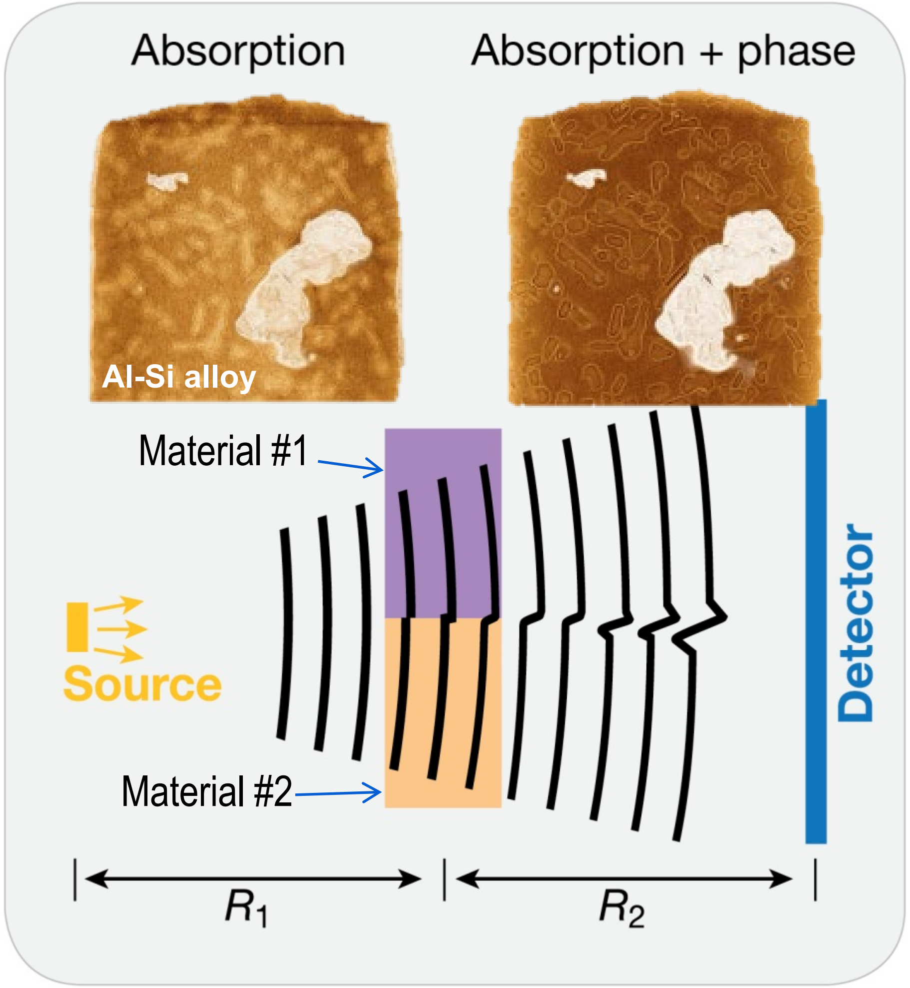


FPX is used to identify localized cracks in high curvature regions of shaft

Zooming in with the **0.4X** objective shows many cracks between fiber plies

Higher resolution imaging with the **4X** objective reveals crack geometries relative to individual fibers

Xradia Versa: Tunable Propagation Phase Contrast



Phase Contrast

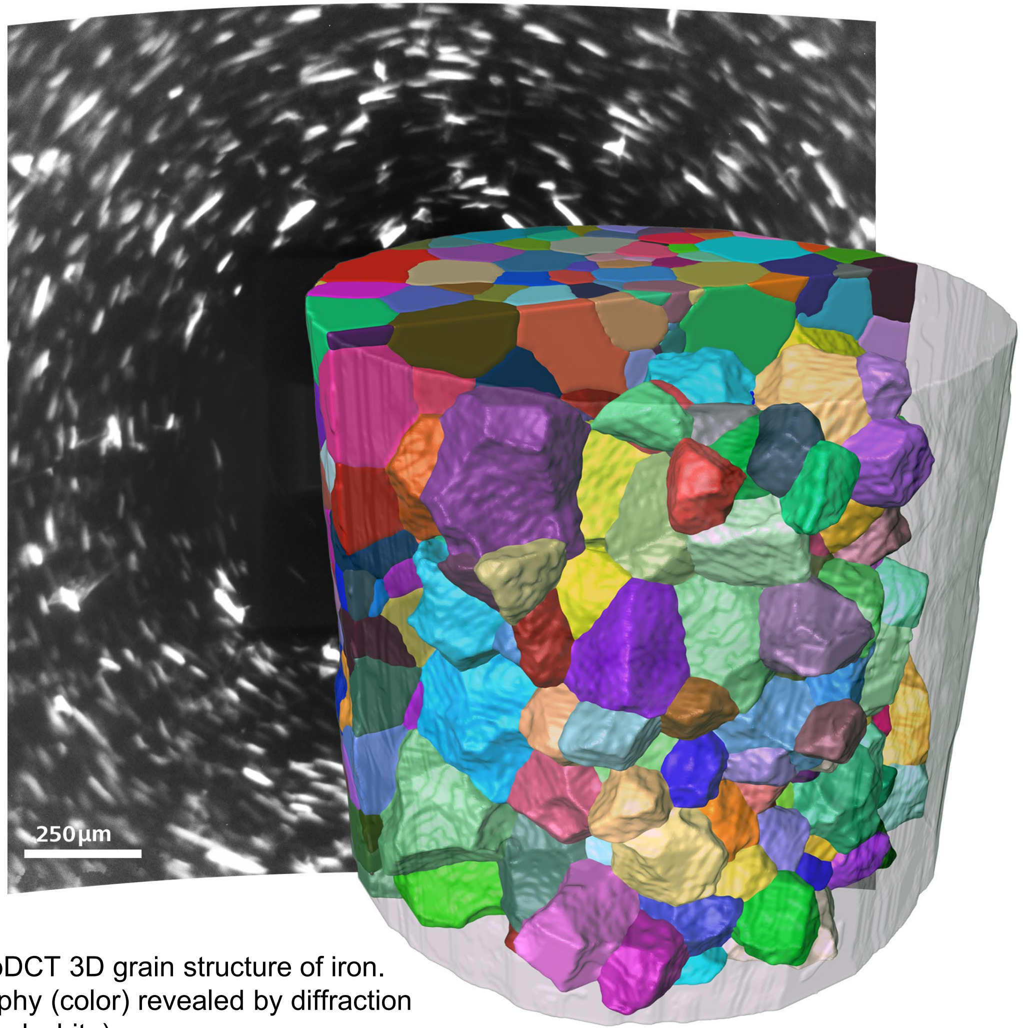
- Effect depends upon refraction rather than absorption
- Phase shift related to refractive index differences between materials
- Fringes are very small (microns) and require small detector pixels to detect

Enabling Technology

- **Small detector pixels** (0.34 µm on 40X) to capture fringes
- Both source and detector have large travel lengths to **maximize fringe**

LabDCT
Unlocking crystallographic information

- Non-destructive 3D grain imaging for mapping orientation and microstructure in 3D for polycrystalline samples - metals and alloys
- Combining 3D grain orientation with 3D microstructural features such as defects or precipitates observed in tomography
- Routine tool access enables logitudinal evolution (‘4D’) experiments studies such as metal corrosion
- Coupling between 3D/4D experiments and microstructure modeling for grain growth kinetics
- Routinely acquire grain statistics on larger volumes to complement correlative investigations (e.g. EBSD)



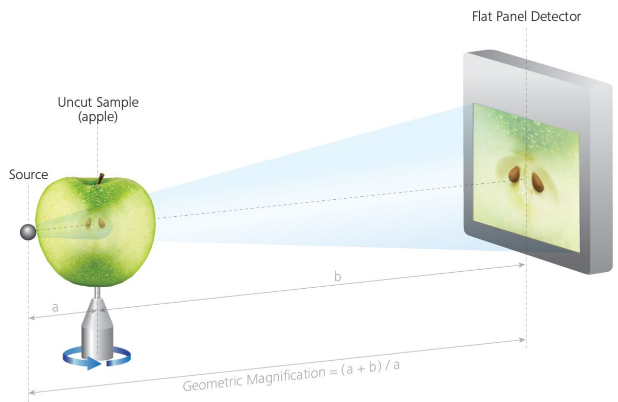
Non-destructive LabDCT 3D grain structure of iron. Internal crystallography (color) revealed by diffraction information (black and white).

Xradia Versa with FPX
Overview

FPX – Flat Panel Extension

ZEISS has developed a world-class flat panel system for the Xradia 5XX & 6XX Versa.
FPX combines microCT with RaaD™, offering the best of both technologies in one system

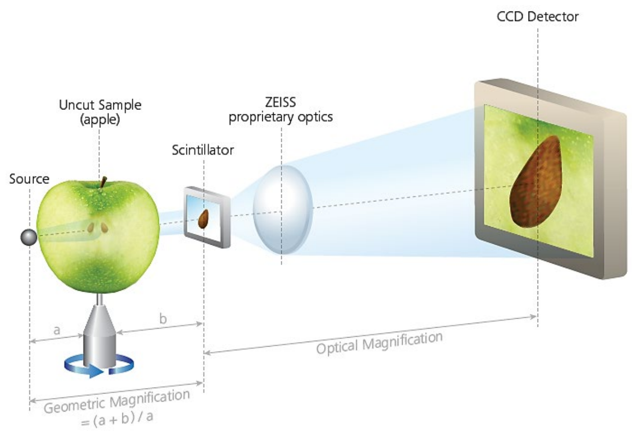
FPX



FPX: "Scout"
Image large samples with speed

+

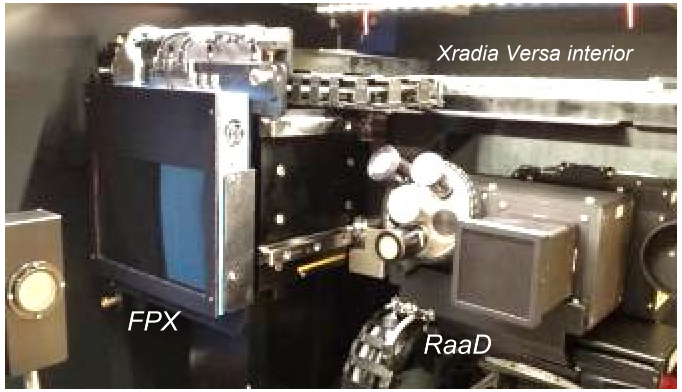
Versa RaaD™
2-Stage X-ray Microscopy



RaaD™: "Zoom"
Image with highest resolution

=

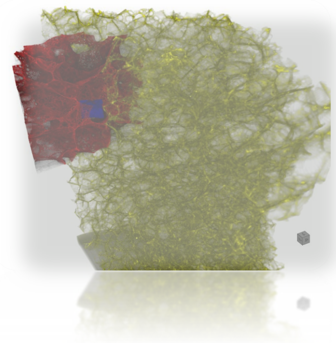
Xradia Versa
FPX and RaaD™ systems



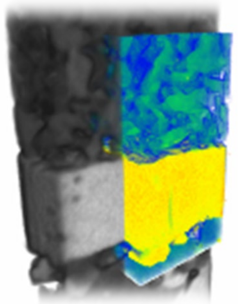
Xradia Versa with FPX
All-in-one imaging solution

Agenda

Overview of ZEISS X-ray Microscopy in Materials Science



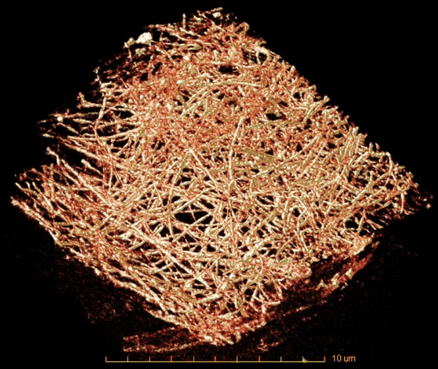
ZEISS Xradia Versa – Technology and Applications

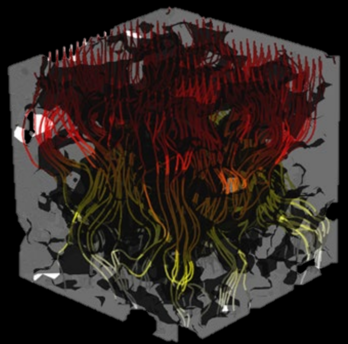


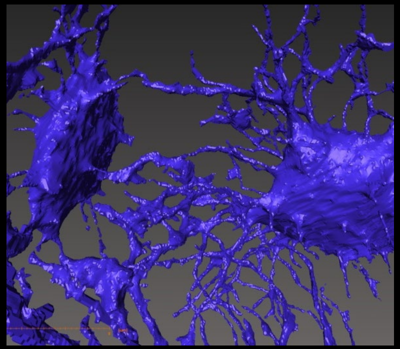
ZEISS Xradia Ultra – Technology and Applications

Xradia Ultra
Applications for Research

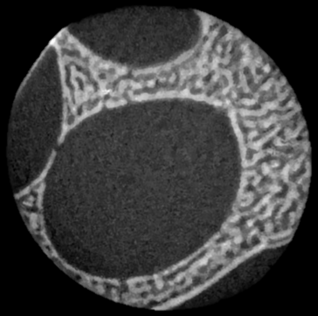
Diverse interests and applications…

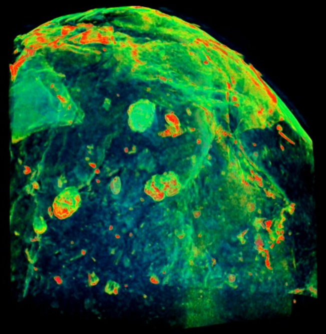

Polymers

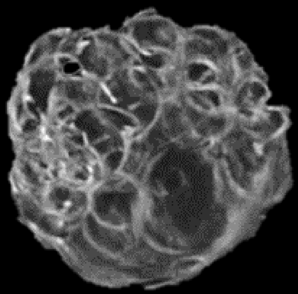

Fuel Cells/Batteries

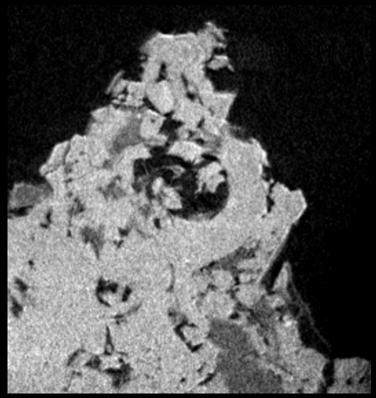

Mineralized Tissue


TSV


Metals


Composites


Pharmaceutical

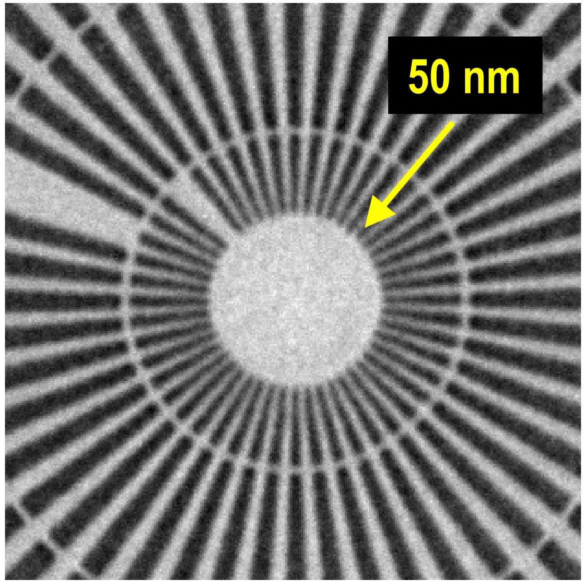

Shale

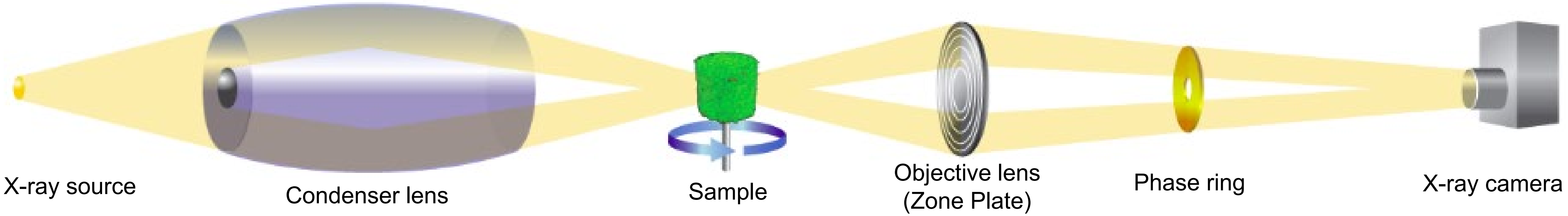
ZEISS Xradia Ultra
3D X-ray nanotomography down to 50 nm resolution

The only non-destructive, laboratory based 3D imaging solution with resolution down to 50 nm: Ideal for 4D and *in situ* studies

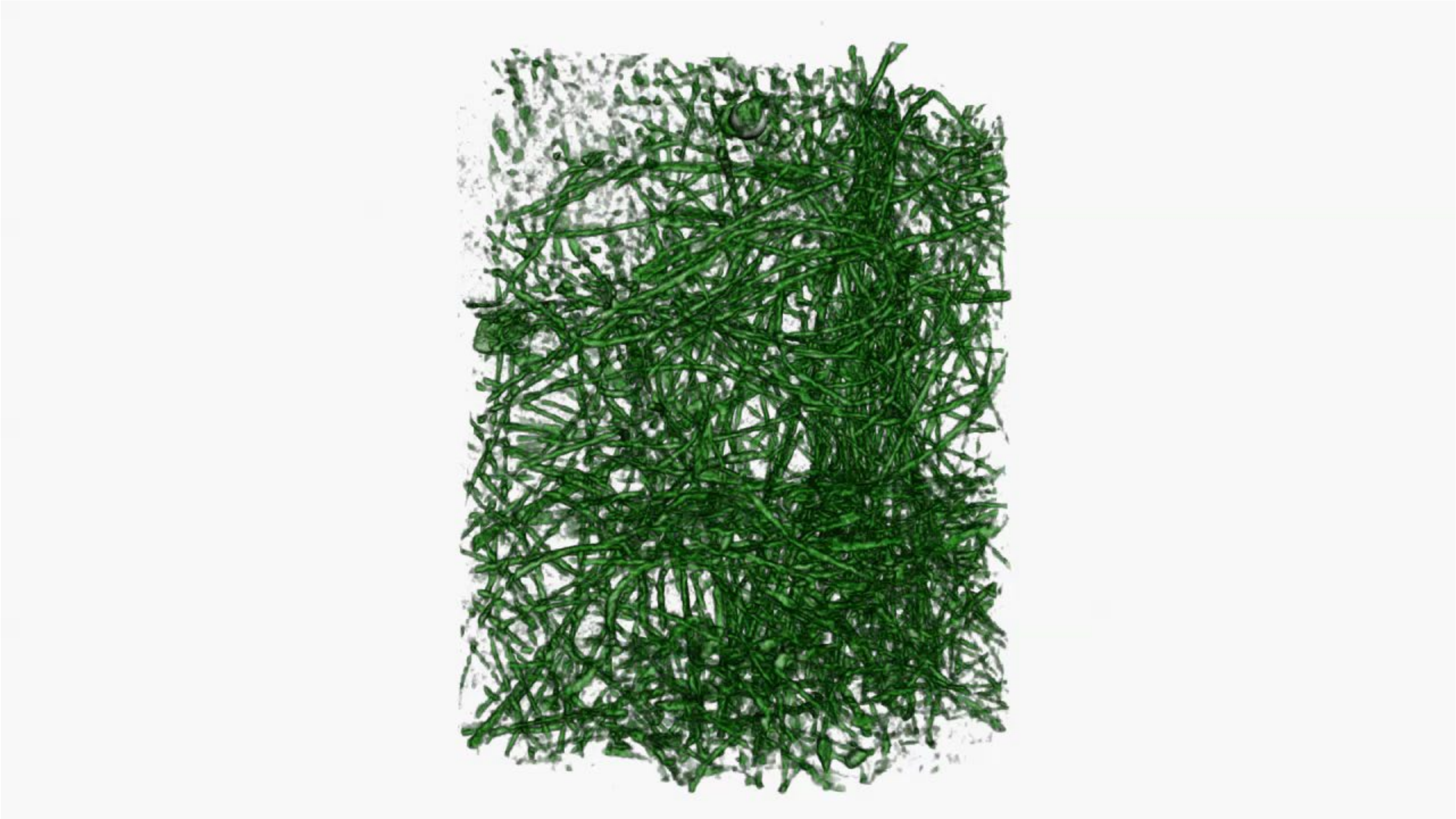
- High brightness X-ray source
  - **Xradia 810 Ultra: 5.4 keV**
  - **Xradia 800 Ultra: 8.0 keV**
- 50 nm spatial (16 nm voxel) resolution
- Advanced X-ray optics
- Absorption and Zernike phase contrast

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



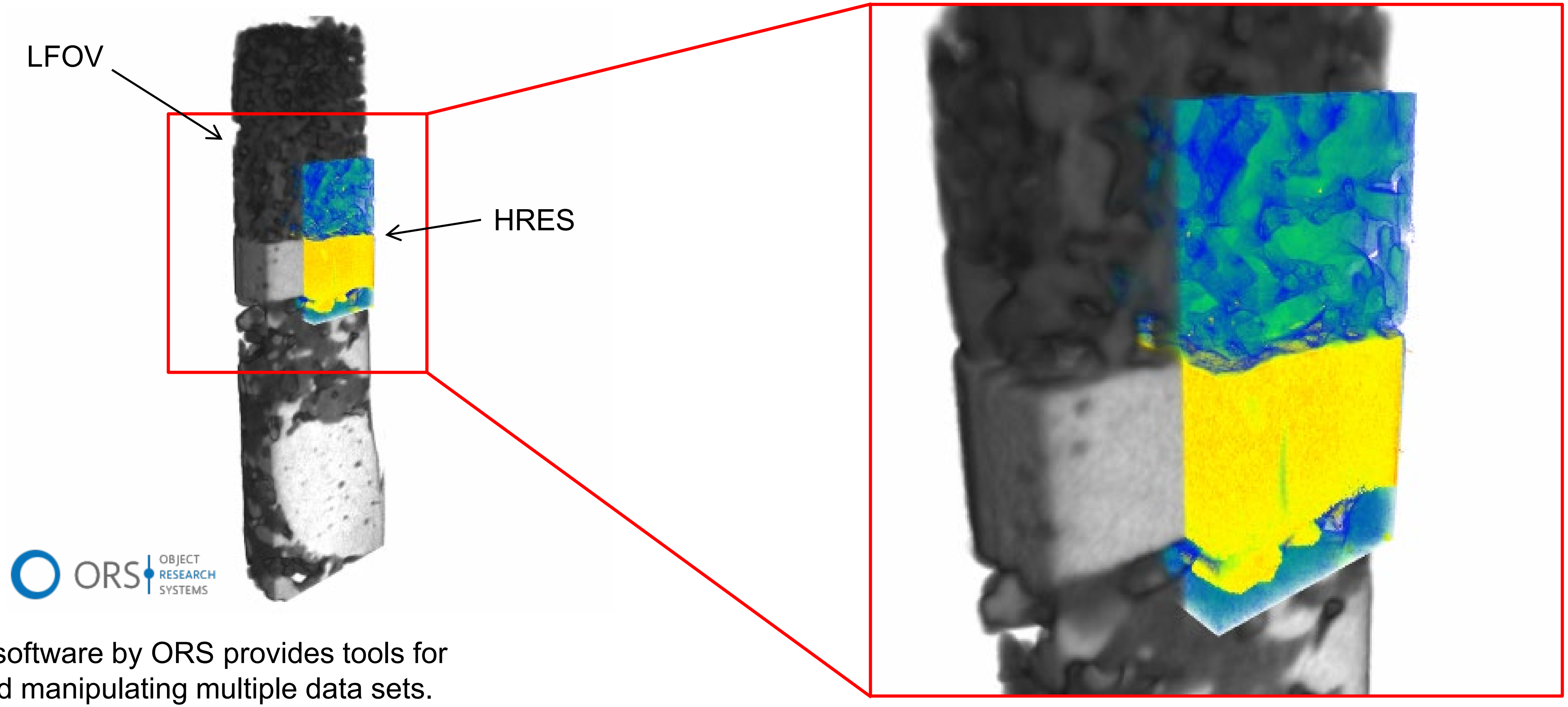


Why Do You Need Zernike Phase Contrast?
Ideal for low density, weakly absorbing materials



Solid Oxide Fuel Cell Multiscale Imaging
Ultra LFOV & HRES data registration

LFOV imaging was first performed with the Xradia 810 Ultra. Then a local region of the sample was scanned in the HRES mode. The images below show the HRES and LFOV data sets overlaid onto each other.



Dragonfly Pro software by ORS provides tools for registering and manipulating multiple data sets.

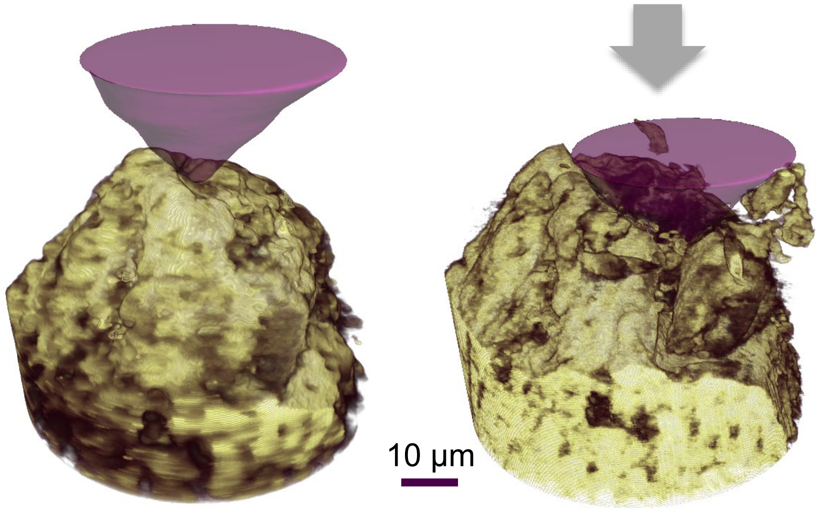
Xradia Ultra Load Stage

In situ nanomechanical test stage for 3D X-ray imaging

- The only solution for *in situ* mechanical testing combined with nanoscale 3D imaging
- Study the evolution of interior structure in 3D, under load, down to 50 nm resolution
- Understand how deformation events and failure relate to local nanoscale features and bulk behavior
- Explore a new length scale – bridge the gap between SEM/TEM and MicroCT
- Operate in compression, tension or indentation mode

Key applications:

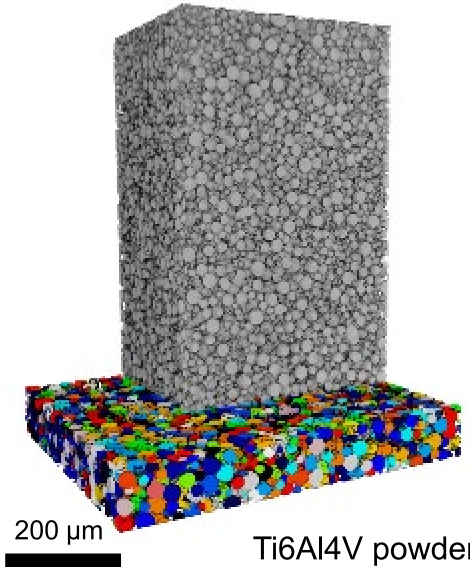
- High strength alloys
- Coatings
- Fibers / composites
- Biomaterials / biomechanics
- Building materials
- Foams



Crack propagation and fracture in dentin
Courtesy Univ. Manchester

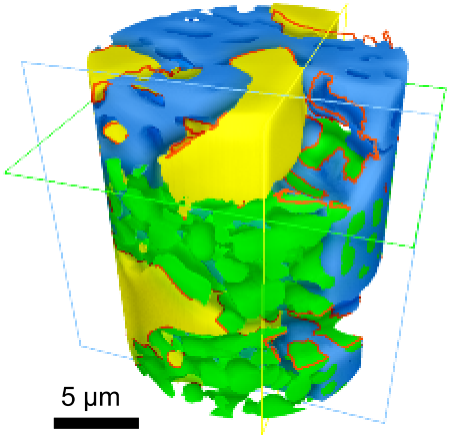
ZEISS X-ray Microscopy for Materials Science
Overview

- **Non-destructive, state of the art 3D imaging**
  - Highest resolution
    - Study structures with down to 50 nm resolution
    - Maintain resolution across a wide range of sample sizes
  - Highest contrast
    - Synchrotron technology adopted for a lab-source
    - Available 24/7
- **Wide range of sample volumes for multi-scale, hierarchical materials (cm3 → µm3)**
- **Unique modalities**
  - 4D material evolution studies, 'time-lapse'
  - High resolution studies within flexible array of *in situ* environments
  - Navigate & correlate:
    - Precursor to complementary and efficient physical sectioning with Crossbeam (3D FIB-SEM)



Ti6Al4V powder for AM

Xradia 620 Versa
Sub-micron 3D X-ray Microscope



Fuel cell electrode

Xradia 810 Ultra
Nanoscale 3D X-ray Microscope

ZEISS