Extending Synchrotron X-ray Microscopy to the Laboratory
X-Ray Microscopy as a correlative imaging technique

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Carl Zeiss Microscopy GmbH
Outline

- 3D X-ray Microscopy
  - What is a 3D XRM?
  - Architectures:
    - Submicron:
    - ZEISS VersaXRM
    - Nanoscale:
    - ZEISS UltraXRM

- XRM Applications
  - Materials Science

Merkle & Gelb, Microscopy Today, March 2013
World’s Largest Installed Base of Synchrotron 3D Microscopes

Synchrotron Source Products

Full field tomography

Cryo tomography

STXM

Res down to ~30 nm

Broad portfolio of X-Ray imaging technologies
Tomography in 3D X-ray Microscopy: How it Works

![Diagram of tomography setup with labels: Source, Sample, Objective, Scintillator, Camera, Projektionen, 2D Slice Views, 3D reconstruction.]
ZEISS
A complete 3D-Microscopy portfolio

- **Xradia Versa**: Sub-micron 3D X-ray Microscope
- **Xradia Ultra**: Nanoscale 3D X-ray Microscope
- **MicroCT**: Sample size [m]
- **Metrotom**: X-ray CT
- **AURIGA**: Cross-Beam 3D X-ray Microscope
- **ORION Nanofab**: HIM

Resolution [m]:
- $10^{-9}$
- $10^{-8}$
- $10^{-7}$
- $10^{-6}$
- $10^{-5}$
- $10^{-4}$
- $10^{-3}$
- $10^{-2}$
- $10^{-1}$
- 1

Sample size [m]:
- $10^{-9}$
- $10^{-8}$
- $10^{-7}$
- $10^{-6}$
- $10^{-5}$
- $10^{-4}$
- $10^{-3}$
- $10^{-2}$
- 1

Unit:
- micron
- mm
ZEISS Xradia Versa – What’s Unique?

- **Projection** based architecture
- **Resolution degrades substantially** as the sample moves away from the source
- Large detector pixel

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**Conventional MicroCT**

**Xradia Versa Family**

- **Two-Stage Magnification** for unprecedented **Resolution** and **Contrast**
- **High resolution is maintained** as sample moves away from the source
Polymer composite fiber bundle defects at 40X
Working Distance Defined

Traditional CT / µCT architecture (*non-XRM*):

![Diagram showing the traditional CT/µCT architecture with a source, sample, and detector. The working distance formula is given as $GeoMag = \frac{(Dss + Dds)}{Dss}$]

Working distance increases as sample size increases
XRM Maintains High Resolution at Large Working Distances

Geometric Mag Based MicroCTs Resolution rapidly degrades with increasing sample size.

XRM Maintains High Resolution at Large Working Distances.
Polymer Foams – In Situ compression

B. Patterson, LANL, M&A March 2012
Scout and Zoom
Local (interior) Tomography

Measurement at varying length scales through Interior Tomography

<1 µm imaging on 10 mm sample
Microporosity & microfractures between grains
Conventional CT
XRM

2.2 μm/voxel
Positioning for
**Absorption Contrast**

Positioning for Absorption and
**Propagation Phase Contrast**
ZEISS Xradia Versa: Tunable Propagation Phase Contrast

- **Phase Contrast**
  - Refraction rather than absorption
  - Phase shift related to refractive index

- **VersaXRM Design**
  - Small detector pixels (0.34 µm on 40X) to capture fringes
  - Both source and detector have large travel lengths to maximize fringe
Imaging low-Z or similarly dense materials: Propagation Phase Contrast vs. Absorption

Al-Si Alloy
Phase contrast mode highlights interfaces, revealing details not present in absorption image.

Al/Si sample courtesy of A. Shahani and P. Voorhees (Northwestern University)
Adjust energy for each scan: Automated Filter Changer

- **Select energy produced by source**
  - Spectrum from source is broad band
  - Range is $30 \text{ kV} \rightarrow 160 \text{ kV}$
  - User selects source energy from this range

- **Automated Filter Changer**
  - Filters selectively tune energy bands from the broad band source
  - Auto filter wheel allows users to select filters directly from the scan recipe
Single Energy Tomography

Example:
Al and Si are very difficult to distinguish from one another in single scan.
Compositional Contrast Process
Flow Example

Scan #1 – High Energy
Scan #2 – Low Energy

Dual Scan Contrast Visualizer (DSCoVer)
Both scans combined into one using easy, interactive user interface

Both scans combined together

Aluminum (green)
Silicon (Red)

Aluminum separated from Silicon

VersaXRM-520
Better imaging for flat samples: HART

What
- Optimized imaging mode for high-aspect ratio samples / features

Value
- **Up to 50% higher throughput** for Semi-type high aspect ratio samples with equivalent detectability

How
- Angle dependent (non-uniform) projection spacing
- More densely spaced projections along long views
- Less densely spaced projections along normal view

Tomography without HART: uniformly spaced projections

HART projection spacing and density optimized for feature-rich short side
**FPX – Flat Panel Extension**
CZXRM has developed a world-class flat panel system for the Xradia 5XX Versa. FPX combines MicroCT with RaRaD™, offering the best of both technologies in one system.
Xradia Versa with FPX
Enable solutions that require range

FPX – Enhancing “Scout-and-Zoom” workflow
Fast, large FOV imaging with FPX enables a broadened, more efficient multi-scale workflow in combination with RaaD™

FPX “Scout”  4x “Zoom”  20x “Zoom”
Find regions of interest (ROI)  Versa RaaD™  Versa RaaD™

Xradia Versa with FPX
Packaging/Assembly – Large Scale Assembly to Small Scale Defects

Mobile phone battery imaged in 3D with **520 Versa (FPX option)** without sectioning or opening the package.

Results showed more cracks in the bent regions (fold of jelly roll) than in the straight regions (middle of battery), suspected to be due to high tensile stresses in those positions.
Hockey stick - Fiber Reinforced Composite
“Scout-and-Zoom” workflow for defects

Quickly Scout with FPX

Zoom with Multiple Objectives

Extend Versa Scout-and-Zoom workflow to examine damage over multiple length scales down to crack and fiber geometry without sectioning hockey stick

Now available as an Applications Note
Catalytic Converter
Versa FPX now accommodating larger samples in FOV

- **Scout**: Larger sample imaged in FPX using multiple FFOV scans
- **Zoom**: Interior tomography imaged of ceramic monolith (40 mm diameter) without sectioning steel casing
- **Non-destructive**
Image Your Electronic Devices or Components:
FPX Option Allows Large Field of View and Fast Scans

- Electronics Board to Module to Package to Interconnect
- Motherboard for phone with one workflow on package
- Three important modules in phones today
  - Processor module
  - Battery module
  - Camera module

FPX: Complete Smartphone

Carl Zeiss Microscopy
Scout and Zoom Workflow Examples

Survey (Scout) Scan

FPX Scout Scan

High Resolution RaaD (Zoom) Scans

ROI

RaaD Scan
Electronics Example: Processor Module in Cell Phone
Scout and Zoom Offers Enhanced Flexibility and Quality

**Step 1:** FPX Scout Image

**Step 2:** Zoom with RaaD
Interconnect Image (1.6 µm/voxel)

**Step 3:** Analyze Interconnect Quality with RaaD: Virtual Planar Cross Sections

- Cu Post on Die with Solder
- Solder (die side)
- Cu Pad (substrate)
LabDCT

1. Acquisition on Xradia 520 Versa

Uniquely enabled by ZEISS X-ray Microscopes
- Utilizes white/polychromatic divergent X-ray beam
- Configured in Laue focusing geometry
- Specialized apertures, beam stop, and dedicated detector
- LabDCT recipes integrated in Scout-and-Scan
- Available as optional module for Xradia 520 Versa

Patent Pending
Laboratory Diffraction Contrast Tomography (DCT)
ZEISS Xradia 520 Versa

Bringing synchrotron technology to the laboratory

- 3D crystallographic information obtained on a laboratory XRM (ZEISS Xradia 520 Versa)
- Turns non-destructive 3D grain mapping into a routine tool.
- Enables extended evolution ('4D') experiments impractical at the synchrotron

Lab-DCT reconstruction of grain positions and orientations of over 500 Ti-grains. Grain orientation and center of mass shown.
Application Case
Sintered copper spheres

Sample description:

<table>
<thead>
<tr>
<th>Material</th>
<th>Copper</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size</td>
<td>50-150 micron</td>
</tr>
<tr>
<td>Space group</td>
<td>225 (Fm-3m)</td>
</tr>
<tr>
<td>#grains</td>
<td>176</td>
</tr>
</tbody>
</table>

In collaboration with the University of Manchester
ZEISS Xradia Ultra 810: 3D X-ray Microscope using X-ray Optics

50 nm spatial (16 nm pixel) resolution

High brightness x-ray source (5.4 kV)
High efficiency condenser
High-resolution objective zone plate
Zernike phase contrast optics
Precision tomography system

<table>
<thead>
<tr>
<th>Mode</th>
<th>Mag</th>
<th>2D Res</th>
<th>Field of View</th>
</tr>
</thead>
<tbody>
<tr>
<td>Large Field of View</td>
<td>200X</td>
<td>150 nm</td>
<td>65 µm x 65 µm</td>
</tr>
<tr>
<td>High Resolution</td>
<td>800X</td>
<td>50 nm</td>
<td>15 µm x 15 µm</td>
</tr>
</tbody>
</table>
• In the past, nanoscale 3D X-ray imaging was only available at the synchrotron
  • Resolution of most instruments are micro scale, typically lower res than Versa family (albeit faster)
  • Select few have nanofocus architecture, featuring res down to 30 nm (Xradia only commercial supplier)
• With ZEISS Xradia Ultra, near-”nanofocus synchrotron” resolution and image quality is available in a lab based instrument
Materials Science Examples

Polymers

Batteries/Fuel Cells

Ceramics

Composites

Superconductors

Metals

Glass

Coatings
The sample was mounted on an Aluminum rod and placed in a standard pin vice holder.

<table>
<thead>
<tr>
<th>Scan</th>
<th>Voxel size (µm)</th>
<th>FOV (mm)</th>
<th>Energy/Power (kV/w)</th>
<th>Total scan time (Hrs.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-phase polymer</td>
<td>2.4</td>
<td>2.4 x 2.4</td>
<td>40/3</td>
<td>2</td>
</tr>
<tr>
<td>Interface 1</td>
<td>1</td>
<td>1 x 1</td>
<td>40/3</td>
<td>3</td>
</tr>
<tr>
<td>Interface 2</td>
<td>1</td>
<td>1 x 1</td>
<td>40/3</td>
<td>3</td>
</tr>
</tbody>
</table>
## Multiphase Polymer

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Scan Objectives</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 phase polymer</td>
<td>Observe 3 phases and each interface. Calculate particle size distributions of each phase and volume content.</td>
<td>Successfully imaged and identified interfaces and different sized particles in each phase, using 1 μm voxel resolution scans.</td>
</tr>
</tbody>
</table>

![Image of multiphase polymer sample](image)
Multiphase Polymer
Low Resolution Scan

Image on the left shows reconstructed 2D slices. Each of the quadrants represents a different orthogonal virtual slice.

Scanned area shown in red

2.4 mm
Orthogonal slices of the sample, showing interface and different sized particles. Top layer is EDPM (ethylene propylene diene monomer) rubber with some hydroxyl-terminated polybutadiene (HTPB) and a small amount of toluene diisocyanate (TDI) as curing agent. Bottom layer (polyurethane) consists of the filler and metal oxides.
3D model of shark skin denticles was constructed using ZEISS Xradia Versa of shortfin mako (*Isurus oxyrinchus*).

High resolution XRM dataset inputed into 3D printer to fabricate arrayed model (12X-scale).

Biomimetic skin underwent hydrodynamic testing to exploring effects of denticle surface roughness.

XRM part of first study to design, fabricate and test synthetic shark membrane.
Polymeric materials with engineered pore structures are useful as catalyst support and biomedical scaffolds, but are challenging to fabricate.

Sai et al. discovered a simple way to make a ‘hierarchical structure’ – having both large several micron-sized and ~tens of nanometer-sized pores.

**ZEISS Xradia 500 Versa** was used non-destructively (“requires no alteration to sample”) and for quantitative 3D network analysis: polymer (blue) and pores (red).

Novel functional 3DOM polymeric structures for CO$_2$ capture are prepared by colloidal crystals templating thru inexpensive/rapid preparation.

“...this demonstration showed the feasibility of using nanoscale 3D XRM for the visualization of the 3D morphology of 3DOM materials down to 50 nm resolution (16 nm voxels), representing up to three orders of magnitude improved resolution from the previous micro-CT experiments.”
Nanofiltration membrane imaged with ZEISS Xradia Ultra 810 using Zernike phase contrast (64 nm voxel)

- XRM visualized the different layers of the membrane: interpenetrating small fiber layer, fibrous backing and interface region.

Sample Courtesy of ITRI
ZEISS Xradia Ultra:
Imaging and Analysis of Electrical Trees
Schurch, R. et al., Imaging and Analysis Techniques for Electrical Trees using X-ray Computed Tomography. IEEE Transactions on Dielectrics and Electrical Insulation Vol. 21, No. 1; February 2014

- Electrical treeing is one of the main mechanisms of degradation in polymeric high voltage insulation, a precursor of power equipment failure
- Previously used methods for the characterization of electrical trees include optical microscopy, SEM, TEM, Ultrasound, NMR and others
- Schurch et al. used ZEISS Xradia Ultra and nanoXCT to acquire high resolution 3D datasets. Subsequent quantitative analysis of tree diameter, tortuosity, volume and other parameters was performed.
- High resolution, non-destructive XRM provides “new valuable information” and “is expected to enable a deeper understanding of electrical treeing phenomenon”

Figure 9. Cross-sections of the Sample 3. Distances from the starting point of the tree in µm: a. 0, b. 7, c. 14, d. 21, e. 31, f. 42.
EB-TBC: Buckled Coating Failure

Versa: Non invasive 2D and 3D imaging of delamination @ 0.7 um voxel resolution

Sample courtesy of Dongming Zhu, NASA GRC

Lau et al, ICACC 2010
Versa XRM used to nondestructively observe cracks in dense ceramic TBC layers at sub-micron resolution with good contrast between cracks, top coat, bond coat, TGO, and substrate layers.

Results reveal crack size, shape, aspect ratio, opening width, and position with respect to bond coat surface.

Discovered linkage of multiple cracks and preferential crack orientation
YSZ
LFOV and HRES Data Registration

The 3D rendering below shows HRES and LFOV absorption data sets overlaid onto each other. The yellow boxes show the extent of the two data sets, with the LFOV data clipped away to reveal the HRES data.

Visual SI Advanced software by ORS provides tools for registering and manipulating multiple data sets.
In Situ Experiments: Corrosion (Temperature/Humidity-Induced)

80 mm diameter *in situ* corrosion chamber. 500 µm wire exposed to 40ºC / 30% relative humidity for atmospheric induced SCC (stress corrosion cracking).

Type 304L Wire + MgCl₂ Spray

Source: Dirk Engelberg, University of Manchester
Commercial 18650 Battery
Full Battery Inspection

- Specimen imaged **non-destructively** with **520 Versa XRM**.

- The XRM technique allows specimens to be imaged intact, providing **high-resolution data without sectioning**.

- **Workflow:**
  - **Inspect** the specimens
  - **Identify** regions for higher resolution analysis
  - **Drive** the microscope to enlarge those regions
Defects Observed After 100 Charge Cycles
Commercial 18650 Li-ion Battery Cell Cathode
1.8 μm Voxel Size

Non-destructive imaging with 520 Versa XRM
Laser Welds

in situ tension testing and evolution experiment

Evolution of Voids (1.5 μm Voxel)

Deben CT5000-TEC
Load stage

Tension = 50N  Tension = 140N  Tension = 186N

Sample courtesy of Sandia National Lab
4D Measurement of Cracks in Ti-SiC

The morphology of the crack at Kmax at various crack growth stages.

with Permission from Withers, et al.
Correlative Imaging – An ultimative Workflow

ZEISS Xradia Versa
3D Imaging, Scout & Zoom
Sample size: ten's of mm
Resolution down to 700 nm

CrossBeam/LASER
3D Imaging, Analysis, Sample/Target Preparation

ZEISS Xradia Ultra
3D Imaging, Scout & Zoom
Sample size: ten's of μm
Resolution down to 50 nm

CrossBeam
3D Imaging, Analysis
Al-Cu Eutectic Alloy
Multi-scale XRM to SEM Workflow

XRM:
- Quantify microstructure evolution in 3D over time (4D)
- Non-destructive identification of buried ROI

FIB-SEM:
- Confirm dendritic fine structure with nanometer-resolution
- Analytical evaluation (EDS, EBSD)

Sample courtesy of B. Patterson, LANL
XRM Reconstruction reveals several areas with an unexpected high specific mass

- Correlative examination of position 1 and 2
Correlative examination of e.g. position 1 reveals agglomerated grains containing Lanthanum.
ZEISS X-ray Microscopy Core Advantages

Core Advantages

**Highest resolution**
- Versus any Micro/nanoCT
- At largest working distance (RaaD™)
- Unique < 50nm spatial res

**Highest contrast**
- Unique detector design
- Unique phase contrast imaging
- Unique dual energy

**Non-destructive**
- Image same sample multiple times

Unique Modalities

**SCOUT & ZOOM:**
Switchable magnifications on the VersaXRM

**In Situ & 4D:**
Highest res for *in situ* due to RaaD™

**Correlative Multi-scale**
Scout and Zoom to FIB