In Situ & 4D Science
Observing and quantifying the evolution of 3D microstructure
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Since the advent of the microscope, engineers and scientists have refined imaging techniques to observe 2D and 3D microstructures and quantify evolution over time, or resulting from stimuli such as temperature, force, etc. The vast majority of characterization techniques require samples to be destructively cut or consumed so that they could not be observed under realistic conditions over time.

ZEISS 3D X-ray microscopes (XRM) are uniquely suited to image materials in situ under variable environments in 4D (3D plus time) in order to non-destructively characterize and quantify the evolution of 3D microstructures. From mechanical materials testing to carbon sequestration investigations, innovative ZEISS features such as Dual Scan Contrast Visualizer (DSCoVer) and Automated Filter Changer facilitate a broad range of in situ research and discovery.

A new materials science has emerged in which samples are analyzed “in situ” to facilitate essential characterization of performance under relevant operating or environmental conditions. Measuring critical microstructural variation (e.g., pore size, crack propagation) in the same sample advances R&D scientific investigations and material reliability improvements by evaluating the impact of varying levels of:

- Tensile or compressive stress
- Temperature
- Electrical bias
- Pressure
- Chemical environment
- Gas or fluid flow

The non-destructive nature of X-ray microscopy is uniquely suited to studying materials in situ under variable environments with controlled conditions over time (4D). ZEISS’s unique “resolution at a distance” (RaaD) architecture and In Situ Interface Kit deliver the highest performance in situ 3D XRM characterization, maintaining high imaging resolution as the distance between the X-ray source and sample increases within in situ rigs. Overcoming the resolution limits of conventional micro-CT, Xradia Versa ensure peak performance.

Enabling New Discoveries
Continuing to push the limits for scientific advancement, ZEISS has emerged as the industry’s leader for in situ solutions, enabling the use of the widest variety of rigs, from high pressure flow cells to tension, compression and thermal stages. Conducting in situ studies at submicron resolution in 3D enables unprecedented capabilities for a diverse range of investigations essential for new discoveries in:

- **4D microstructural evolution:** to non-destructively determine the effect of environmental, stressed or real operating conditions on microstructure over time with samples repeatedly imaged to observe developments critical to material performance: crack propagation, corrosion or grain growth critical, etc.

- **Failure analysis studies:** to inspect semiconductor chips that appear structurally intact upon manufacture but can reveal weak interfaces under thermal stresses that may crack or deform under the heat of operation

- **Iteration between physical experiments and computational models:** to assess changes in structural evolution as samples with complex 3D pore pathways become permeated by fluid or gas, or experience temperature and compression changes. These changes can be correlated and iterated with finite element analysis models for 3D microstructure and even external stress or flow models

- **In operando or under operation device studies:** to understand aging and failure mechanisms in order to improve production processes for devices such as semiconductor packages under electrical bias or batteries and fuel cells during energy conversion
Why ZEISS

Spatial resolution, image contrast, and working distance are all key parameters characterizing the performance of an X-ray microscope. ZEISS has leveraged technology from its synchrotron heritage to revolutionize research with laboratory-based X-ray microscopy. Xradia Versa routinely demonstrate superior imaging performance (<700 nm spatial resolution, <70 nm voxels) compared to conventional laboratory- and synchrotron-based micro-CT solutions. Tunable propagation phase contrast, compositional contrast and absorption contrast on the submicron scale help to achieve superior results for imaging low-Z materials.

ZEISS in situ solutions solve the resolution and sample load issues of micro-CT to deliver robust stages with load capacities up to 15 kg and is the only X-ray solution able to maintain submicron resolution within larger chambers.

An innovative Dual Scan Contrast Visualizer (DSCoVer) allows flexible side-by-side tuning of two distinct tomographies for features normally indistinguishable within a single scan, while Automated Filter Changer capabilities enhance workflow efficiency.

Application Examples

Hydration and Crystal Formation

Understanding Wet and Dry States. A carbonate rock imaged in hydrated and non-hydrated states in salt water for water evaporation studies. Using DSCoVer, the combined resultant image can be tuned to highlight the regions of water evaporation and even salt crystal formation.

In Situ Crack Propagation

Rat mandible study: In situ compression study. Mandible was placed inside a tensile loading rig within the MicroXCT-400 and scanned while undergoing compression to examine crack initiation and propagation. Sample courtesy of Sunita Ha, University of California San Francisco.

In Situ Crack Propagation

Hydrated Carbon Fiber: 4D evaporation study. Scans over time noting evolution of structure as water evaporates. Sample courtesy of Dr. Ugur Pasaogullari, University of Connecticut.
In Situ Thermal and Mechanical Study

High Contrast Imaging of Porous Polymer In Situ Under Heat and Stress. Volume rendering of polymer used in biomedical applications demonstrates segmented struts. Flow simulation demonstrates permeability of polymer in X, Y, Z directions. Color map of blue to red demonstrates fluid velocity, showing permeability an order of magnitude higher in the Y direction. Images courtesy of Dr. M. Badiger, National Chemical Laboratories, India.

In Situ Foam Compression

Silicone Foam (PDMS) in 3D. Bulk properties such as void volume measured under 20% (left) and 50% (right) loads of compression. Image courtesy of Dr. Brian Patterson, Los Alamos National Lab

Nanoscale Polymer Electrolyte Fuel Cell (PEFC) Study

Fuel Cell Electrode. PEFC characterized by nanoscale Xradia 800 Ultra provided accurate distributions of particle agglomerate sizes and pore dimensions needed to feed computational models potentially including effects of environmental humidity. Image courtesy of Shawn Litster and William Epting, Carnegie Mellon University

In Situ Fluid Flow

Limestone Flow Cell Study. Supercritical CO\textsubscript{2} in keton limestone, before segmentation and after showing CO\textsubscript{2} storage capability. Image courtesy of Matthew Andrew, Qatar Carbonates and Carbon Storage Research Centre, Imperial College London
Resolution at a Distance (RaaD)
Resolution can degrade as working distance increases on conventional micro-CT machines that rely solely on small spot size and geometric magnification. ZEISS’s unique Xradia Versa architecture maintain high resolution over large working distances. Xradia Versa use a patented detector system with scintillator coupled with visible light optics, a design rooted in our synchrotron heritage, and has a dual magnification capability that reduces dependence on geometric magnification. This enables imaging of large samples (100 mm in diameter), including samples contained in in situ chambers, to be imaged at submicron levels the microscope maintains high resolution at large working distances, as indicated in the graph below.

Optional In Situ Interface Kit
Available with the Xradia Versa family of instruments, this optional kit contains: a mechanical integration kit, robust cabling guide, facilities feed-throughs, and recipe-based software capability that simplifies operation from within the Xradia Versa user interfaces.

Xradia Versa combine the highest level of stability, flexibility and controlled integration of such in situ devices, which benefit from an optical architecture that doesn’t compromise resolution in imaging variable environmental conditions.

Suggested Reading:


