Technical Note

In Situ & 4D Science

Observing and Quantifying the Evolution of 3D Microstructure

Seeing beyond

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 their evolution over time, or as the result of external stimuli such as temperature, force, etc. The vast majority of 3D characterization techniques require samples to be destructively cut or consumed, rendering observation under realistic conditions over time impossible. 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 materials testing under mechanical load or at elevated temperatures, to observing flow in porous media, ZEISS environmental sample holders and innovative features such as "resolution at a distance" (RaaD) facilitate a broad range of *in situ* and 4D 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. The non-destructive nature of X-ray imaging uniquely enables measuring and quantifying the microstructure of the same sample (e.g. pore size, crack propagation) as it changes under the impact of varying levels of:

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

Overcoming the resolution limits of conventional micro CT, ZEISS Xradia Versa is uniquely suited to meet the requirements of the most demanding experiments. Studying materials *in situ* under controlled conditions is made possible by ZEISS Xradia's unique "resolution-at-a-distance" (RaaD) architecture, which facilitates high resolution imaging even as the distance between X-ray source and sample increases to accommodate environmental chambers. *In situ* interface kits facilitate the integration of both off-the-shelf sample environments available from ZEISS, or customer-designed solutions for novel applications.

Extending *in situ* and 4D imaging to the nanoscale, ZEISS Xradia Ultra are the only non-destructive 3D X-ray microscopes offering laboratory resolution down to 50 nm. This provides a unique portal between non-destructive submicron 3D X-ray imaging and serial sectioning using electron microscopy, which provides nanometer resolution but where the sample is destroyed in the process of imaging.

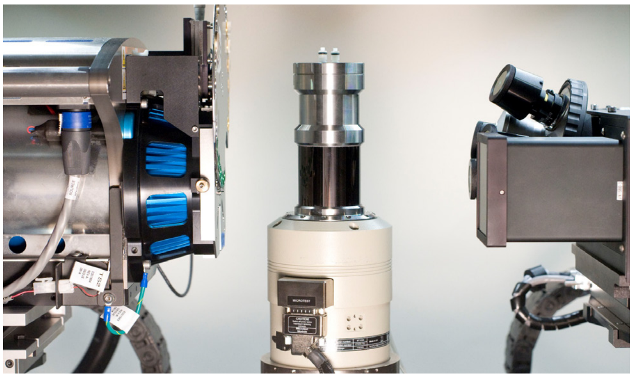


Figure 1 *ZEISS Xradia Versa with X-ray source (left), sample stage with tension/compression stage (center), and detector (right). Even with source to sample distances of several centimeters, required to accommodate* in situ *sample holders, voxel sizes below one micron can be achieved.*

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 sample holders, from high pressure flow cells to tension, compression and thermal stages.

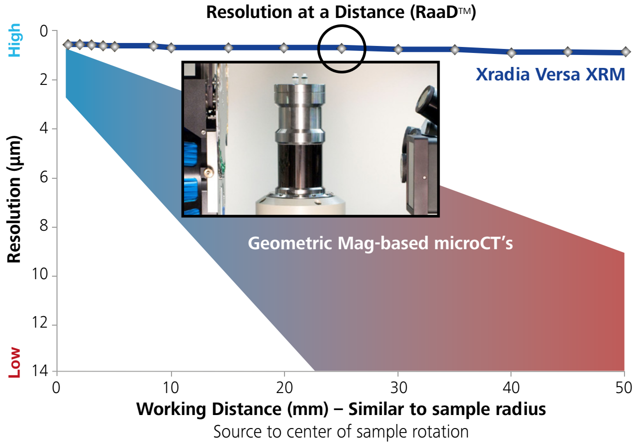
Cover Image In situ *foam compression study, rendering of pore structures.*
Images courtesy of Brian Patterson, Los Alamos National Laboratory

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 perfor mance, i.e., crack propagation, corrosion, grain growth.
- *Failure analysis studies:* to inspect electronics devices that appear structurally intact upon manufacture but can reveal weak interfaces under thermal stresses that may crack or deform under the heat of operation.
- *Fluid flow studies:* examine how multiple fluid phases flow and interact within a porous medium at representative process conditions, and compare with complex multiphase computational models to understand how fundamental processes such as fluid wettability interact with pore structure to influence macroscopic flow behavior.
- *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.

Resolution at a Distance (RaaD)

Conventional micro CT instruments use large pixel (~100 µm) flat panel detectors and rely solely on small spot size and geometric magnification to achieve high resolution. Thus, resolution degrades quickly as working distance increases. ZEISS's unique Xradia Versa architecture uses a patented detector system rooted in our synchrotron heritage, which provides small pixels down to <500 nm enabled by scintillators coupled to visible light optics.[1,2] The optical magnification in the detector reduces dependence on geometric magnification and preserves high resolution (small voxels) at large working distances. Large samples around 100 mm in size, or samples contained within *in situ* devices, can be imaged at submicron resolution (see chart below).



Application Examples

Evolution of Surface Defects (1.5 µm Voxel)

Evolution of Voids (1.5 µm Voxel)

Figure 2 *Tensile testing of a steel laser weld under increasing load. The data reveals a crack initiating and propagating from a rough surface imperfection, as well as the elongation of internal voids.* Performed in collaboration with Sandia National Laboratories. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Application Examples

In situ *Compression and Study of Microstructural Evolution*

Average porosity values under rib and channel at 0, 1, 2, 3 MPa non-uniform compression pressure.

Compression Pressure	0 MPa	1 MPa	2 MPa	3 MPa
Porosity (%)	Rib: 72.20 Channel: 72.20	Rib: 71.54 Channel: 71.54	Rib: 67.68 Channel: 71.00	Rib: 60.30 Channel: 70.00
Pentration length into the channel area (µm)	0	28	61	132

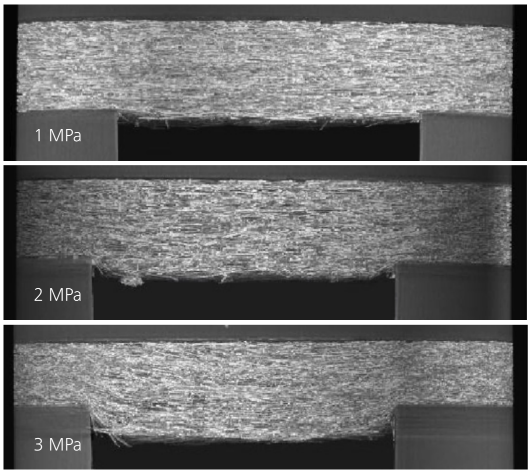


Figure 3 *XRM virtual cross sections of a carbon paper gas diffusion layer (GDL) used in a polymer electrolyte fuel cell electrode. The GDL layer is examined* in situ *under varying states of non-uniform compressive load to investigate the microstructural effects indicative of an operating cell. The 3D data sets are analyzed to determine porosity change as well as protrusion of the GDL fiber structure into the neighboring gas flow channel.*
N. Khajeh-Hosseini-Dalasm et al. / Journal of Power Sources 266 (2014) 213-221. Image and table reprinted with permission from Elsevier. [3]

Progressive Crack Growth

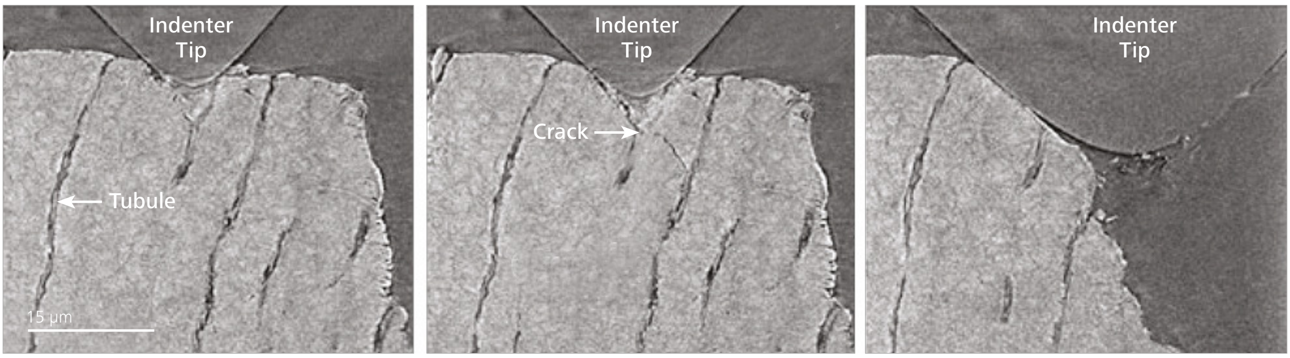


Figure 4 *Progressive crack growth in elephant dentin imaged at 64 nm voxel size (150 nm spatial resolution), engineered by Ultra Load Stage in indentation mode.*
Courtesy of The University of Manchester. [4]

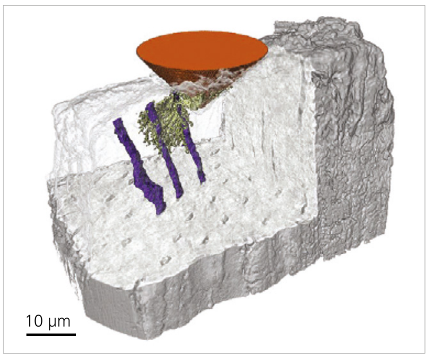


Figure 5 *Rendering showing the 3D morphology of a selected crack (yellow) in relation to neighboring tubules (blue) and the indenter tip (orange).*
Courtesy of The University of Manchester.

Multi-phase Flow

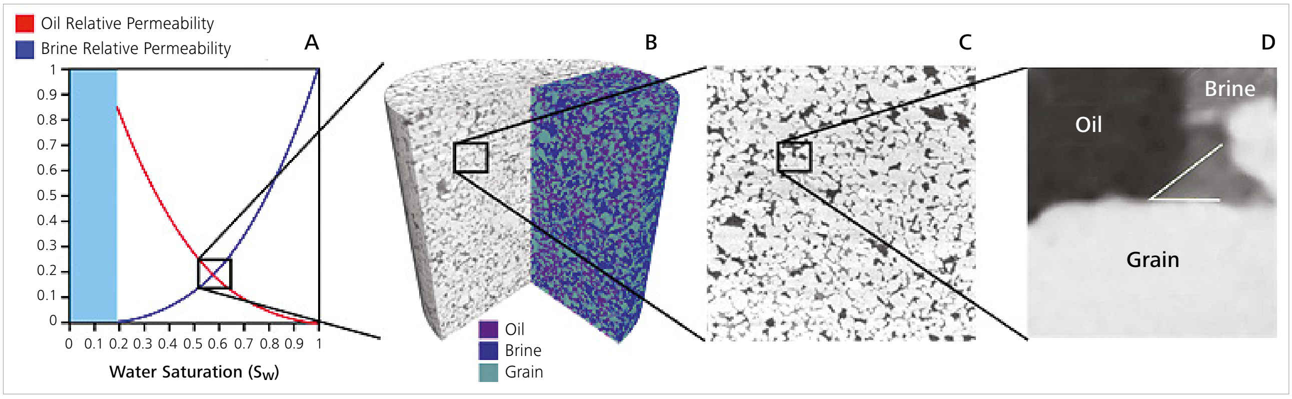


Figure 6 *Correlate core-scale relative permeability (A) with pore-scale fluid distributions (B-C) and* in situ *measurements of wettability and contact angle (D) at representative conditions all in the same experiment.*

Steady State Flow of Multiple Fluid Phases in Geological Samples

Correlate multiple scales of multi-phase flow behavior from core to pore on the same sample, allowing for core-scale changes in relative permeability to be described in terms of changes in pore-scale fluid distribution. This can be done by conducting full steady state flow experiments *in situ* within the ZEISS Xradia Versa. These data can then be compared with computer simulation results to validate and inform a new generation of pore-scale models. Fundamental controls on multi-phase fluid displacement, such as contact angle, can be measured directly on real samples at representative subsurface conditions by imaging fluid-fluid interfaces at high resolutions using the unique dual-stage architecture present within the ZEISS Xradia Versa.

Optional *In Situ* Interface Kit

Available with the ZEISS 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 ZEISS Xradia Versa user interfaces. This system simplifies the physical integration of both commercial and custom *in situ* chambers, along with their associated cables and connections, to enable easy sample rotation and tomography acquisition even with complex experiments.

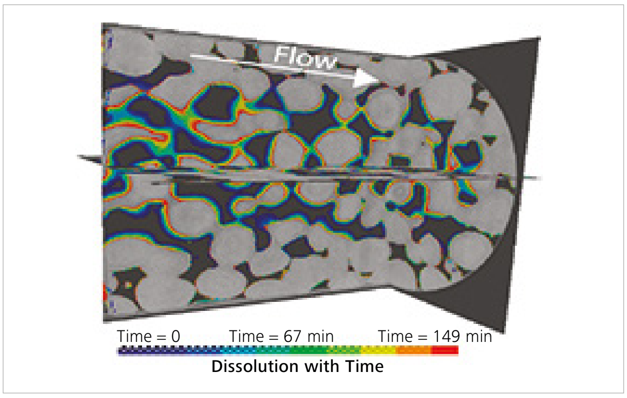


Figure 7 *Reactive fluids injected into a limestone, allowing for the progress of fluid-solid reaction to be tracked in a 4D manner with a time resolution of around 20 minutes, and porosity and permeability evolution to be examined.* [5]

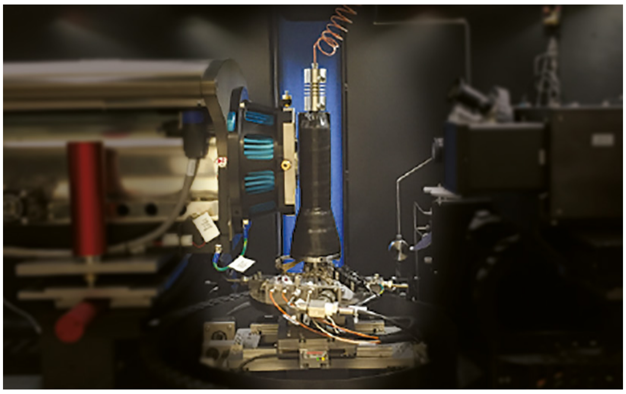


Figure 8 *Flow cell installed in ZEISS Xradia Versa.*

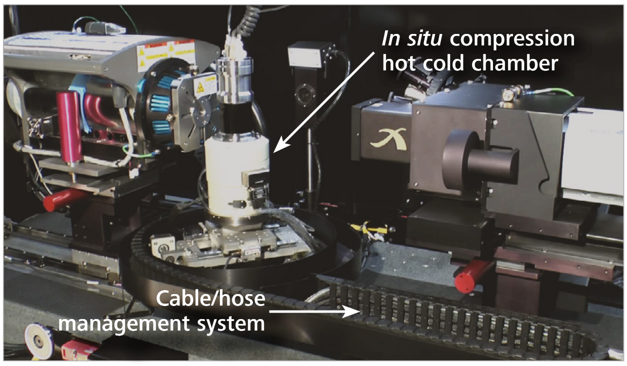


Figure 9 In situ *interface Kit for ZEISS Xradia Versa: the most flexible* in situ *and 4D solution accommodating the widest variety of* in situ *chambers*

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

Tension, compression and temperature stages for ZEISS Xradia Versa

A suite of tension, compression and temperature stages is available for use with ZEISS Xradia Versa, offering load ranges up to 5 kN, and temperature up to 180°C or down to -15°C. Leveraging ZEISS Xradia Versa's unique RaaD capability, *in situ* mechanical or thermal test-ing can be performed with resolution below one micron. ZEISS's user friendly Scout-and-Scan™ software interface enables automated acquisition of multiple tomograms to understand microstructural evolution at different load stages or temperatures.

ZEISS Xradia Ultra Load Stage

ZEISS Xradia Ultra Load Stage uniquely enables *in situ* nanomechanical testing – compression, tension, indentation – with non-destructive 3D imaging. Study the evolution of interior structures in 3D, under load, down to 50 nm resolution.

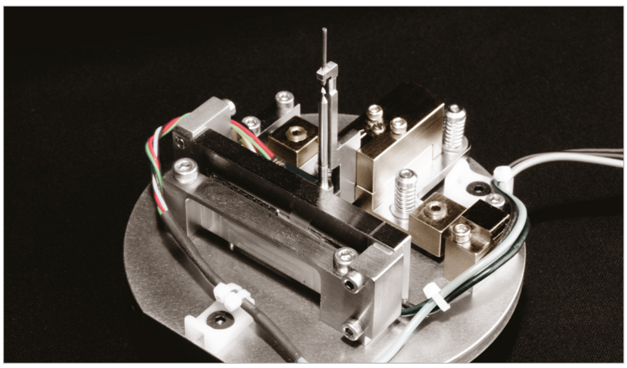


Figure 10 *ZEISS Xradia Ultra Load Stage for in situ nanomechanical testing*

Understand how deformation events and failure relate to local nanoscale features. Complement existing mechanical testing methods to gain insight into behavior across multiple length scales.

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.

ZEISS Xradia Versa routinely demonstrate superior contrast and resolution (500 nm spatial resolution, <40 nm voxels) compared to conventional 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 by delivering robust stages carrying up to 25 kg. ZEISS Xradia Versa is the only X-ray solution able to maintain submicron resolution within larger chambers.

ZEISS Xradia Ultra are the only laboratory based 3D X-ray microscopes achieving resolution down to 50 nm, offering both absorption and Zernike phase contrast to image a wide range of materials. Together with Ultra Load Stage, ZEISS Xradia Ultra is the only solution for *in situ* mechanical testing in a laboratory based 3D X-ray microscope.

ZEISS *in situ* solutions for 3D X-ray microscopy

ZEISS Xradia Versa submicron 3D XRM

- Tension and compression stages with load up to 5 kN
- Heating / cooling options from -15°C to 180°C
- *In situ* interface kit: cable/hose management system
- Reservoir condition flow cell for multiphase flow

ZEISS Xradia Ultra nanoscale 3D XRM

- Tension, compression and indentation stage with load up to 9 N

References and Suggested Reading

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