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Title: Uniaxial Compression of Cellular Materials at a 10-1 s-1 Strain Rate Simultaneously with Synchrotron X-ray Computed Tomographic Imaging

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Uniaxial Compression of Cellular Materials at a 10^{-1} s^{-1} Strain Rate Simultaneously with Synchrotron X-ray Computed Tomographic Imaging

Brian M. Patterson
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Multi-Institutional Collaborative Effort

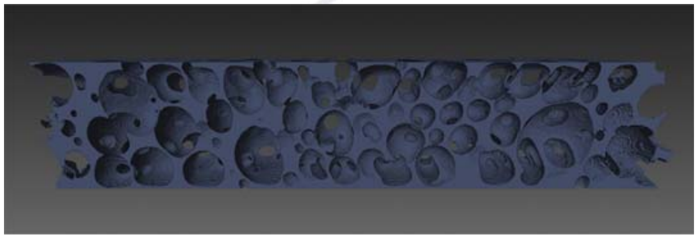
- **Los Alamos National Laboratory**
  - Brian M. Patterson, Robin Pacheco, Kevin Henderson, Nikolaus L. Cordes, James CE Mertens
- **Arizona State University**
  - Nikhilesh Chawla, Angel R. Ovejero, Tyler Stannard, Jason Williams, Sudhanshu Singh, S. Singaravelu, C. Shashank Kaira
- **Atomic Weapons Establishment**
  - Mathew Robinson
- **National Security Campus**
  - Carol Putman, Stephanie Schulze, John Dirvage
- **Argonne National Laboratory, Advanced Photon Source**
  - Xianghui Xiao

Motivation

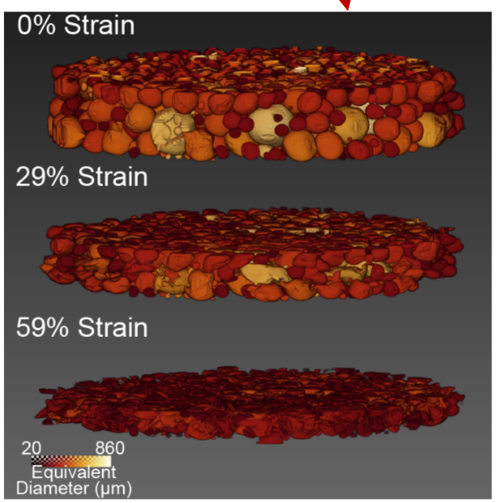
- X-ray tomography is a fantastic technique for characterizing a material's starting structure as well as for non-destructive, *in situ* experiments to investigate material response.
- 3D X-ray tomography is needed to fully characterize the morphology of cellular materials
- Lab-based micro-CT can provide initial conditions of the material.
  - Imaging is too slow to capture the stress relaxation
  - Experiment cannot be paused
- Synchrotron micro-CT can capture 3D images without pausing experiment.
- Provide data for LANL weapons modeling codes; model validation at STS conditions.

Understanding the mechanical response of hyper-elastic materials

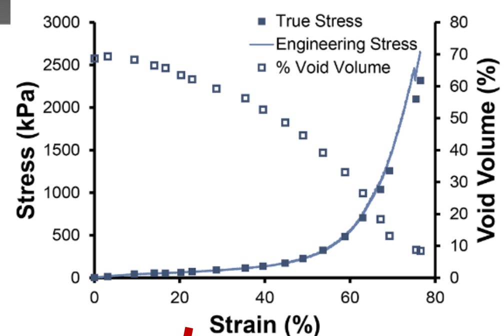
Microstructure



Segmentation / Morphology

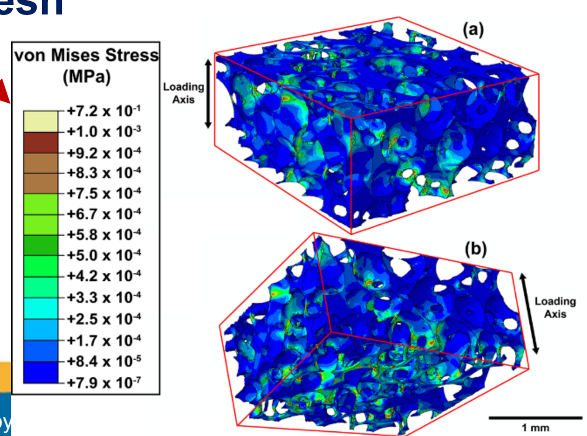


Structural Mechanical Properties

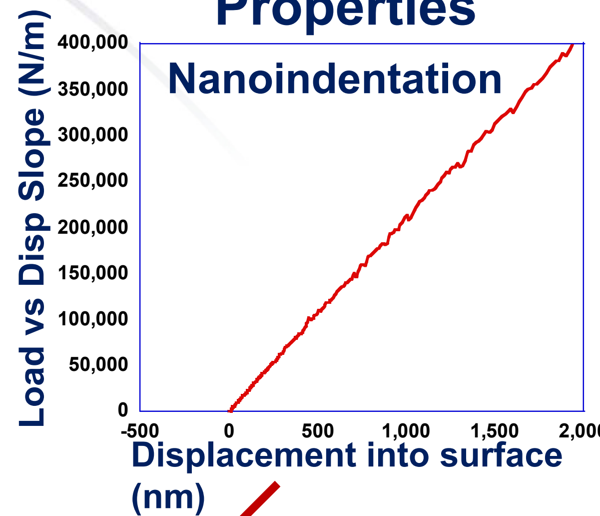


FEM

Mesh



Micro Mechanical Properties



Modulus / Hardness

Materials strategy framework

Goals: Performance prediction and controlled functionality

Themes: Defects & Interfaces | Extreme Environments | Emergent Phenomena

- Materials Dynamics
- Energetic Materials
- Integrated Nano-materials
- Complex Functional Materials
- Actinides & Correlated Electron Materials
- Materials in Harsh Env.
- Manuf. Science

Cross cuts: Making | Measuring | Modeling

Weapons Materials Strategic Planning Goals

- Facilitate reductions in uncertainty and improvements in agility for the US NWC
  - Improved understanding of materials (processing/structure/prop/performance)

- Improve collaborations among LANL/NWC
  - Subgroups organized by material/component
  - Generate proposals and work packages → DSW
  - Include all relevant organizations (T, W, etc.)

- Provide a planning framework for future program, research, and collaborations
  - Funded program to support R&D needed to develop new materials / manufacturing processes in support of future LEPs

Mesoscale Analysis (HE, foams, metals)

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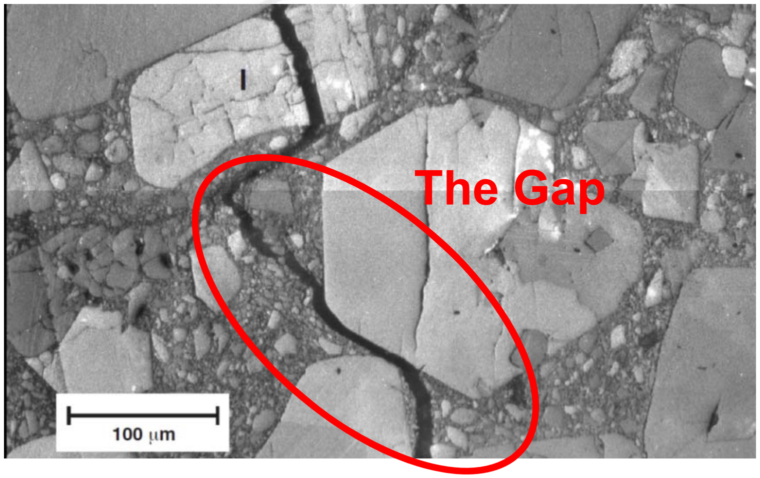
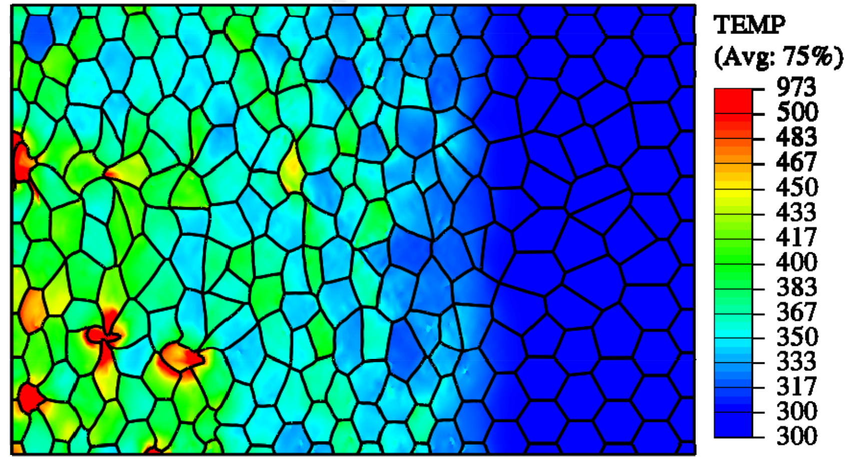
9501 Microstructure Image by P. Rae



Microstructural Simulation Image by D.J. Luscher

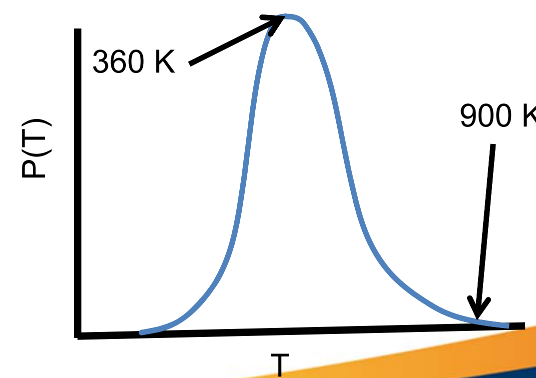


<u>The Gap is due to uncertainties in determination</u>:

- Grain size and orientation distribution (properties are anisotropic)
- Porosity
- Constituent properties (anisotropy, phase, history, temperature & rate dependence)
- Interface between constituents
- Small scale internal measurements for validation

Initiation and onset of damage are **local** phenomena

Temperature distribution from Microstructural Simulation



Micro/Nano CT Laboratory

Xradia Micro Computed Tomography

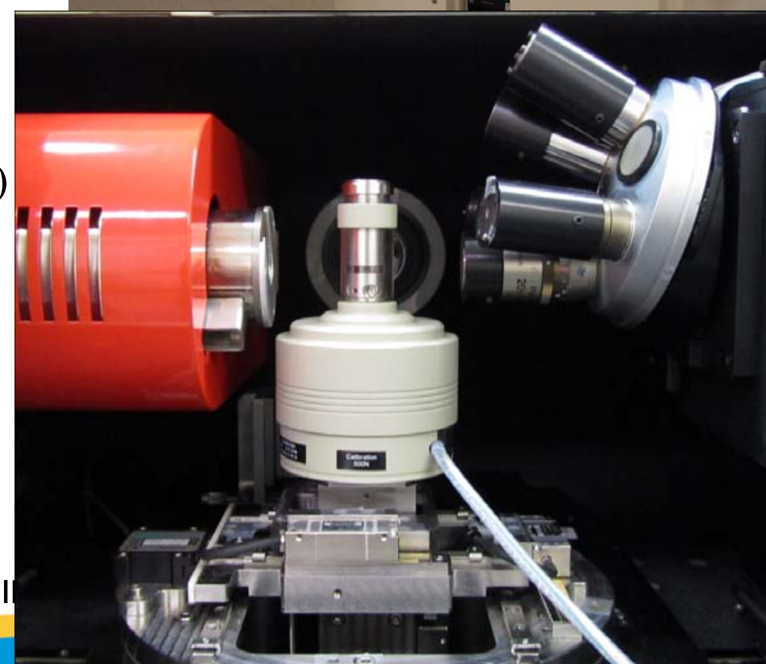
X-ray Source: Micro Focus Hamamatsu 5 µm spot, 40 – 150 kV acceleration voltage, 10 W total power (4W nominal)

Detector: 2k x 2k Electrically Cooled CCD, 4 objectives

Available Objectives:
2X 6 µm/ pixel, 12 mm FOV
4X 3 µm/ pixel, 6 mm FOV
10 X 1.2 µm/ pixel, 2.4 mm FOV
20X 0.6 µm/ pixel, 1.2 mm FOV (1.5 µm resolution)

In-situ studies:
Sample may be imaged in states of either tension or compression

Dynamic changes can be tracked in 3D



Highly Utilized LANL Capability

- Polymers
  - Foams (silicone, PU, PS), full density, in-situ, aged, irradiated, filled
- High explosives
  - Damage, crystal sizes, distributions
- Carbon fiber composites
  - Voids, fiber orientations
- Metals
  - Alloys, damage, eutectics, foams
- Physics targets
  - Low density, coated materials
- Additive Manufacturing
  - Polymers and metals, voids, surface finish
- High surface area materials

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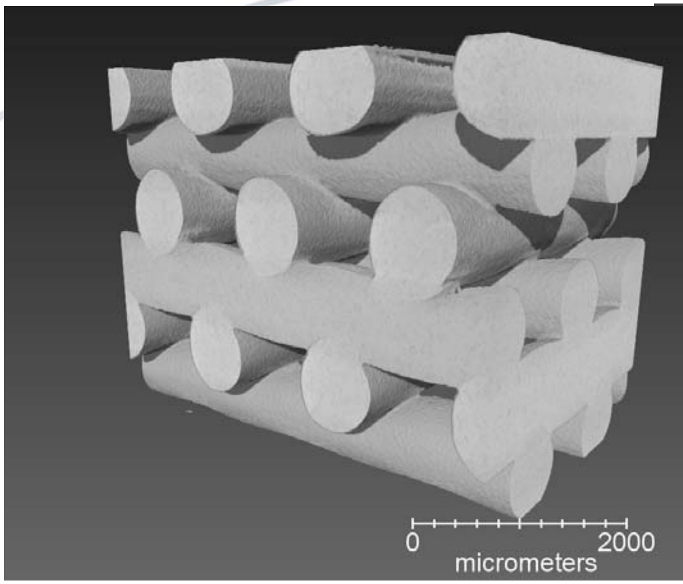
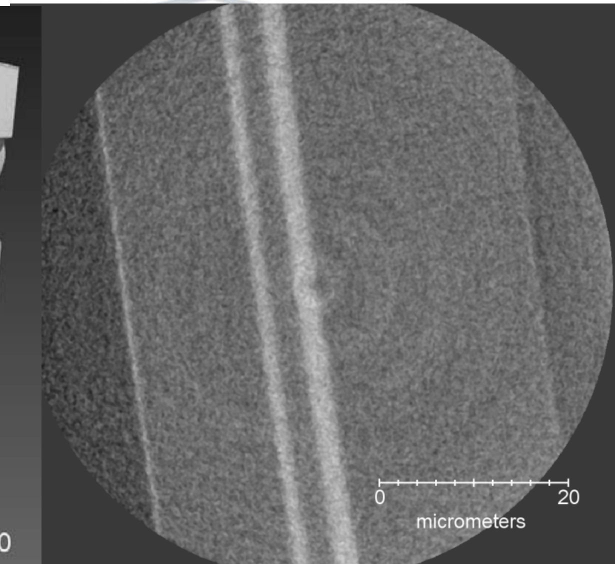
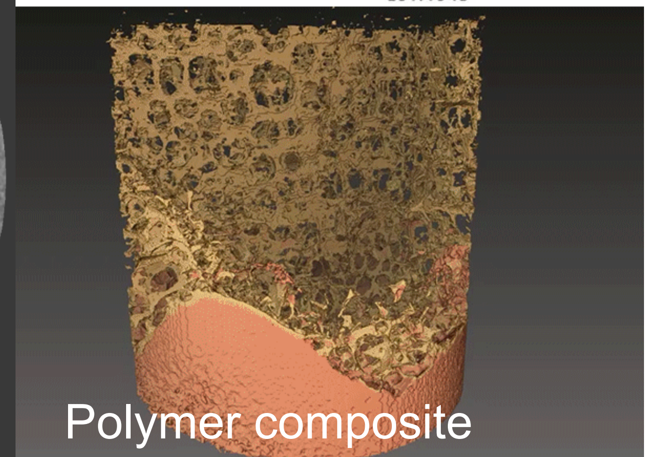
3D printed polymers

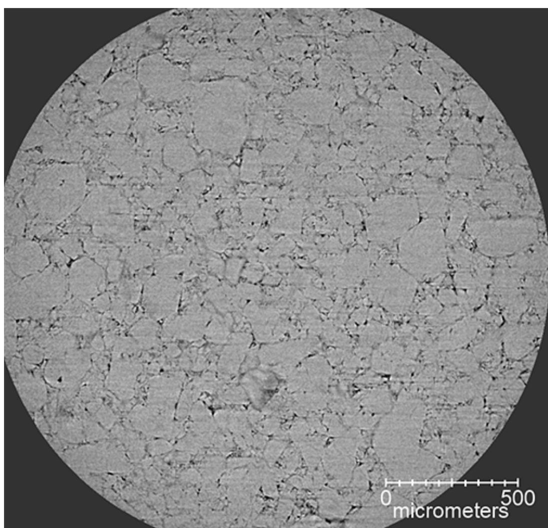


Fusion capsule shell

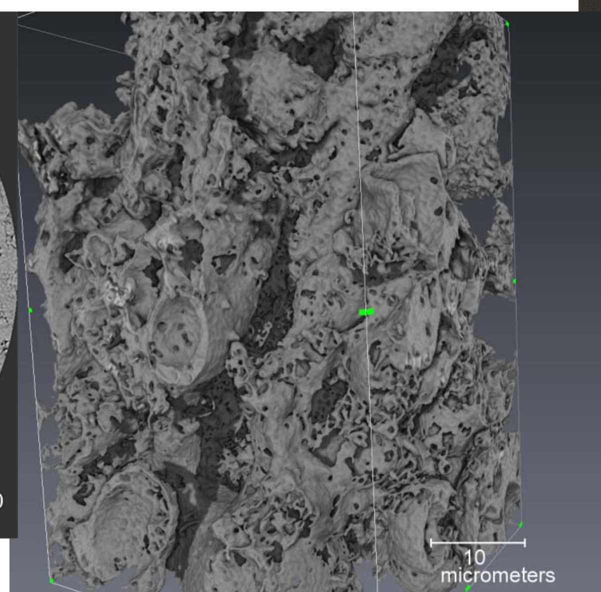




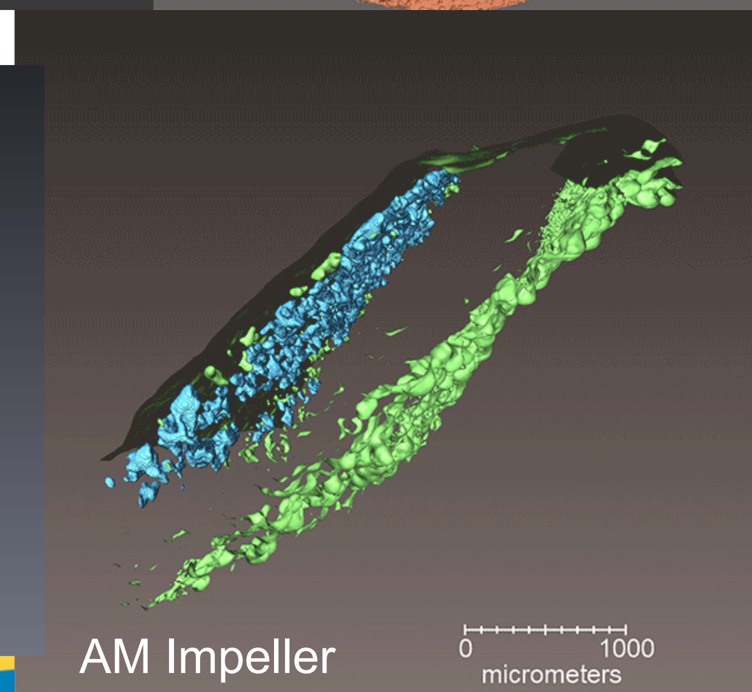
Polymer composite



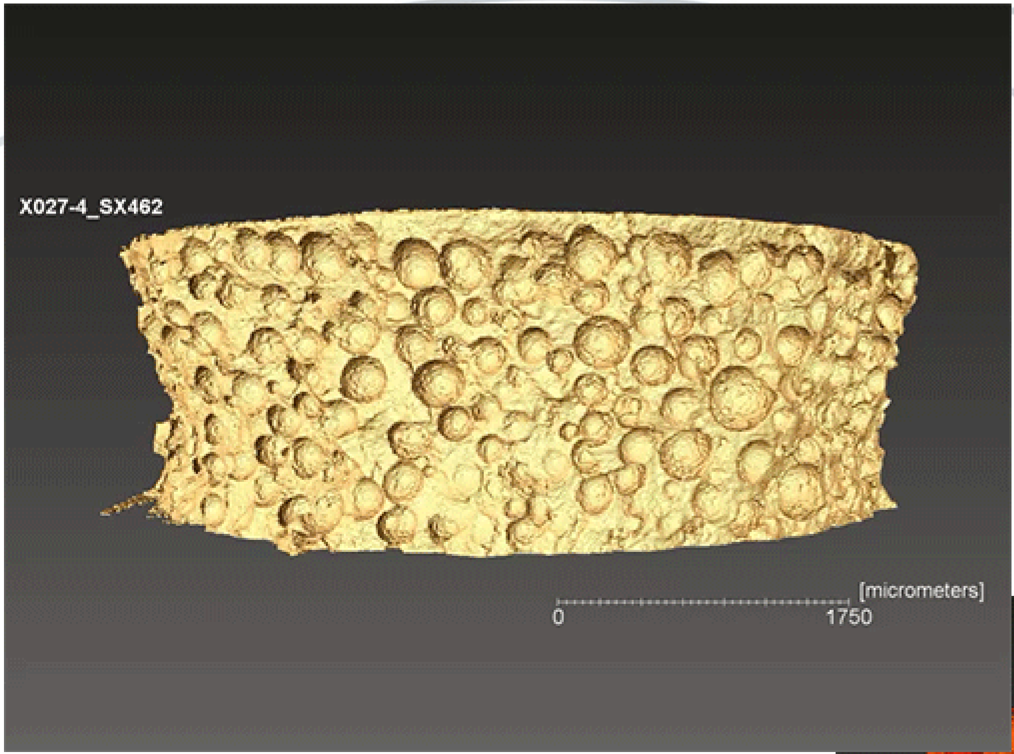
High Explosives

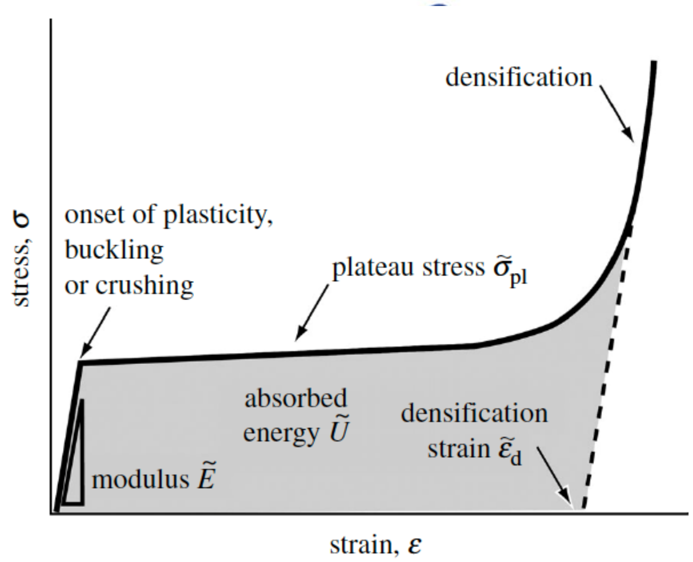


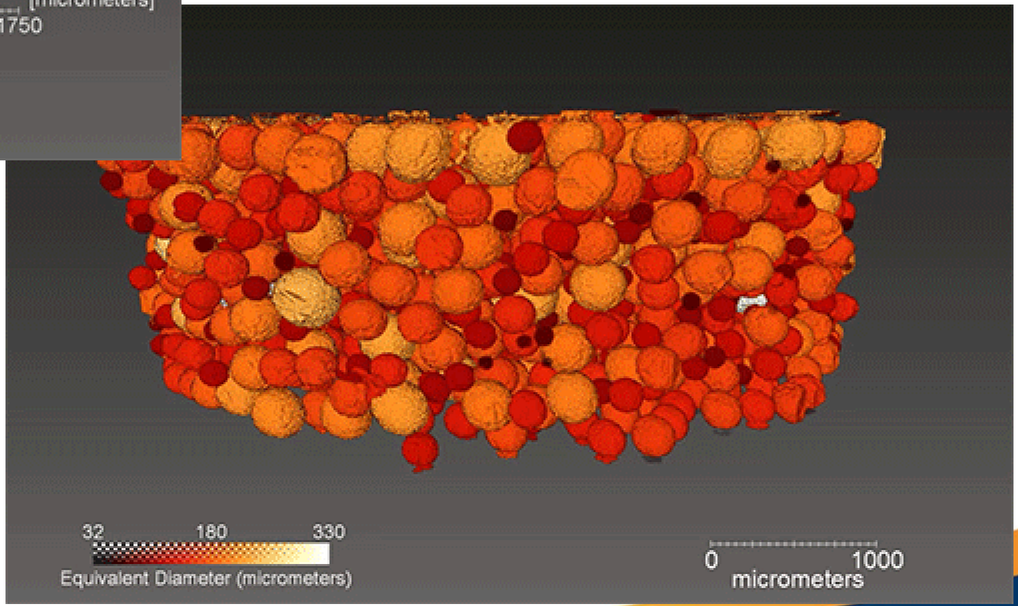
Low density structure



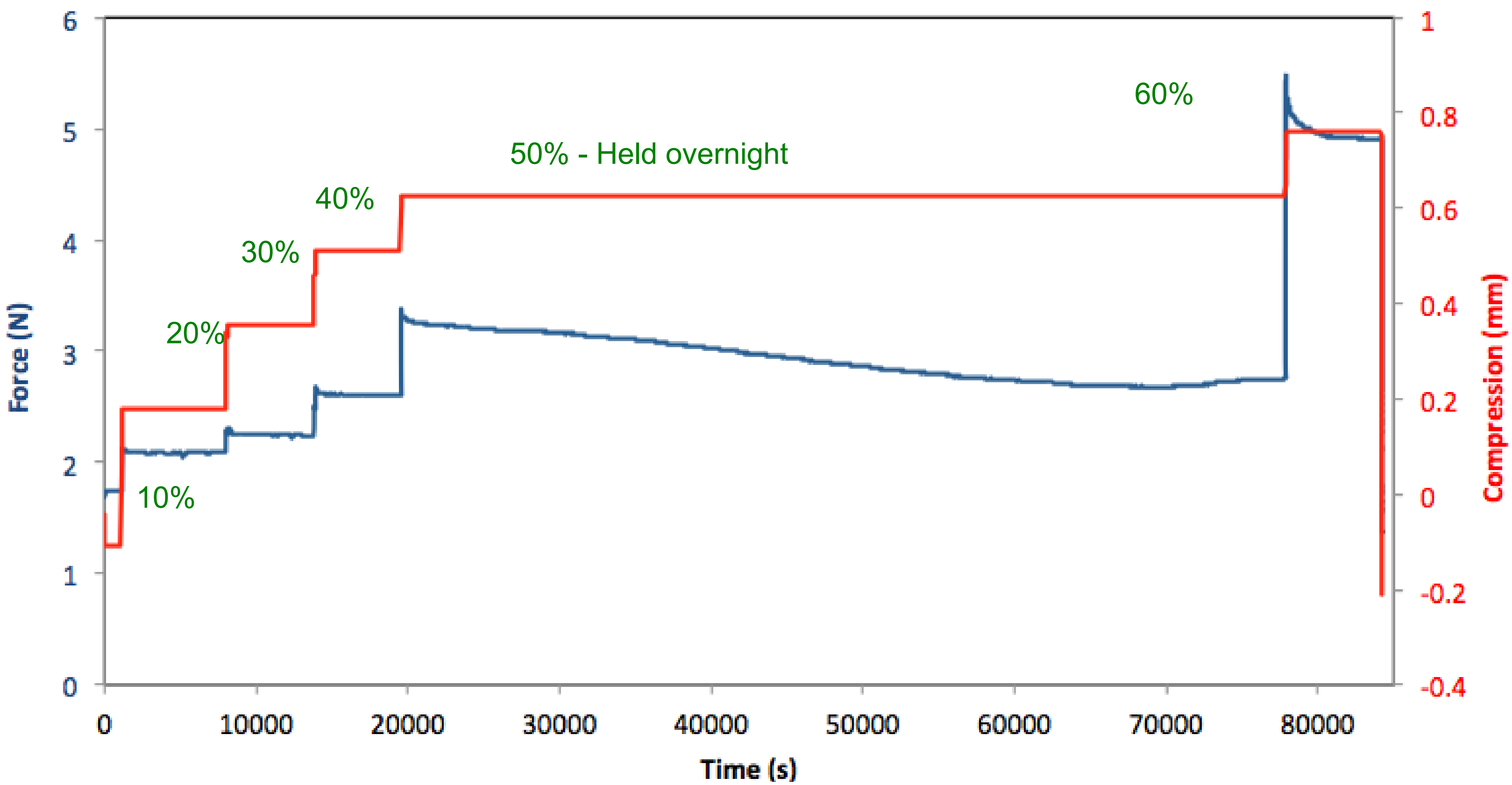
AM Impeller


X027-4_SX462
[micrometers]
0
1750


densification
onset of plasticity,
buckling
or crushing
plateau stress $\tilde{\sigma}_{pl}$
stress, σ
absorbed
energy $\tilde{U}$
densification
strain $\tilde{\varepsilon}_d$
modulus $\tilde{E}$
strain, ε


32
180
330
Equivalent Diameter (micrometers)
0
1000
micrometers

Stress Relaxation



B. M. Patterson, Kevin Henderson, Zachary Smith, J. Mat. Sci., (2013), 48(5), 1986-1996

Synchrotron Imaging During Loading

- Understanding the mechanical properties of hyperelastic materials is a complex materials science challenge

- The non-linear (neo-Hookean) response is difficult to capture
  - Bending
  - Buckling
  - Densification
  - Crack initiation and growth
  - Recovery

- Characterization must not stop the experiment; elastic response cannot be paused

Synchrotron Tomography

- High speed 3D images of materials during loading (novel capability)
- Improved understanding of fracture initiation and growth
- Directly feed morphological structure into the models; model robustness
- Strain rates of relevance
- Materials: silicone foams, AM materials, HE
- Organizations: NSC, AWE, Sandia

The Advanced Photon Source, Argonne National Laboratory

Beamline 2-BM

Beamline Specs	
Source	Bending Magnet
Monochromator Type	Multilayer monochromator
Energy Range	11-35 keV
Resolution (ΔE/E)	1 x 10^{-2}
Flux (photons/sec)	1 x 10^{12} @17 keV
Beam Size (HxV)	25mm x 4mm
Unfocused	
Monochromator Type	Pink-White Beam
Energy Range	10-170 keV
Flux (photons/sec)	1 x 10^{14} @ keV
Beam Size (HxV)	
Unfocused	25mm x 4mm

Meso Scale Dynamic CT Imaging of Polymeric Foam Materials

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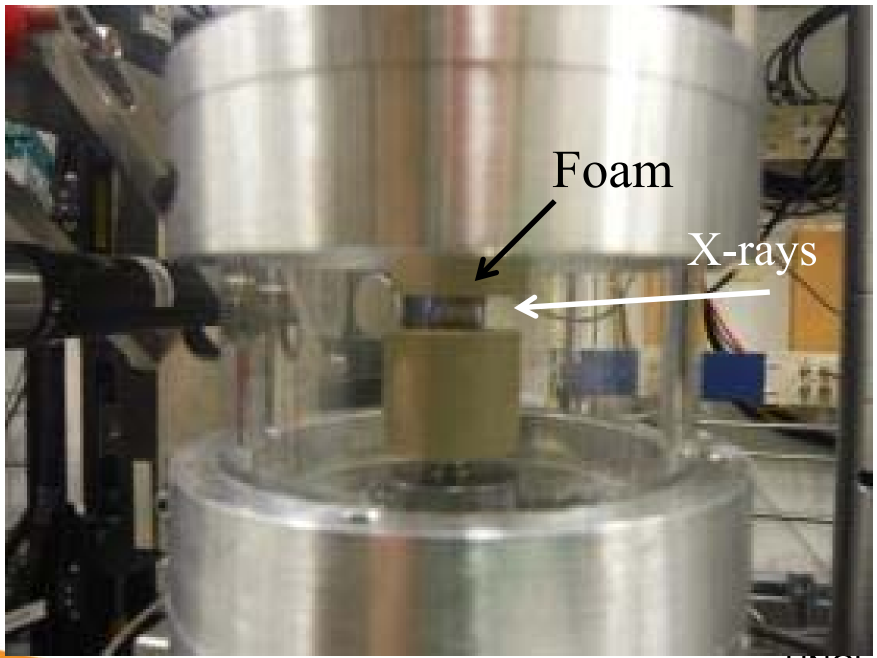
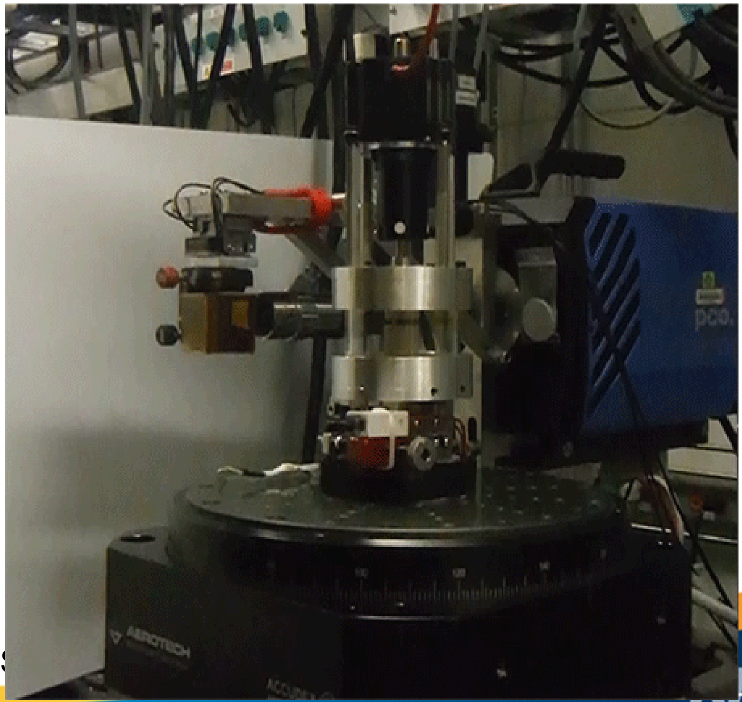
Using an *in-situ* load cell, we can acquire a full 3D image of a polymer foam in **one second**. Stage is rotated in a 'washing machine' approach back and forth. Each CT was separated by 4 seconds of 'spin up/ spin down'.

20 sets of dynamic CT's were successively collected as samples were simultaneously compressed up to ~60 %.

This data will be critical to providing true structural starting points and the tracked response will help validate our 3D models of their various formulations, structures and the resultant mechanical performance.





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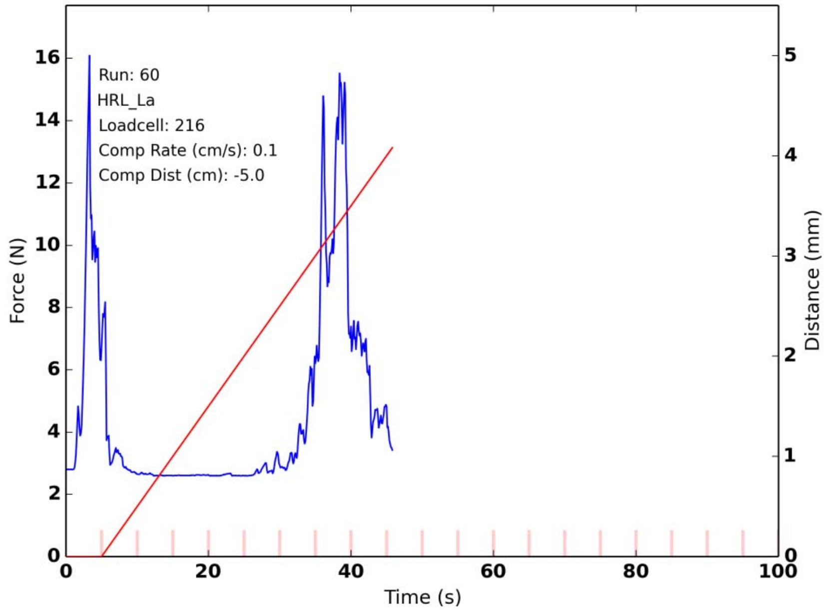

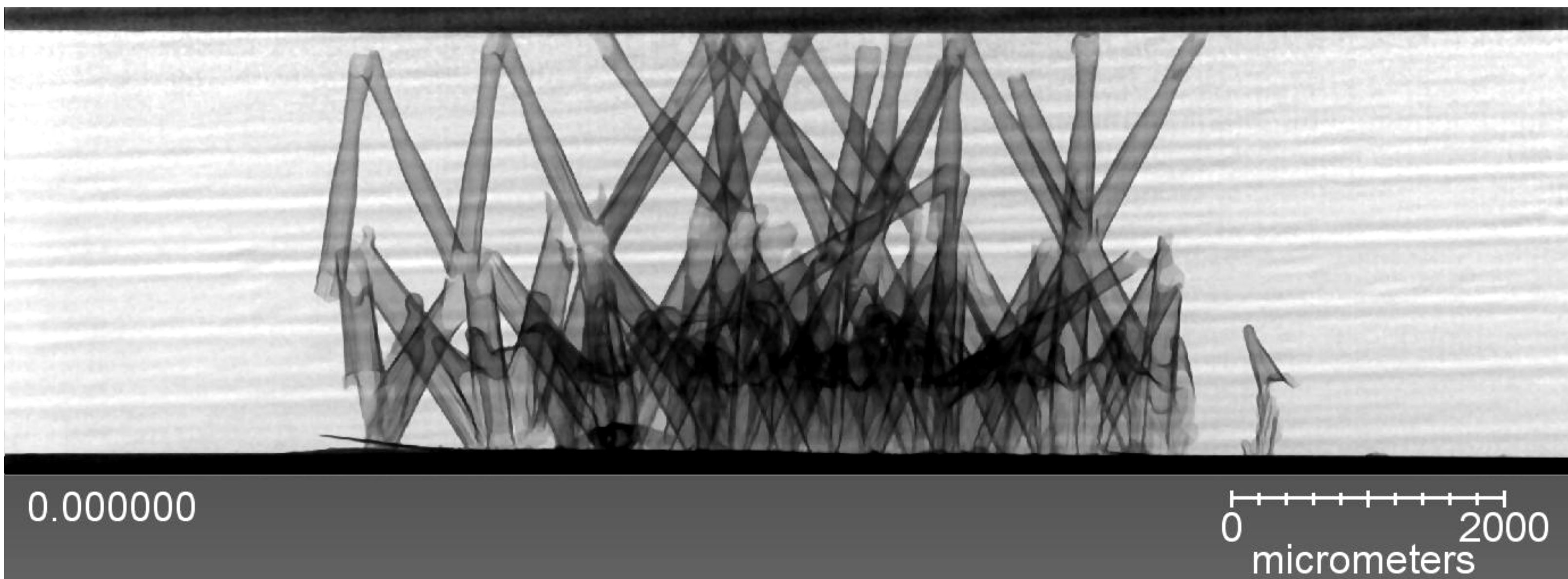

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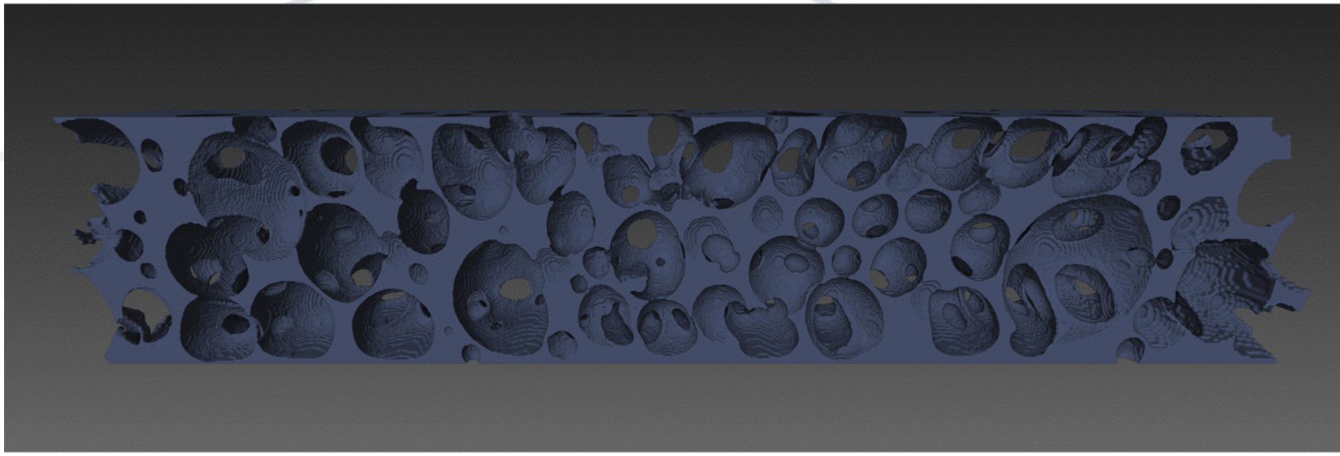
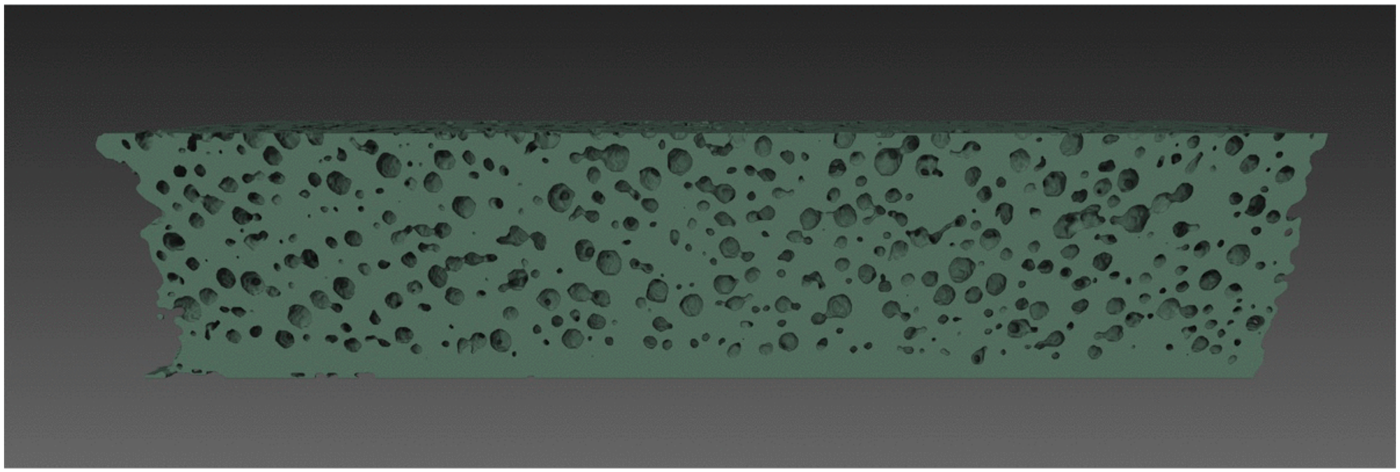
In situ compression

9x10^{-3}

LK3626



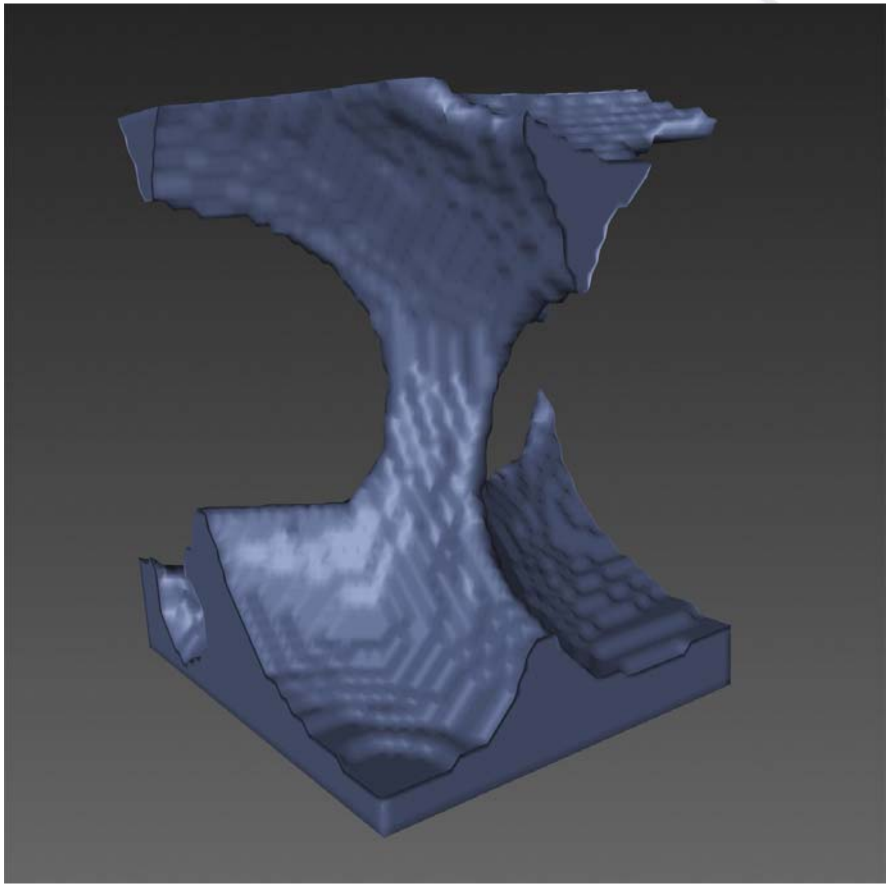
1.1x10^{-2}



Syntactic sylgard

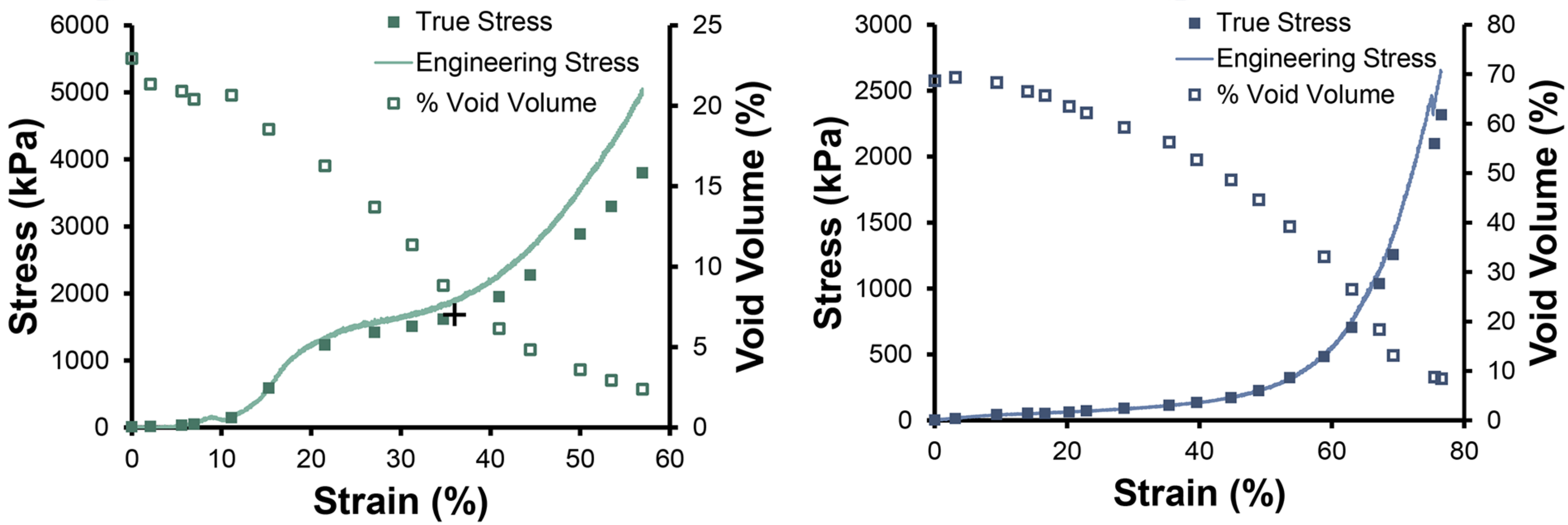
1 mm

Single ligament bend/buckle

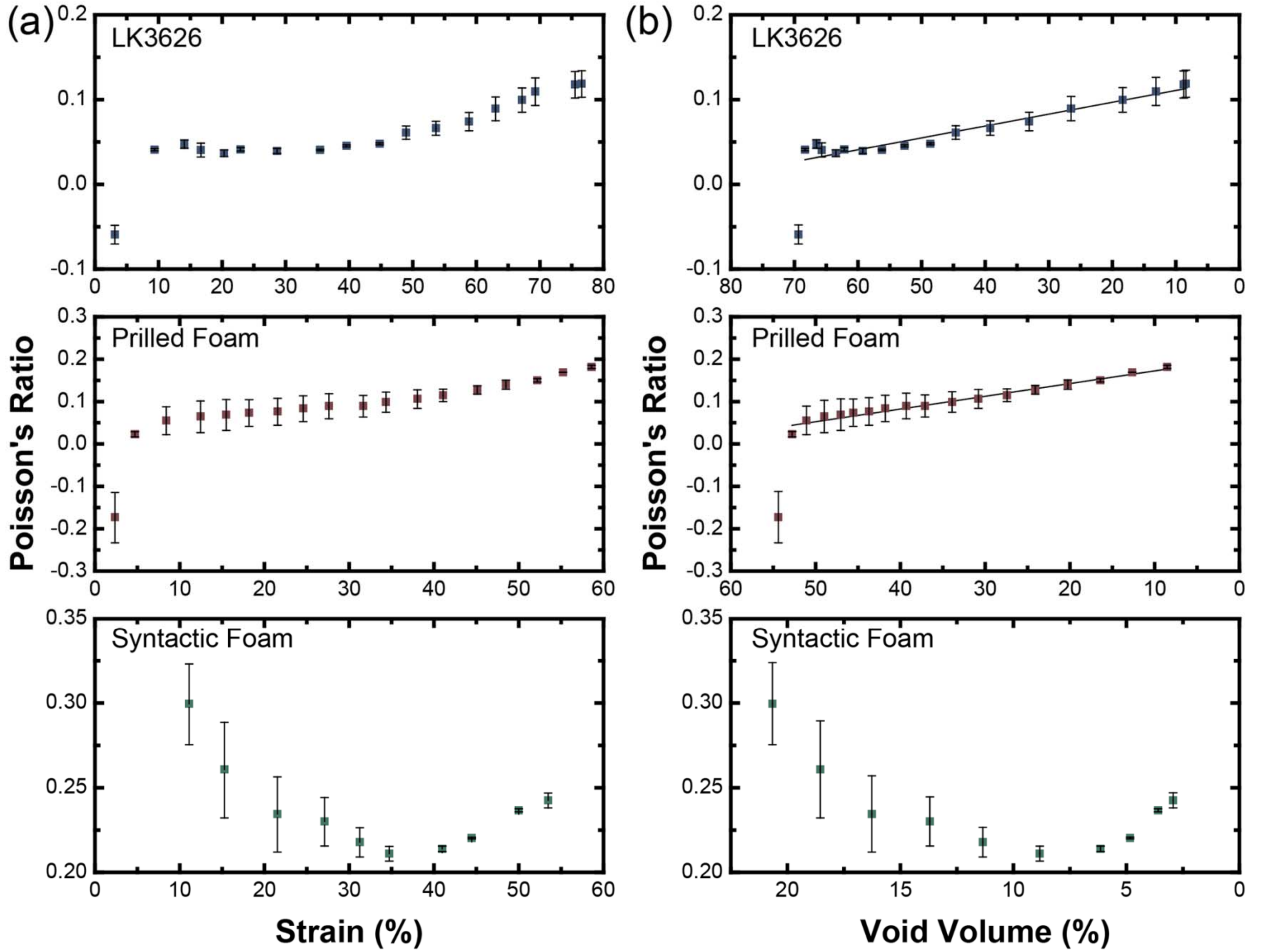


200 micrometers

CT can measure true stress

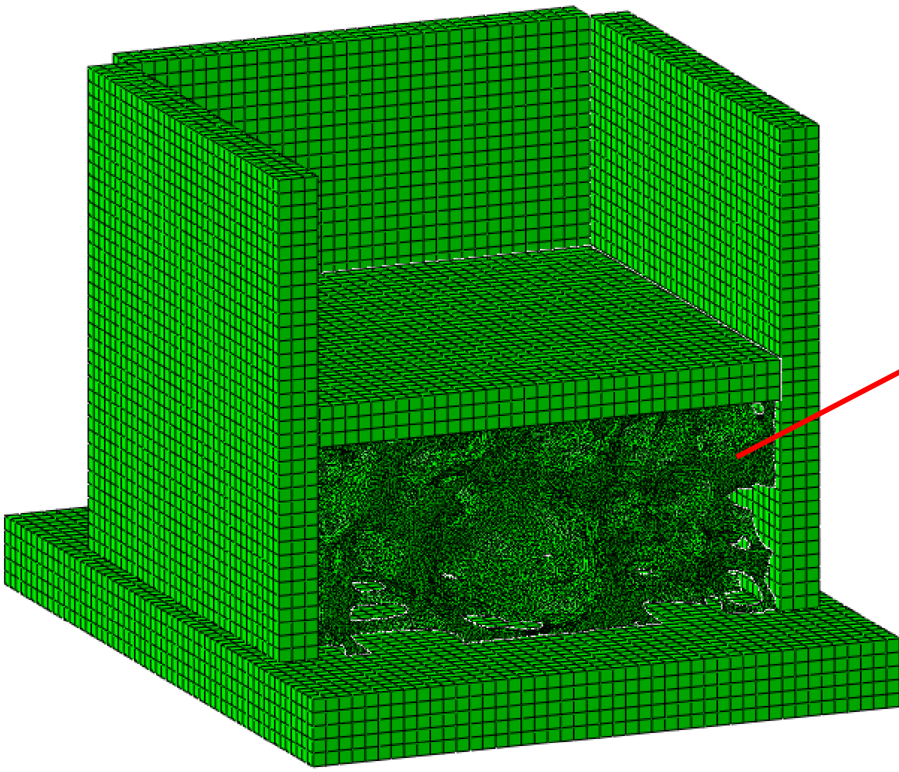


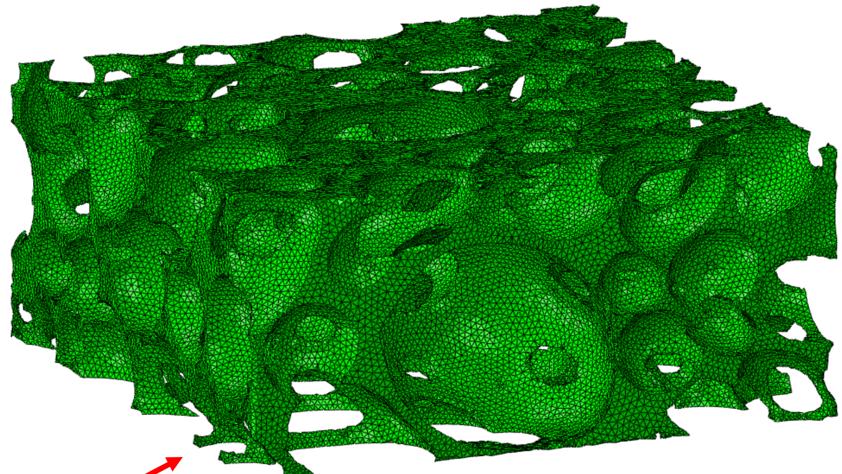
Poisson Ratio



Modeling in Abaqus

The meshed volume was imported in Abaqus to model the deformation





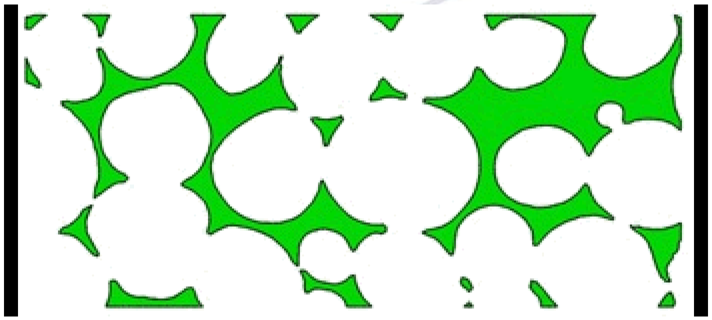
CT3 3D Model Information

- Abaqus elements used C3D4 (Tet's)
- Foam size 2 x 2 x 1 – mm
- E = 50 MPa and υ = 0.4
- Note: Front plate has been removed for visualization purposes

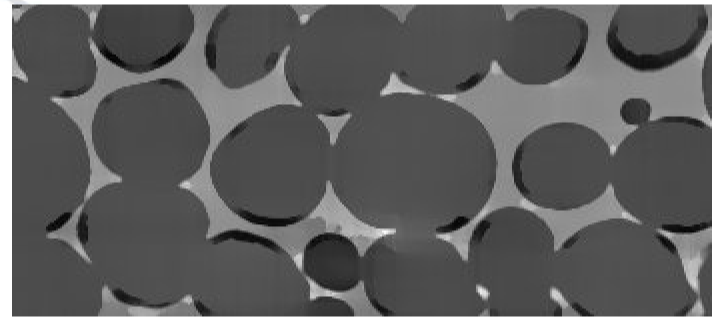
Deformation Comparison at Different Locations

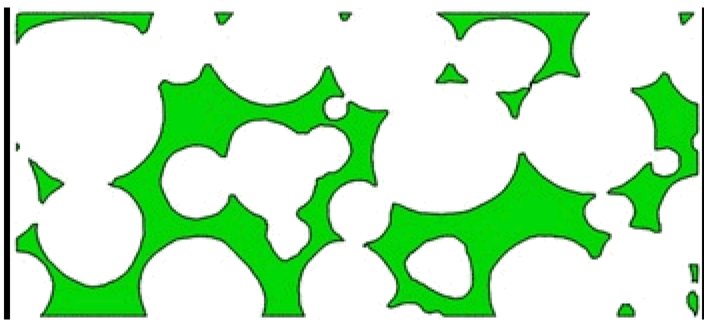
FEM

Gray scale images

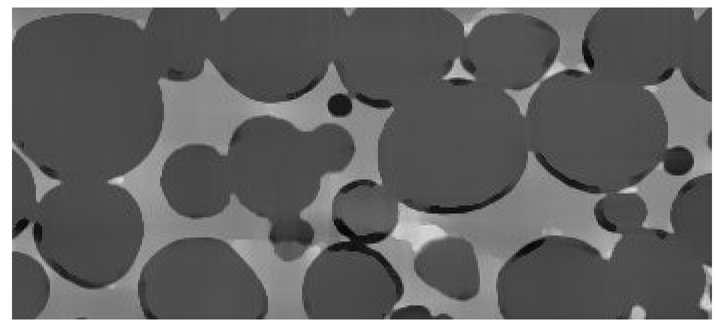


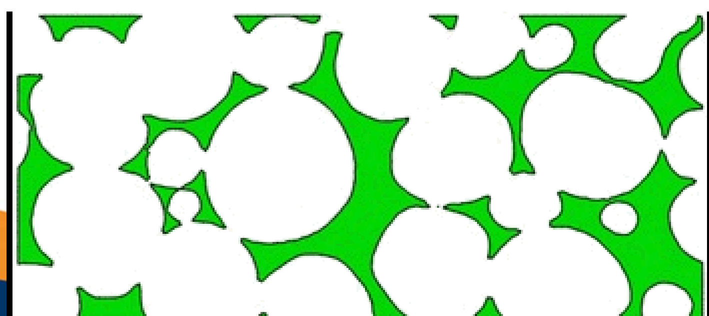
7th slice



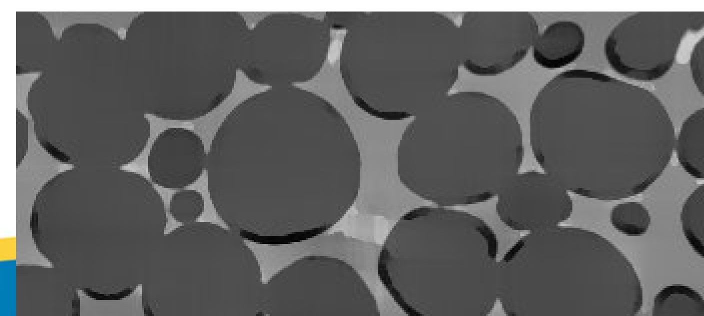


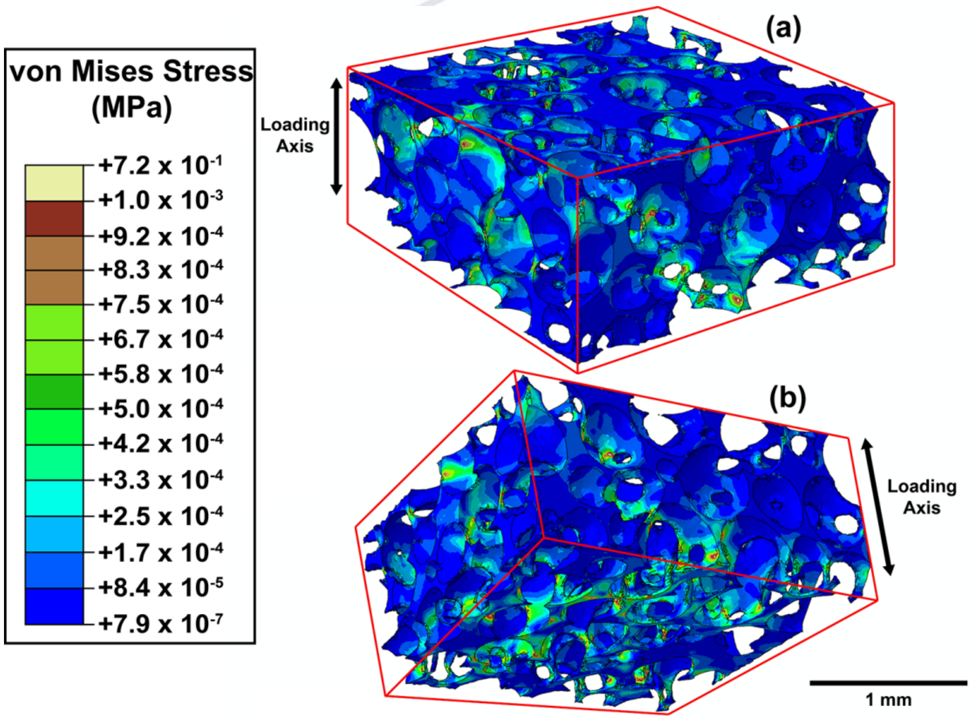
101st slice

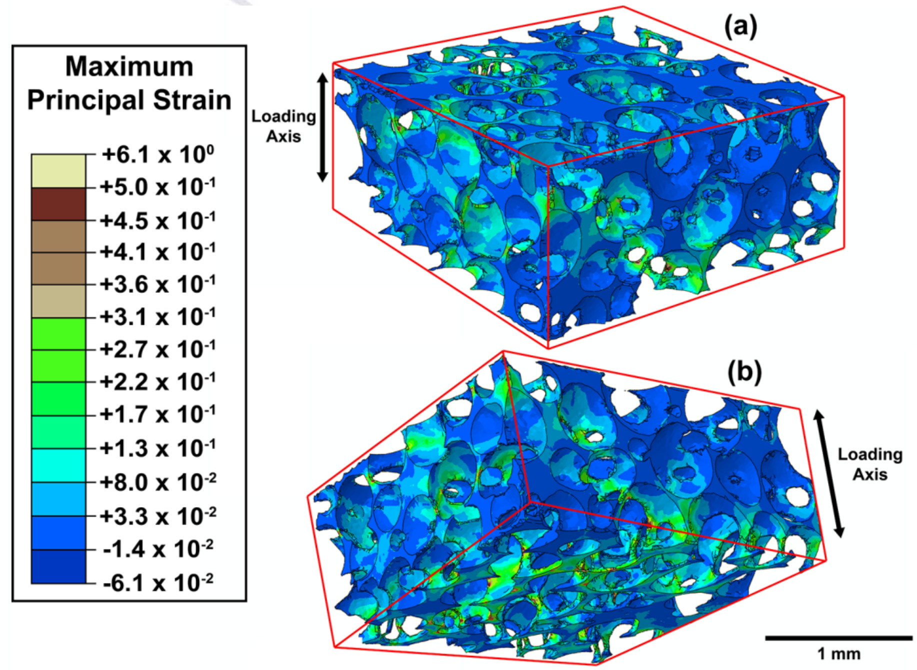




119th slice



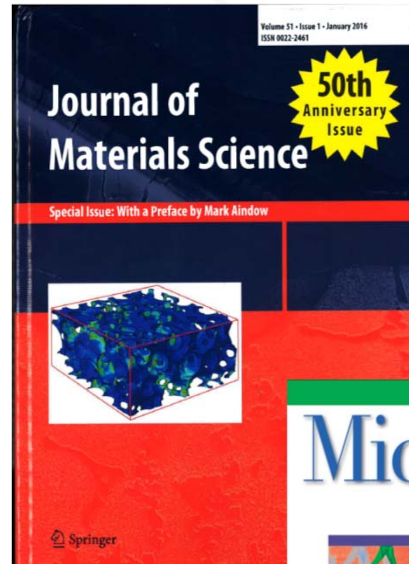

von Mises Stress (MPa)
+7.2 x 10^{-1}
+1.0 x 10^{-3}
+9.2 x 10^{-4}
+8.3 x 10^{-4}
+7.5 x 10^{-4}
+6.7 x 10^{-4}
+5.8 x 10^{-4}
+5.0 x 10^{-4}
+4.2 x 10^{-4}
+3.3 x 10^{-4}
+2.5 x 10^{-4}
+1.7 x 10^{-4}
+8.4 x 10^{-5}
+7.9 x 10^{-7}
Loading Axis
(a)
(b)
Loading Axis
1 mm

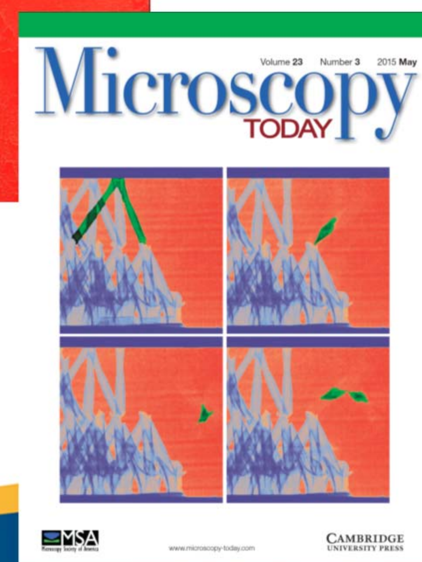

Maximum Principal Strain
+6.1 x 10^{0}
+5.0 x 10^{-1}
+4.5 x 10^{-1}
+4.1 x 10^{-1}
+3.6 x 10^{-1}
+3.1 x 10^{-1}
+2.7 x 10^{-1}
+2.2 x 10^{-1}
+1.7 x 10^{-1}
+1.3 x 10^{-1}
+8.0 x 10^{-2}
+3.3 x 10^{-2}
-1.4 x 10^{-2}
-6.1 x 10^{-2}
Loading Axis
(a)
(b)
Loading Axis
1 mm

Conclusions of completed work

Successfully demonstrated 10^{-2} strain rates while completed 3D imaging. We have applied a linear model to the data which agrees reasonably well with the collected images.

- Two manuscripts
  - Patterson, B. M., et al. (2016). "In situ X-ray synchrotron tomographic imaging during the compression of hyper-elastic polymeric materials." Journal of Material Science **51**(1): 171-187.
    - Nominated for the Cahn Prize by the Journal of Materials Science
  - Cordes, N. L., et al. (2015). "3D Imaging of Advanced Cellular Materials using in-situ Synchrotron-based X-ray Computed Tomography during Uniaxial Compression Loading." Microscopy Today.

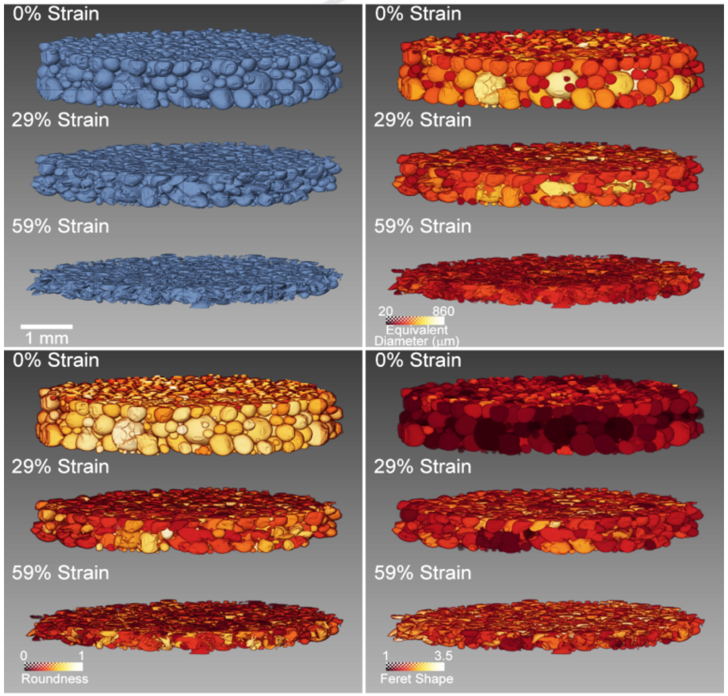


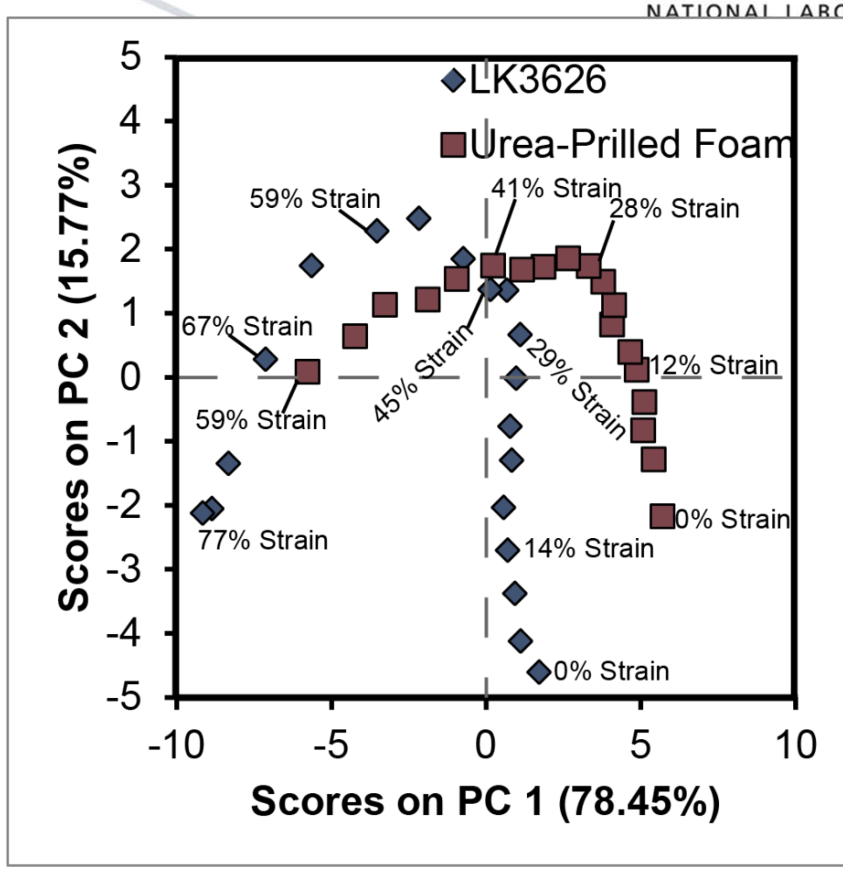


Current Work

- Pattern recognition
- Nano-indentation to measure material properties
  - Measure the modulus of the bulk material
- Faster data acquisition
- Collected data on:
  - Irradiated/aged and carbon fiber filled SX358 foams
  - 3D printed materials
  - HE

Principal Components Analysis and Pattern Recognition





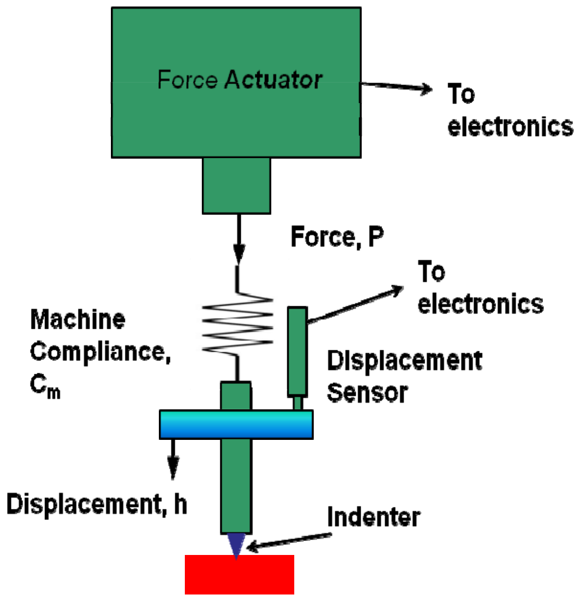
3D void shape descriptors (e.g. voids eccentricity) can be used as inputs into Principal Components Analysis, which is a pattern recognition method.

This model adequately differentiates 2 different foams at separate stress-strain states.

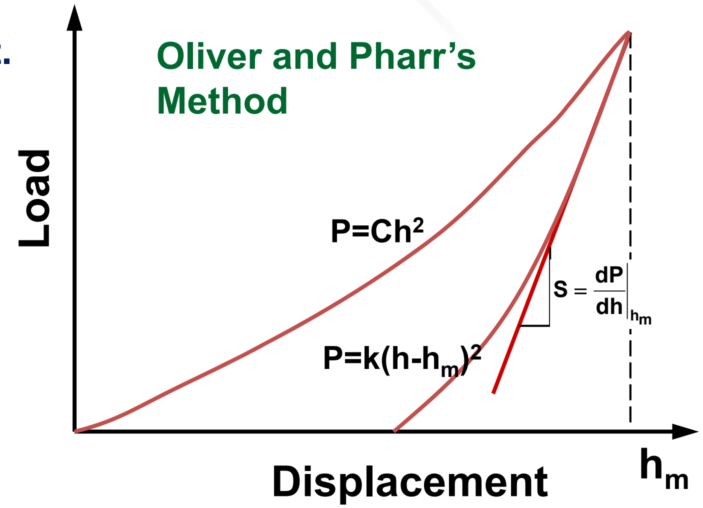
This method can be used next to compare the performance of unaged vs. aged polymer foams.

Mechanical properties using Nanoindentation

- Young's modulus and hardness can be obtained for small microstructural features.
- Stiffness is measured as the slope of unloading part of load-displacement curve. (Oliver and Pharr's method)
- Single modulus value is obtained from each test.



Basic Components of Instrumented Indentation Tester



$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}}$$

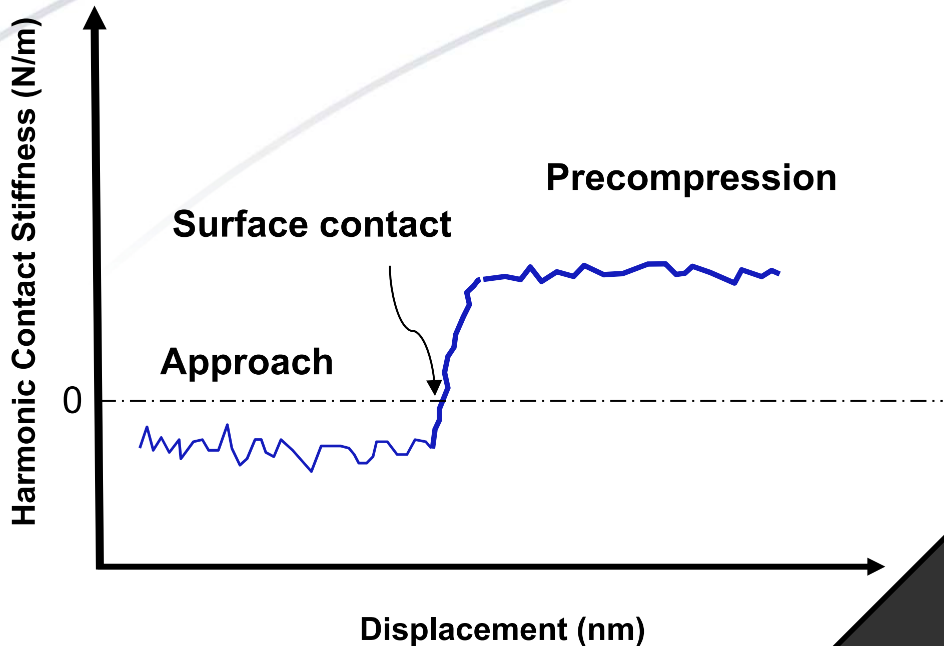
$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$$

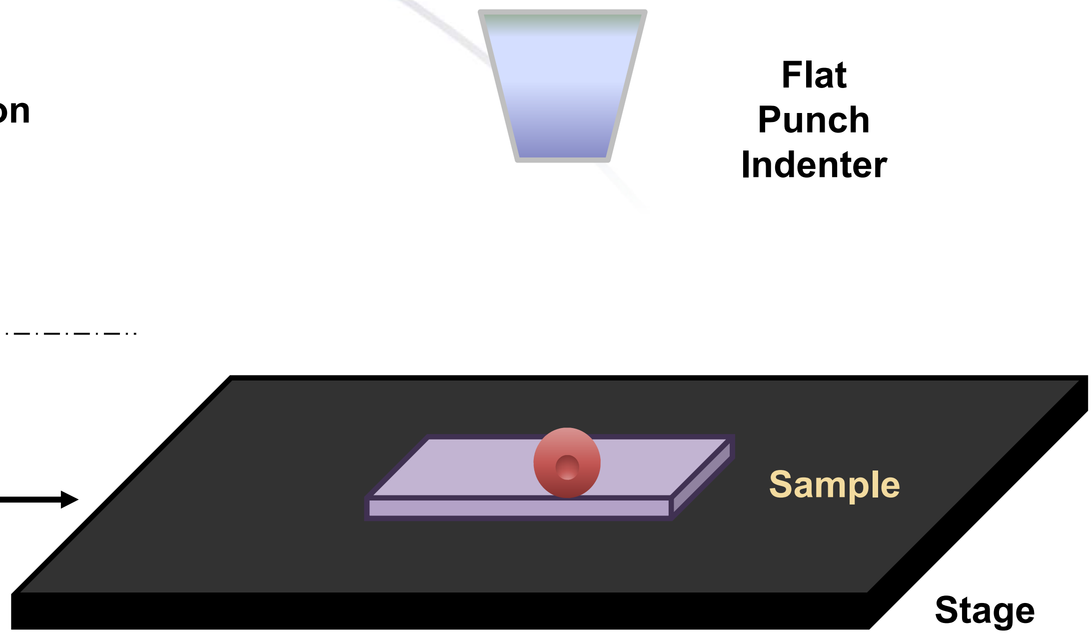
A = Projected Area
E_r = Reduced Modulus
E_i = Modulus of Indenter
E = Modulus of Material

S = Contact Stiffness
β = Indenter Geometry Factor
ν = Poisson's Ratio

Oliver and Pharr, J. Mater. Res., (1992)

Nanoindentation – Polymer testing method





- **Approach:** Harmonic contact stiffness is less than zero
- **Surface Contact:** Harmonic contact stiffness increases (75-200 N/m)
- **Further indentation:** Precompression to specified depth and testing at various frequencies

Nanoindentation results of foam (SX 358)

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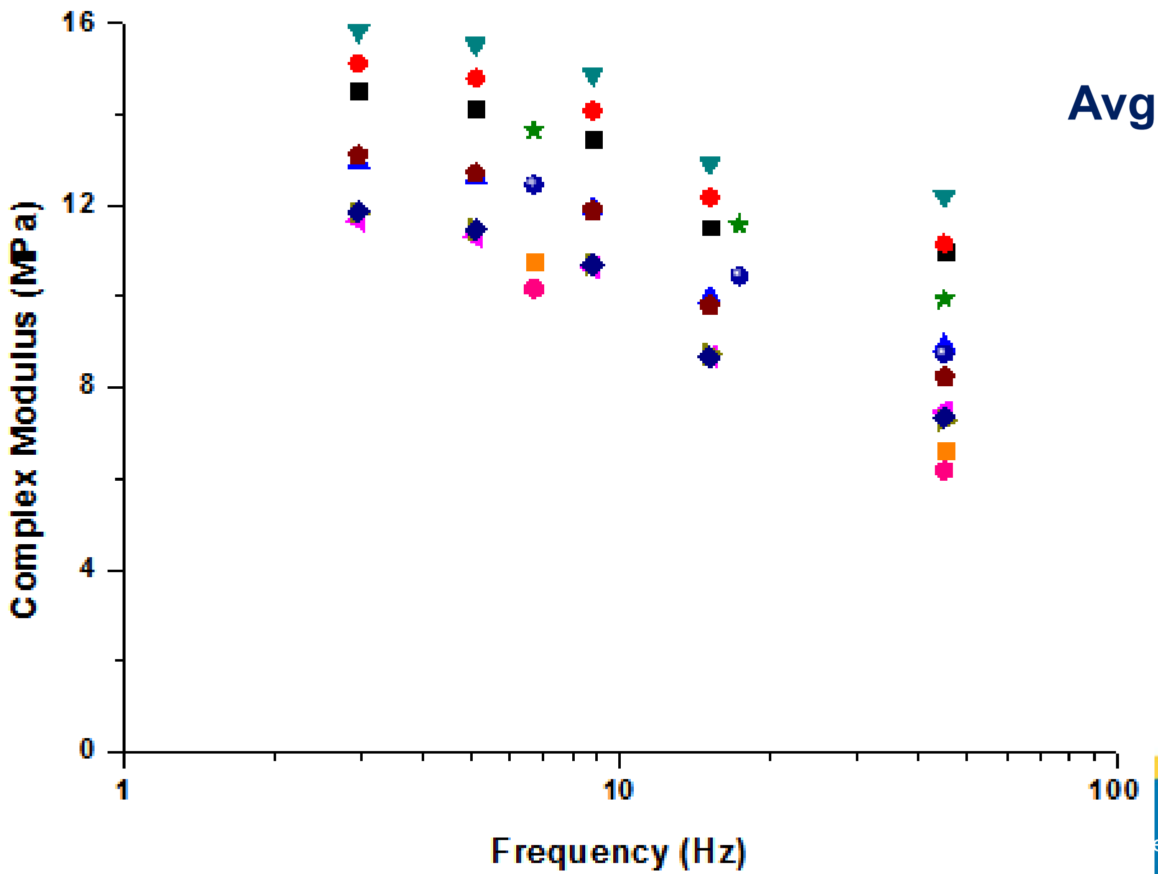
•The storage modulus dropped from a mean value of 13.4MPa to 8.8MPa in the range of frequency during testing.

•Mean loss modulus was less than 1MPa and the complex modulus E* = E' + iE'' can be approximated as E* ~ E'.

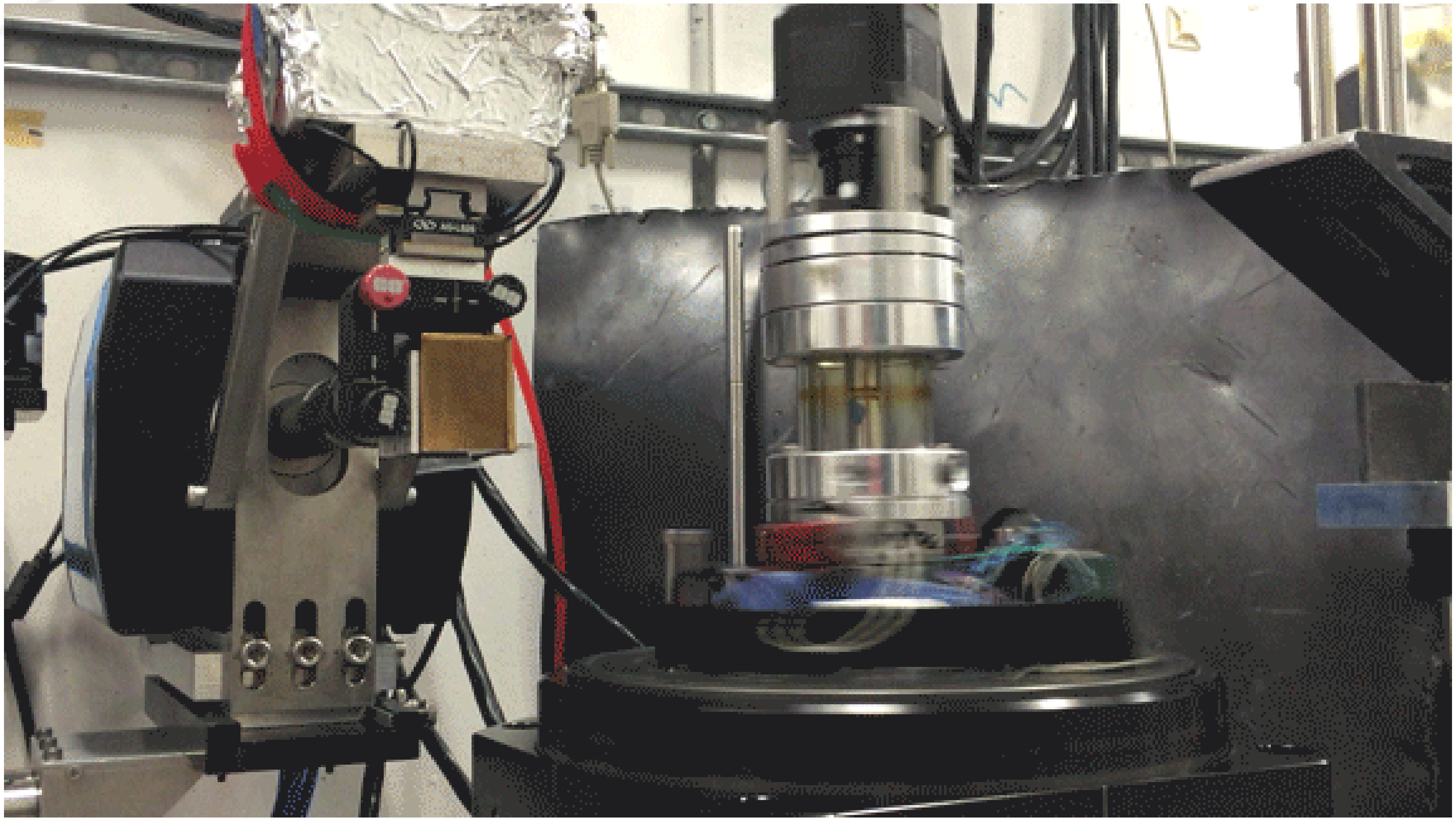


Avg. Complex Modulus – 11.3 MPa

ent of Energy's NNSA

Even Faster!!

0.25 seconds per 901 image CT, continuous

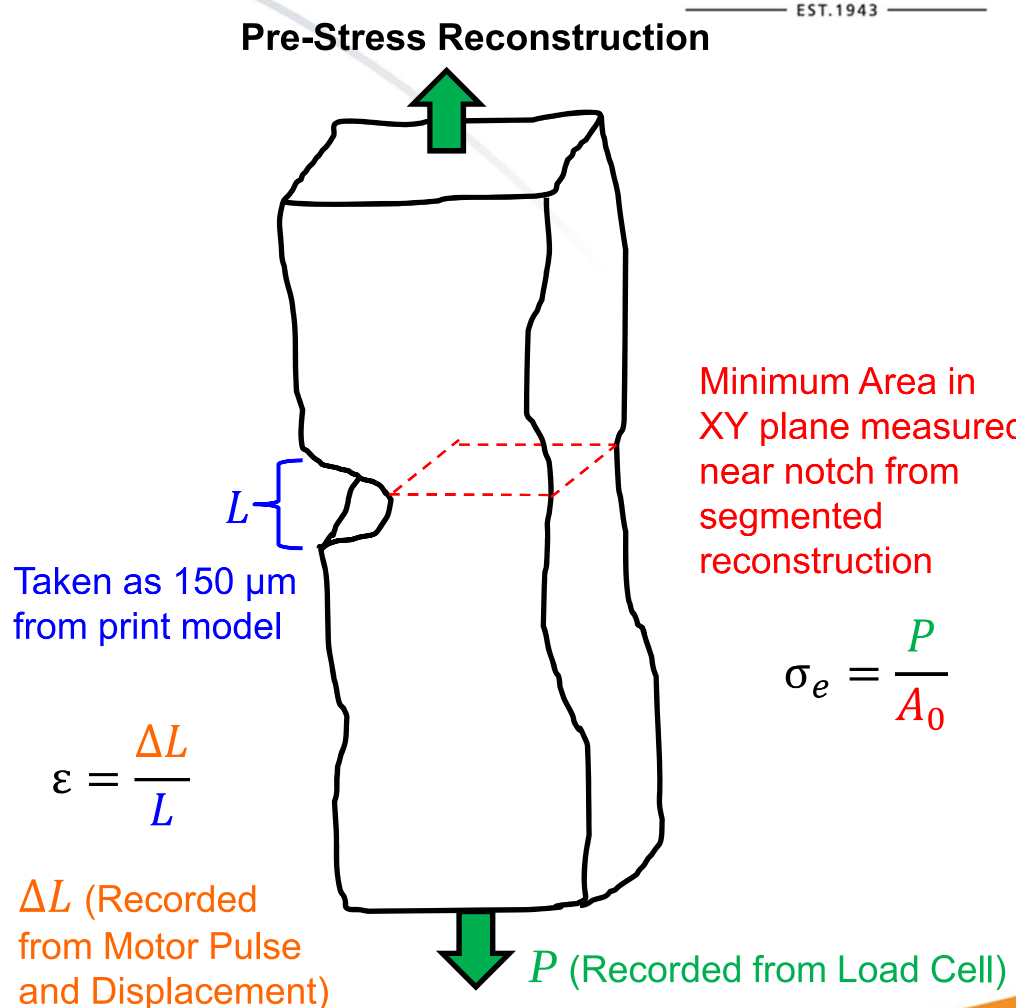


Aged, filled, and irradiated SX358

- Study to understand the mechanical performance SX358
  - Irradiated using gamma rays at the Sandia GIF
    - Pristine;
    - Room temperature; N_2; gamma
    - Room temperature; air; gamma
    - 70^{o}C, N_2; gamma
    - 70^{o}C; air; gamma
- Carbon fiber filled SX358

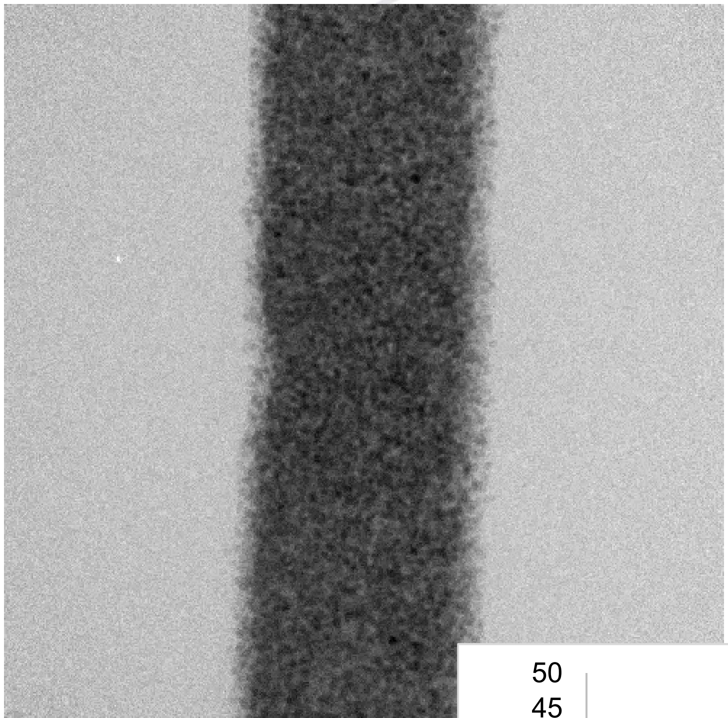
Stress and Strain Measurement Method

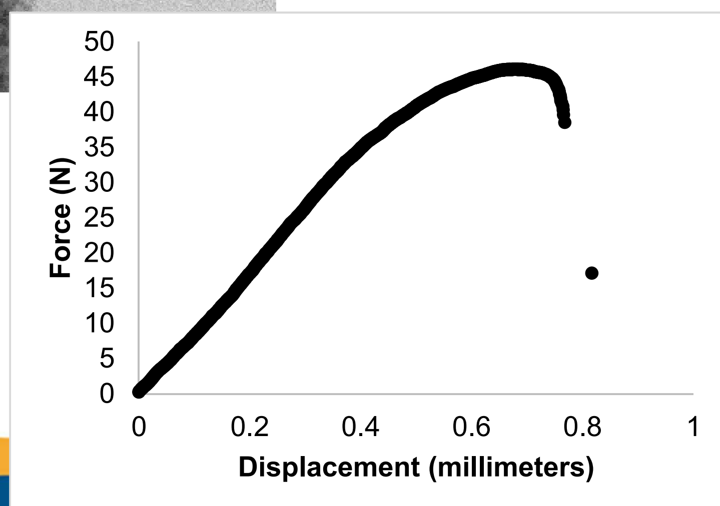
- One reason for variation in strain-to-failure in two different EOS100_X samples may be variation in the actual notch geometry
- 150 µm used for all samples to calculate strain
  - Actually quite non-uniform in appearance, complicated by presence of residual print material)
- Assume loading direction is parallel to CT Z-direction
- Only the initial cross-sectional area was taken for stress calculation (i.e. engineering stress was used)

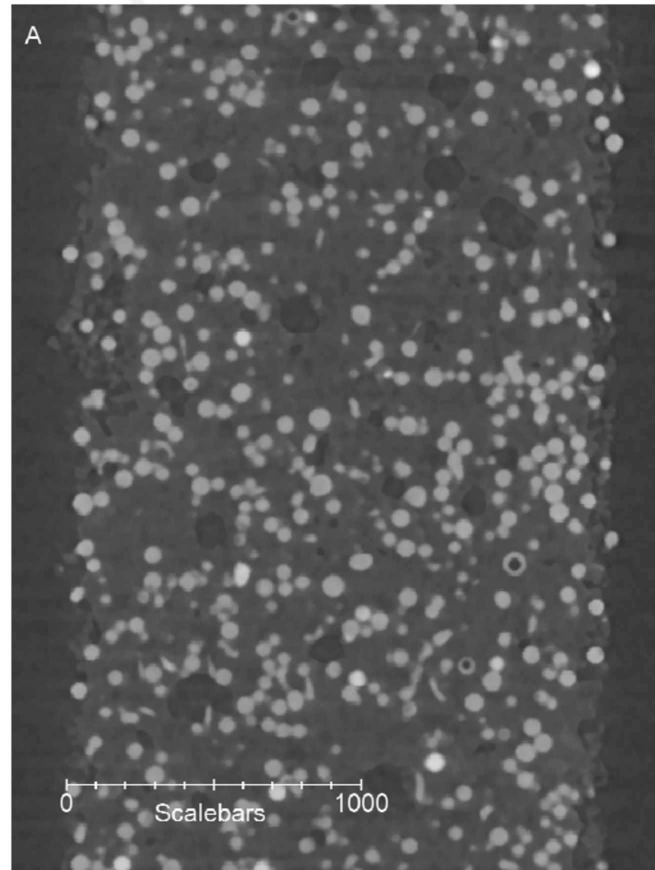


Laboratory radiography and tomography

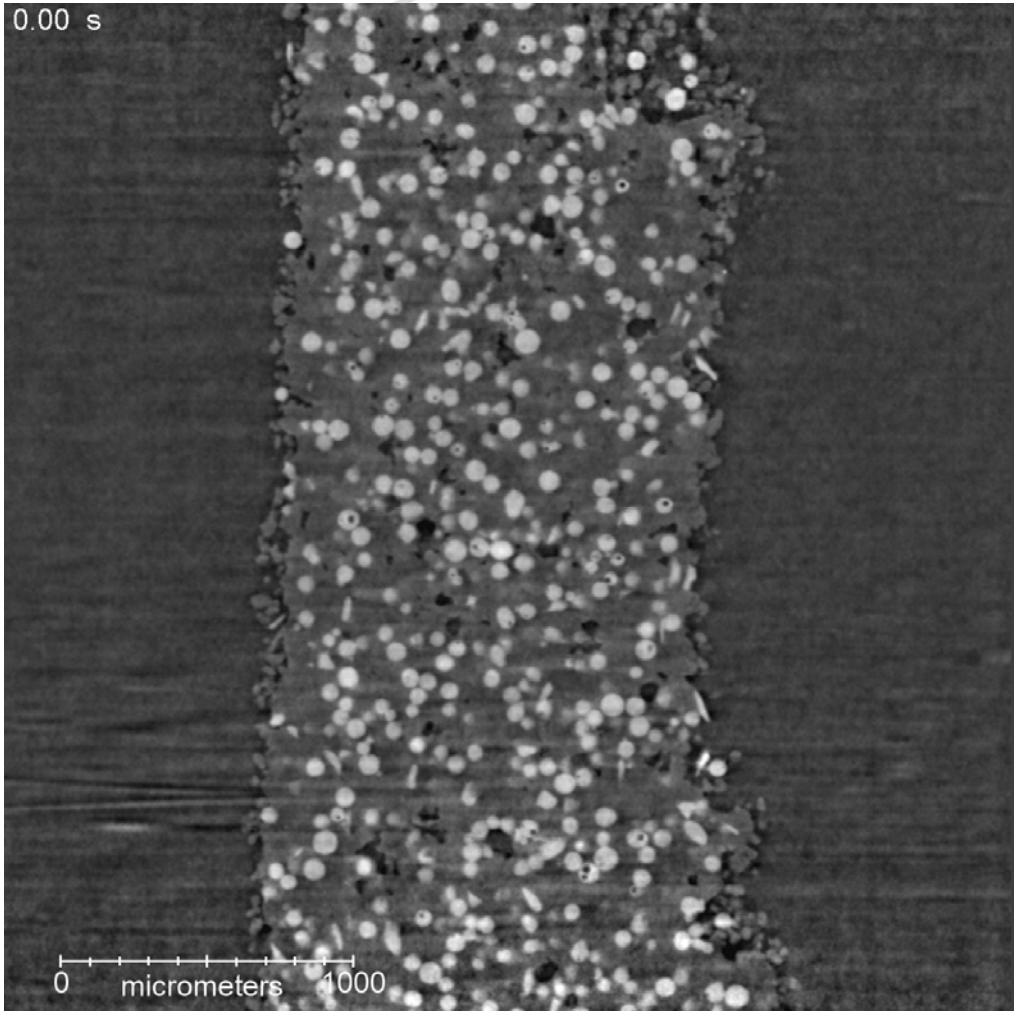
3200 GF 3X recycled

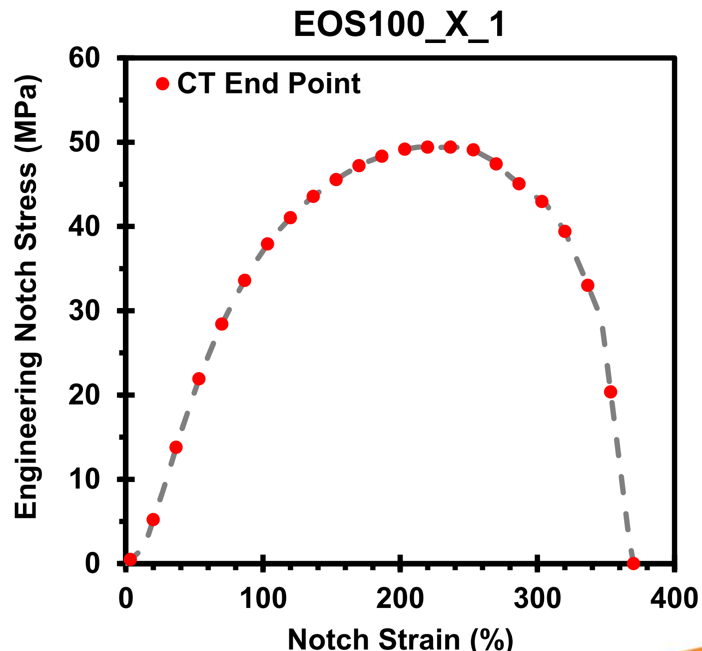




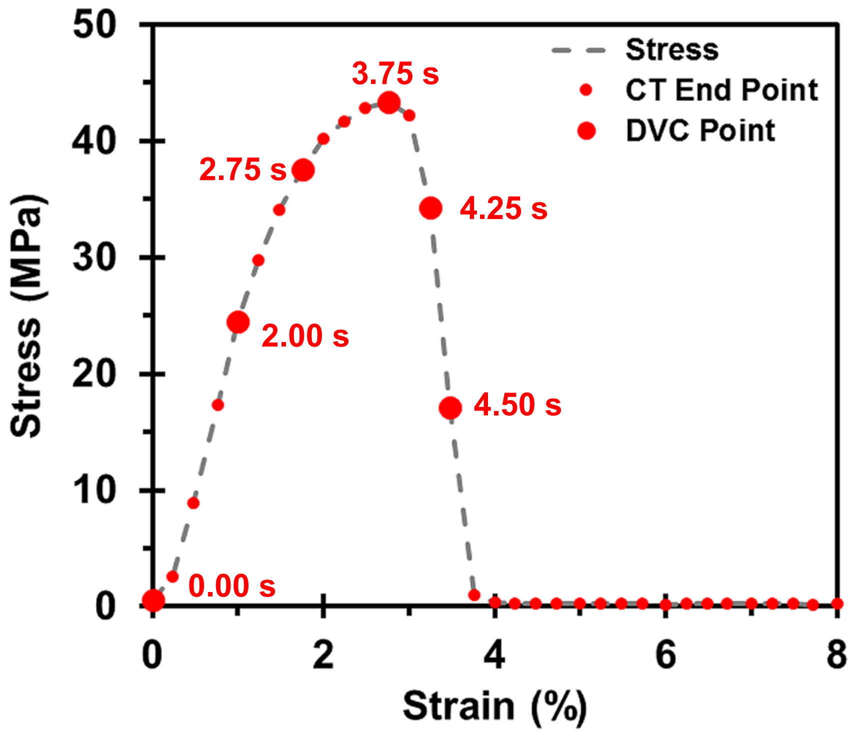


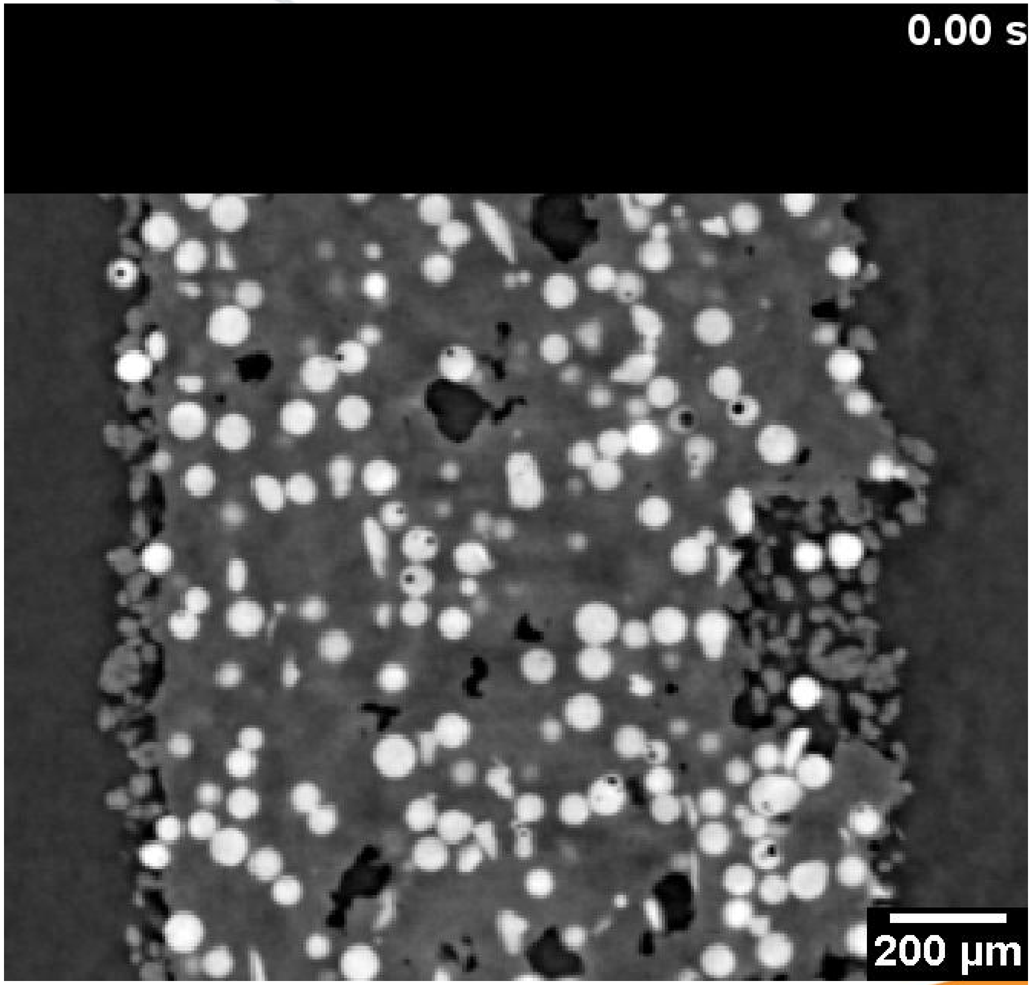
3D printed EOS 3200 GF tensile specimen



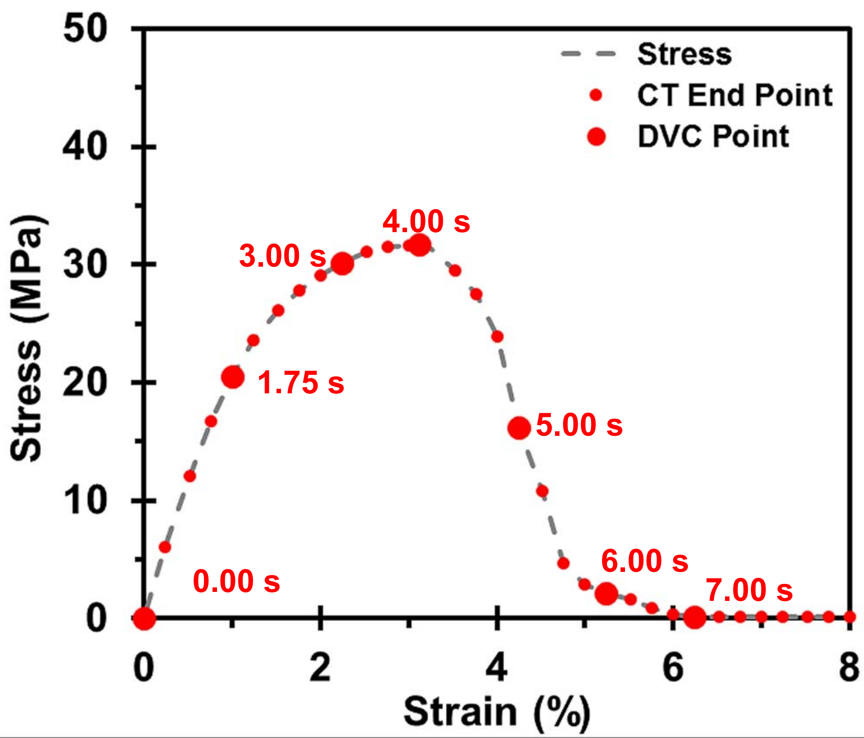


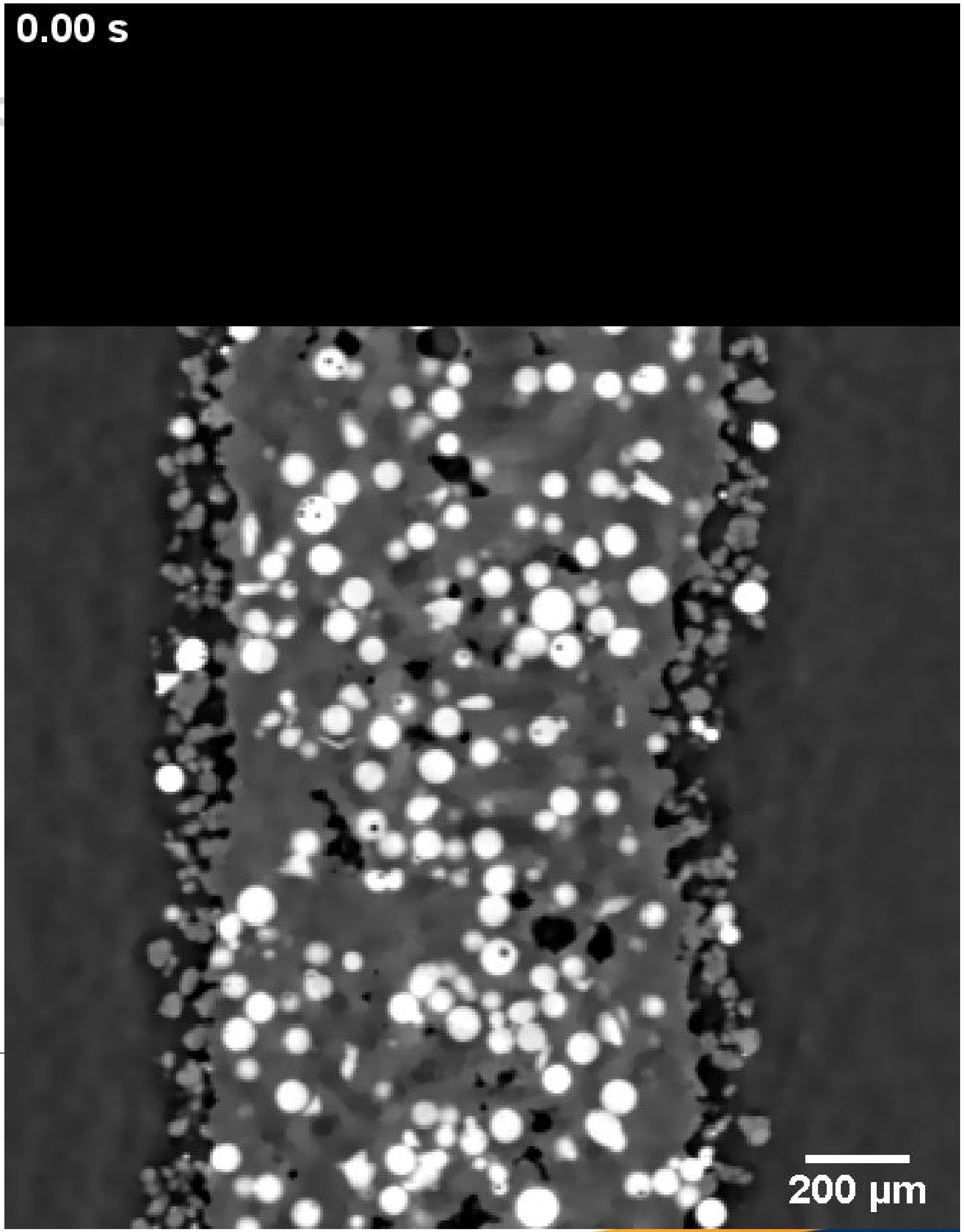
EOS100_X_2


Stress (MPa)
Strain (%)
Stress
CT End Point
DVC Point
0.00 s
2.00 s
2.75 s
3.75 s
4.25 s
4.50 s
0
10
20
30
40
50
0
2
4
6
8

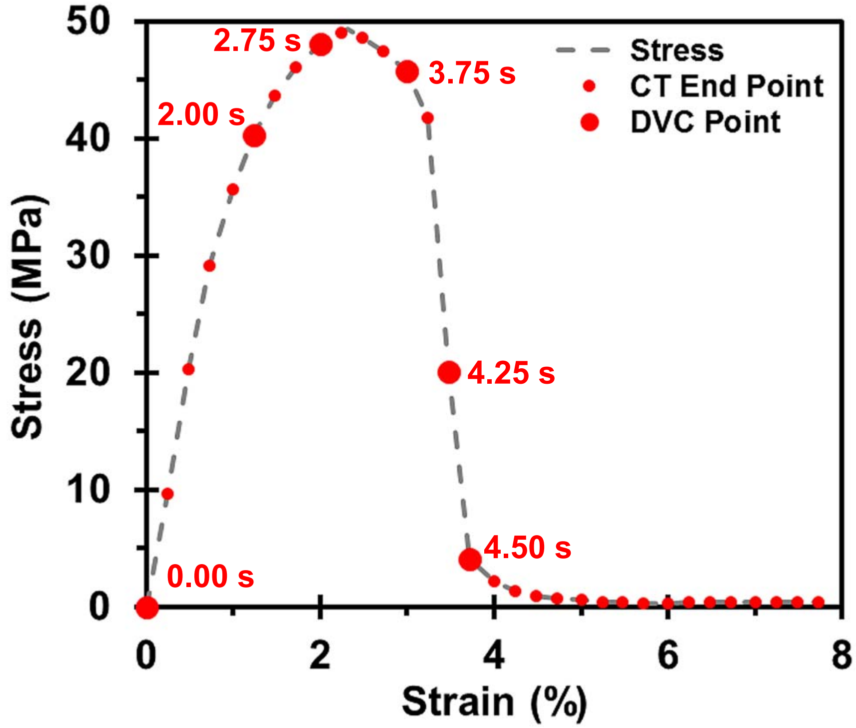

0.00 s
200 µm

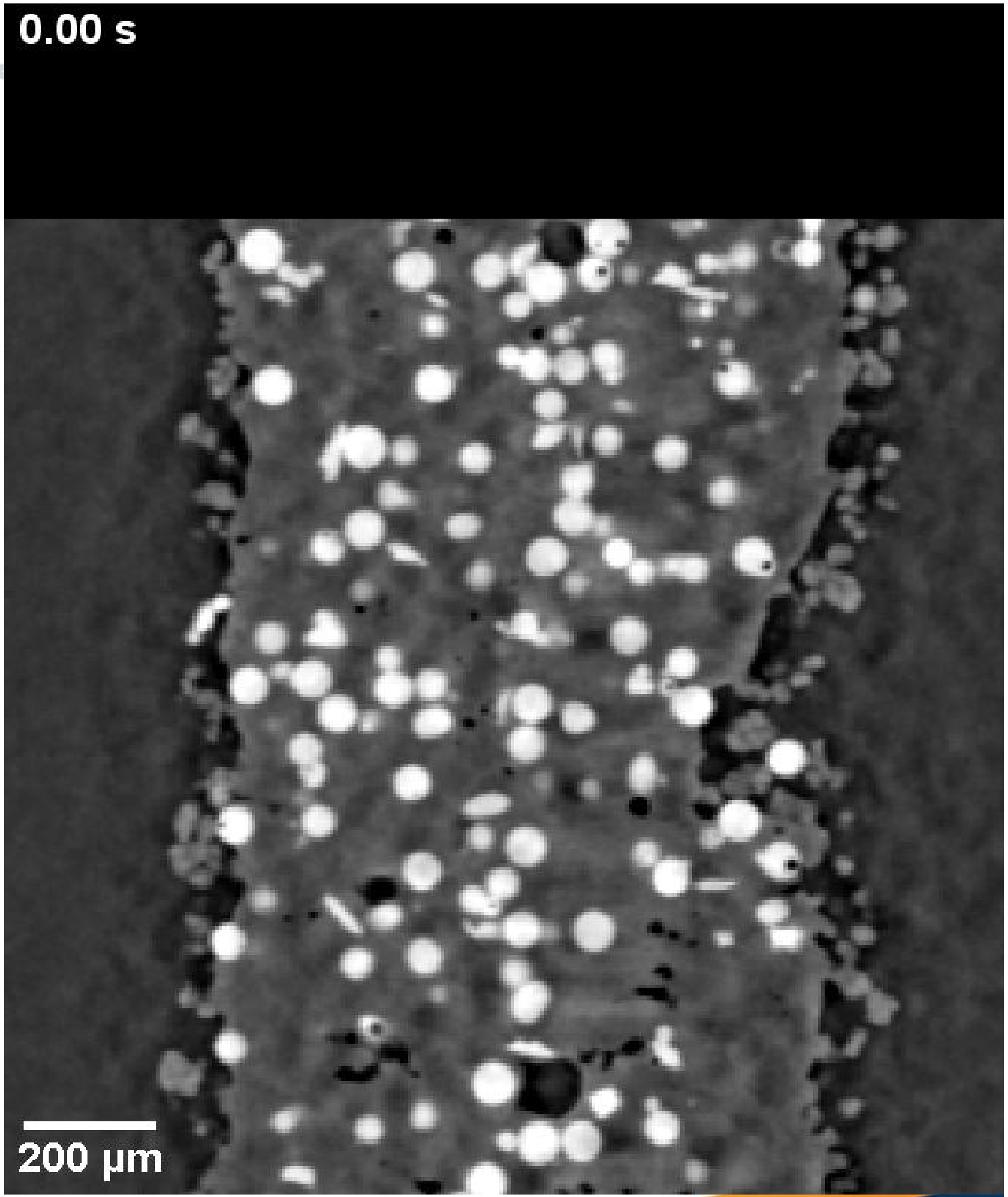
EOS100_Y_1


Stress
CT End Point
DVC Point
50
40
30
20
10
0
Stress (MPa)
0
2
4
6
8
Strain (%)
0.00 s
1.75 s
3.00 s
4.00 s
5.00 s
6.00 s
7.00 s

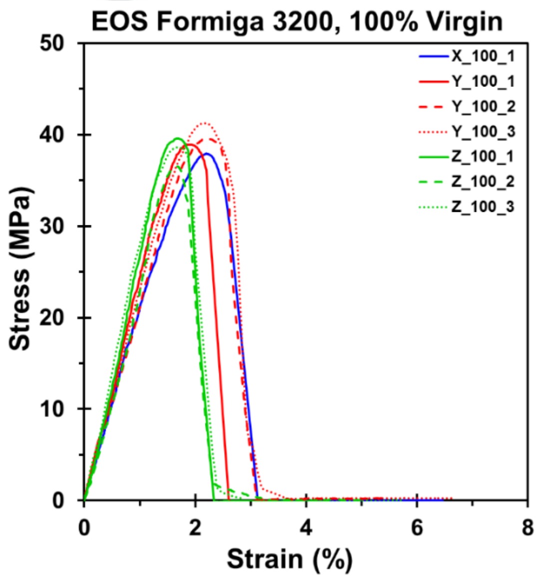

0.00 s
200 μm

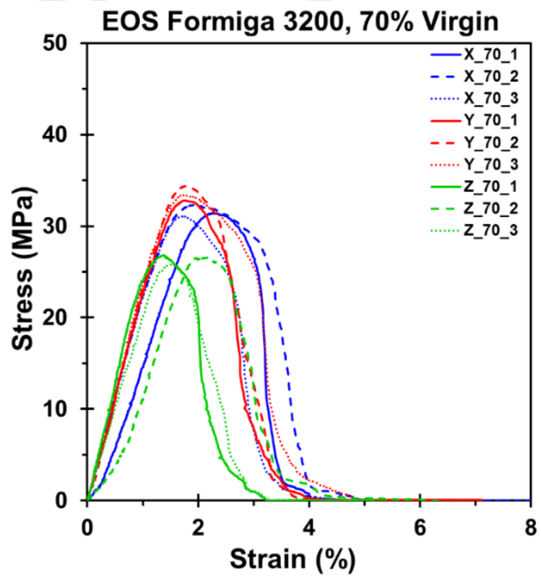
EOS100_Z_1

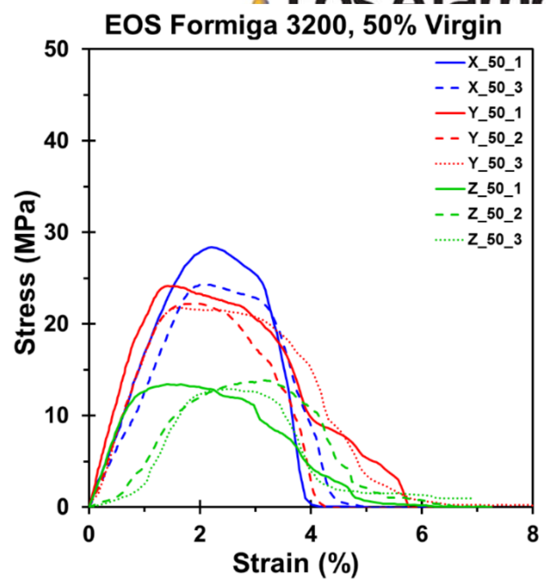

Stress
CT End Point
DVC Point
2.75 s
3.75 s
2.00 s
4.25 s
4.50 s
0.00 s
Stress (MPa)
Strain (%)
0
10
20
30
40
50
0
2
4
6
8


0.00 s
200 µm

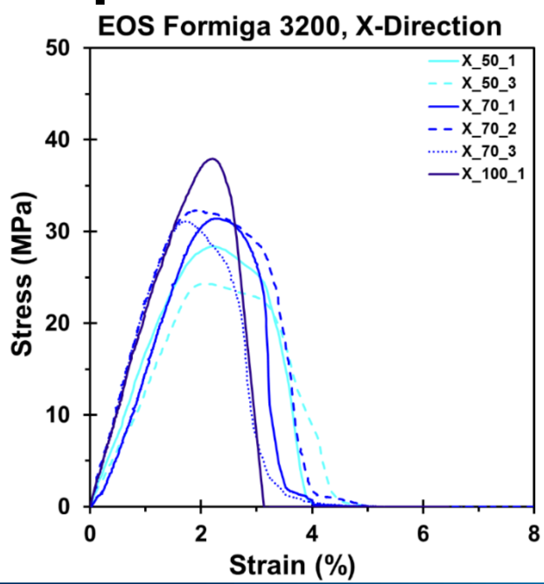
Direction Effect 3200 GF (Deben)

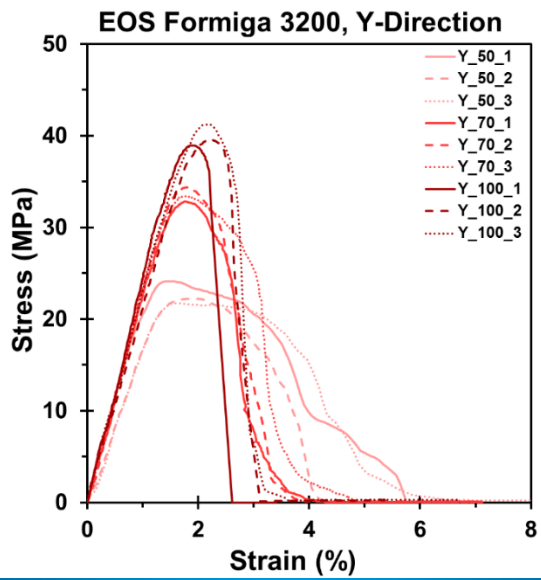


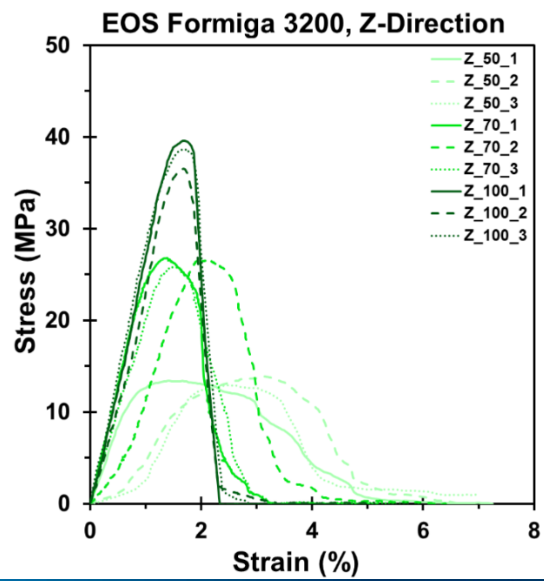




Composition Effect

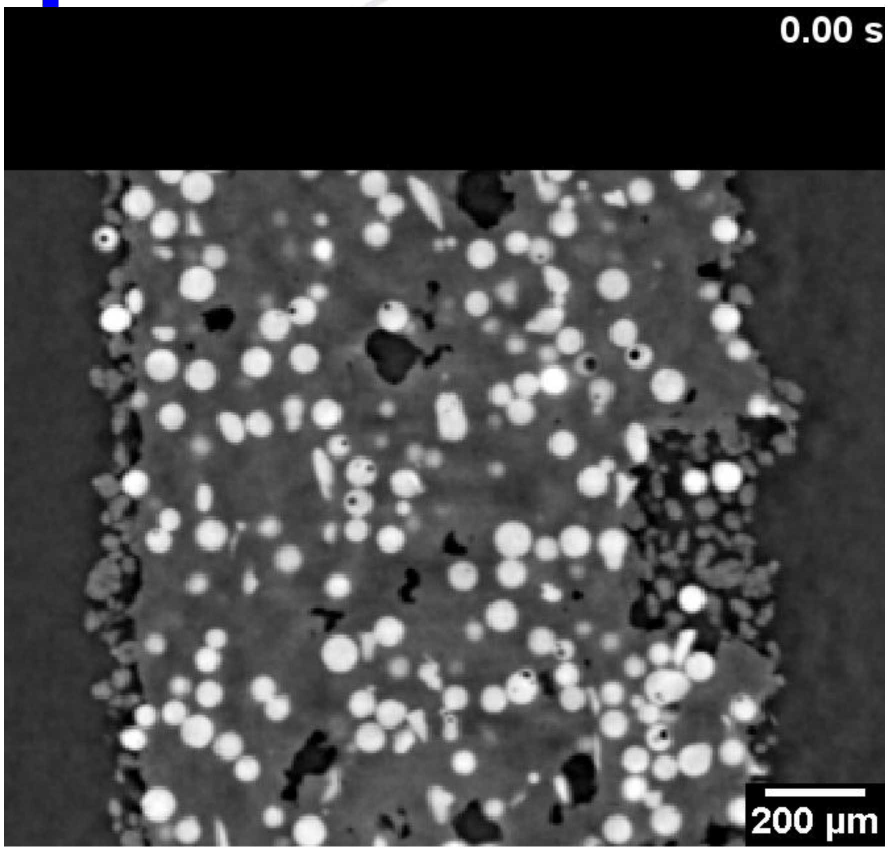






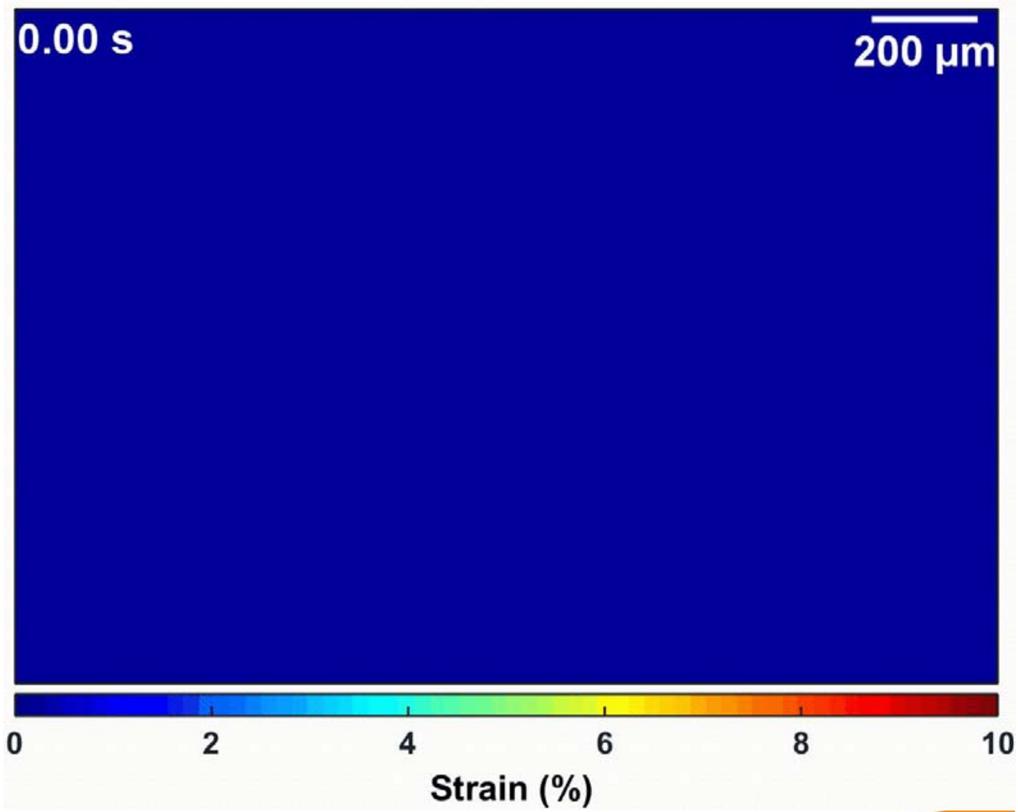
EOS100_X_2

In situ CT



σ_{zz}

Digital Volume Correlation (ε_{zz})



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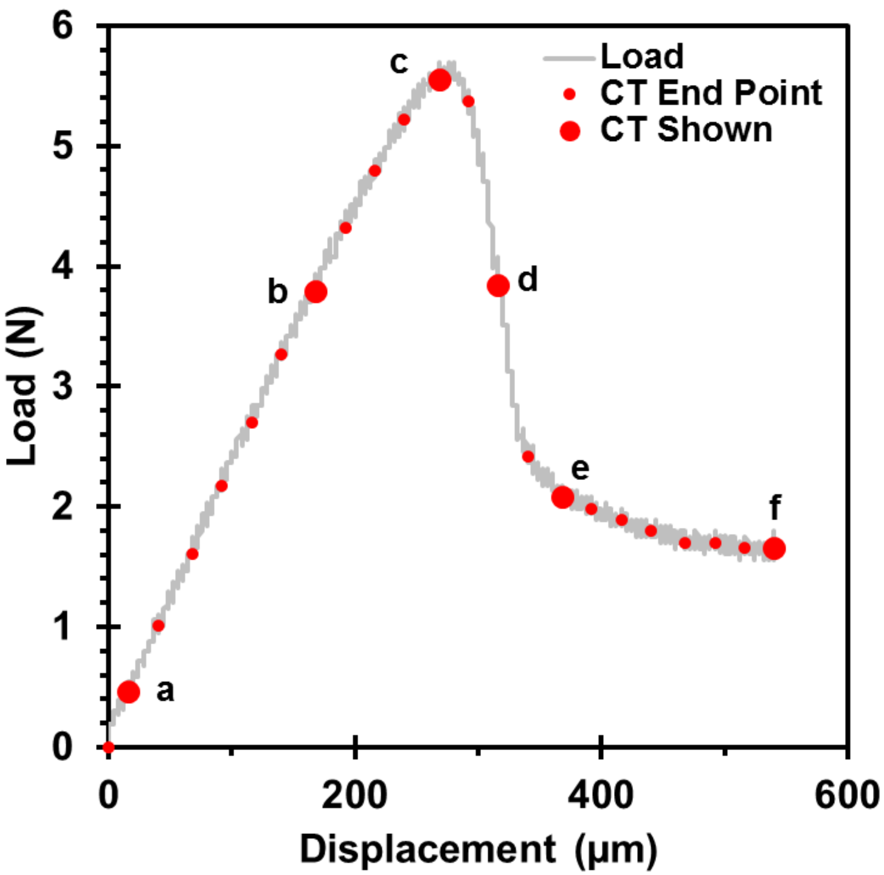
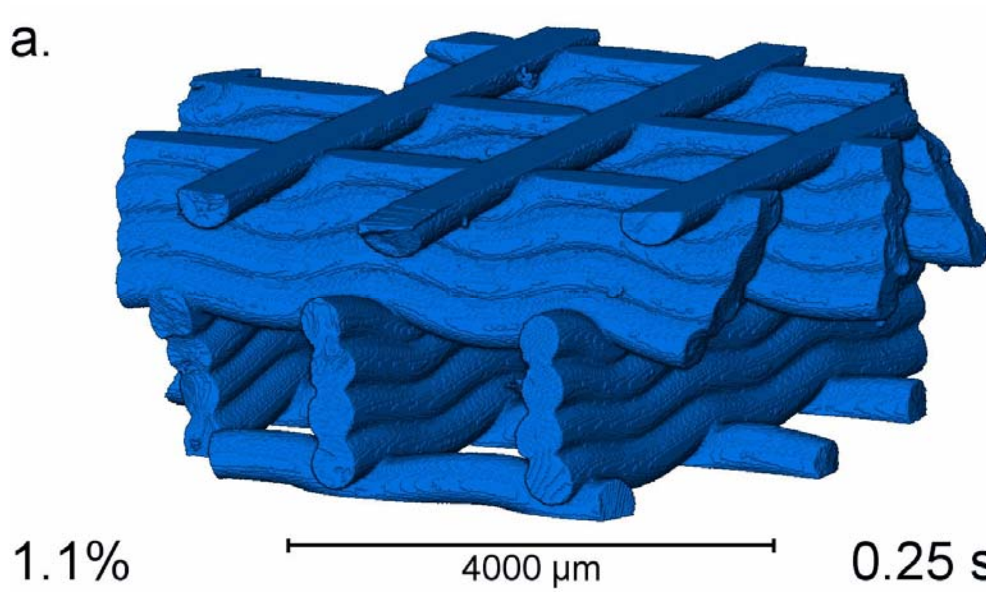
Summary

- Stress calculations approximate
  - Residual particles included in cross sectional area measures
  - Stress intensity of notch radius not considered
- Notch makes strain calculations approximate
  - Used 10 mm gage length to calculate strain
- ASU/APS shows slightly higher load-displacement data than Deben (fairly consistent)
- Higher recycled content seems to increase strain-to-failure (ductility)
- Higher recycled content seems to decrease UTS (strength)
- UTS for Z print-direction converges with X/Y at 0% Recycled

Direct Ink Write Materials

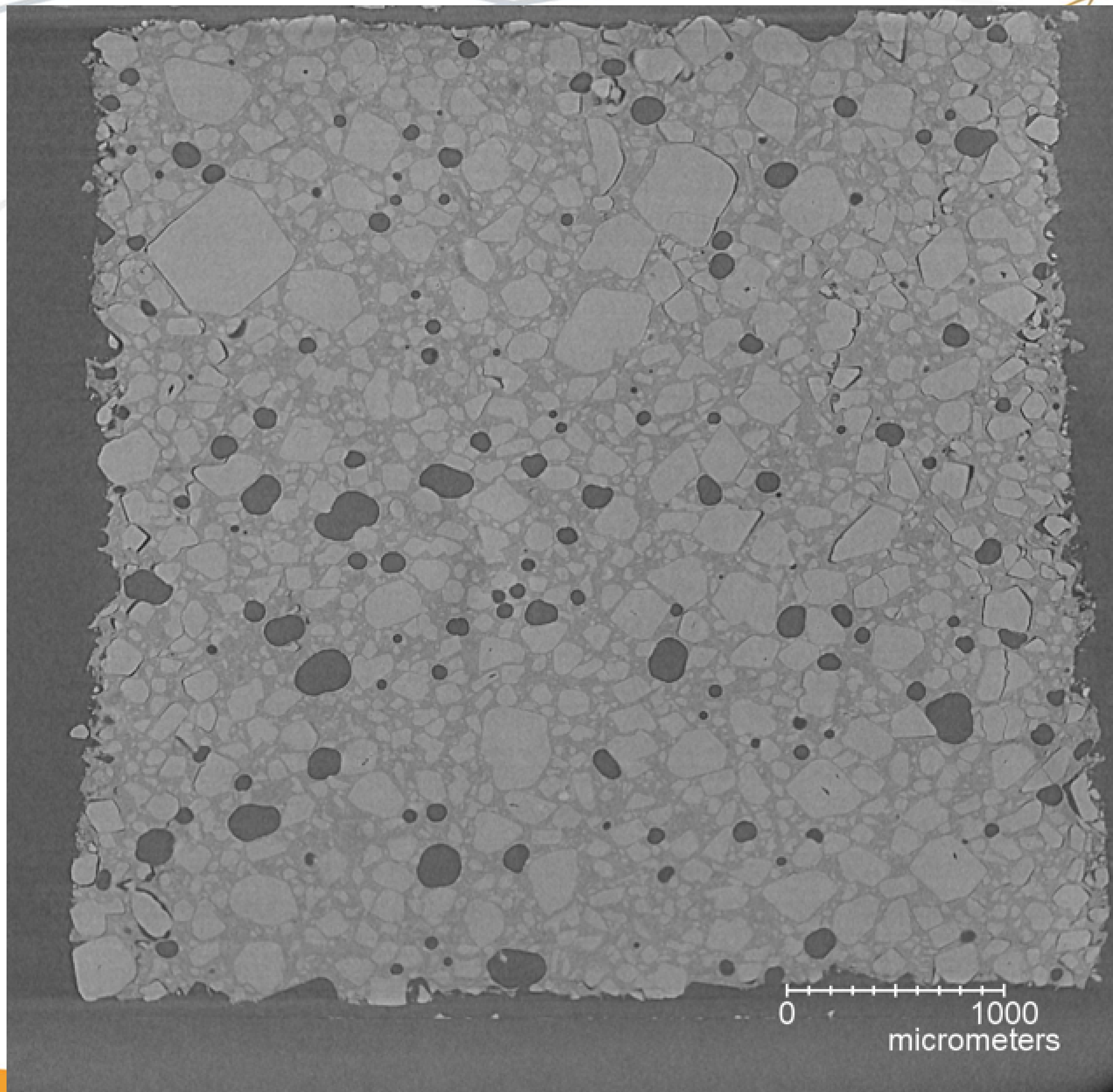
4x2 SE1700



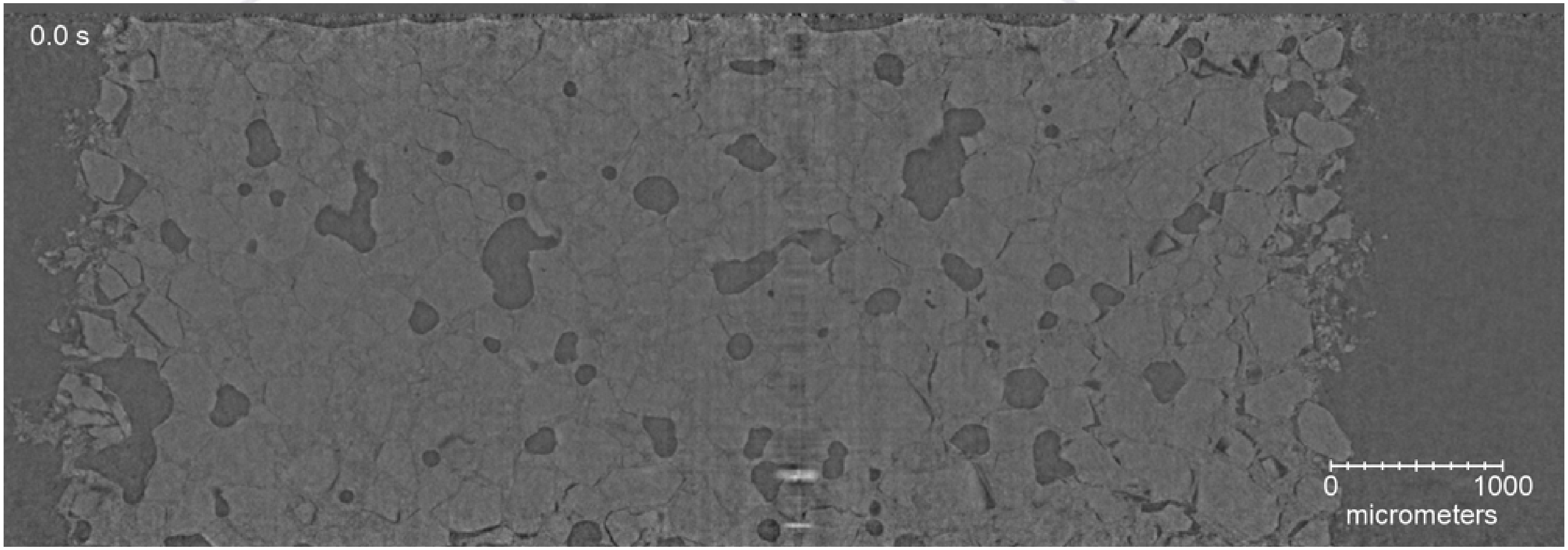


(Sample courtesy John Dirvage and Carol Putman, NSC)

Extra high MDI, HMX-HTPB Composite



Sugar-HTPB at APS



5 mm

Material caramelized due to high intensity X-ray exposure

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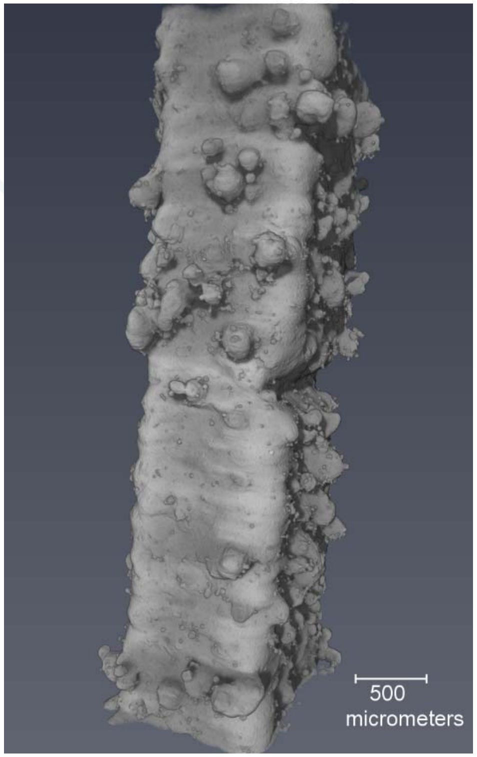
Future Directions (April 2016)

- Improved reconstructions
- 3D printed aluminum (KCP)
- Even faster imaging using a spindle

- Aerotech ABS2000 direct-drive spindle (100 rps)
- Use 32ID
- FOV limited to ~2 x 2 mm



Should be able to get to 2 x 10^0 strain rate imaging of polymer foams.

Conclusions:

High rate radiographic and tomographic imaging (0.25 s 3D frame rate) using synchrotron CT can capture full 3D images of hyper-elastic materials at a 10^{-2} strain rate.

Dynamic true in situ uniaxial loading can be accurately captured.

The three stages of compression can be imaged, bending, buckling, and breaking.

Implementation of linear modeling is completed.

Meshes have been imported into LANL modeling codes, testing and validation is underway.

Direct comparison and validation between in situ data and modeled mechanical response.

Cross cutting technology applicable to other material systems (AM polymers & metals, CF, ceramics, HE)

Faster imaging and strain rates (100x faster) forthcoming.

Funding was provided by the
- Enhanced Surveillance Campaign, Tom Zocco, PM
- Engineering Campaign 6, Eric Mas/Antranik Siranosian, PM

Los Alamos National Laboratory is operated by Los Alamos National Security LLC under contract number DE-AC52-06NA25396 for the US Department of Energy. This research used resources of the Advanced Photon Source, a U. S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

Extra Slides

- Nanoscribe
- HE Modeling (Moore, LDRD-DR)
- Nano computed tomography load cell

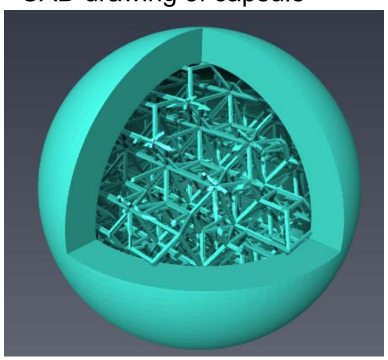
3D printed capsule for the MARBLE campaign

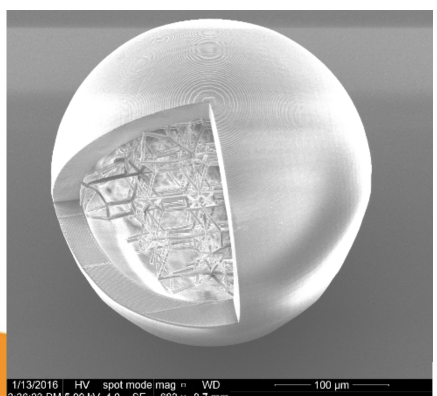
300 µm diameter capsule was 3D printed using the Nanoscribe two-photon polymerization system.

Simultaneously the capsule was printed containing a hexagonal lattice structure.

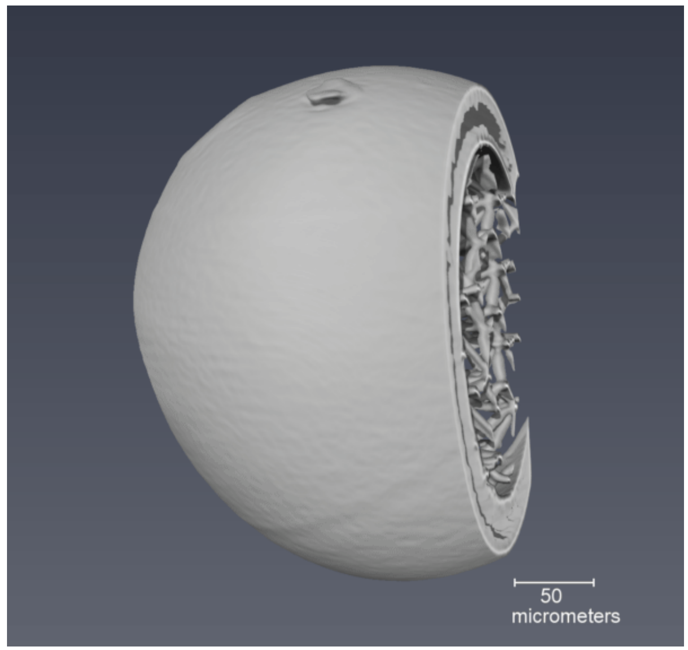
Interior lattice ligaments are ~1 µm in diameter.

CAD drawing of capsule





SEM image of printed capsule.



MicroCT image of the printed capsule. Ligaments are below the resolution of the microscope.

Nano-scale X-ray CT reconstructed slice through a few ligaments confirming the high quality of the print build.

Contact: Brian M. Patterson, John Oertel, Tana Cardenas, Matthew Herman

Detection of explosives at home and abroad is difficult – Need new capabilities.

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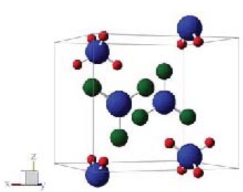
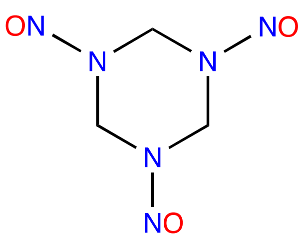
No method is yet able to solve the simultaneous challenges of rapid screening, areal coverage, sensitivity and selectivity necessary to unequivocally identify threats (terrorists, insurgents, in-theater vs. airport)

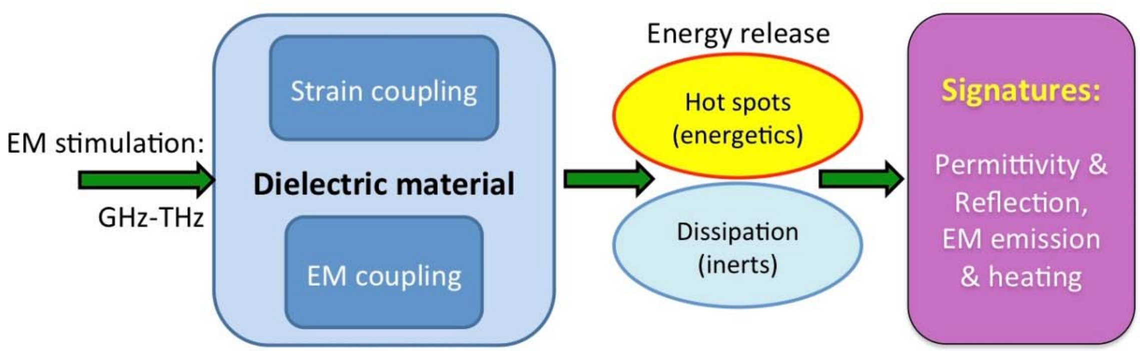
A new science-based methodology is needed that detects the bulk explosive directly, preferably at a safe stand-off distance, and when camouflaged or hidden – Use GHz to THz.

The ground breaking innovation of this LANL proposal is in recognizing and utilizing the innate properties of explosives for detection – by coupling energy to microstructures to affect localized chemical energy release

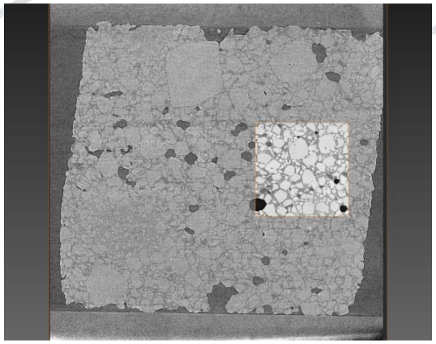


We capitalize on chemistry and microstructure to revolutionize detection

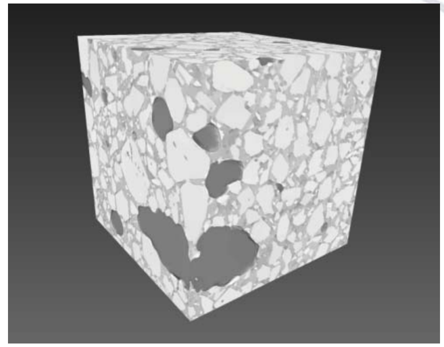




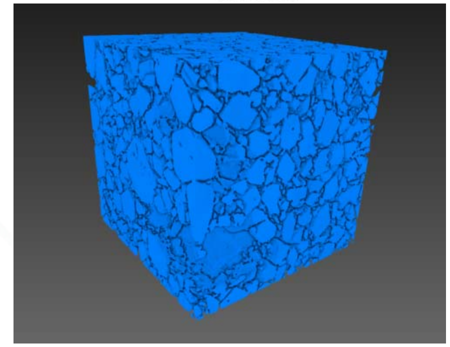
CT processing



Data was cropped then smoothed with an edge-preserving smoothing filter



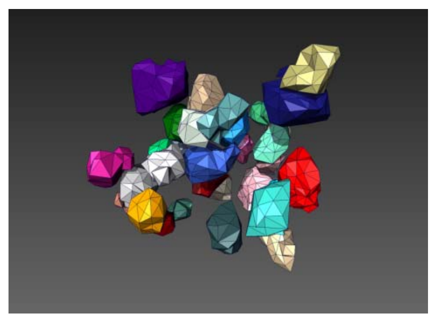
Data was segmented for HMX crystals

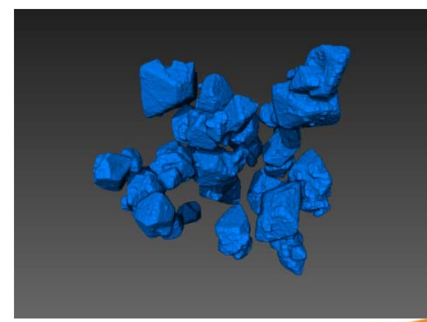


1. Border crystals removed
2. Small crystals removed (less than 150 micrometer equivalent diameter)
3. Removed voids and small holes in crystals
4. 35 Crystals

CT data cropped and processed for use in Comsol

- Each crystal individually labeled.
- 1.5 mm isotropic volume





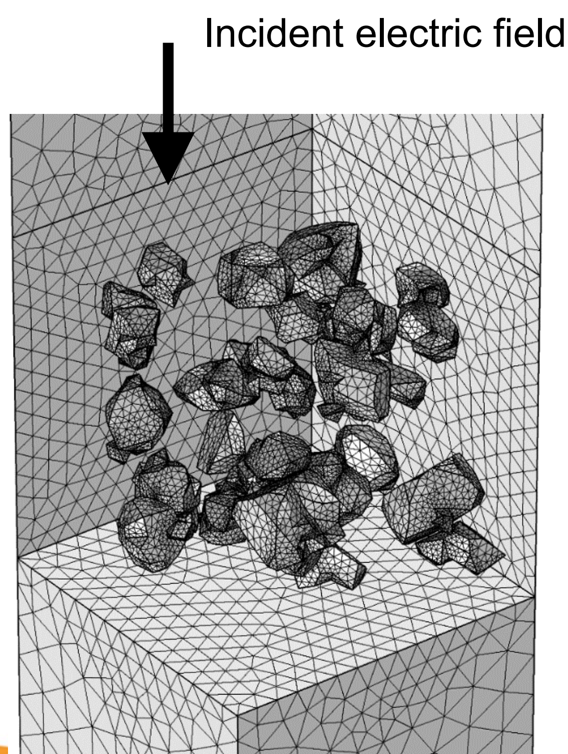
1. Simplified to ~2900 faces
2. Used Avizo’s Surface Tests for intersections, orientation, aspect ratio and closedness

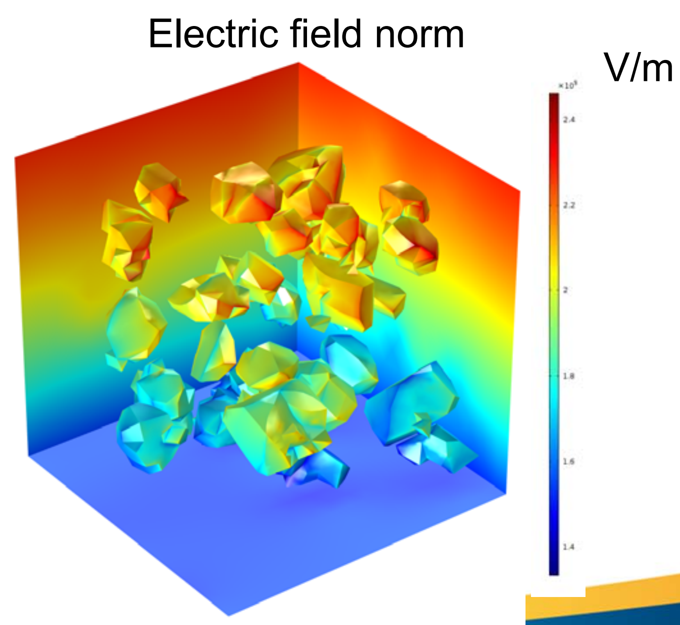
COMSOL simulation of coupled electromagnetic and heat conduction in energetic materials

- Pulse of 10.6 GHz E field from upper surface
- Temperature rise from 20°C as a function of time was measured
- Chemistry ignored

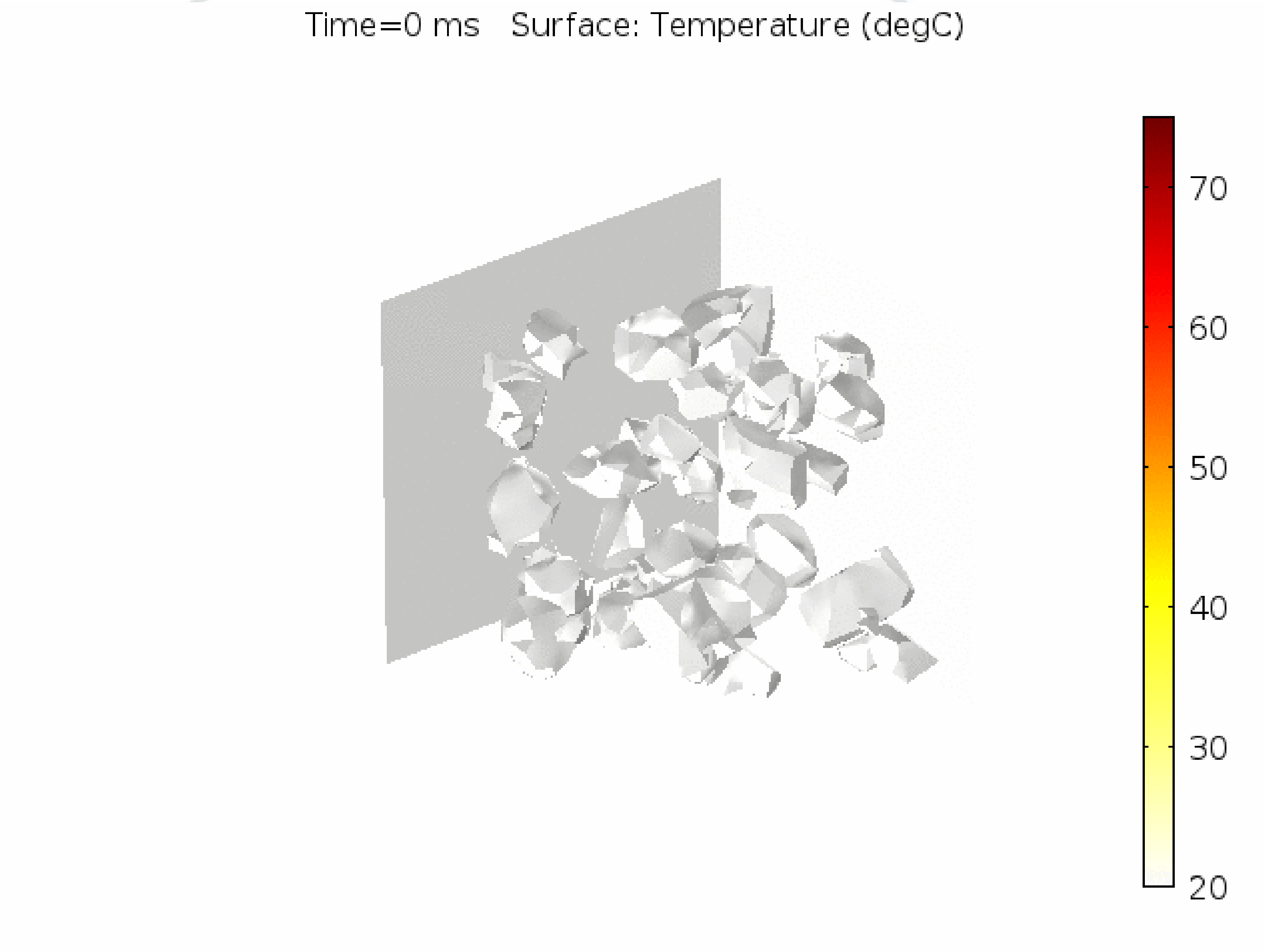
$$-\nabla^2 \mathbf{E}(\mathbf{r},\omega) = \omega^2 \varepsilon(\omega, T)\mathbf{E}(\mathbf{r},\omega)$$

$$\rho C \frac{\partial T(\mathbf{r},t)}{\partial t} = k\nabla^2 T(\mathbf{r},t) + \frac{1}{2}\omega\varepsilon''(\omega,T)|\mathbf{E}(\mathbf{r},\omega)|^2 + Q_{chem}$$



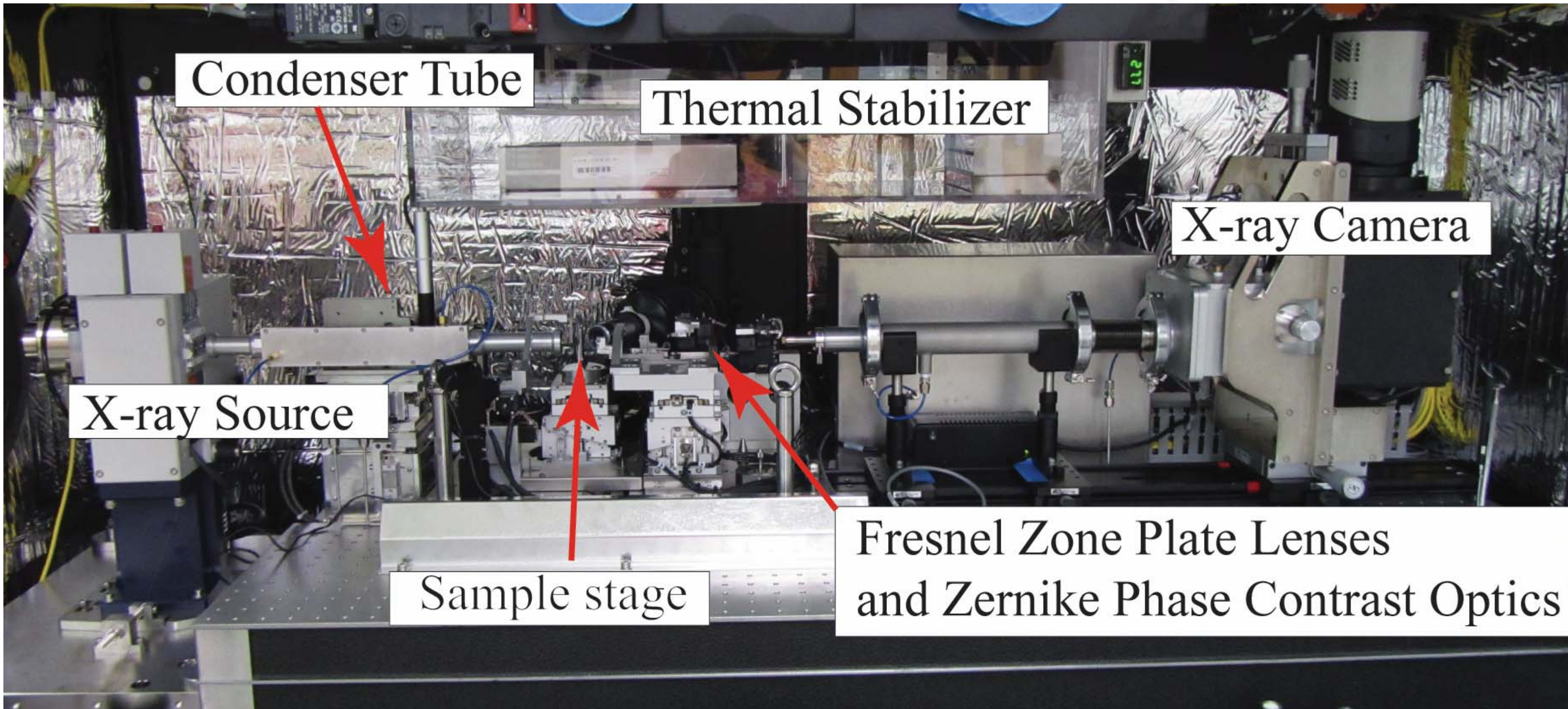


Temperature distribution



Xradia UltraXRM

Large FOV = 65 µm, 150 nm resolution
Small FOV = 15 µm, 50 nm resolution
Absorption or Zernike phase contrast



Absorption and Phase Contrast

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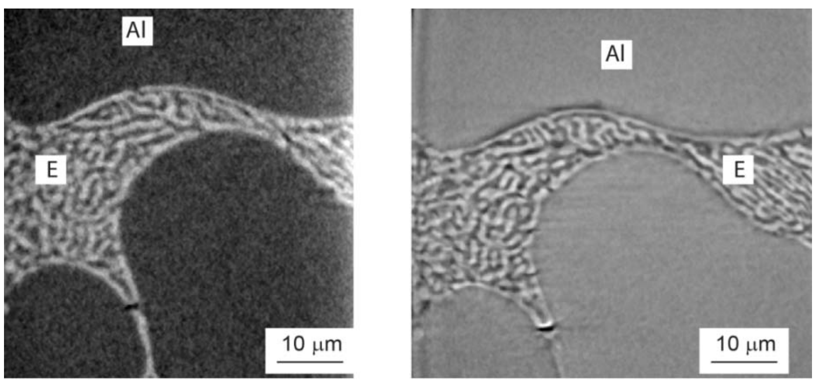
Absorption ("Shadow") Contrast

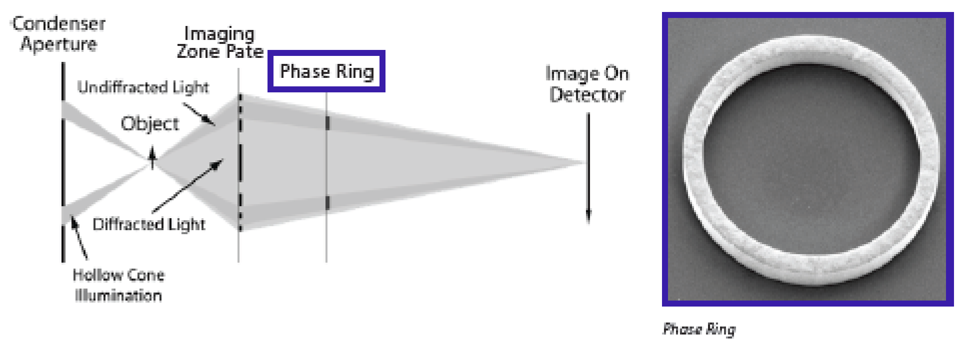
- Based on large density variations, i.e. differences in attenuation
- Examples:
  - Material vs. pore space or voids
  - High density inclusions in medium density material

Phase Contrast

- Based on refraction rather than absorption
- Sensitive to interfaces between materials of similar (typically low) density
- Examples:
  - Organic materials such as polymers, tissue etc.



Absorption contrast (l.) and phase contrast (rt.) of an Al-Cu alloy.



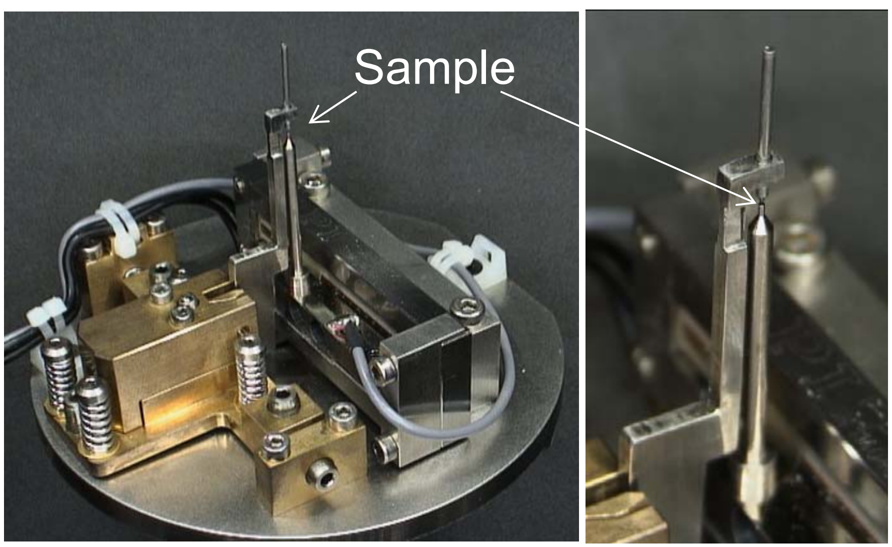
Phase contrast concept based on the Zernike method

Carl Zeiss UltraXRM Load Stage

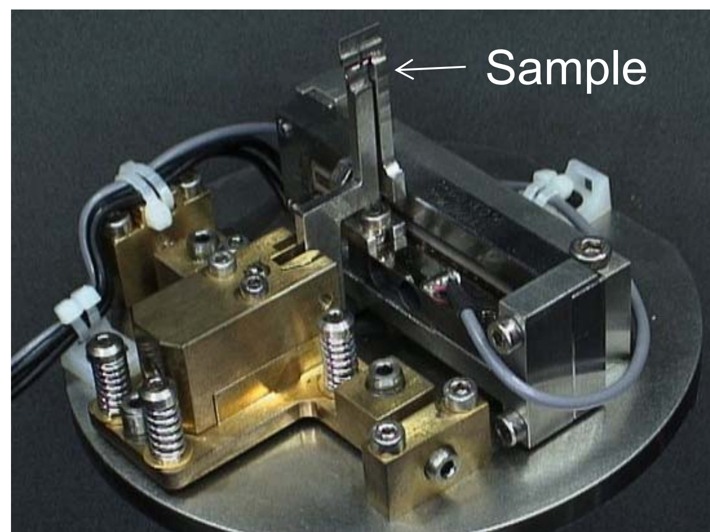
- Opportunity to apply to several materials issues since ~ October.

- Suitable for UltraXRM Nano-CT, 5.4 and 8.04 kVp (800/810) systems.
- 9N piezo, 0.5 mm travel
- Compression, indentation, and tension
- Radiography and limited angle CT

Uniaxial compression



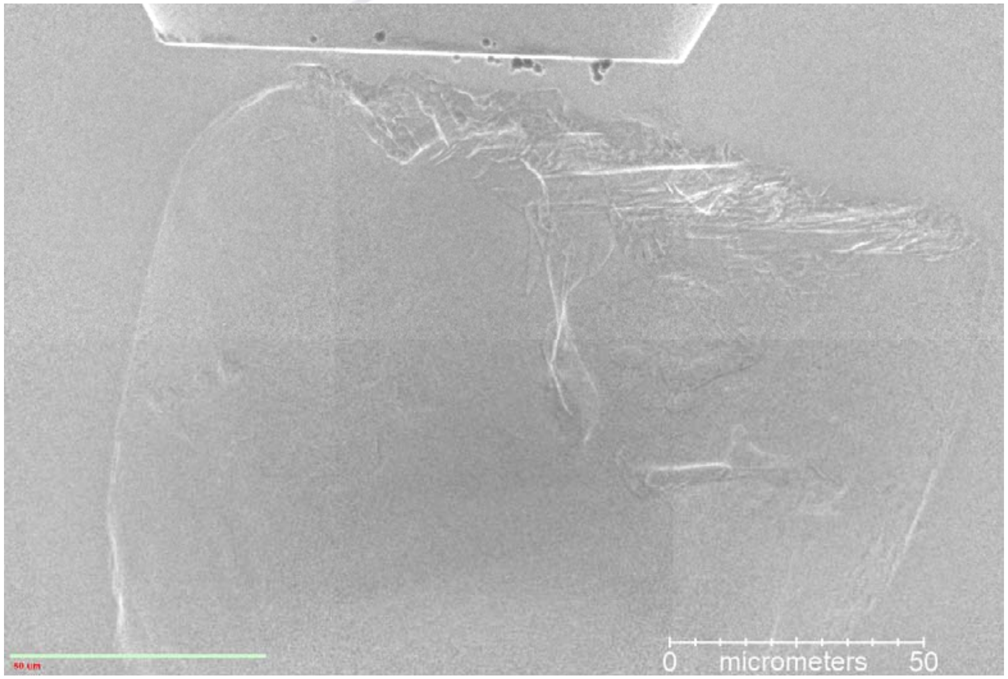
Uniaxial tension

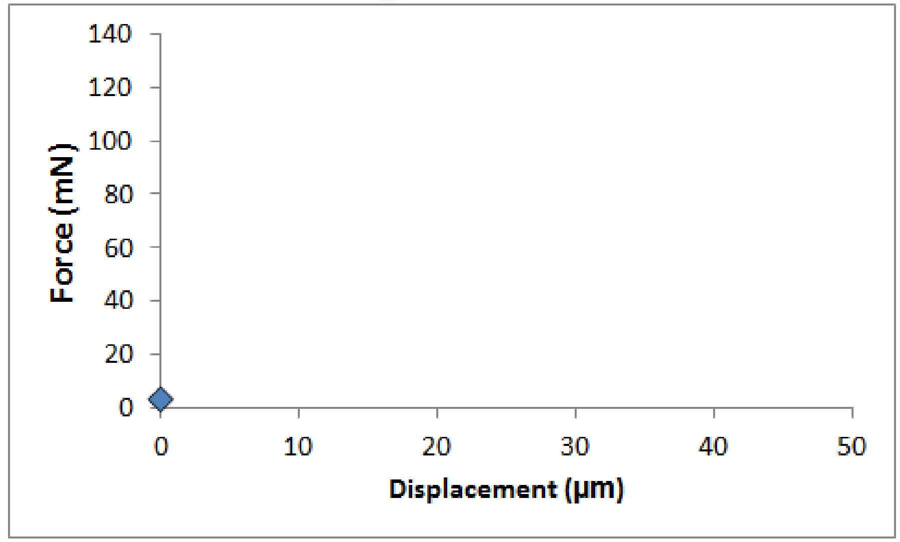


Single Crystal of HMX

- A single crystal of HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) was imaged during compression.
- Phase contrast large field of view.
- Stitched mosaic with load displacement
- Two CT's one before compression, a second after compressing 5 micrometers

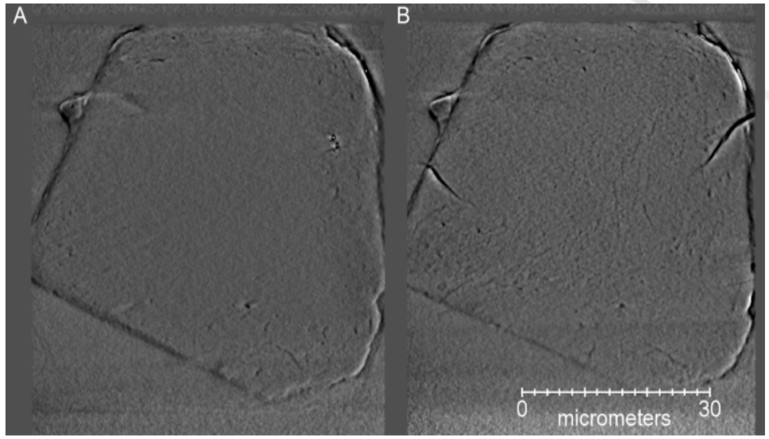
Single crystal radiography



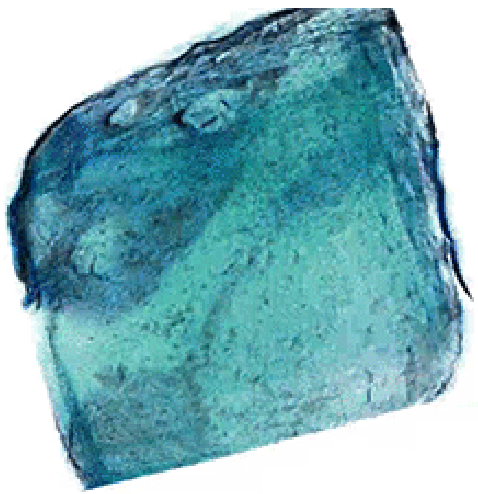


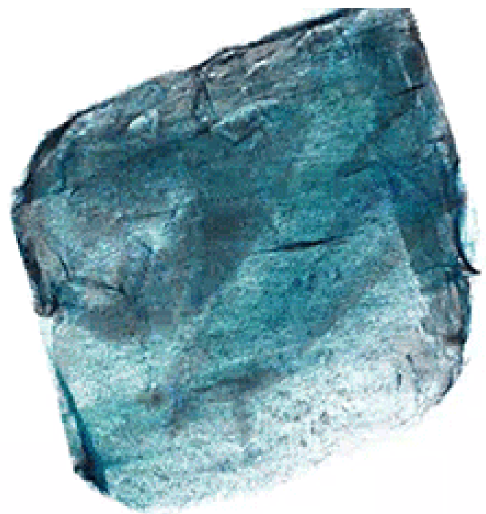
2 x 3 mosaic
Large field of view, Zernike contrast
30 s exposure, 130 nm pixel size
Shatter

High Explosive



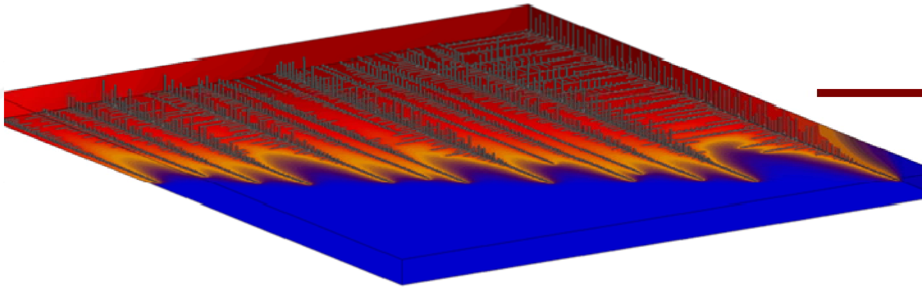
1441 images
60 s exposure
65 nm voxel



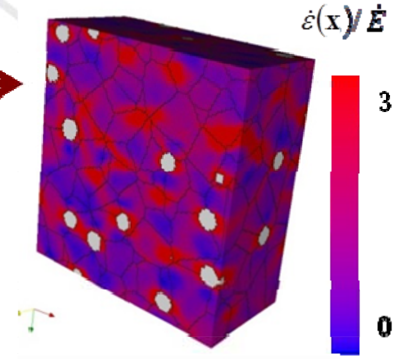


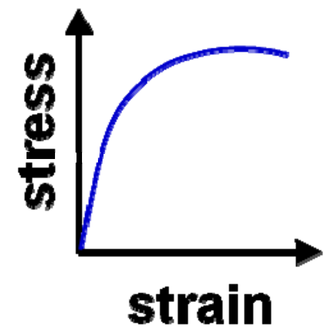
* Rendered by Hrishi Bale using ORS Visual SI

From Solidification to Mechanical Performance: a Modeling and Experimental Campaign

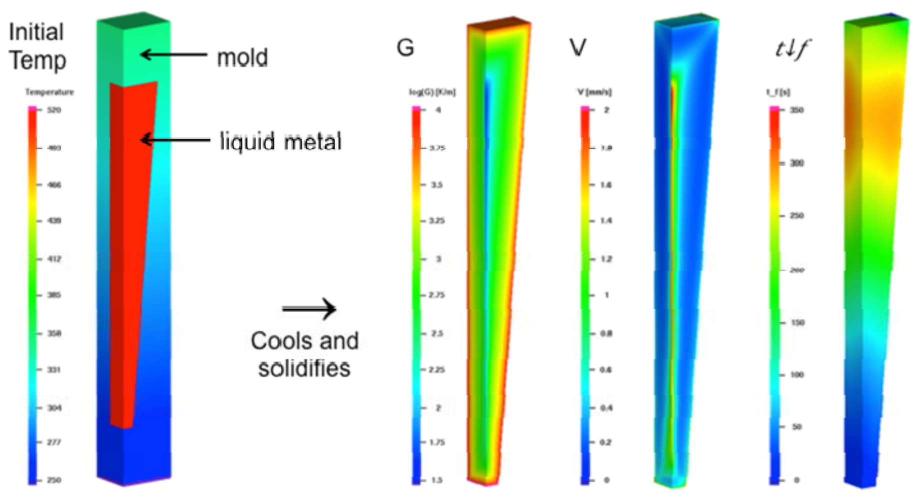


Solidification Microstructure





FFT-based Micromechanical Predictions



Solid-liquid Processing

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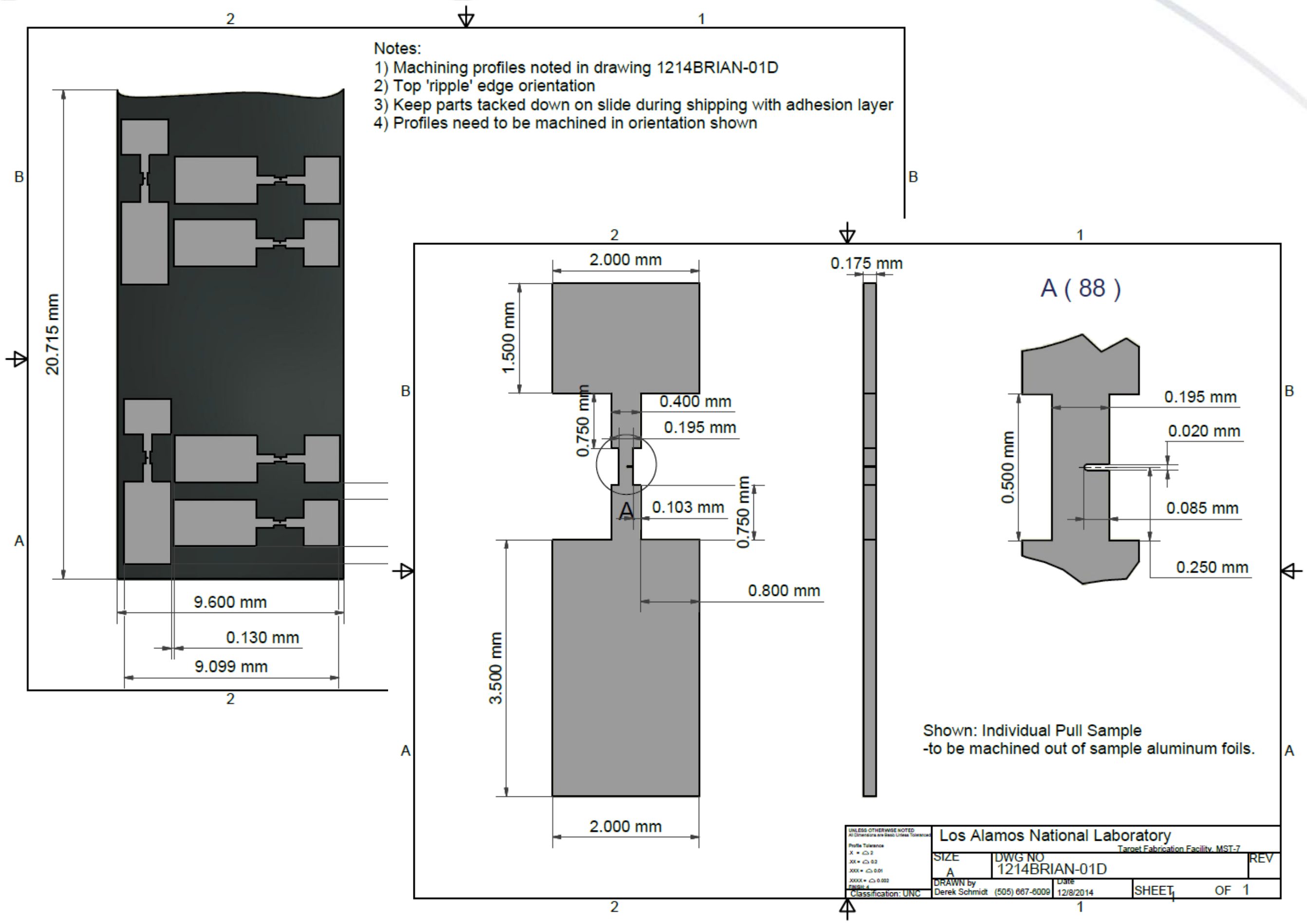
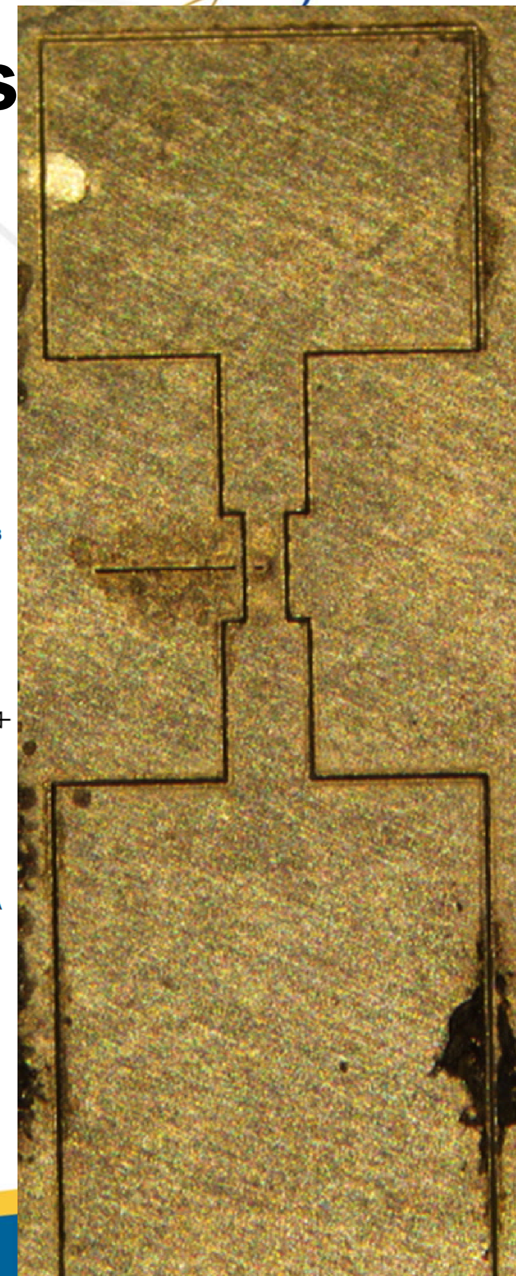
Al-Cu Micro tensile specimen

- Al 88%- Cu 12% thin foil was cyclically melted and cooled.
- Final cooling was 75 K/min with a solidification velocity of 240 μm/s.
- Polished to 150 μm thickness.
- Several tensile specimens were cut from the foil.
- Specimen was imaged in micro CT
- Optical video made of the initial setup of the sample
- Mosaic radiograph collected as the sample was pulled in 2 micrometer increments
- Nano-CT tomograms collected
- Final microCT tomogram collected after breaking.

Tensile specimen specifications





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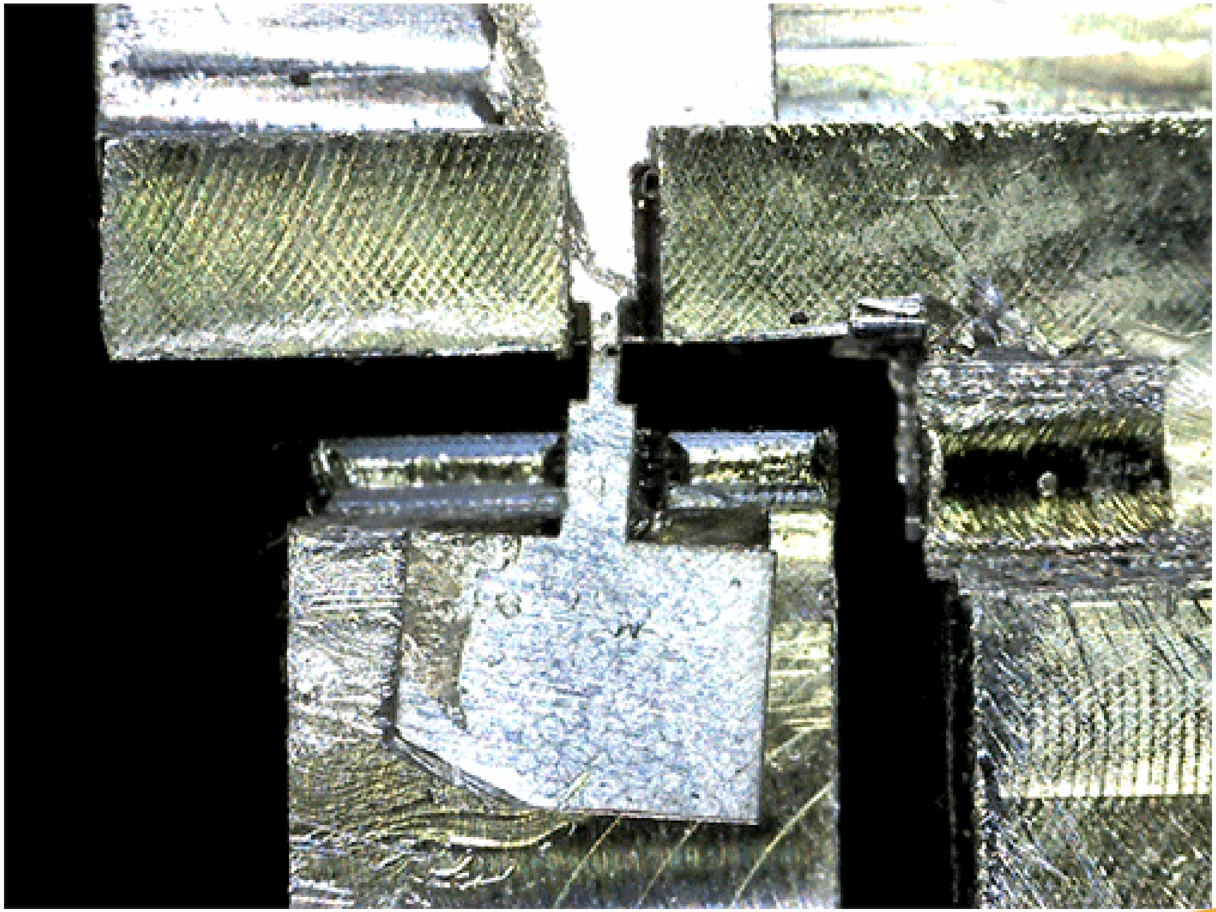
Initial set into load cell

A small amount of silicone was used to tack the tabs into place.



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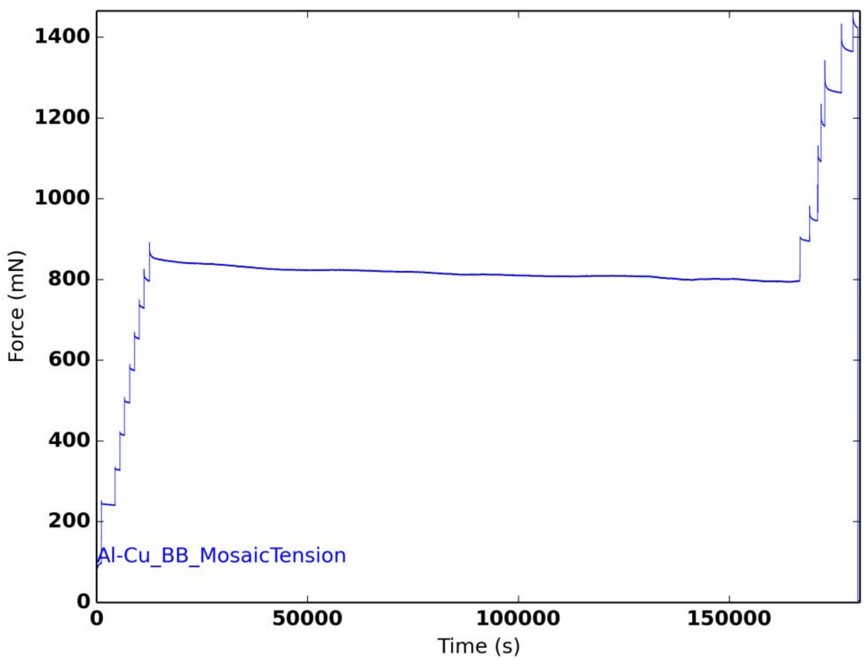
Al-Cu tensile specimen

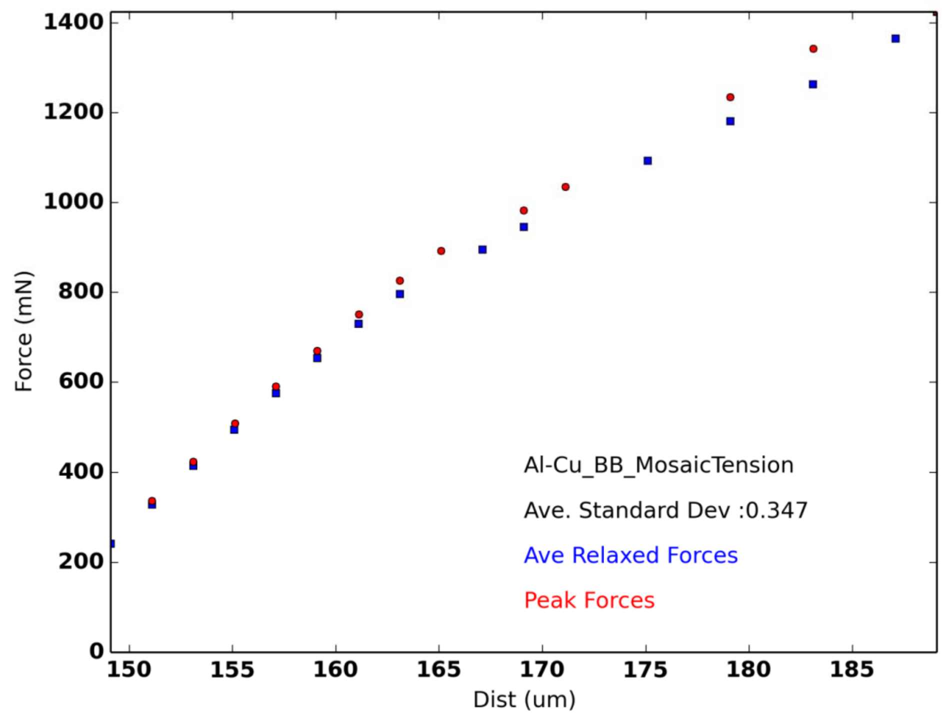
65 micrometers

Mechanical Response

Raw data

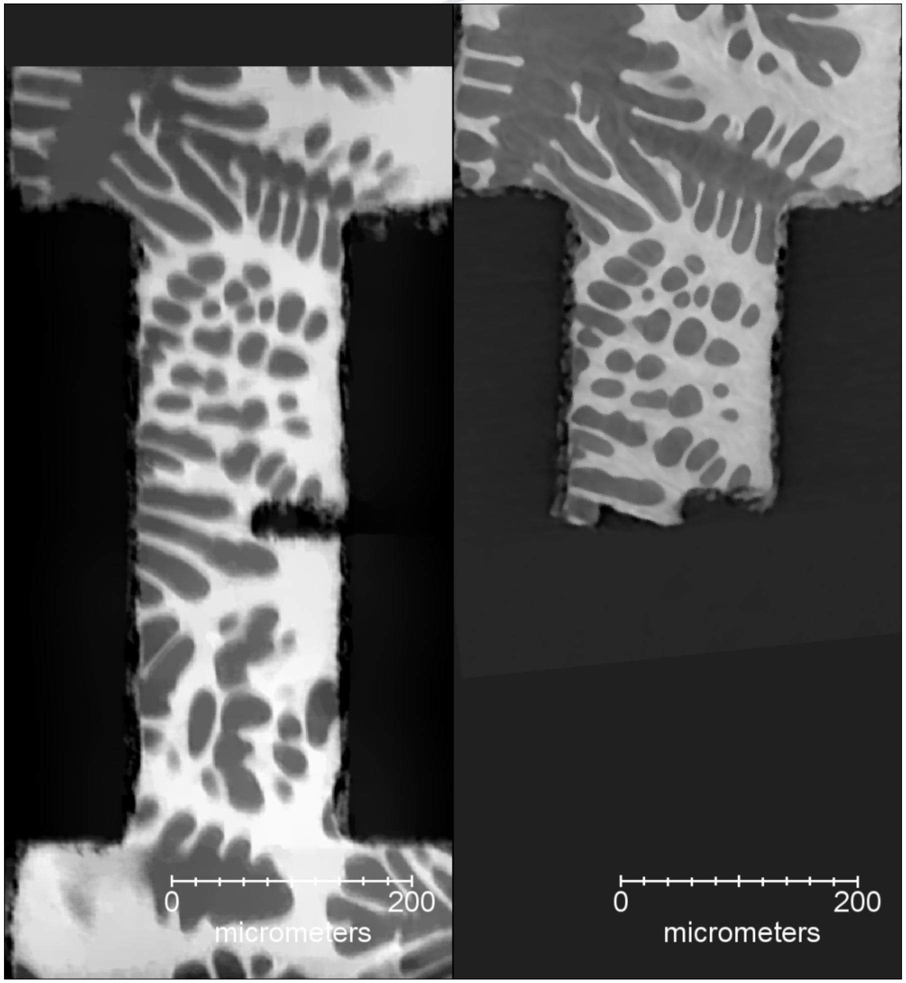


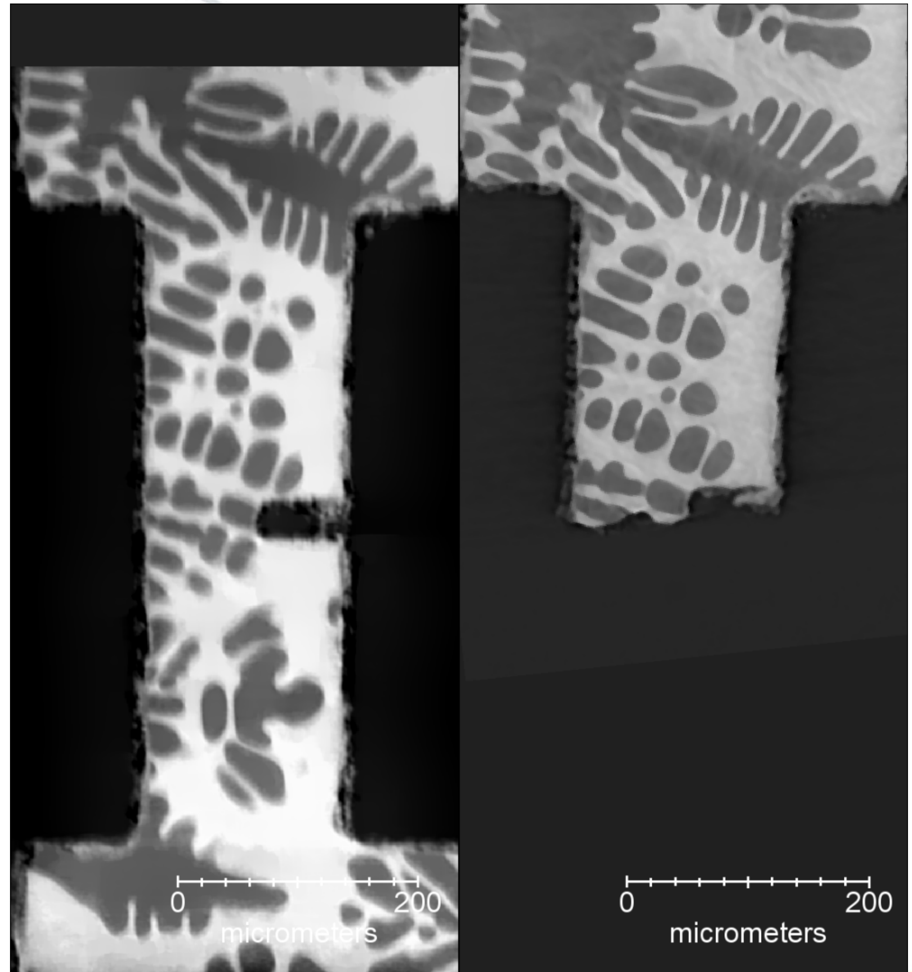
Averaged data



Stress relaxation
- Sample or the stage?

Sample breaks along Al/Cu rich interface; changed direction!





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