# Neutron Imaging @ NIST

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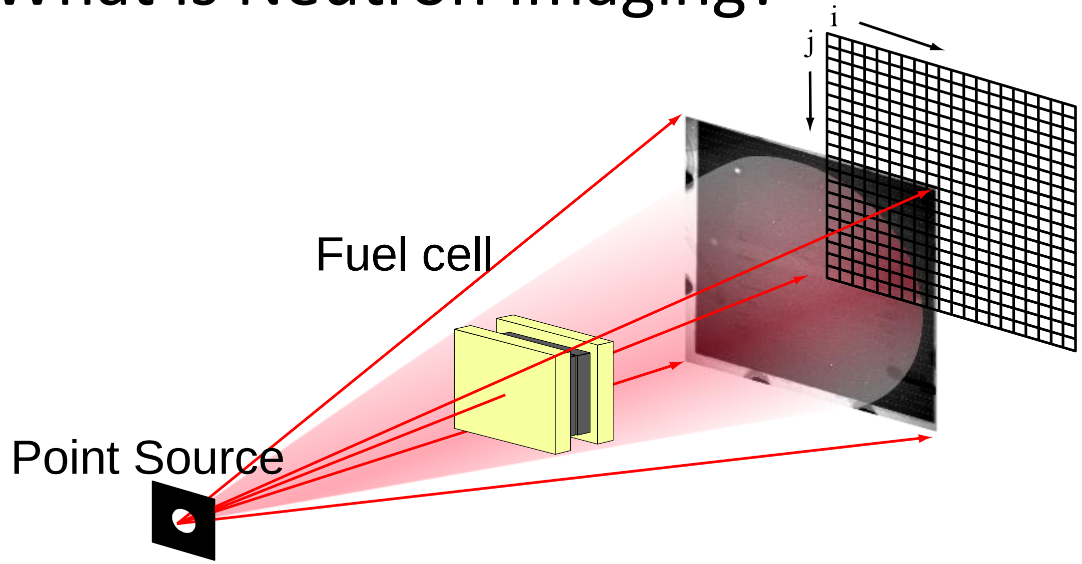
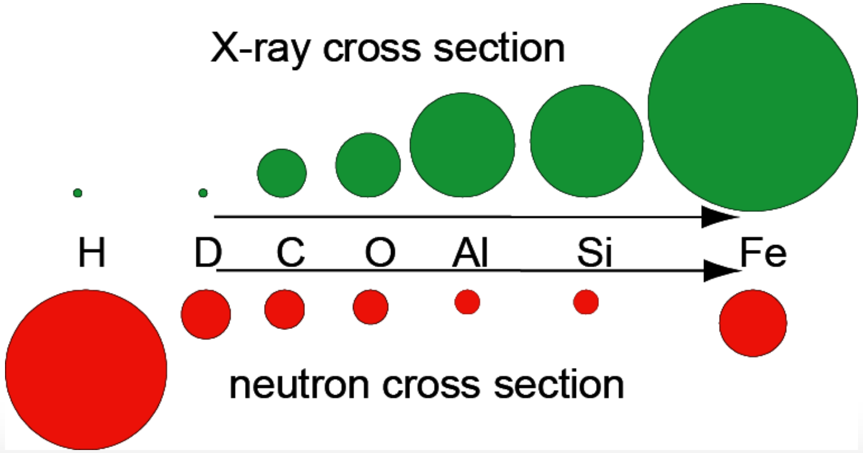
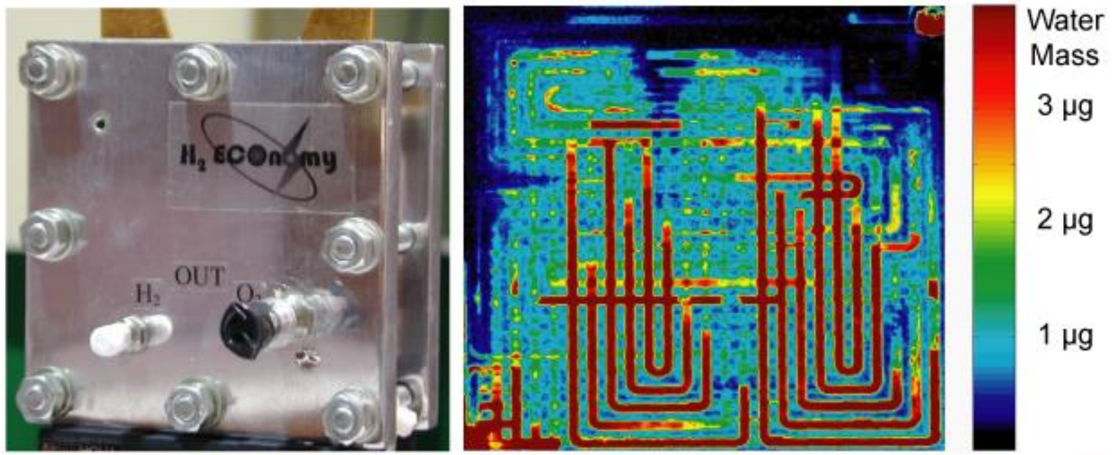
# What is Neutron Imaging?

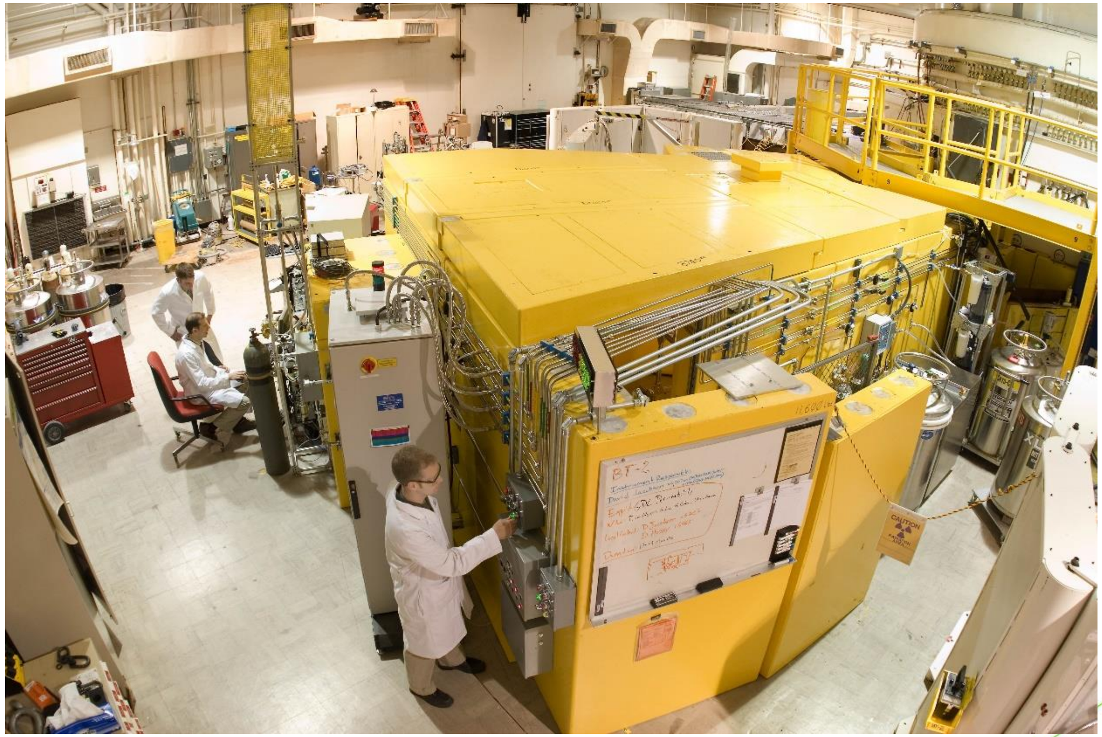


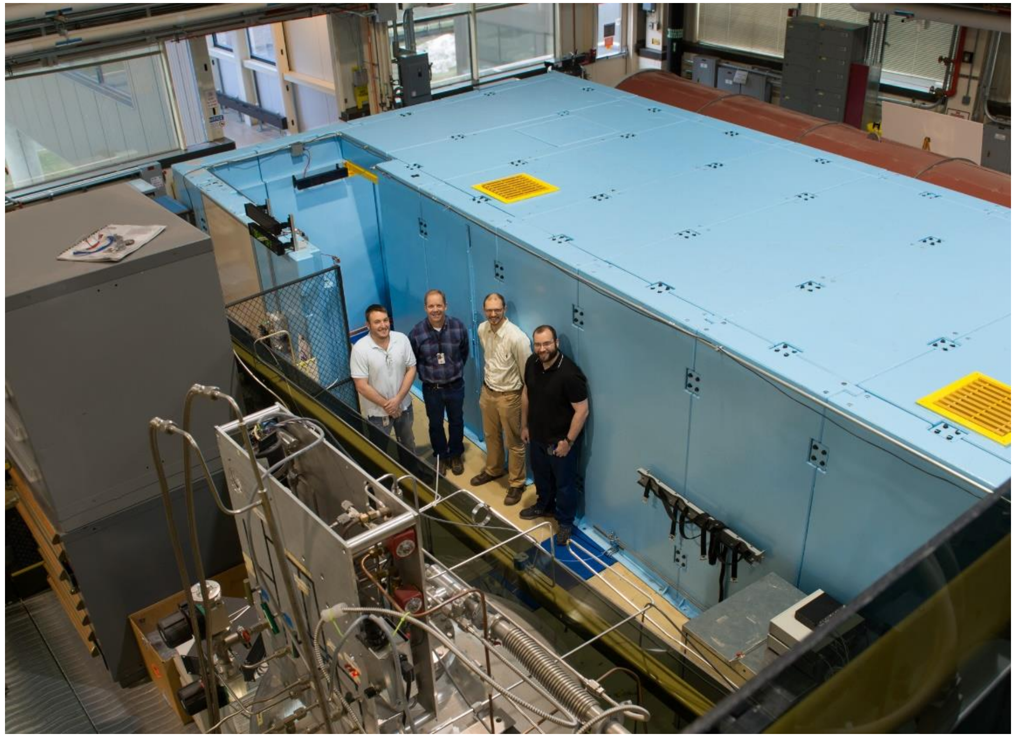




***Hydrogen Fuel Cells: A little water inside a metal matrix - an ideal problem for neutron imaging.***

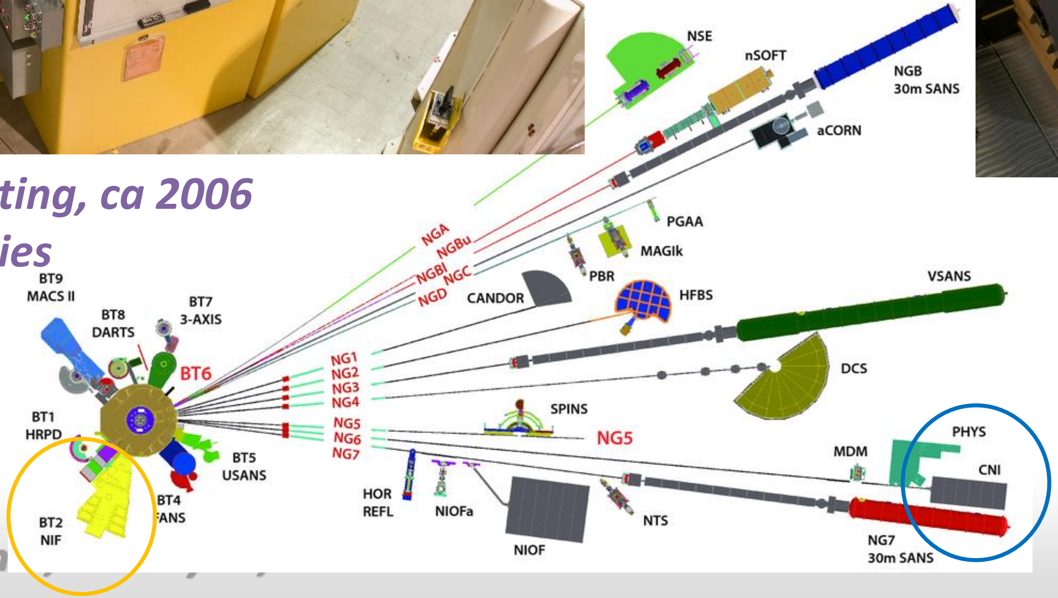
# The NIST Neutron Imaging Instruments



***BT2: Thermal, penetrating, ca 2006***
- ***Fuel cells & Batteries***
- ***Infrastructure***
- ***NeXT***

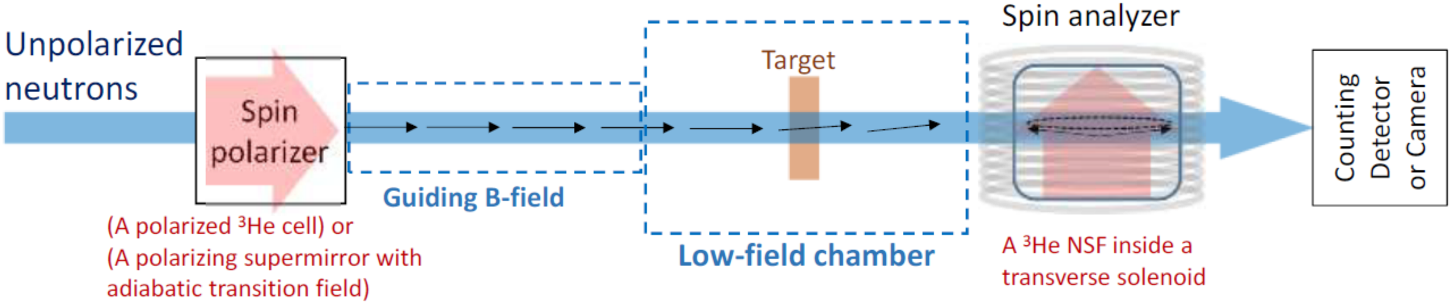


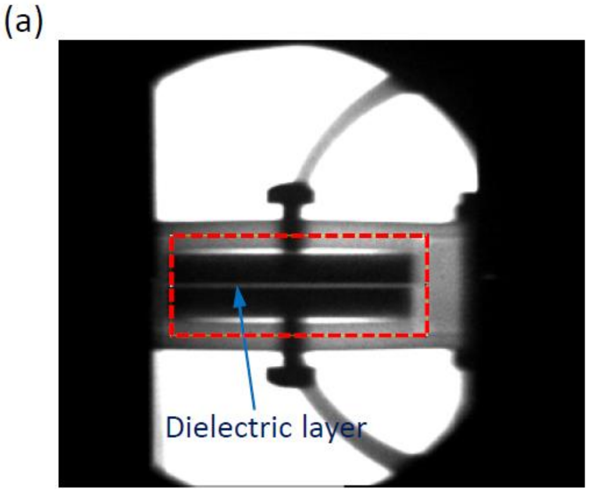
***NG6: Wavelength Selective***
- ***Installed AUG 2015***
- ***Multi-scale Imaging***
- ***Wolter Optics***

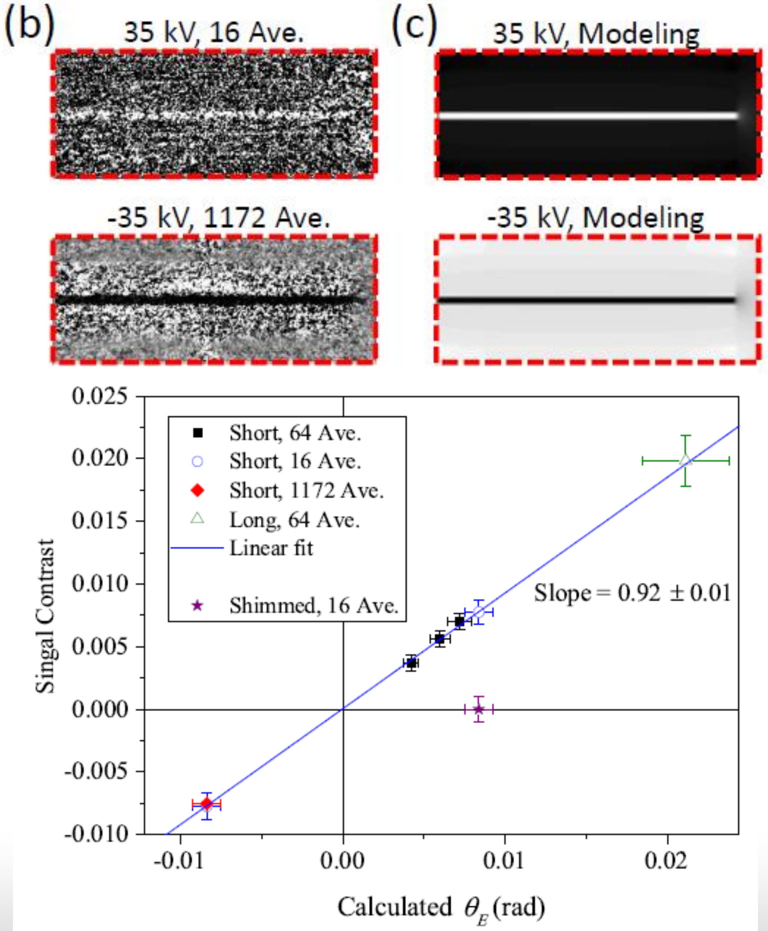


# Cold Neutron Imaging Instrument

CNII is a “neutron optical bench” to develop energy selective imaging methods to produce multi-scale images, from fm to m.

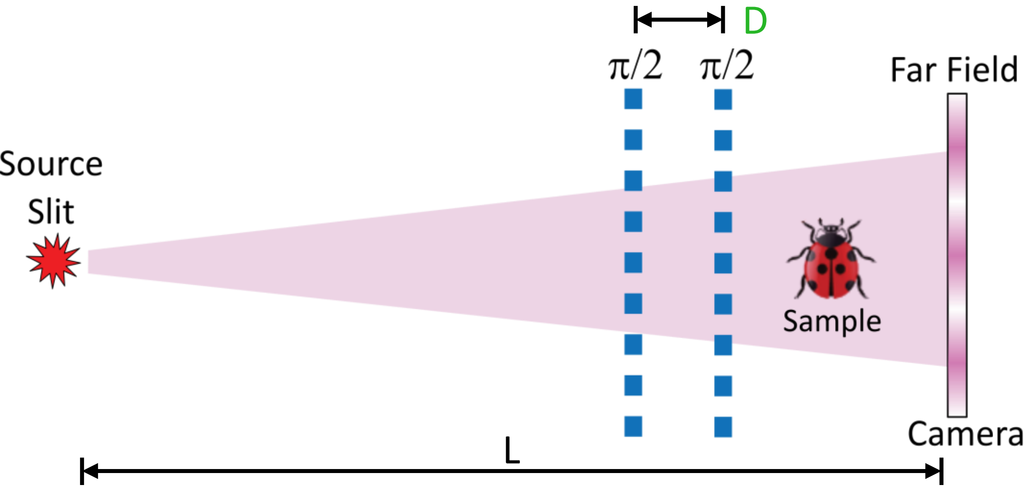


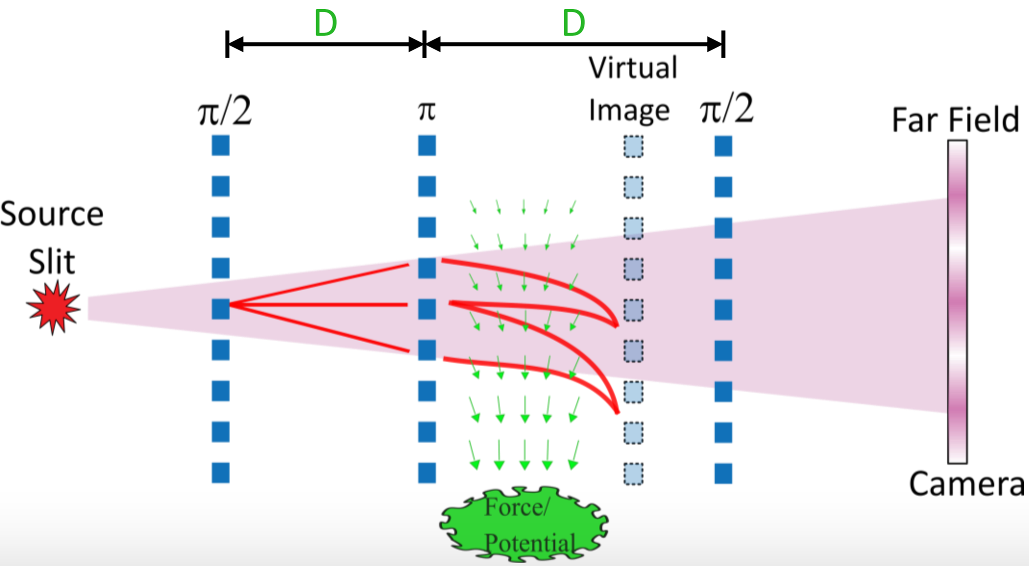

Collaboration with Sandia and NCNR led to first neutron image of an Electric field “Sensitive neutron transverse polarization analysis using a 3He spin filter”, Y.-Y. Jau et al, RSI 91, 073303 (2020)
“Electric-field imaging using polarized neutrons”, Y.-Y. Jau et al, PRL, 125(11), p.110801 (2020).

# Novel Neutron Imaging Far Field Interferometer




## 2-Grating Geometry

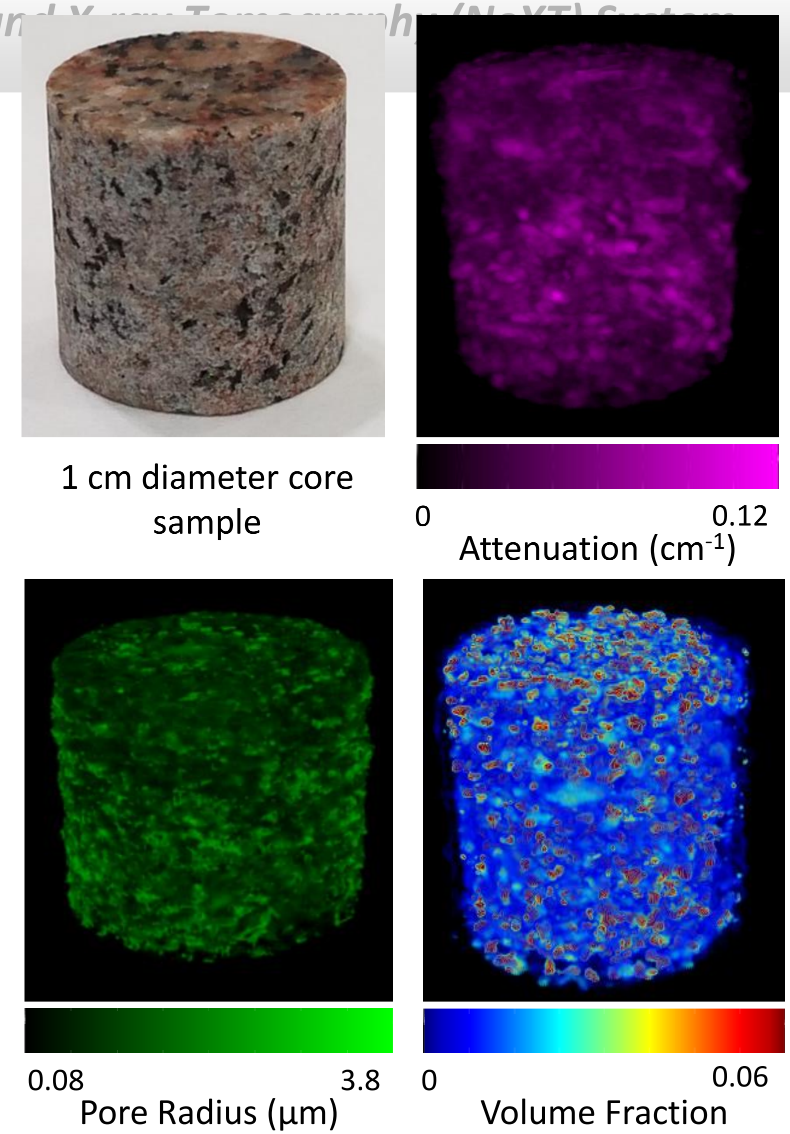
- D = [0.05 to 5 cm]
- $P_D = P_G L / D$
- Sample microstructure reduces visibility
- ***Tunable period probes $1\text{-}10^4$ nm***




## 3-Grating Geometry

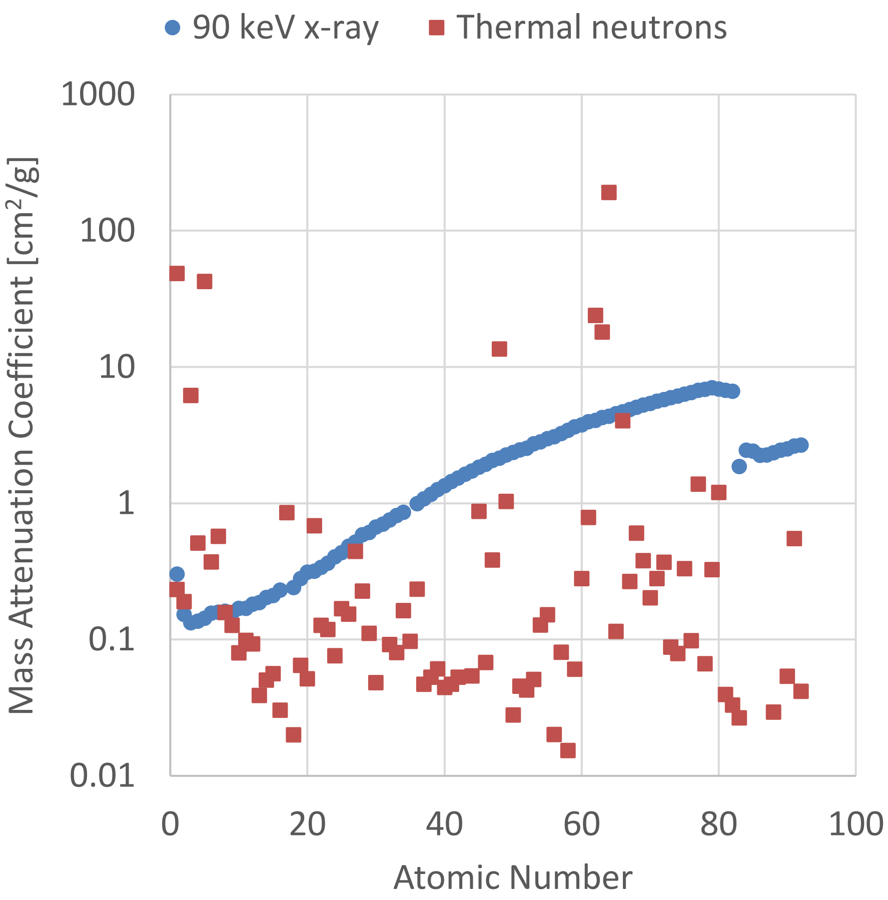
- First practicable meters long neutron interferometer
- ➢ Enables measure of Big "G"

*1st neutron demonstration performed at CNII: PRA **95**, 043637 (2017), PRL **120**, 113201 (2018)*

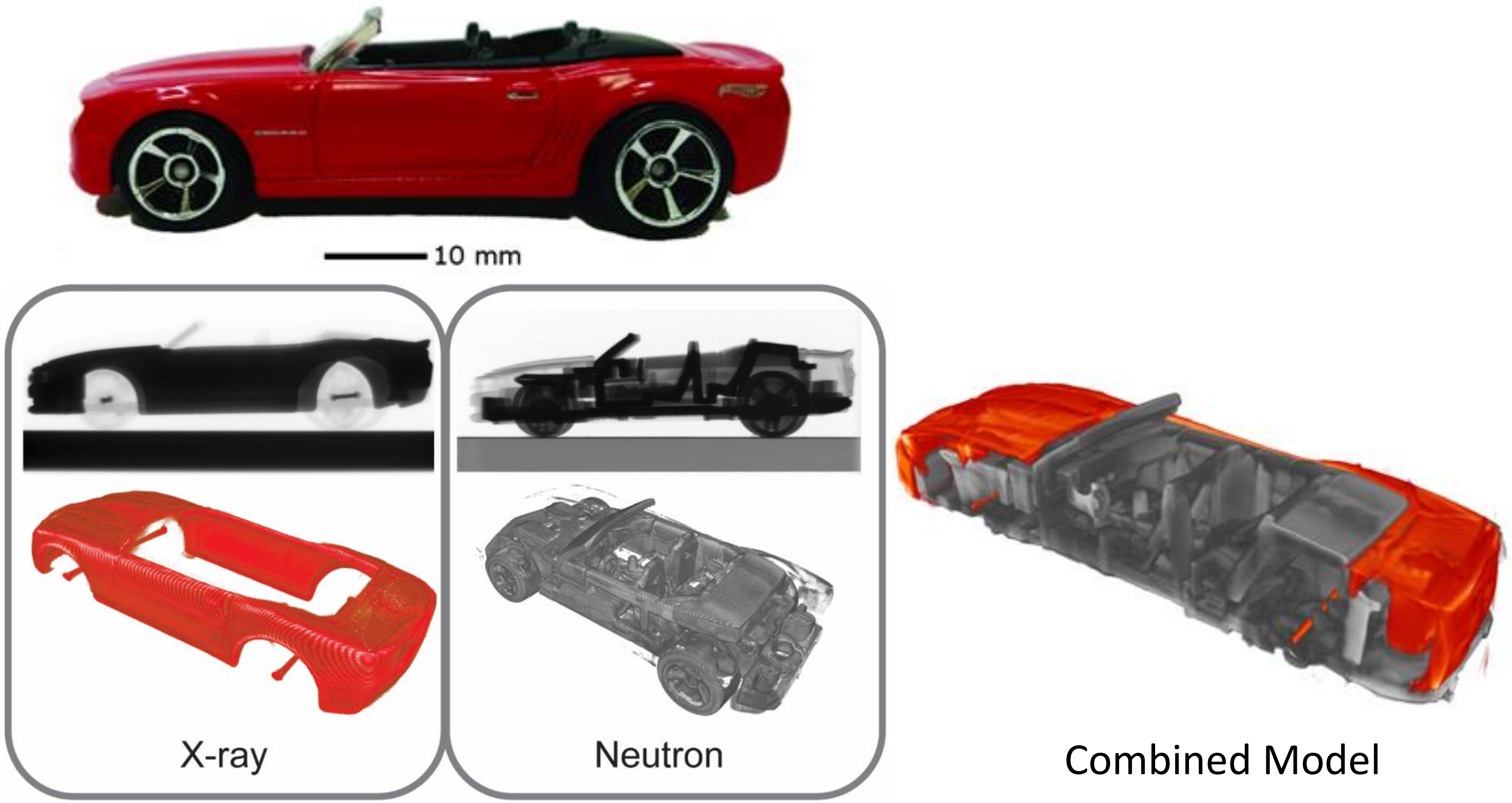



IMS-funded "INFER project" to create NCNR user instrument for "SANS-tomography" and measure G

# Why combine neutrons and X-rays? Awesome complementarity!

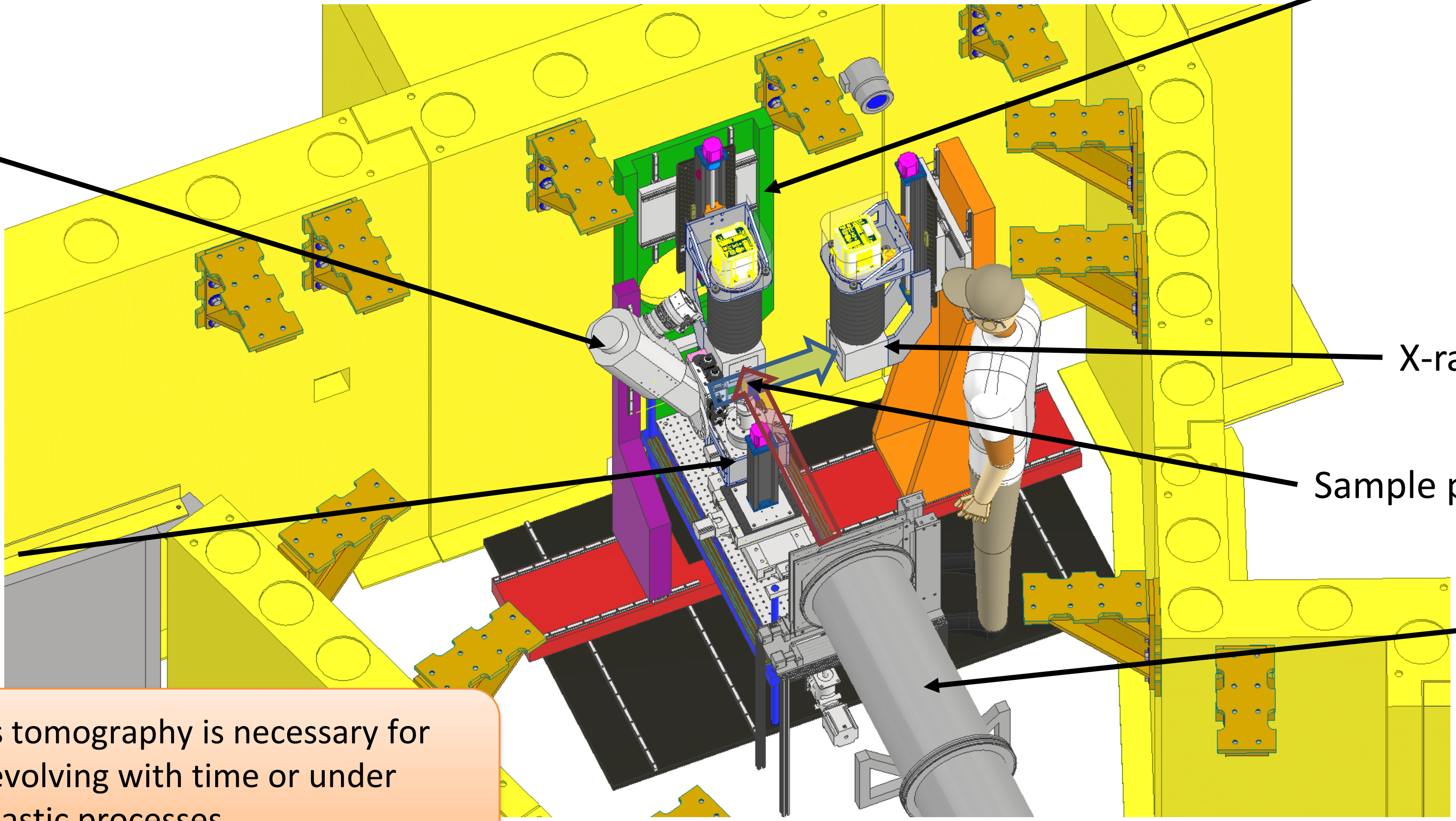



Photograph of Hot Wheels
1:64 scale die cast toy car




Combined Model

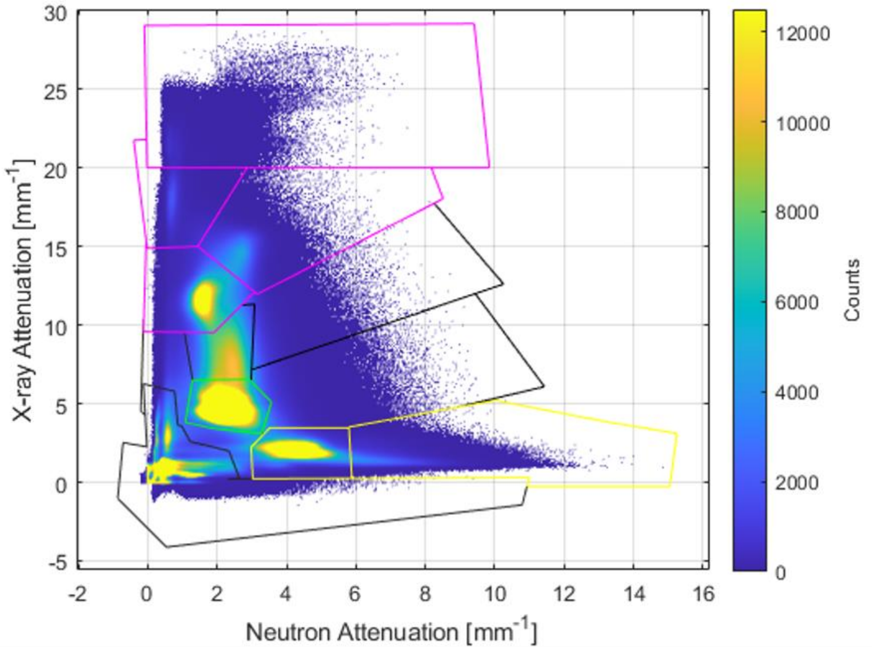
# The NIST **Simultaneous** Neutron and X-ray Tomography System

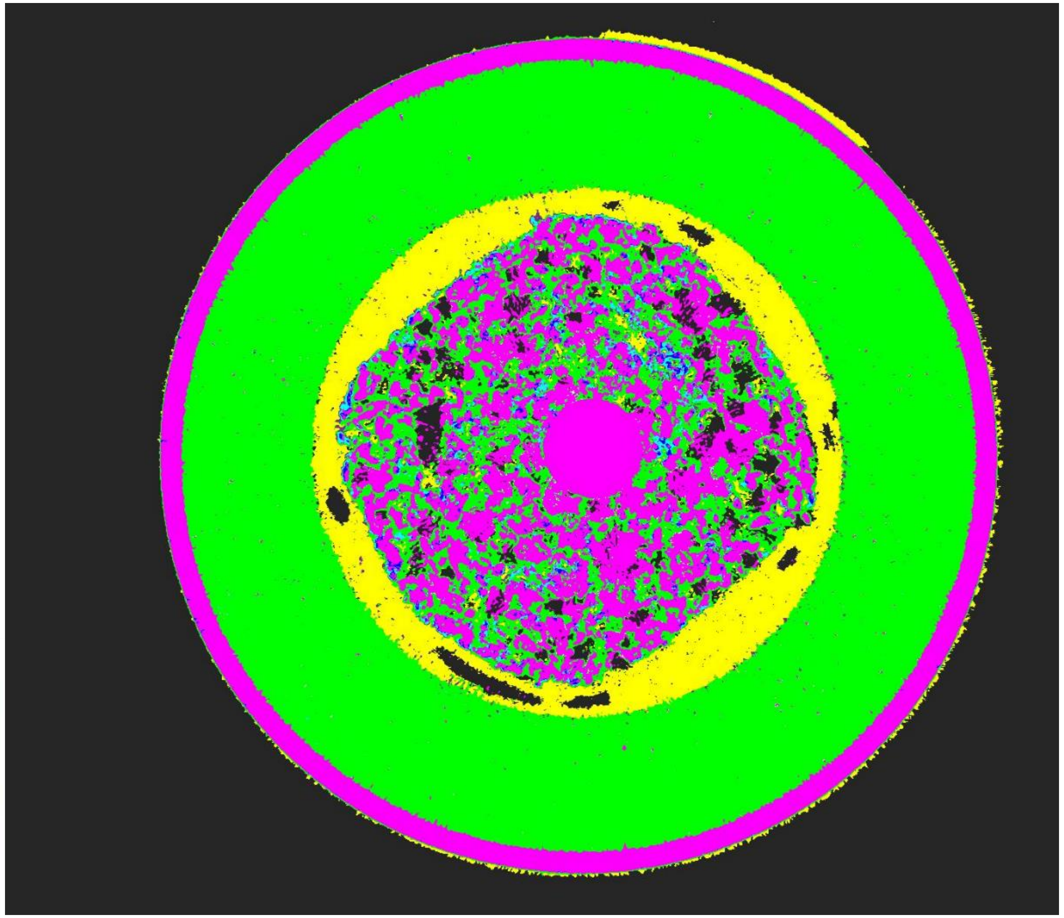


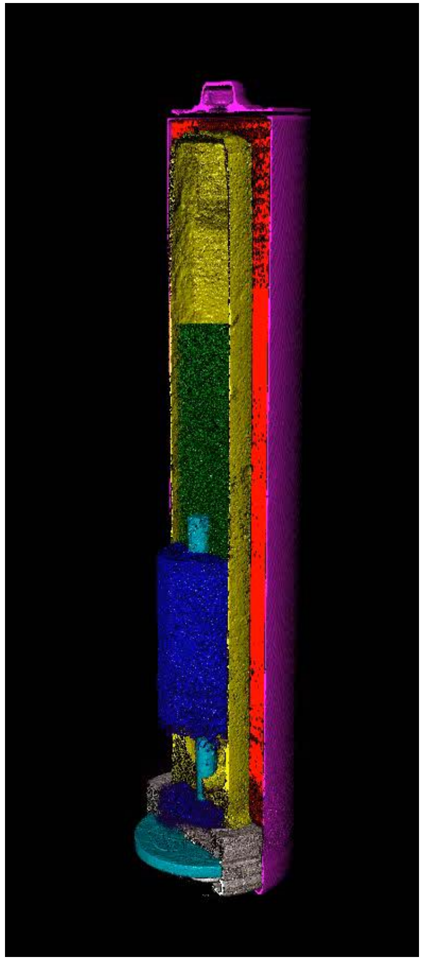

Truly simultaneous tomography is necessary for samples slowly evolving with time or under stochastic processes.

J.M. LaManna et al., “Neutron and X-ray Tomography (NeXT) System for Simultaneous, Dual Modality Tomography”, Rev. Sci. Inst., 88, 113702 (2017).

# Segmentation based on dual histogram of attenuation values





Cathode and electrolyte

Polymer

Can and zinc anode

Void

NIST-developed software acquires, reconstructs, digitally aligns, and co-segments the two modal volumes. NIST bivariate segmentation software provides a simple and fast process for segmentation. Active effort for segmentation can be as low as 10 minutes!

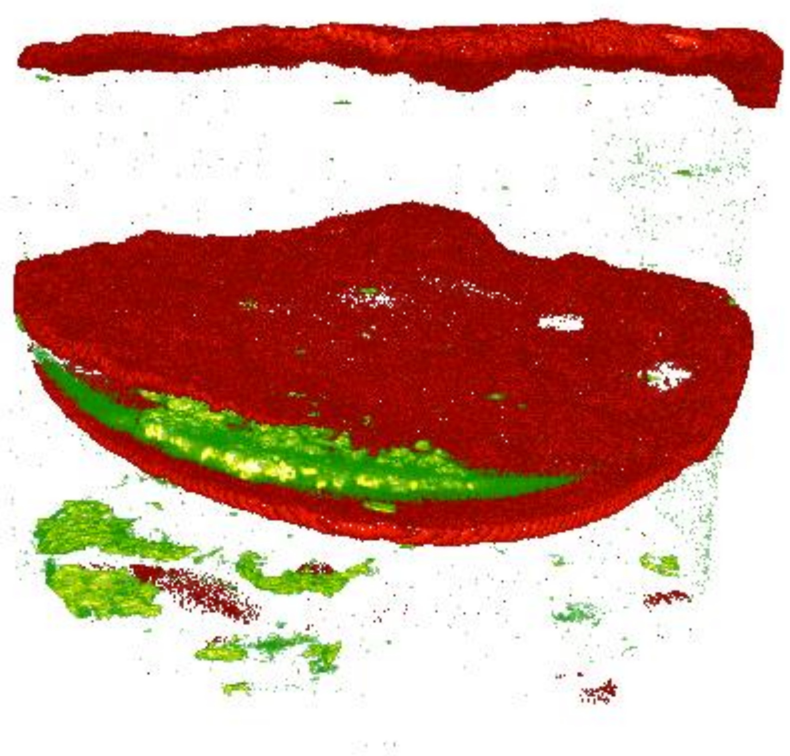
Volume is
1400 x 1400 x 5000

J. M. LaManna et al, Proc. SPIE 11494 (2020); doi: 10.1117/12.2569666

# Improvements by using iterative registration and phase segmentation

**Segmented ϕ25mm shale presented in 2017**

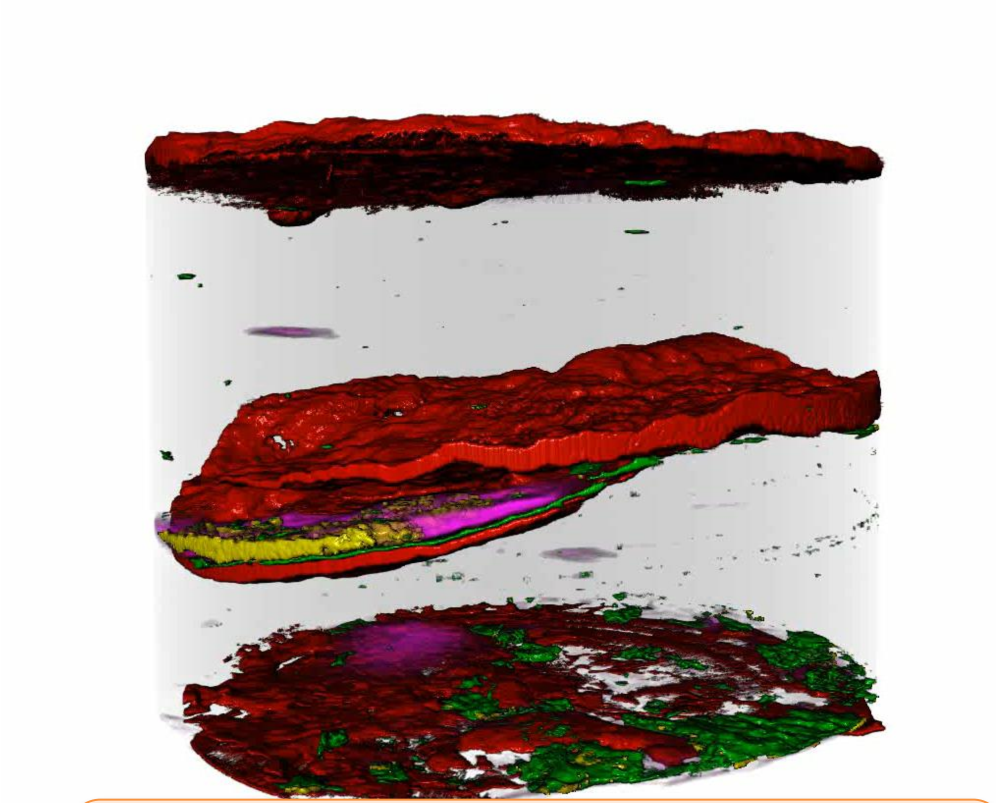
**Independent 1D histogram segmentation, manual alignment**



15 µm pixel, 18 hr scan time

**Current improvements**

**Iterative registration + bivariate histogram segmentation**

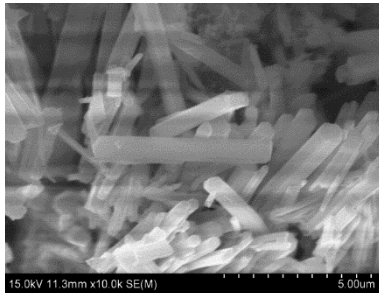


Bivariate histogram segmentation provides better fidelity on strong organic layers and captures additional mineral content (magenta)

# Introduction to Concrete Degradation

- Alkali-Silica Reaction and Delayed Ettringite Formation both form expansive phases that crack and damage concrete
- Important to understand and control to prevent premature failure of critical infrastructure such as nuclear reactors, dams, bridges, etc.


**Figure 1.** Characteristic 'map-cracking' pattern in a bridge column foundation (left); macrocracks and ASR gel exudation at the surface of a core extracted from an ASR-affected concrete structure (middle); and a thin-section showing gel-filled microcrack extending from reactive aggregate through the cement paste (right) (Fournier et. al., 2010).

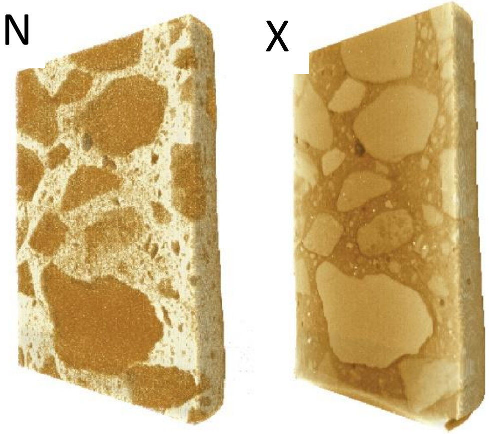



Ettringite crystals

**Multiscale approach to identify detrimental phases at high resolution while also using industrial relevant aggregates**

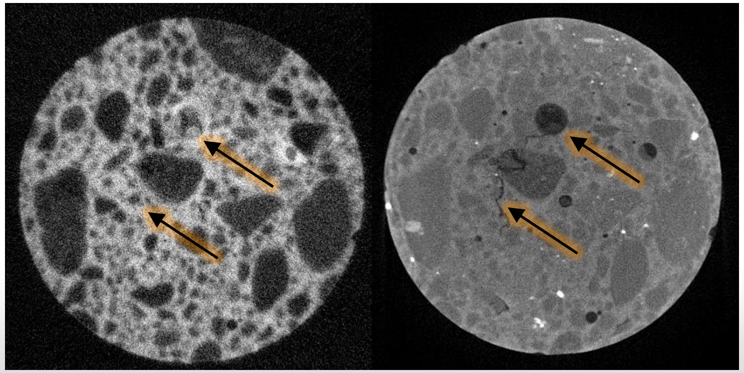
Large 50 mm diameter cores with industry relevant course aggregate, 60 micron resolution

N X

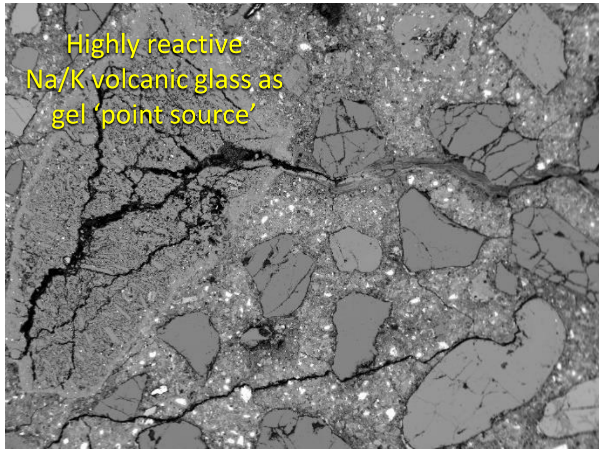


Small 10 mm diameter cores, 15 micron resolution
Highly reactive model system to understand ASR formation

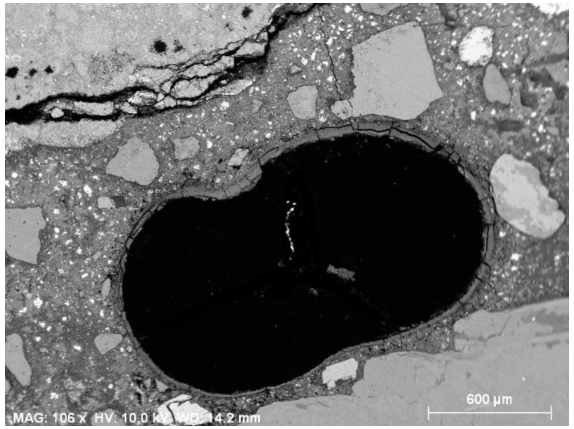
N X

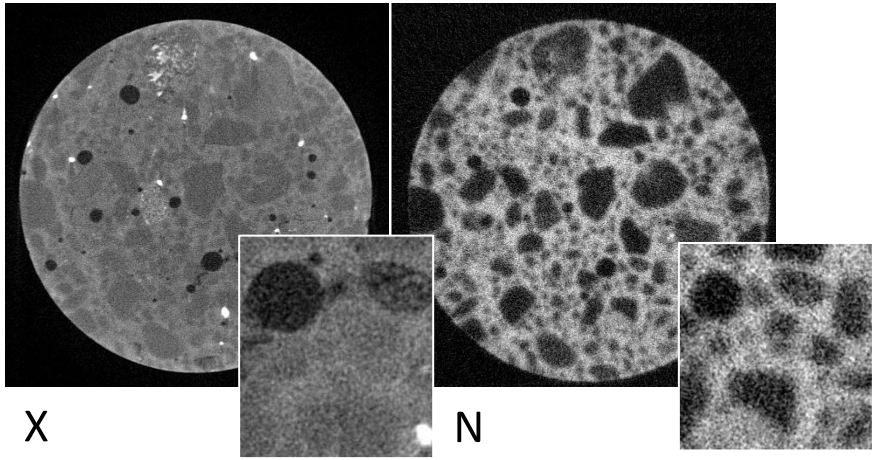


# Identifying structure formed by ASR

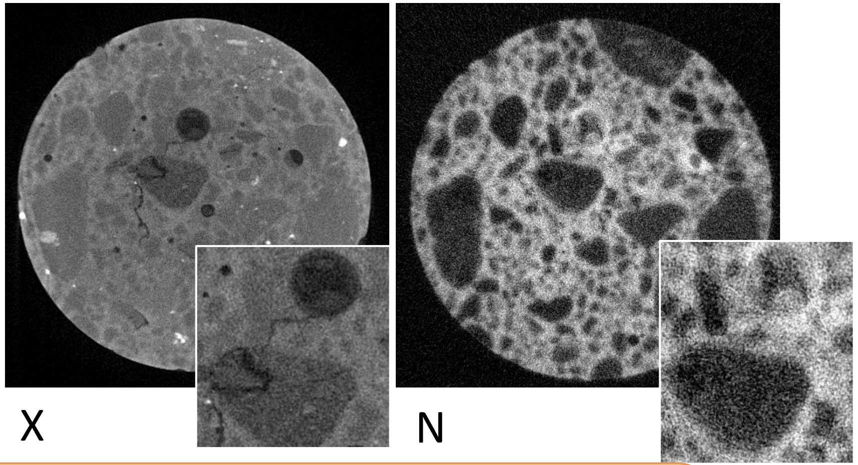



Characteristic structures formed by ASR imaged destructively with SEM



X N

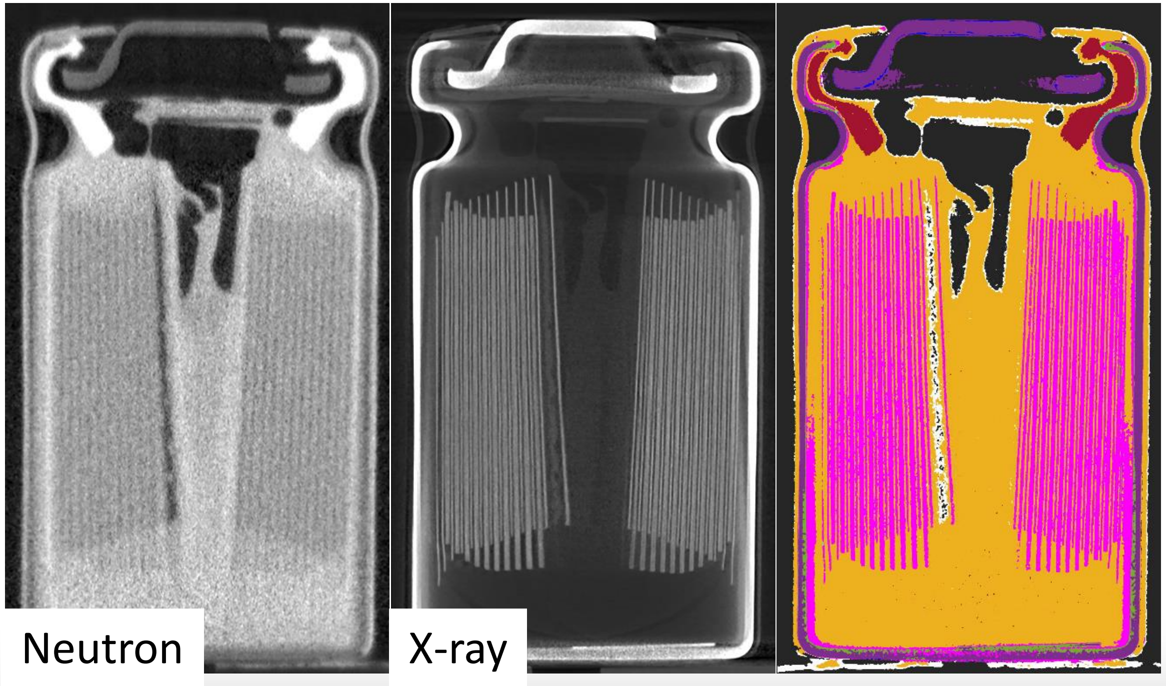


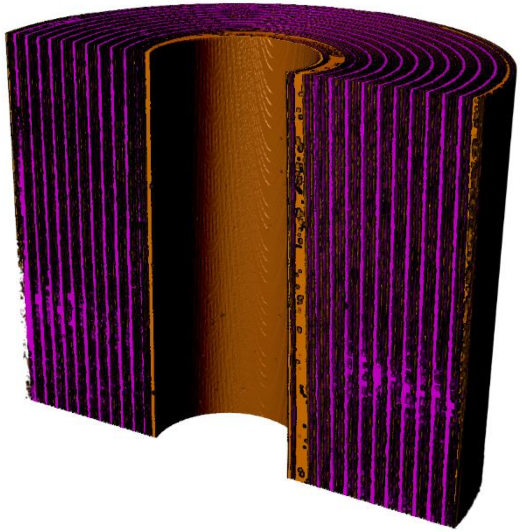
X N

This is the first time ASR has been identified nondestructively through a large volume, i.e. 10 mm dia x 15 mm height vs single slices in SEM

# Detection of interfaces and lithium transport in lithium-ion batteries

- Isotopic sensitivity to lithium allows neutron contrast adjustment
- NeXT improves identification of interfaces when tracking lithium transport
- Neutron and X-ray volumes can be directly correlated during discharge/charge cycling



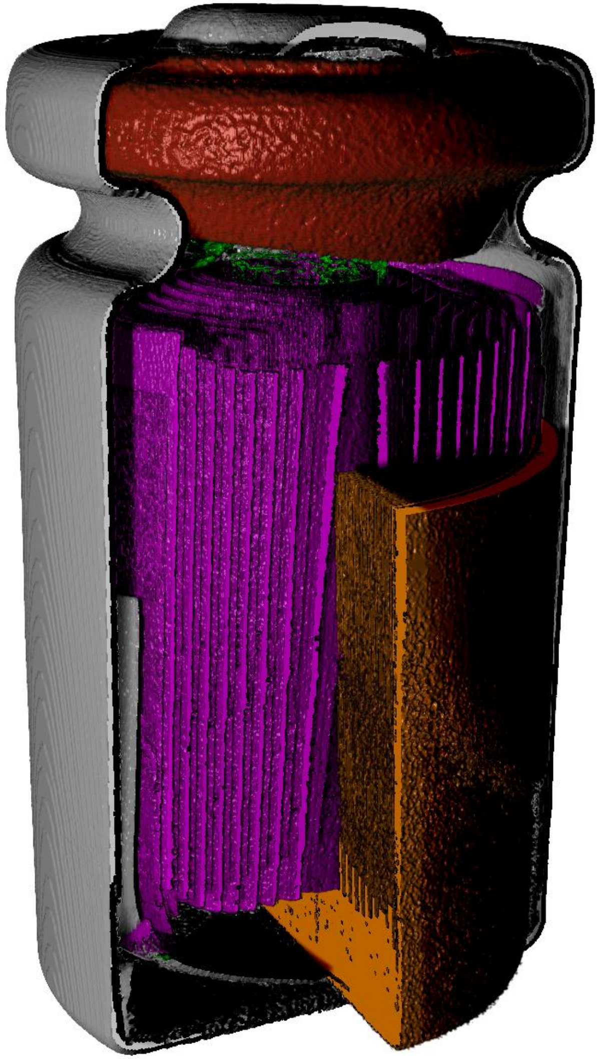



**Copper current collector and NMC electrode**

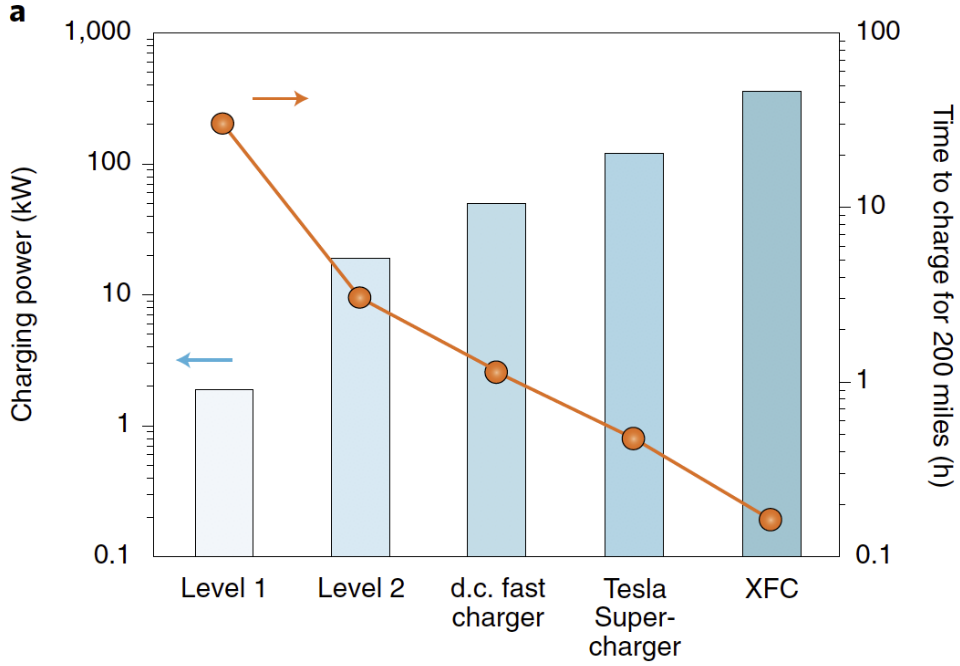
**Graphite electrode and electrolyte**

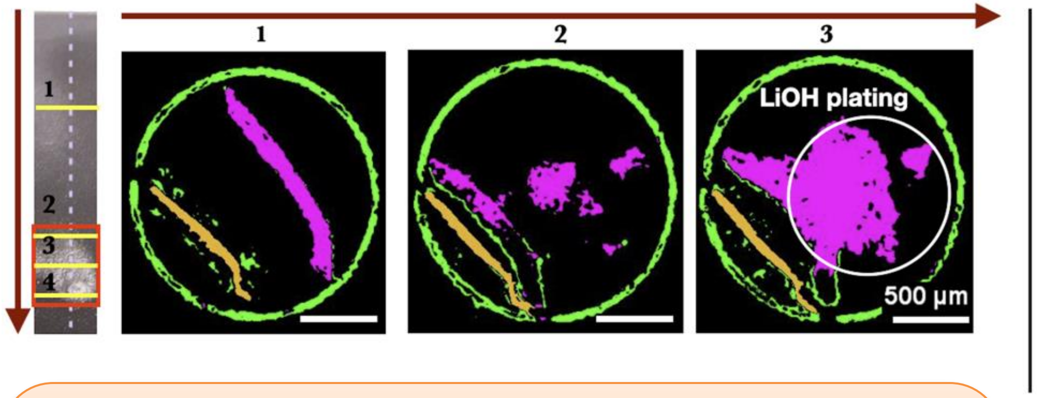
**Gasket**

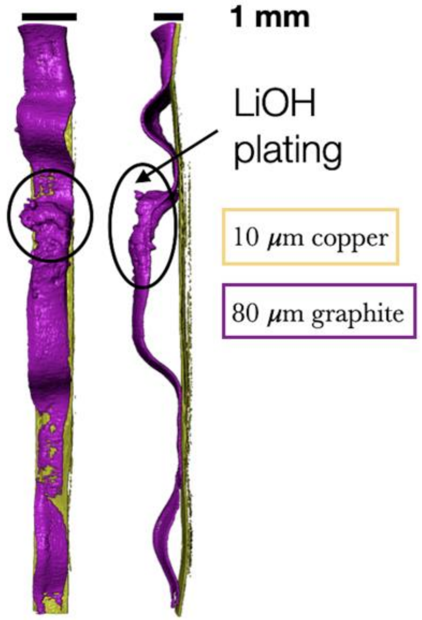
Aluminum current collector



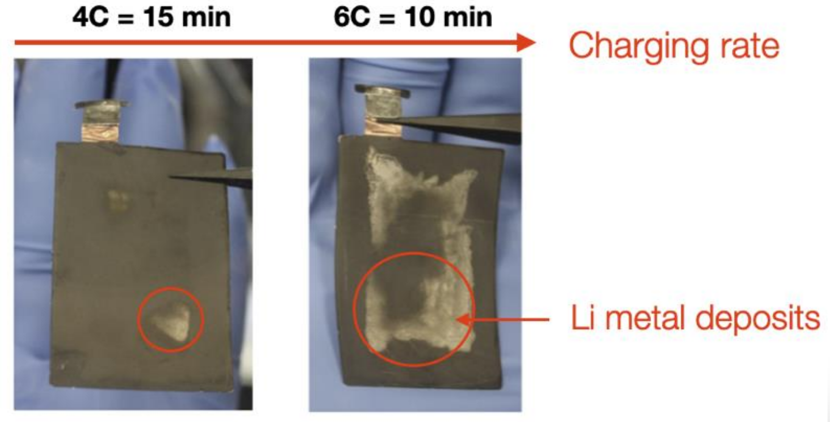
# Lithium Plating During Extreme Fast Charging [Collab: SLAC and Stanford]



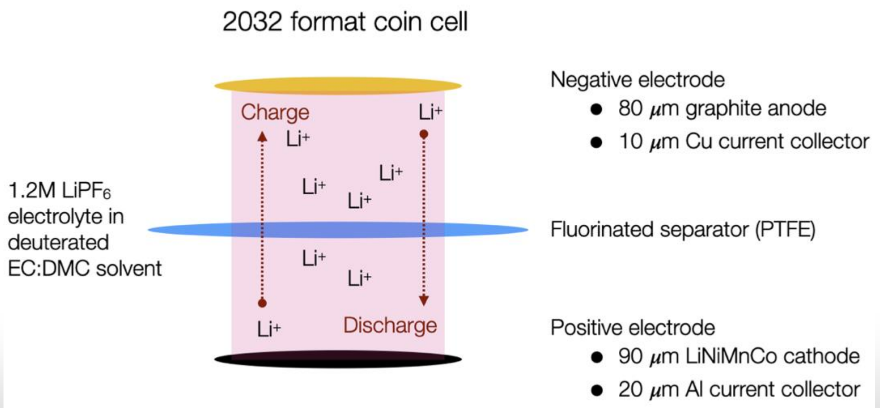



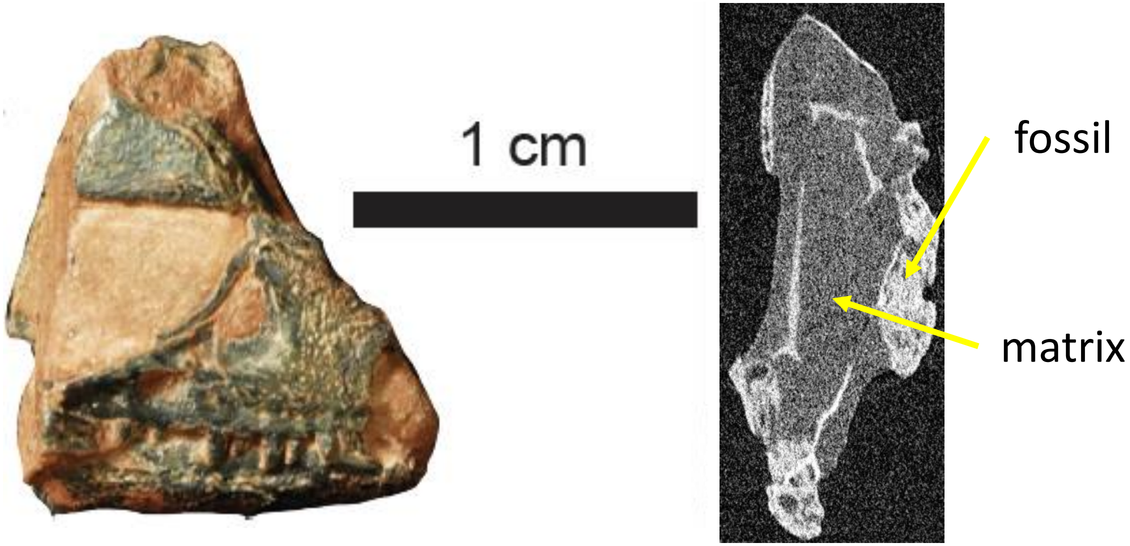

We have demonstrated the ability to see lithium plating in ex situ experiments. We are currently working to develop improved imaging friendly batteries to track plating in situ.




Imaging friendly battery: reduction of H in separator, electrolytes, and gaskets to improve Li detection

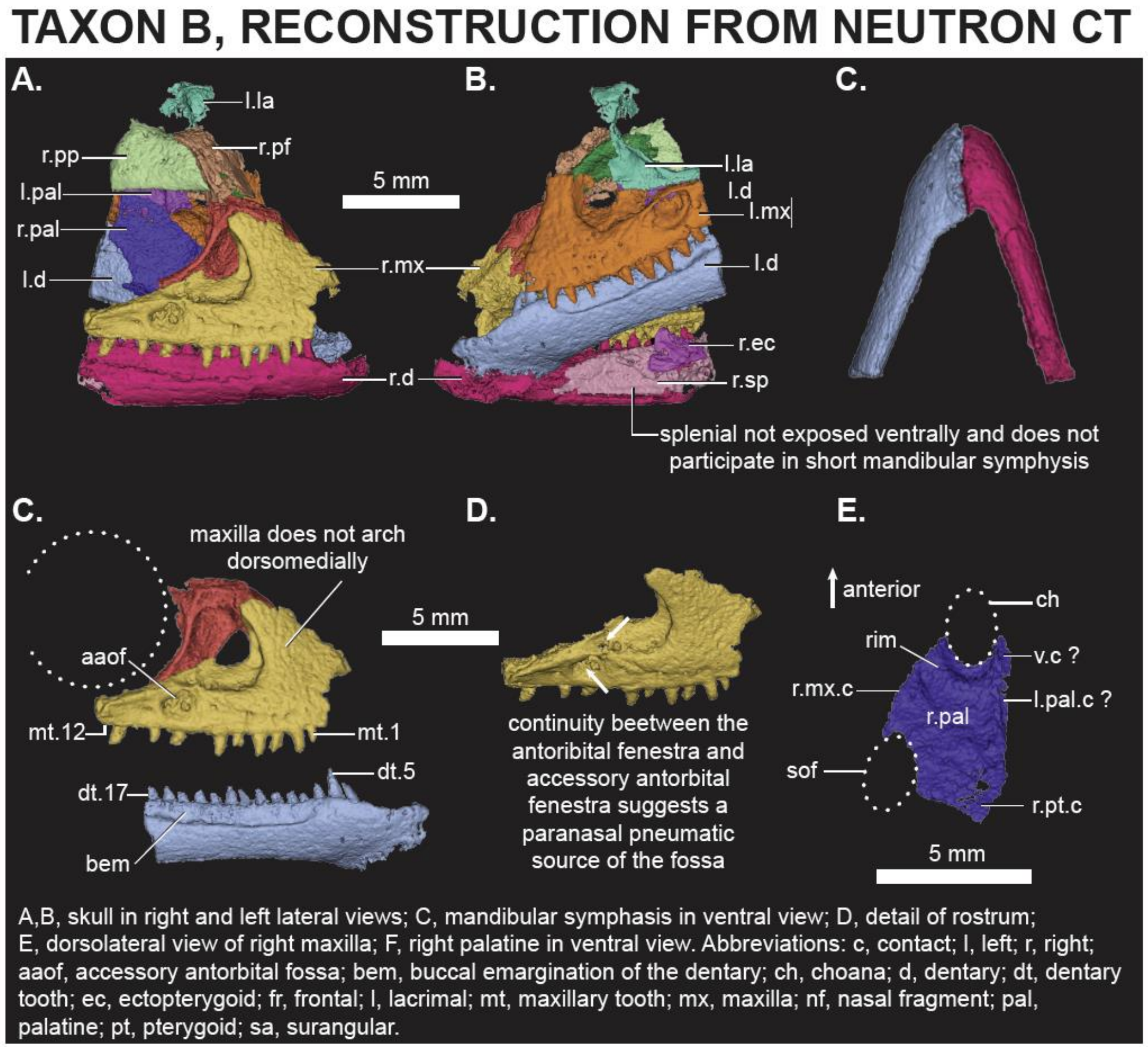


# Anatomical Analysis of Jurassic Crocodyliform in Iron Rich Rock




Fossilized crocodyliform skull from the middle Jurassic located in red mudstone from Huizachal Canyon, Tamaulipas, Mexico

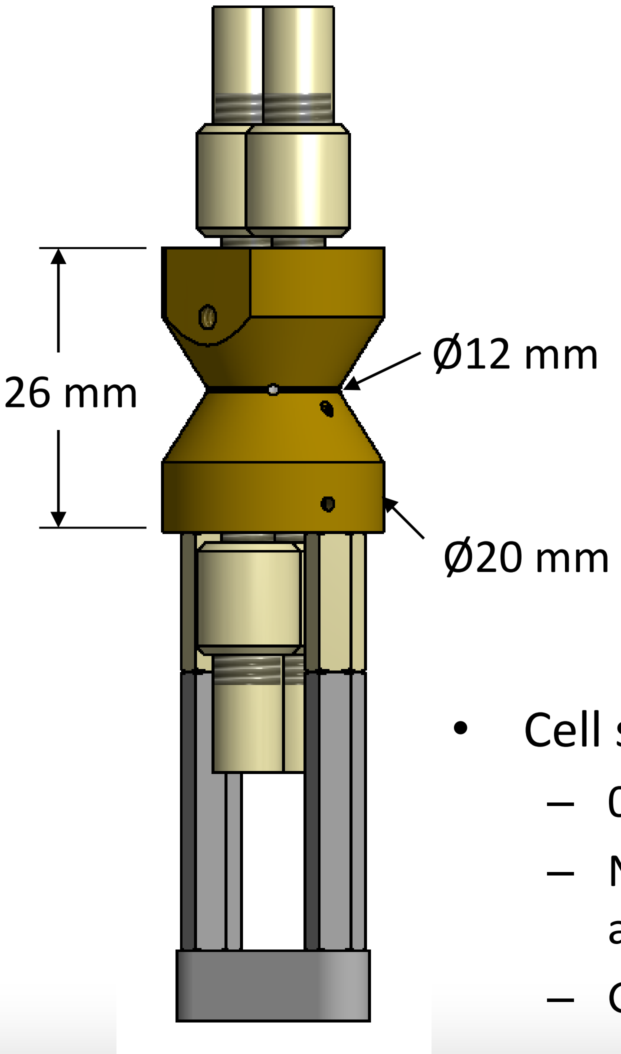
Several attempts to image the fossil with X-ray CT failed to provide sufficient contrast due to the high iron content of the matrix the fossil was located in. Neutron tomography provided excellent contrast and allowed full segmentation of skull fragments to perform anatomical and phylogenetic analysis of this fossil within the family of crocodyliforms.

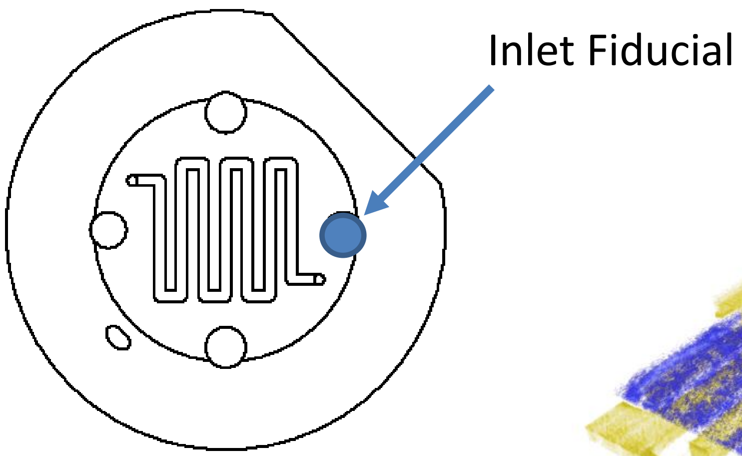



A,B, skull in right and left lateral views; C, mandibular symphasis in ventral view; D, detail of rostrum; E, dorsolateral view of right maxilla; F, right palatine in ventral view. Abbreviations: c, contact; l, left; r, right; aaof, accessory antorbital fossa; bem, buccal emargination of the dentary; ch, choana; d, dentary; dt, dentary tooth; ec, ectopterygoid; fr, frontal; l, lacrimal; mt, maxillary tooth; mx, maxilla; nf, nasal fragment; pal, palatine; pt, pterygoid; sa, surangular.

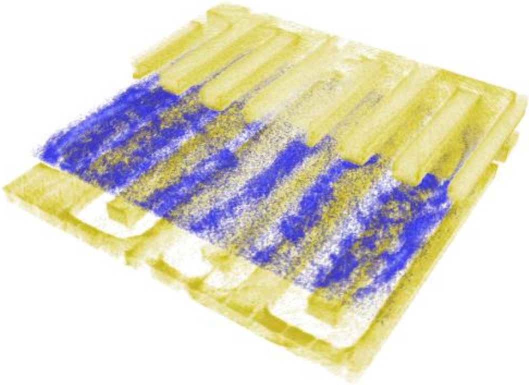
Current work in collaboration with Dr. Josef Stiegler (Stony Brook University) and James M. Clark (George Washington University). Presented at the Society of Vertebrate Paleontology meeting, Oct 2020.

# Update to cell design




Channels 0.5 mm (w) x 0.4 mm (d), 0.5 mm (w) lands



- Cell stats:
  - 0.36 $cm^2$ active area
  - No temperature control due to size and time constraints
  - Gold coated aluminum construction

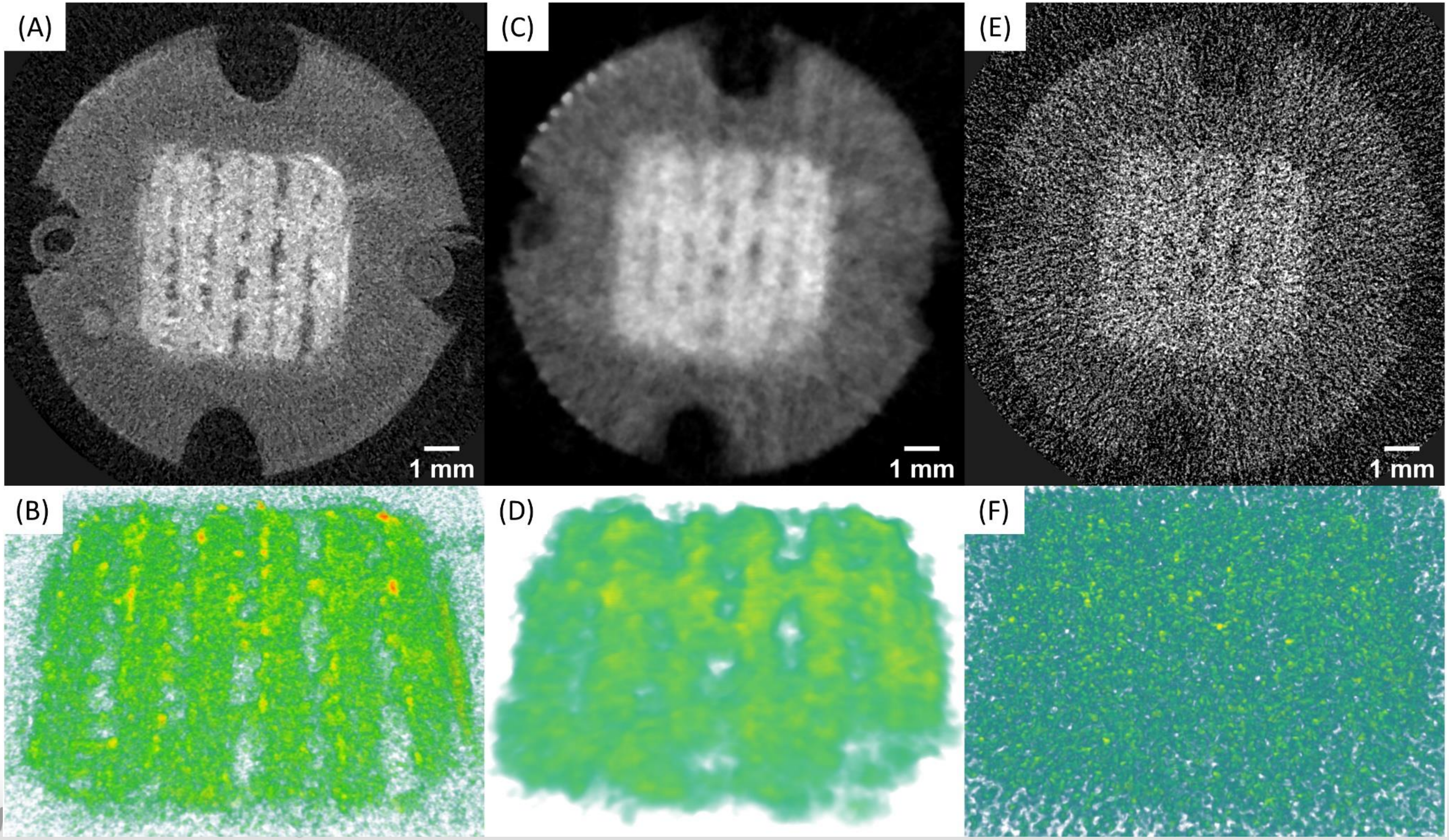
New clamping tube removes stainless steel screw artifacts and increases rigidity to remove motion artifacts. Inlet fiducial improves orientation identification in reconstruction.

# Current Dose Reduction Progress Using ASD-POCS

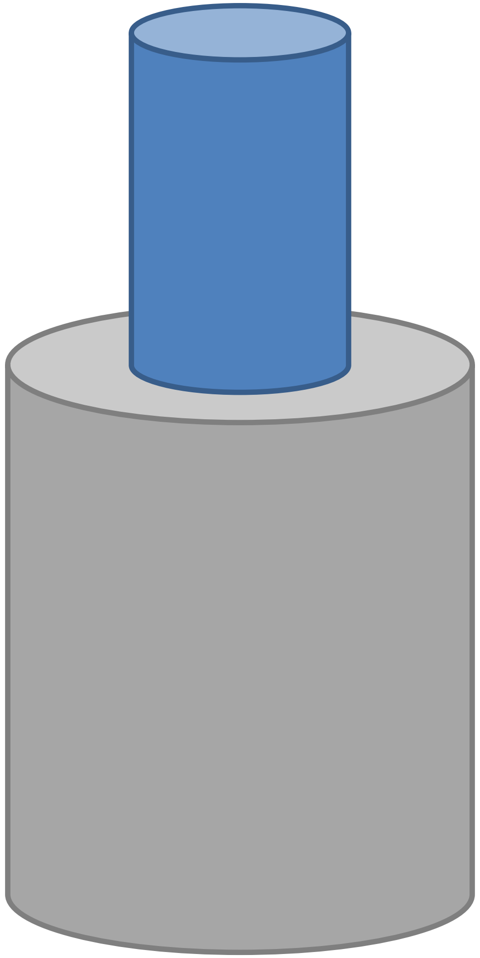
High Res FBP

Simulated 30 minute scan
ASD-POCS w/ seed

Simulated 30 minute scan
FBP



# Water Infiltration into Concrete [Collab: NCSU]

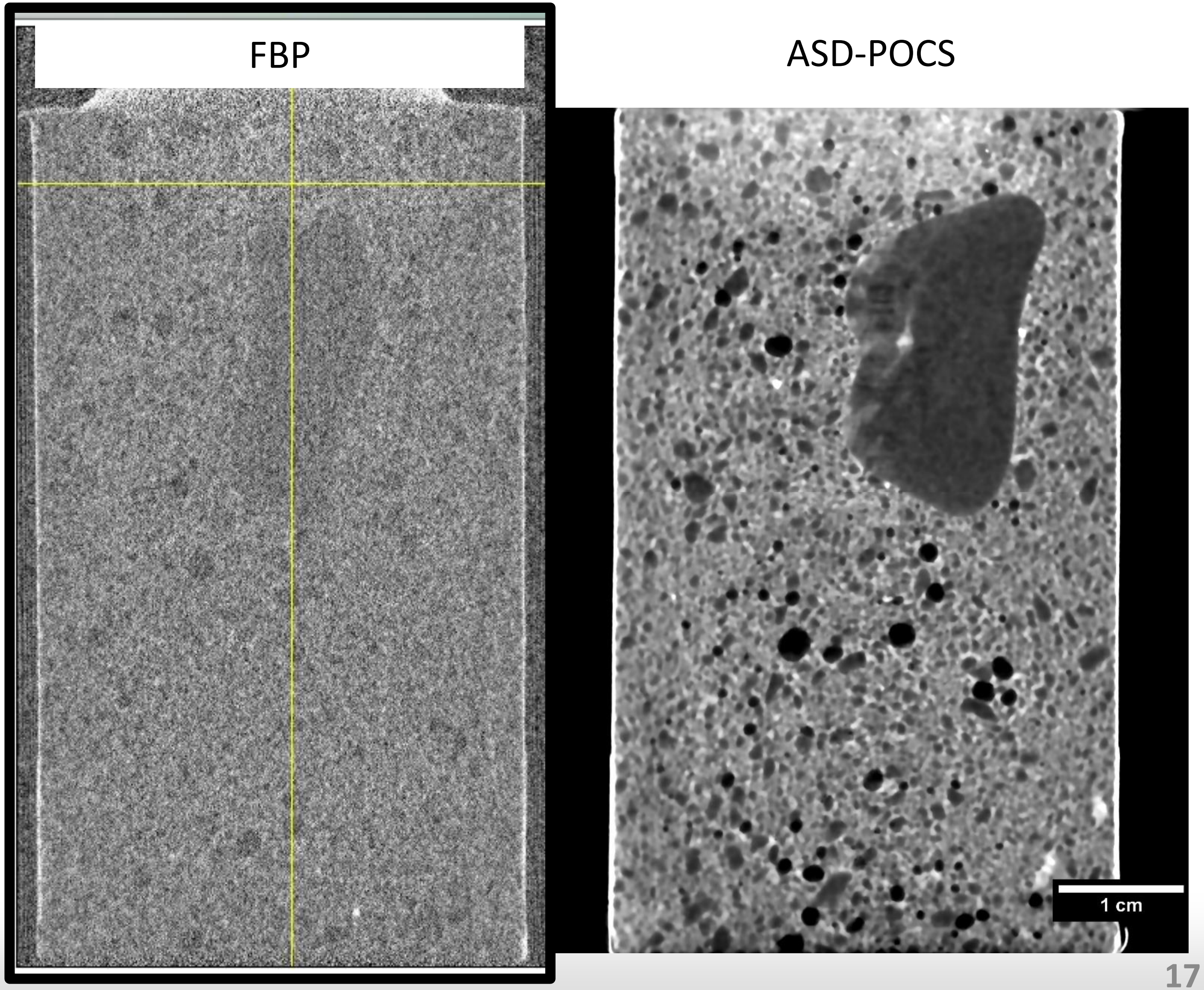


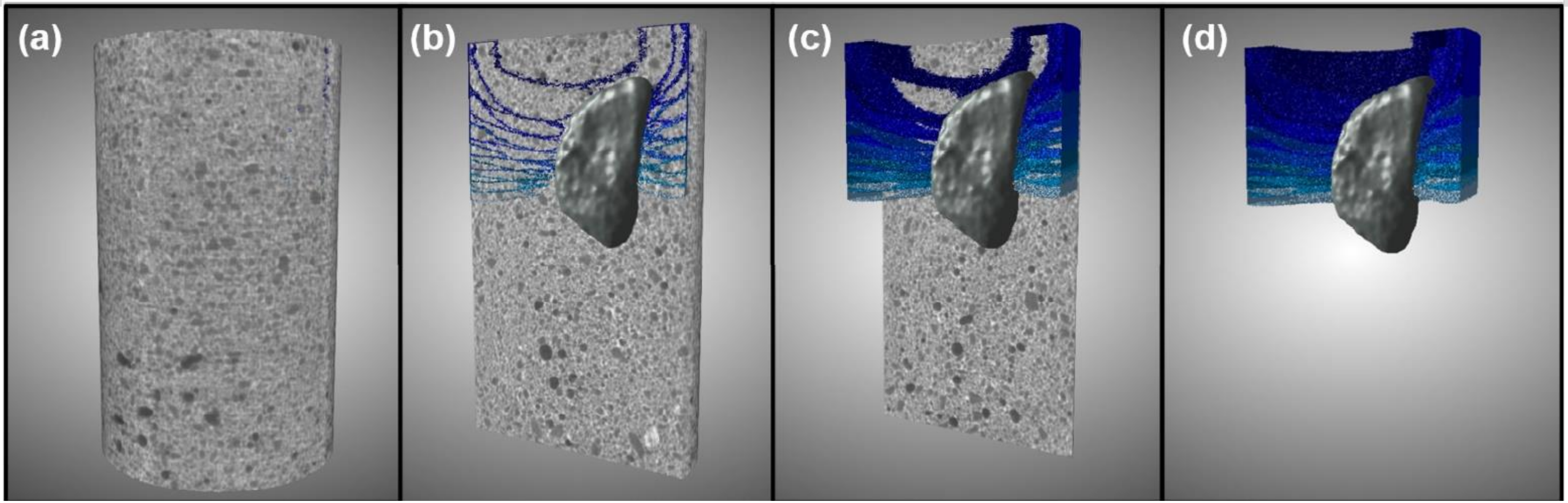
1.5” diameter core

Core made from cement with fine aggregates, one large aggregate placed near center

Column of water placed on top of core to provide reservoir for infiltration experiment

1 h tomography scans acquired with only 60 projections






Legend: (a) 38 mm diameter concrete cylinder, (b) cut-away view revealing large aggregate and contours of progressive water infiltration, (c) 3D water contours, (d) aggregate and water contours only

Water placed on top of 38 mm diameter concrete cylinder made with mostly fine aggregates except for one large aggregate. Sample scanned with NeXT every hour for 8 hours to track water infiltration with time to understand interfacial effects along cement/large aggregate interface.

Simultaneous tomography critical to capture the water infiltration and changes to the concrete. Cement will swell with increases in hydration which can cause deformation in the material. The swelling effect is the primary driving force in the slowing of the infiltration with time.

Current work in collaboration with Prof. Mohammad Pour-Ghaz and PhD student Laura Dalton in the Civil Engineering Department at the North Carolina State University

# Conclusions

- Neutrons provide a unique probe for investigating hydrogenous and/or lithiated systems
- The complementarity of neutrons and X-rays provide additional information for engineering and materials research
- Broad range of research topics can make use of neutron imaging techniques
- System has high demand in the user program

Outlook

- Upcoming improvements will improve our ability to image dynamic systems
- Upcoming improvements to the NeXT system will improve X-ray image quality and increase experiments NeXT can be used for
- The INFER project will unlock new measurement techniques for hierarchical materials

Thank you!

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