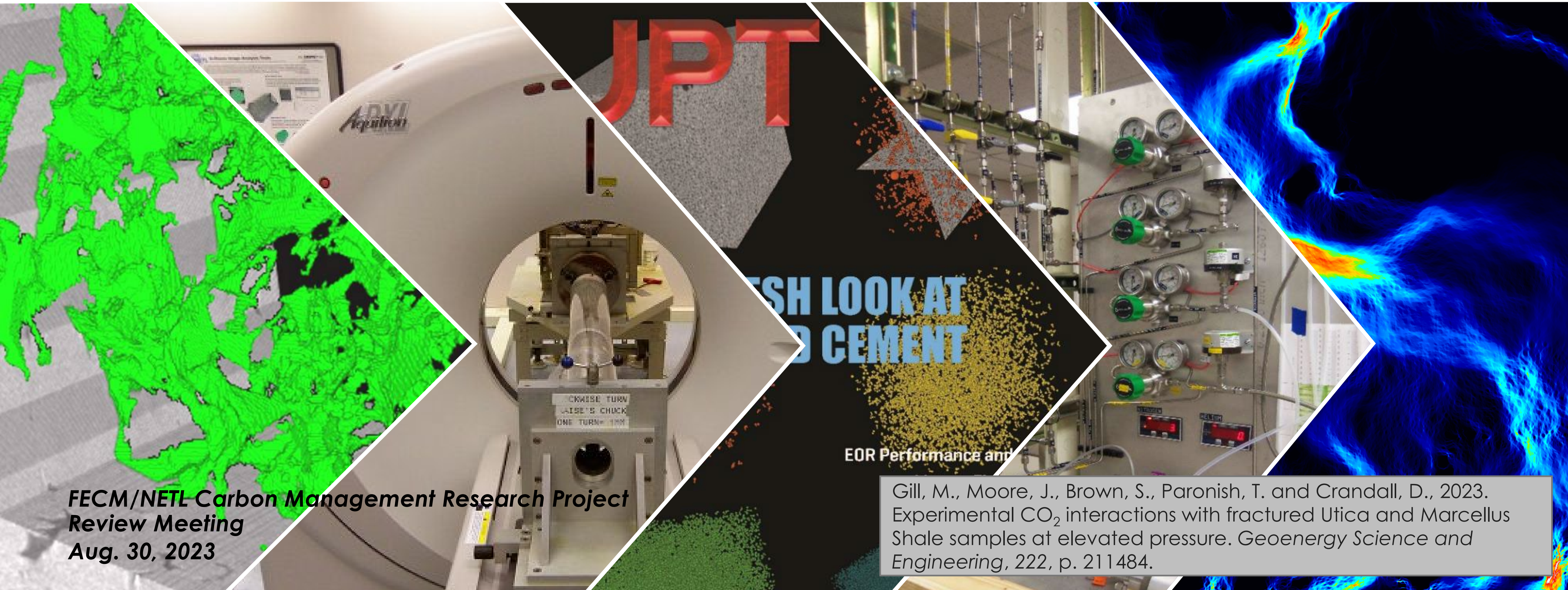
Experimental CO_2 Interactions with Fractured Calcite-Rich Shale Samples at Elevated Pressure

Magdalena Gill
NETL Support Contractor



FECM/NETL Carbon Management Research Project Review Meeting
Aug. 30, 2023

Gill, M., Moore, J., Brown, S., Paronish, T. and Crandall, D., 2023. Experimental CO_2 interactions with fractured Utica and Marcellus Shale samples at elevated pressure. *Geoenergy Science and Engineering*, 222, p. 211484.

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Authors and Contact Information

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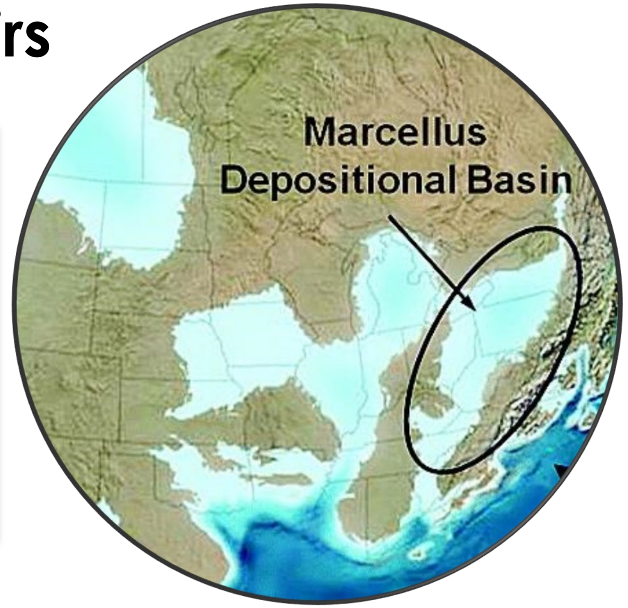
Problem Statement

- **Shales as seals**
  - Low permeability
  - Fractures are primary leakage sources
  - Shale deposition often marine
  - Carbonate content common

- **Shales as reservoirs**



- **What is the impact of carbonate content on fluid flow through shale?**

Experimental Design

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- Samples:
  - Marcellus and Utica Shale
  - 1 inch diameter, 2 inch length cores
  - Brazillian fracture
- Hassler style core holder
  - Temperature ~20 °C
  - Pore Pressure = 1,500 PSI
  - Confining Pressure = 1,700 PSI
- Brine injection over 14 days
  - 5% KI carbonated brine
  - 14 days of continuous exposure
  - Continuous flow 0.1 ml/min
  - Increased flow for permeability measurements
- Elemental composition - Olympus Innov-X® X-Ray Fluorescence Spectrometer (XRF)
- Elemental mapping - Thermo Scientific Scanning Electron Microscope (SEM) with Thermo Pathfinder Microanalysis
- North Star Industrial CT Scanner
  - CT Scan Resolution: 18.2 μm
  - Scaled by 50% for analysis - effective Resolution: 36.4 μm
- TESCAN DynaTOM Micro CT scanner for detailed views of areas of interest at 16.2 μm

CT Scanning at NETL's Geocharacterization Lab

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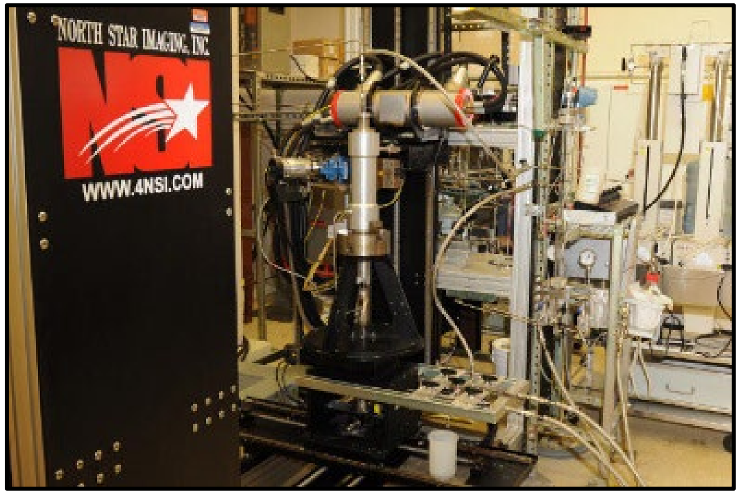
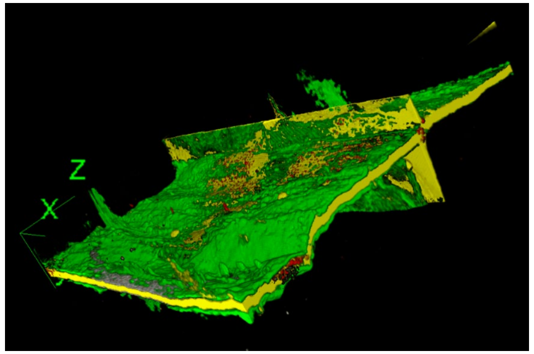
North Star Industrial CT Scanner:

- Workhorse CT
- Pore to core scale resolution range
- Scans at elevated temperature and pressure





TESCAN DynaTOM CT Scanner:

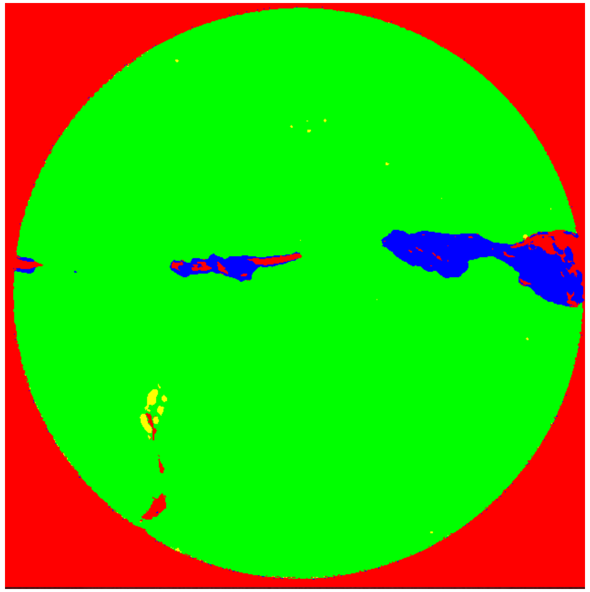
- Installed in 2021
- First of its kind in U.S.
- High-speed, high-resolution scanning
- Resolution ~10 microns

Image Processing

Image processing with **Fiji/ImageJ**
- Noise reduction, image scaling, cropping

Image segmentation performed with **ilastik**
- **Supervised machine learning**
- User defined labels
- Random forest classifier
- User-directed pre-filters and training

Darker: lower attenuation/less dense | **Brighter: higher attenuation/denser**



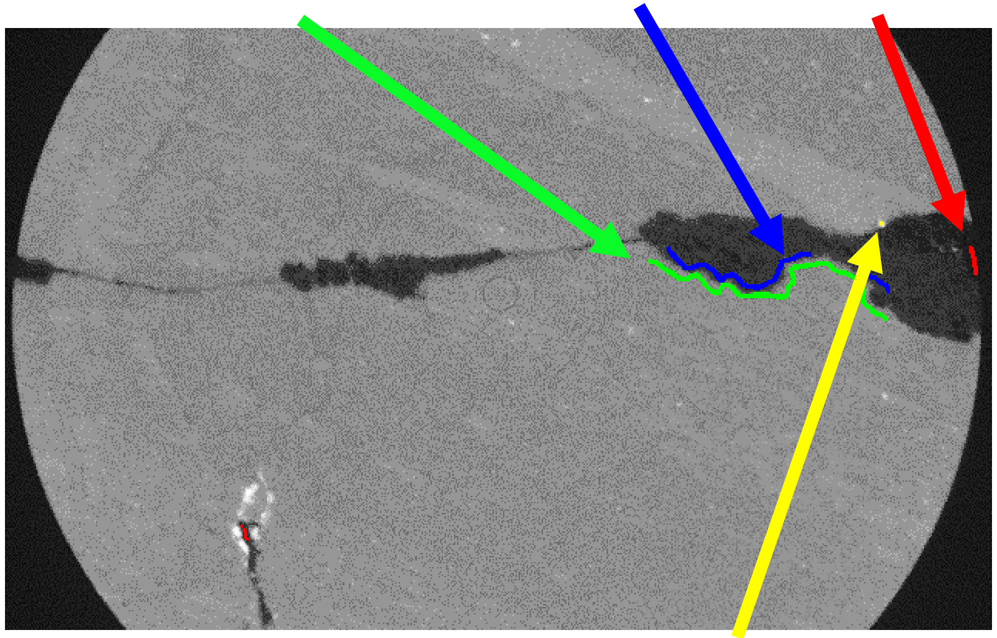
Open Fracture

Reacted Shale Matrix

Unreacted Shale Matrix

Calcite Crystal

Unreacted Matrix   Reacted Matrix   Open Fracture



Calcite crystal

Stuart S, Rudy, M., Eren, K., et al. (2019) *ilastik: interactive machine learning for (bio)image analysis*, in: Nature Methods.
Rueden, C. T.; Schindelin, J. & Hiner, M. C. et al. (2017), *ImageJ2: ImageJ for the next generation of scientific image data*, BMC Bioinformatics 18:529

A Tale of Two Shales

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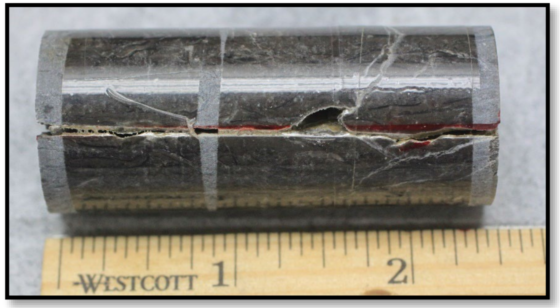
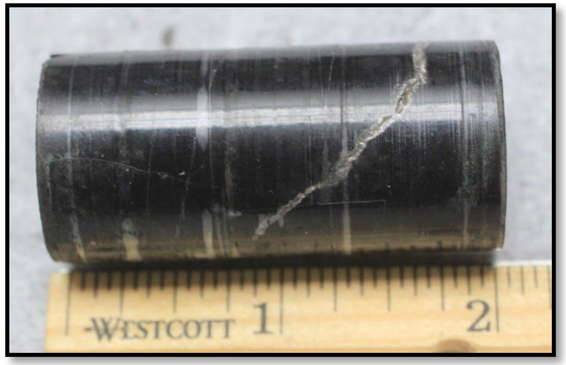
Utica Shale

- Herrick 3H Well
- Monroe, OH (39.6572°N, 80.9847°W)
- Depth: 10,577.6 ft
- Point Pleasant Member
- Fossiliferous, gray, calcareous shale
- Higher Ca content – more calcareous
- Lower Si content – less silty

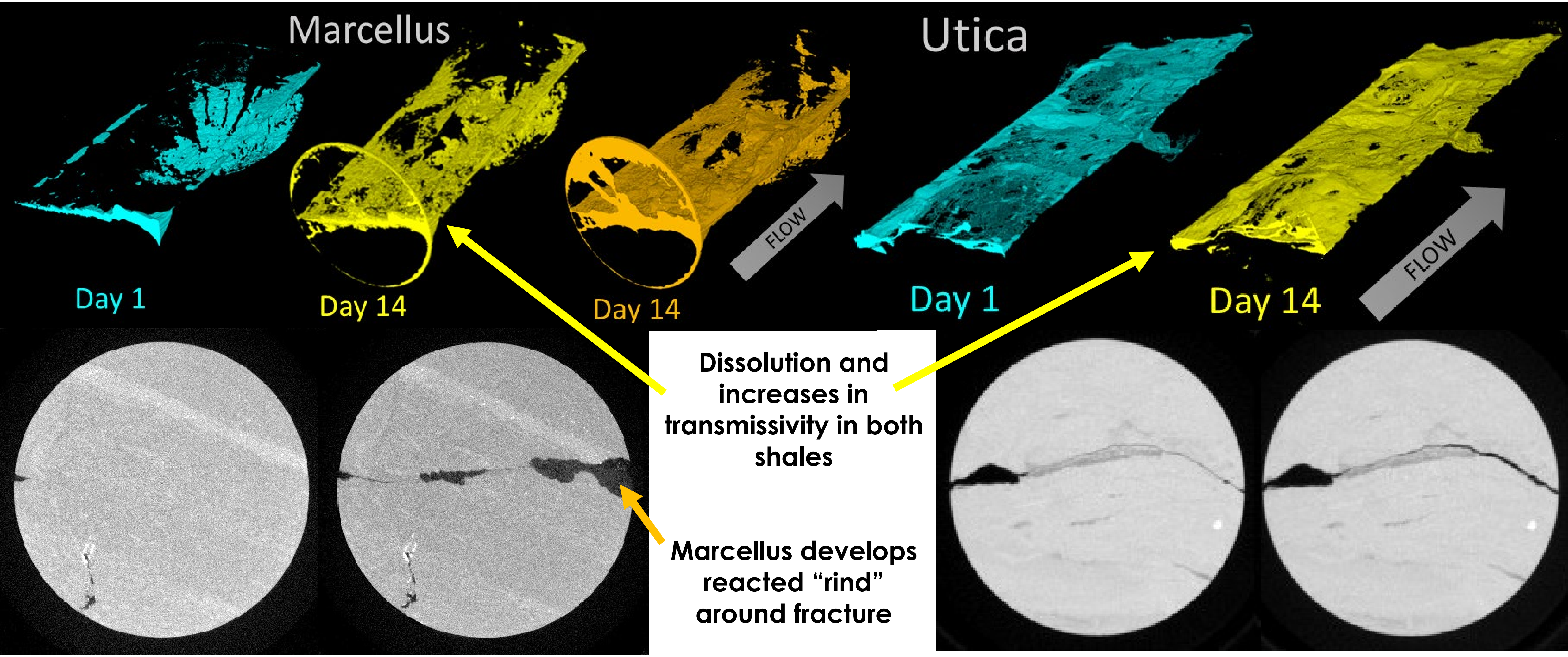


Marcellus Shale

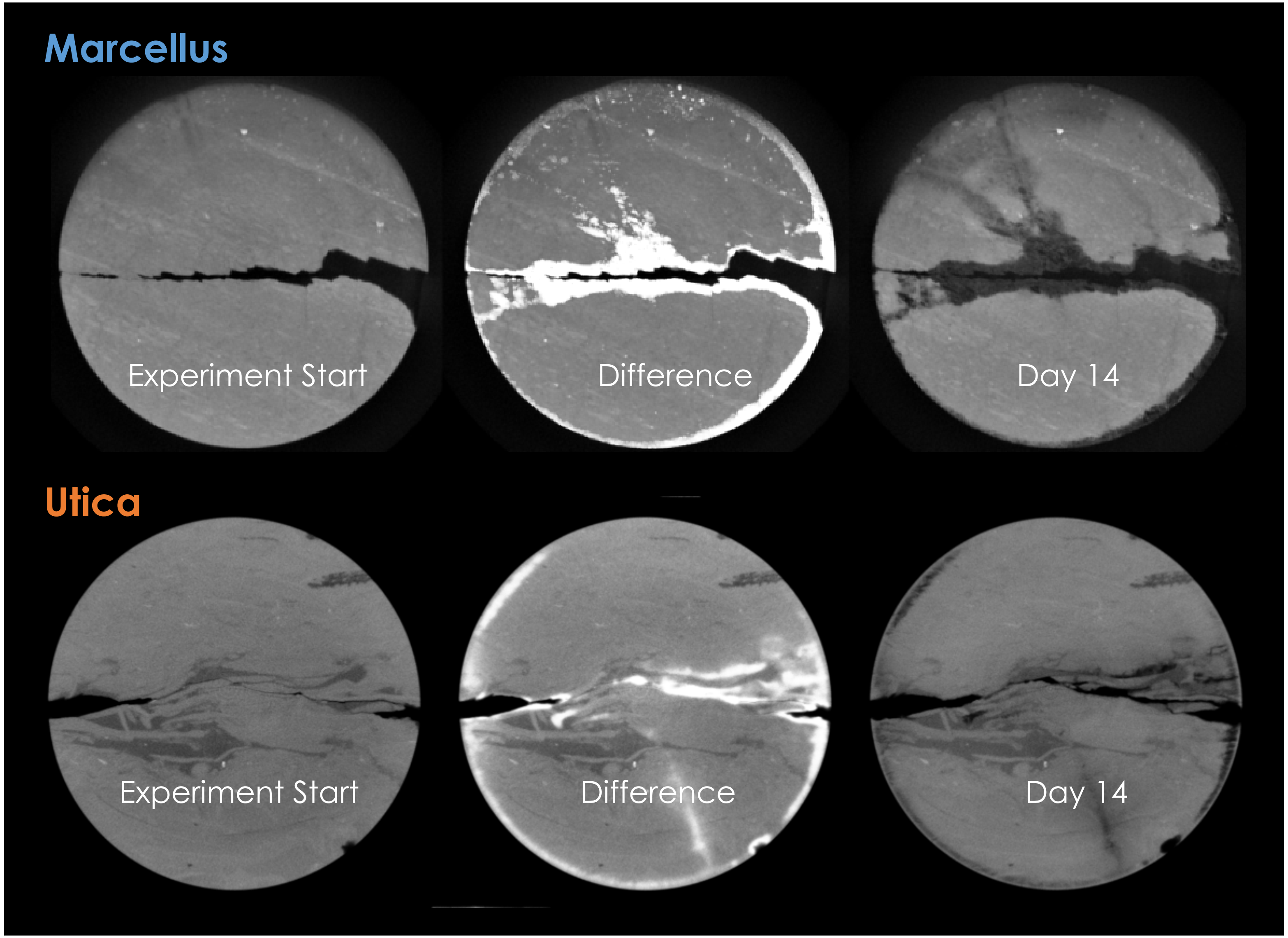
- Outcrop exposure (unweathered rock)
- Bedford County, PA (40.1382°N, 78.5837°W)
- Union Springs Member
- Organic-rich, black, calcareous shale
- Lower Ca content – less calcareous
- Higher Si content – more silty



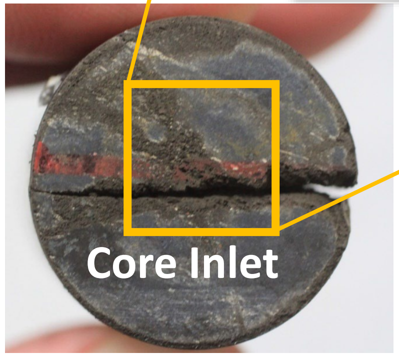
Findings: Different Dissolution Styles

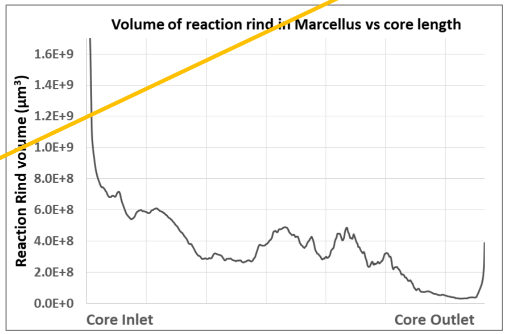


Matrix Dissolution – Change Highlighted in White

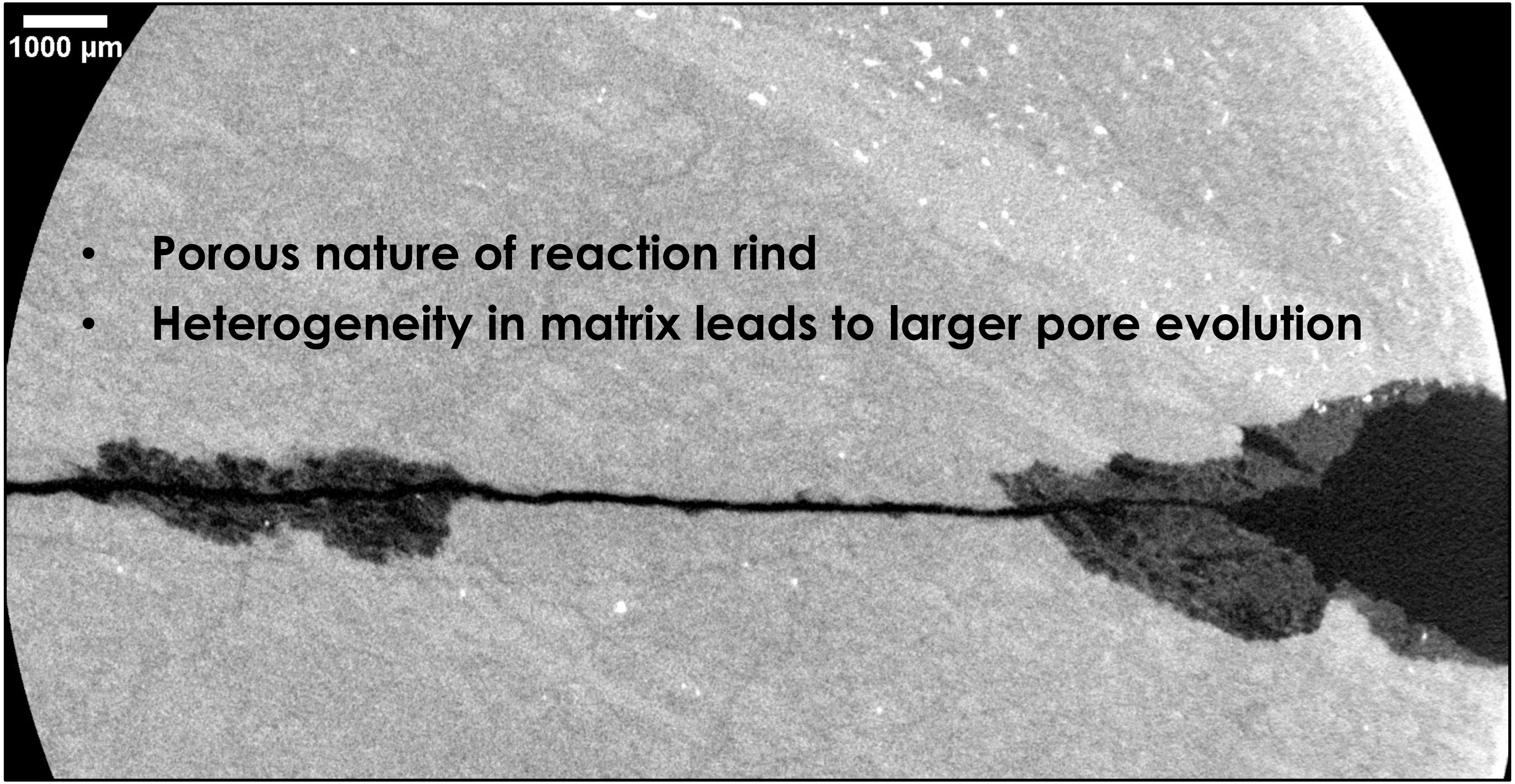


- **Utica: homogenous dissolution**
- **Marcellus: porous reaction rind**



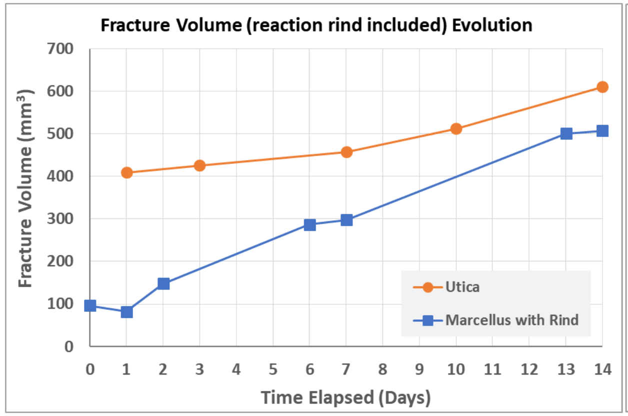


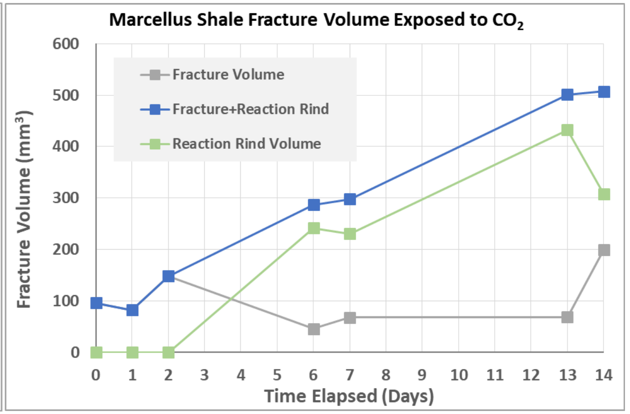
Marcellus - DynaTOM Micro CT

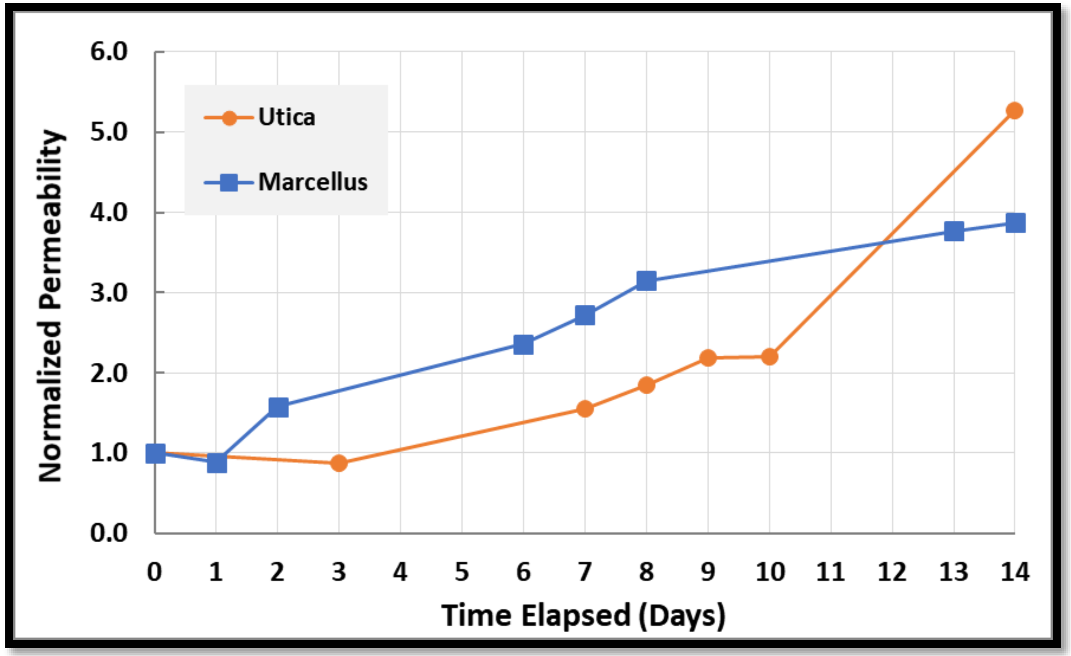


Permeability and Dissolution Style

- Permeability k calculated from differential pressure across core
- Inclusion of reacted zone in Marcellus correlates well with rise in k
- $k_{normalized} = k_{time}/k_0$







SEM, Elemental Mapping, and XRF

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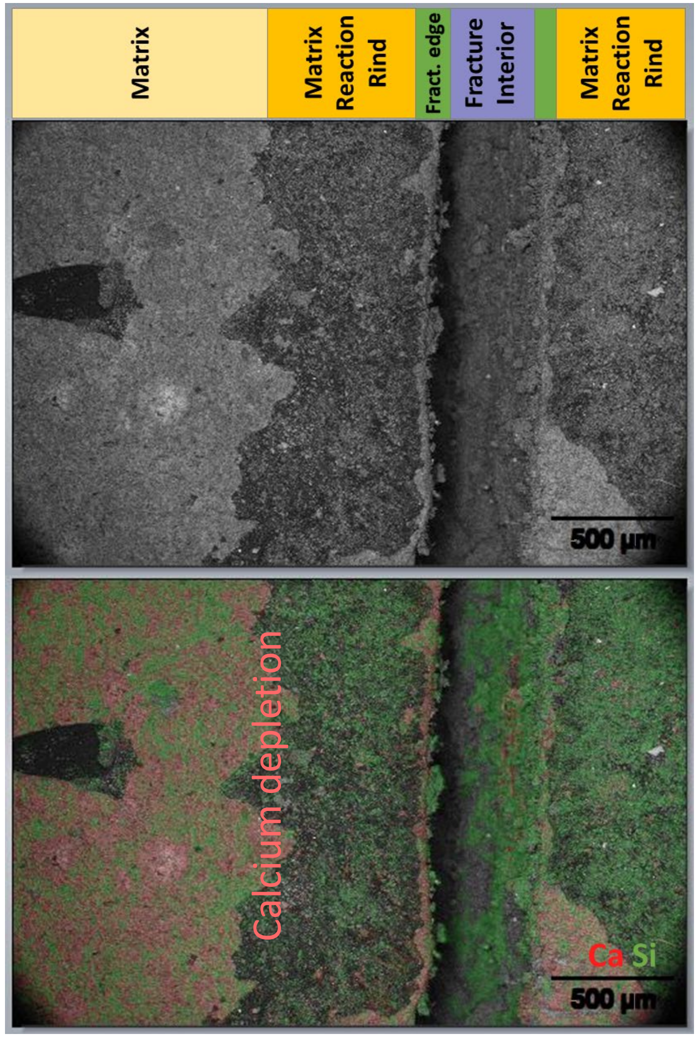
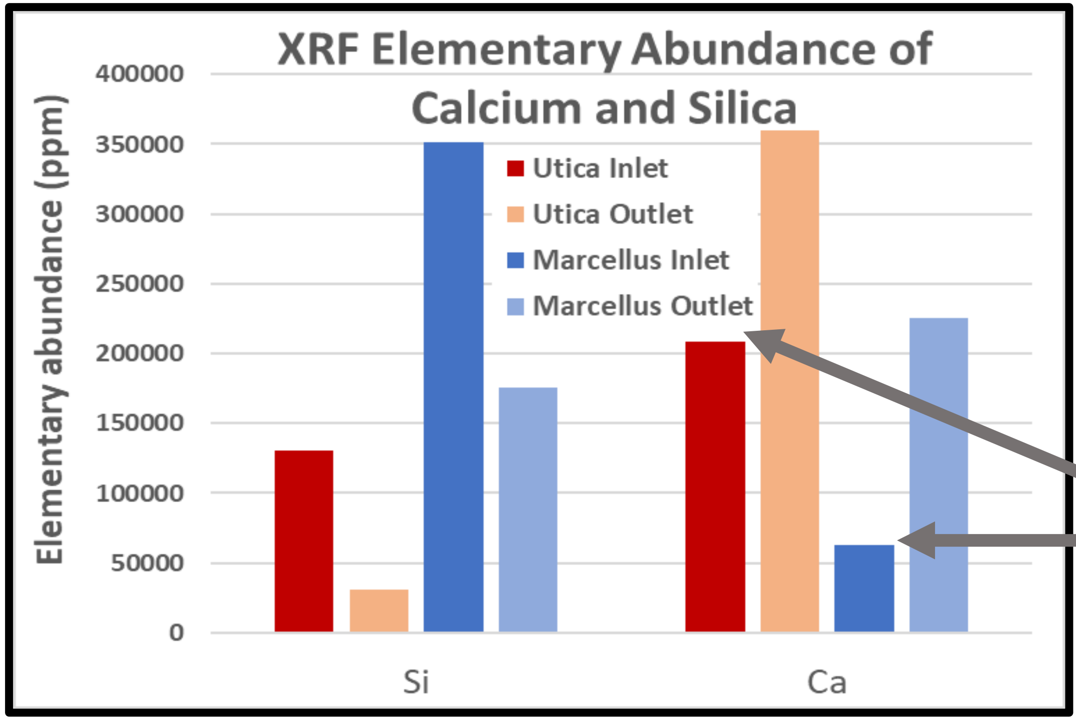
SEM and EDS of Marcellus Shale



- **Utica:** more calcium-rich (higher carbonate content; limey)
- **Marcellus:** more silica-rich (higher siliciclastic content; silty)

- **Ca depletion in reacted areas of both shales**
- **Si exposed wherever Ca dissolved away**



Ca depletion at core inlet in both shales

Conclusions

- Comparison of reactivity of:

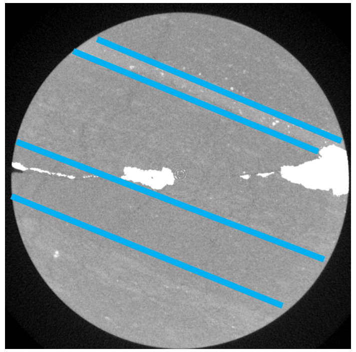
  Marcellus: Low Ca / High Si

  Utica: High Ca / Low Si

- Despite differences in dissolution, both show similar increases in fracture permeability
- A framework of silicious clastic grains facilitates creation of porous and permeable zones upon calcite dissolution
- Less calcareous shales may experience substantial Ca dissolution – leading to increases in permeability comparable to those found in more Ca-rich rocks
- Calcite distribution in shale matrix influences local dissolution rates
- Porous zone has lower mechanical strength: potential for future geomechanical work?

Find more information about this study in:

Gill, M., Moore, J., Brown, S., Paronish, T. and Crandall, D., 2023. Experimental CO2 interactions with fractured Utica and Marcellus Shale samples at elevated pressure. *Geoenergy Science and Engineering*, 222, p. 211484.



Thank You!

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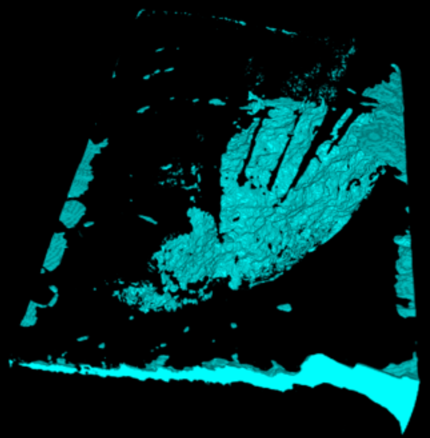
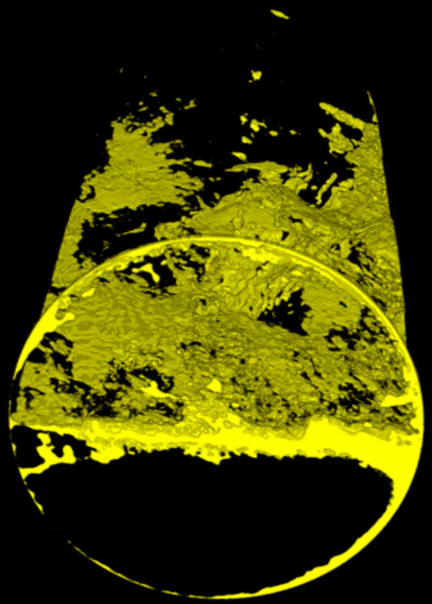
Questions?

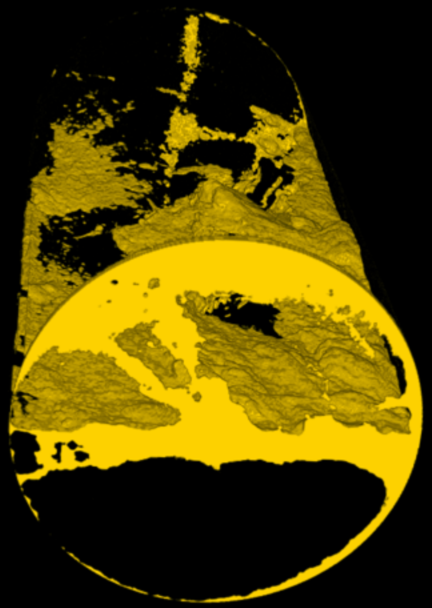
Marcellus Fracture at Experiment Start



Marcellus Fracture at Experiment End



Marcellus Fracture and Reacted Matrix at Experiment End



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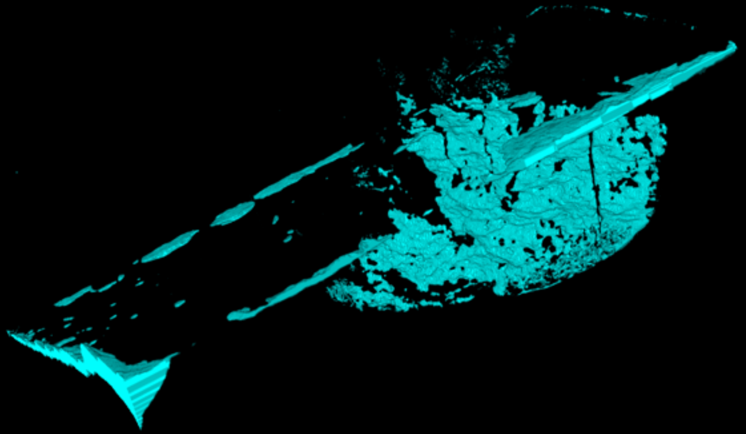

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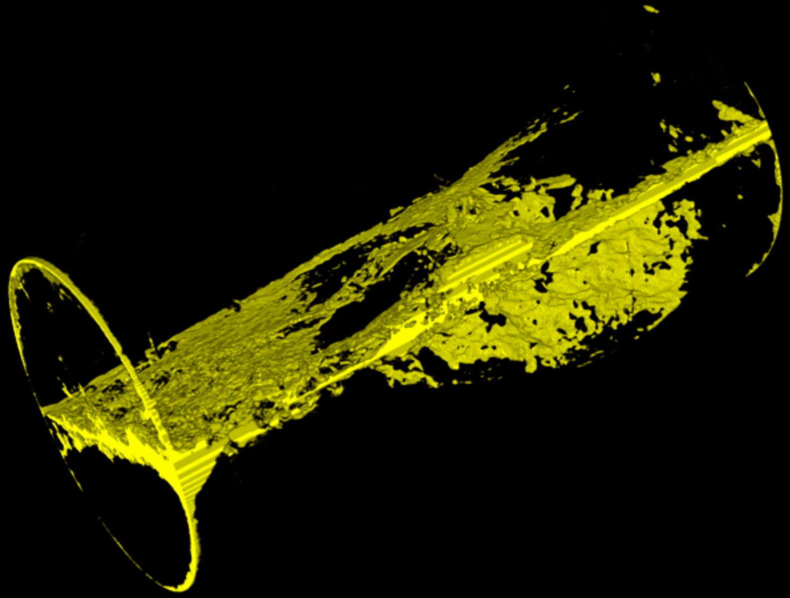

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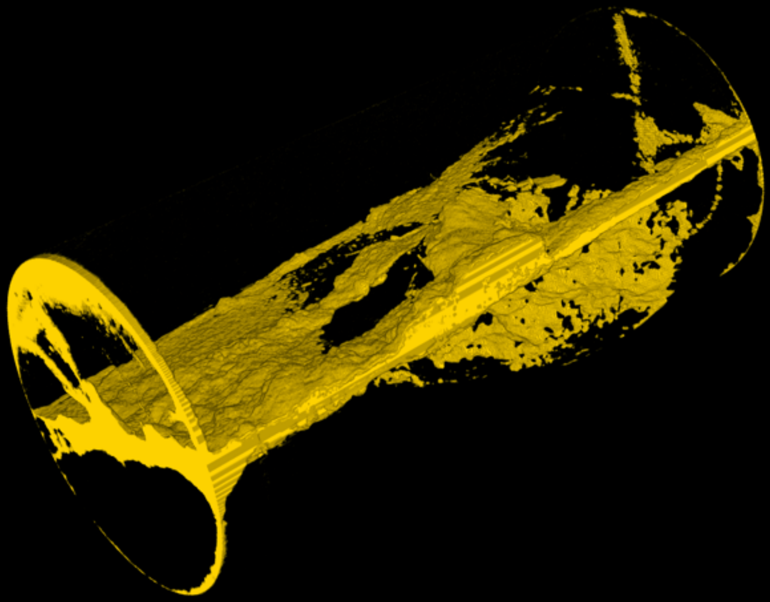

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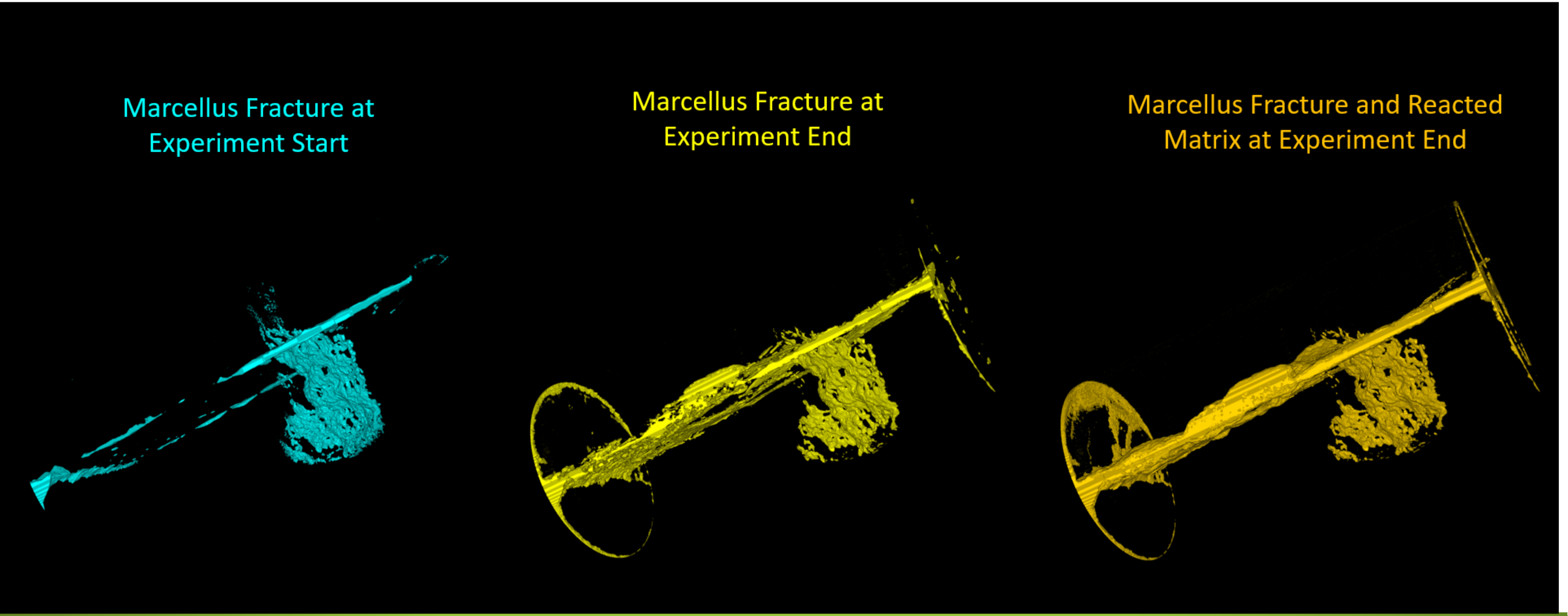

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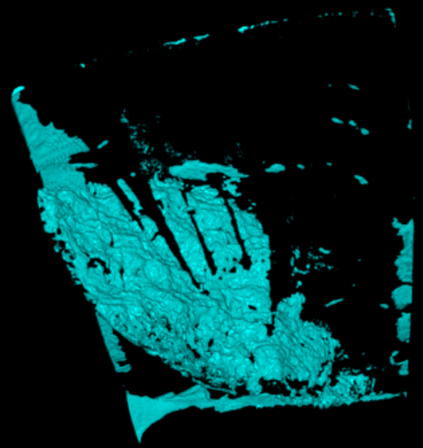

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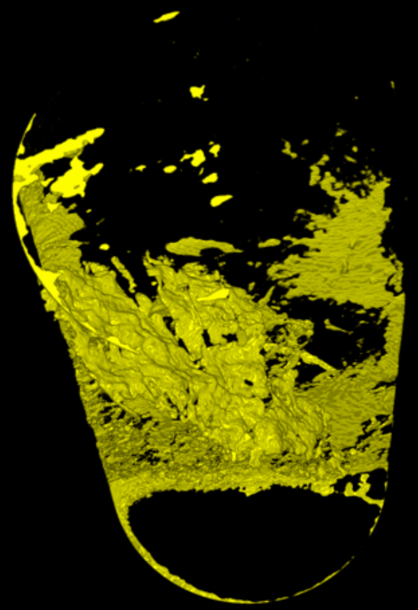

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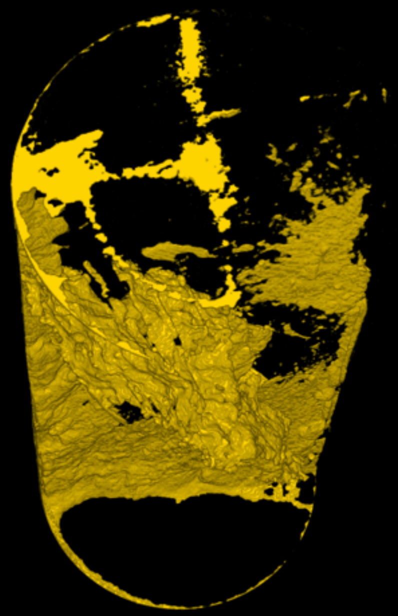

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