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Multiscale characterization of carbonate rock deformation due to dissolution, precipitation, and compaction during core flooding of reactive fluid

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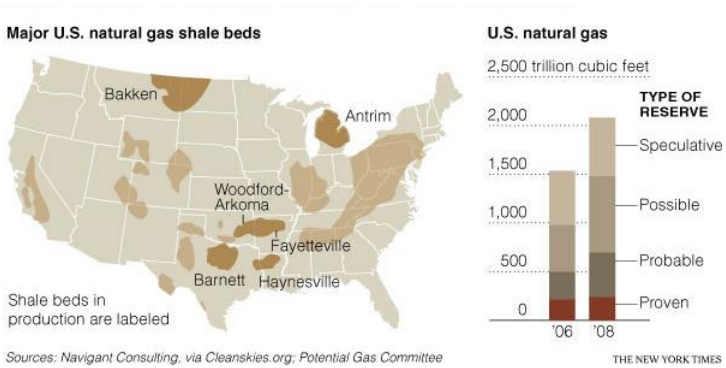
Outline

- Motivations
- Multiscale Characterization of Carbonate Rock - FIB-SEM approach
- Multiscale imaging results
- Summary

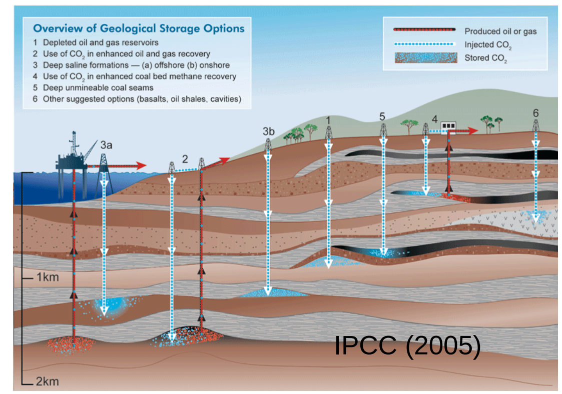
Why study nano-porous materials

- Plenty of pores at sub-micron scale
  - Recent subsurface energy activities highlight the significance of nanopores

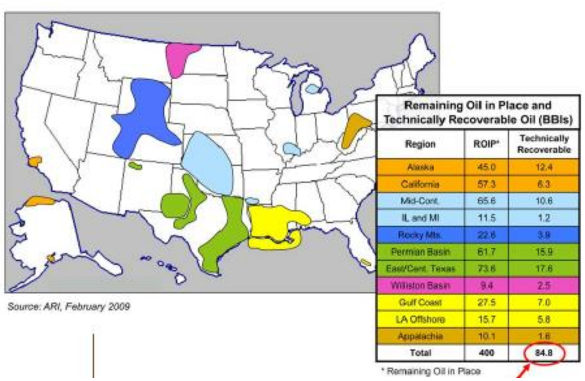
Reservoirs for unconventional resources and …



Caprock of subsurface CO_2 storage and …



…Enhanced oil recovery …



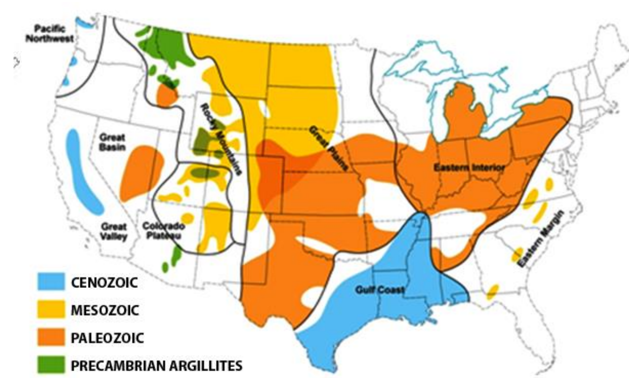
Remaining Oil in Place and Technically Recoverable Oil (BBls)

Region	ROIP*	Technically Recoverable
Alaska	45.0	12.4
California	57.3	6.3
Mid-Cont.	65.6	10.6
IL and MI	11.5	1.2
Rocky Mts.	22.6	3.9
Permian Basin	61.7	15.9
East/Cent. Texas	73.6	17.6
Williston Basin	9.4	2.5
Gulf Coast	27.5	7.0
LA Offshore	15.7	5.8
Appalachia	10.1	1.6
Total	400	84.8

* Remaining Oil in Place

Copied from NETL "small CO2 EOR Primer.pdf"

…and for geologic storage of nuclear waste

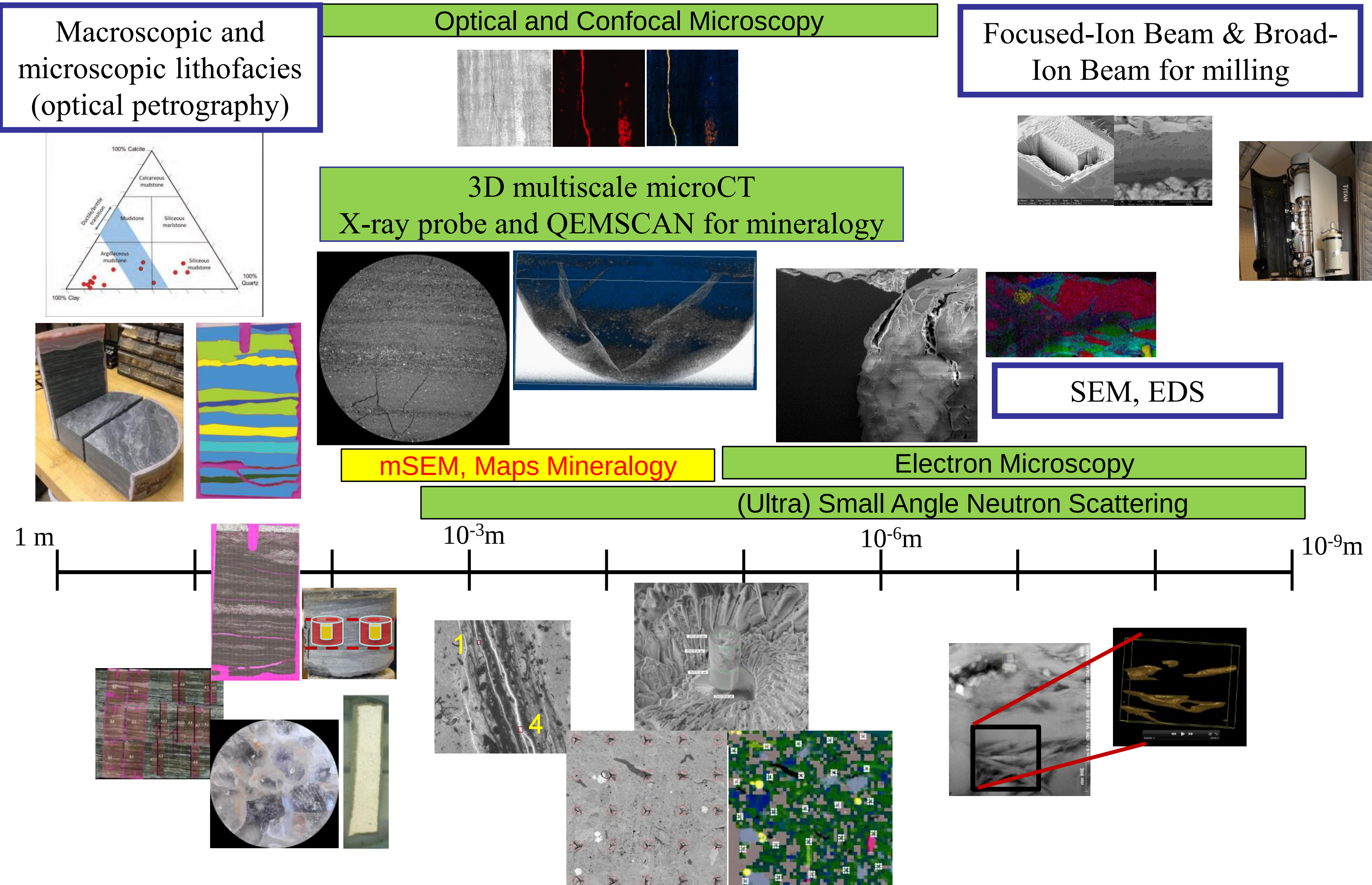


Gonzales and Johnson (1984)

More motivations…

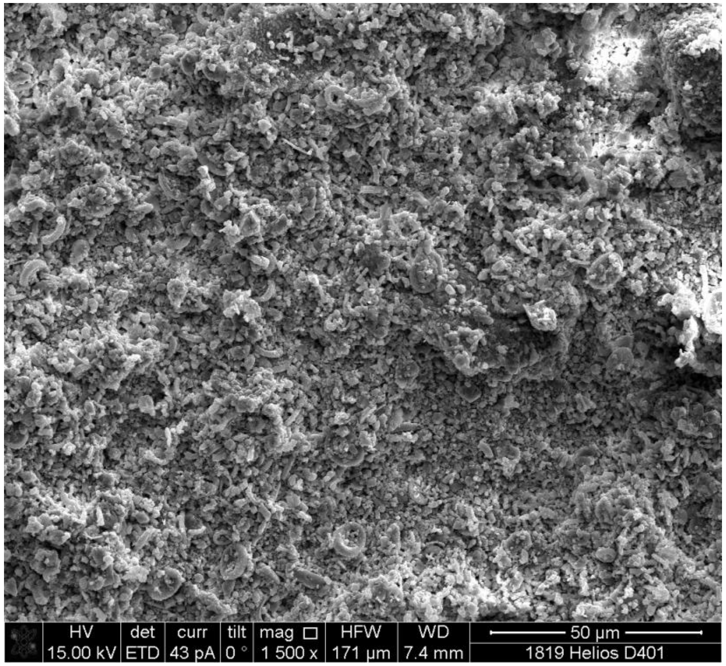
- **Plenty of pores at sub-micron scale** (nano-pores) in shales and carbonate rocks have become increasingly important for emerging problems such as unconventional gas and oil resources, geologic storage of CO_2 and nuclear waste disposal
- **Advances in analytical capabilities** with laser, X-ray, electron, and ion beams offer emerging tools for characterizing pore structures, mineralogy, and reactions at the sub-micron scale
- **Multiscale imaging capabilities –** integration of experimental and numerical tools to probe the structure and properties of materials across scales (e.g., core to nanometer scale) are rapidly advanced
- **Digital rock physics** – data interrogation about how to take nanometer scale information and apply it to the thin-section or larger scale for accurate prediction of coupled geophysical, mechanical, and chemical processes

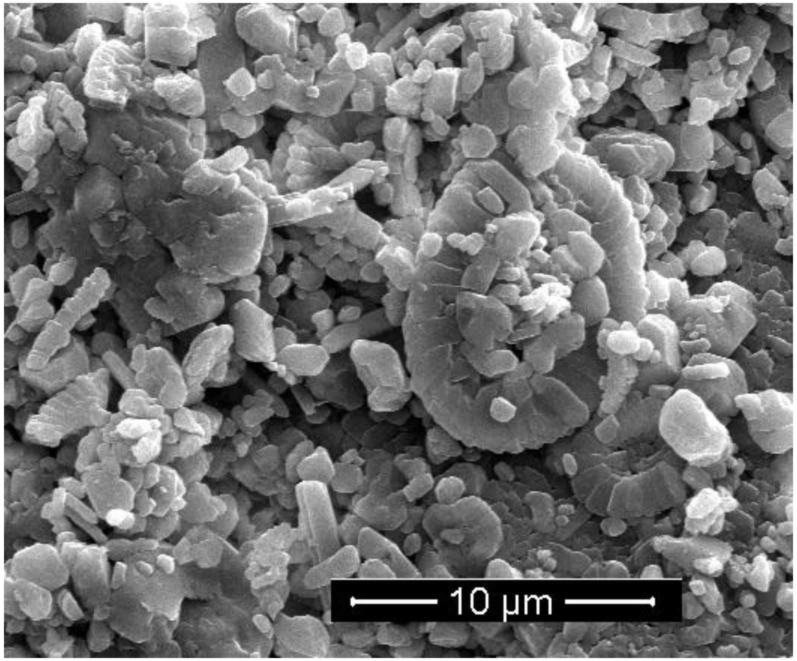
Multiscale characterization of physical, chemical, and mechanical heterogeneity of nano-porous geomaterials



Liège chalk

- Cretaceous Liège chalk (Belgium): Outcrop sample as a surrogate for reservoir rocks in the North Sea
- Clear signs of recrystallization, contact cements, and particle interlocking (Hjuler and Fabricius, JPSE 2009) but well-preserved coccolithophores
- ~95 wt% calcite with clays, quartz and mica
- Long-term chemical flooding testing in a tri-axial system (Nermoen et al., JGR2015; Zimmermann et al., 2015 AAPG Bull)

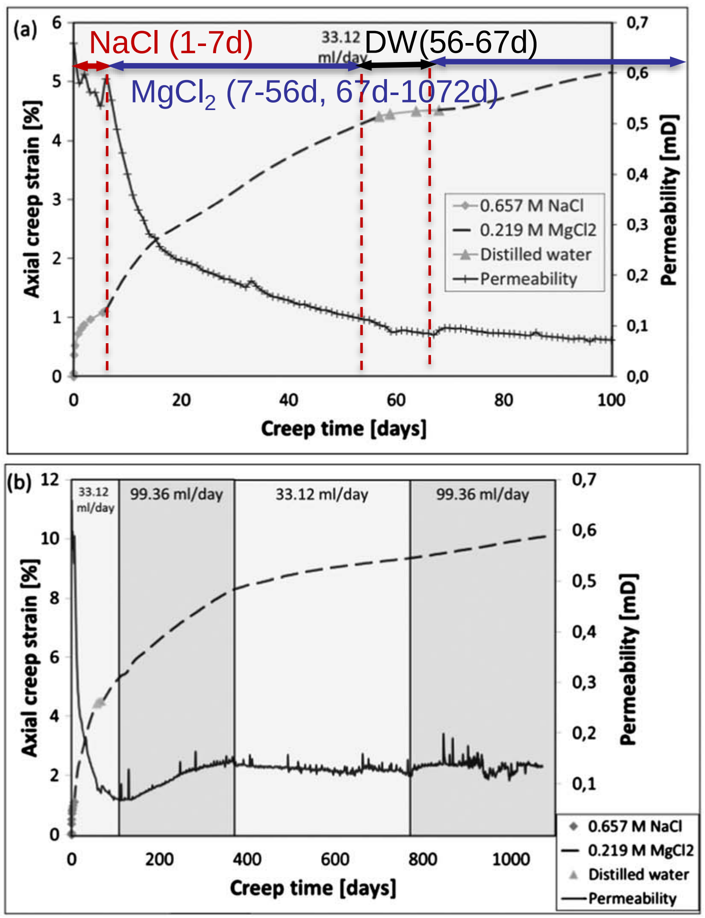




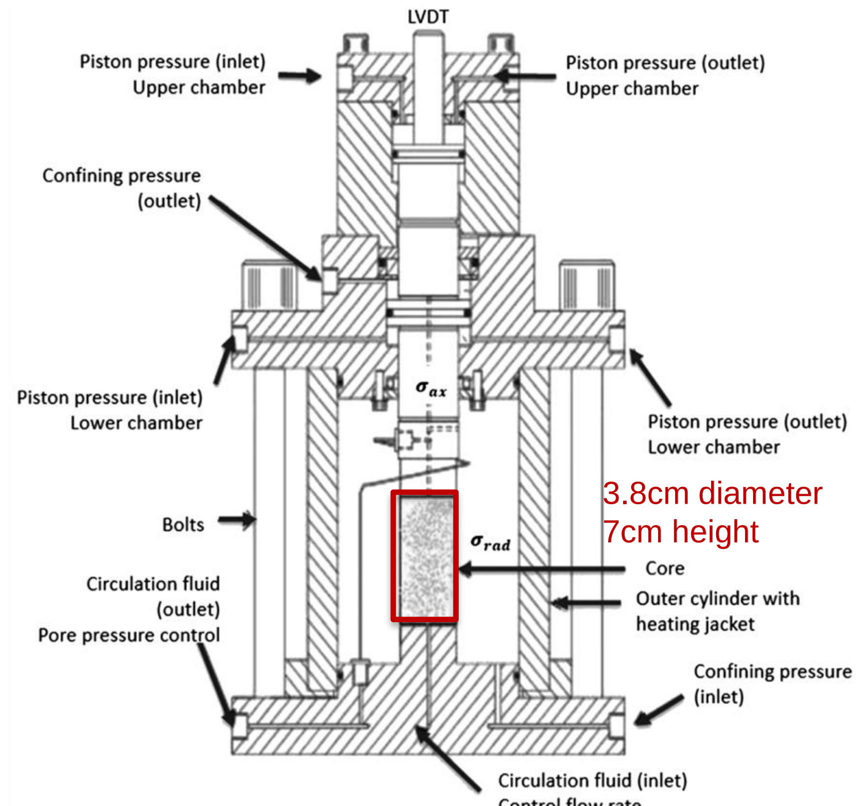
Chemical flooding in the tri-axial cell

Nermoen et al., JGR2015

- $MgCl_2$ (0.22M) flooding following NaCl (0.66M) accelerates axial creep rate
- Permeability initially decreases, rebounded, and then reaches the plateau

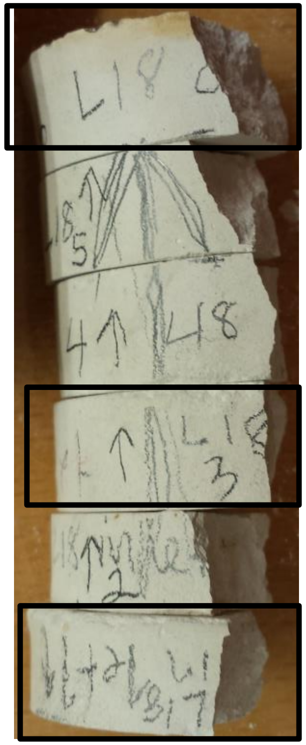


Experimental Setup



Nermoen et al., JGR2015

Multiscale FIB-SEM Analysis

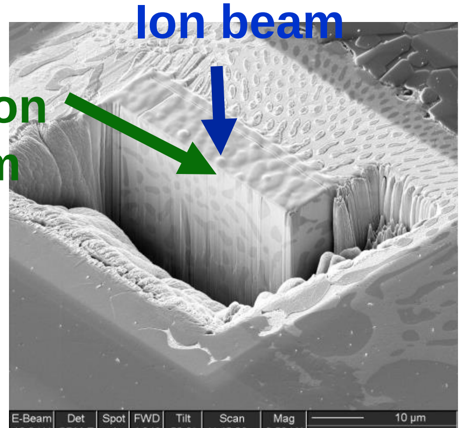


- FIB-SEM analysis of unaltered and altered samples (1000 image slices at 10 nm resolution)
- Energy dispersive X-ray spectroscopy (EDS) mapping of flooded area and 3D EDS mapping
- Plasma FIB over 1-2mm scale at 1 µm resolution

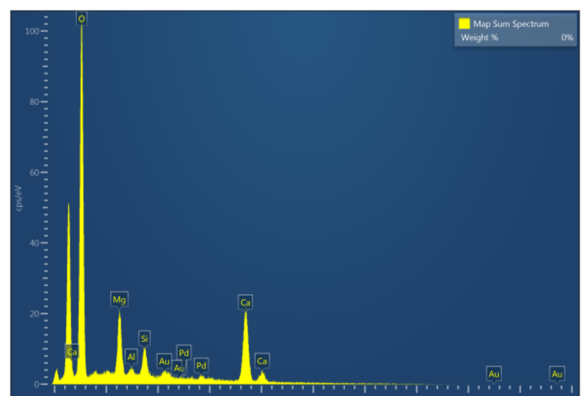
512 days sample

Nermoen et al., JGR2015

Electron beam

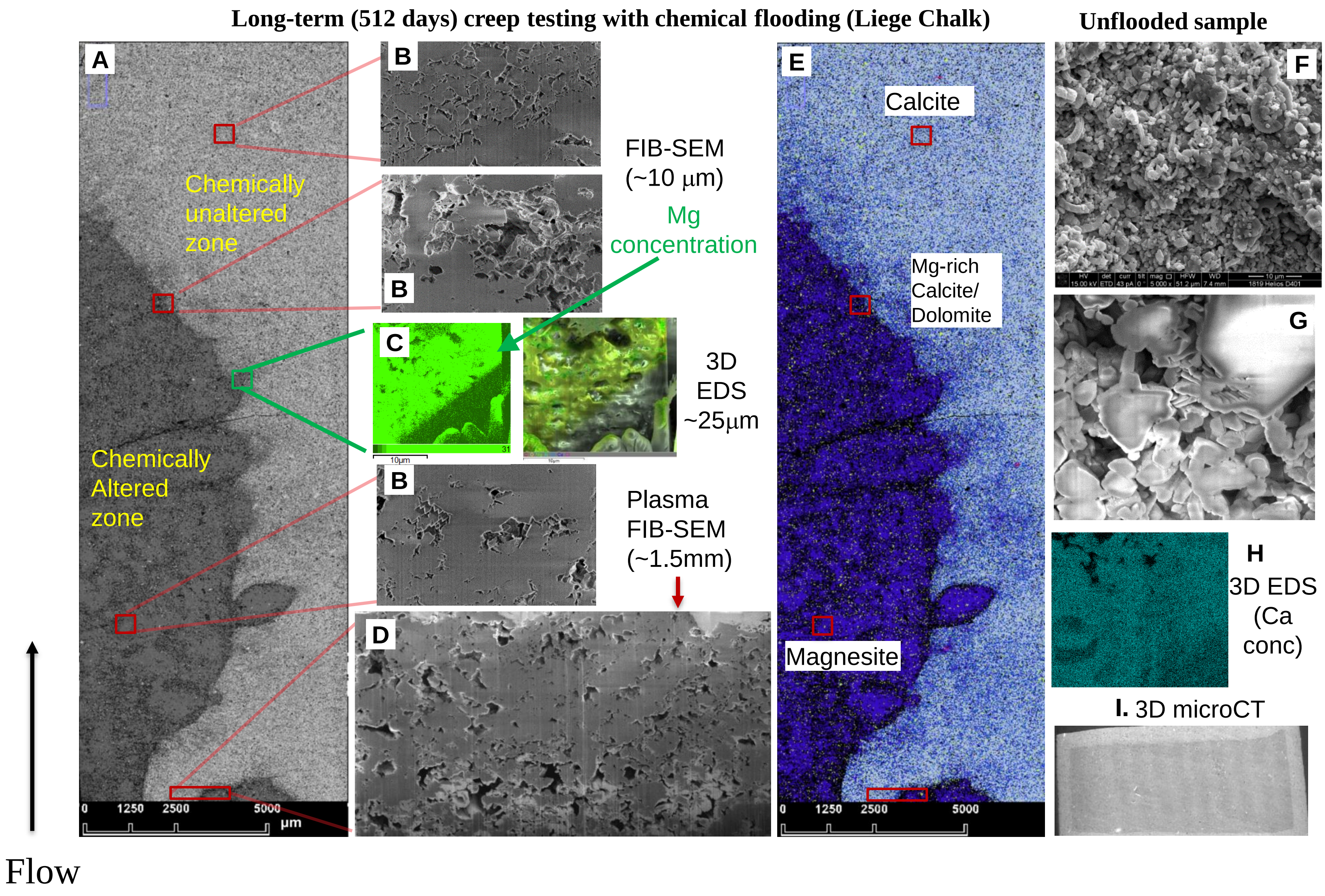


FIB-SEM



EDS analysis
(25 nm resolution)

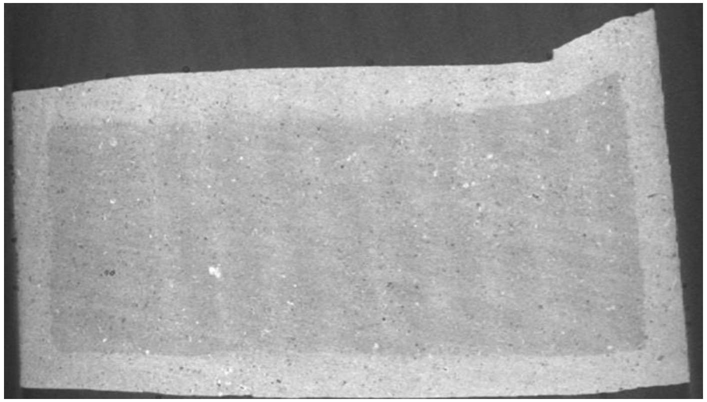
Multiscale Imaging Results



Imaging examples

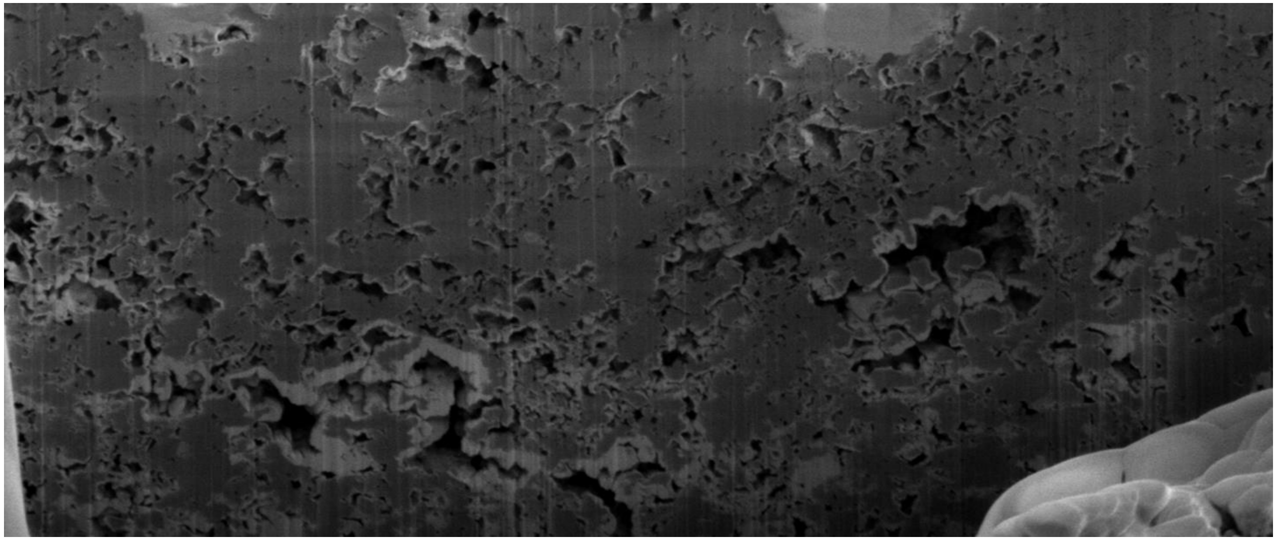
MicroCT image (Unaltered sample)

- ~2 cm x 0.8 cm @ 16 µm res.
- Porosity = ~ 40% and volume rendering image (right)

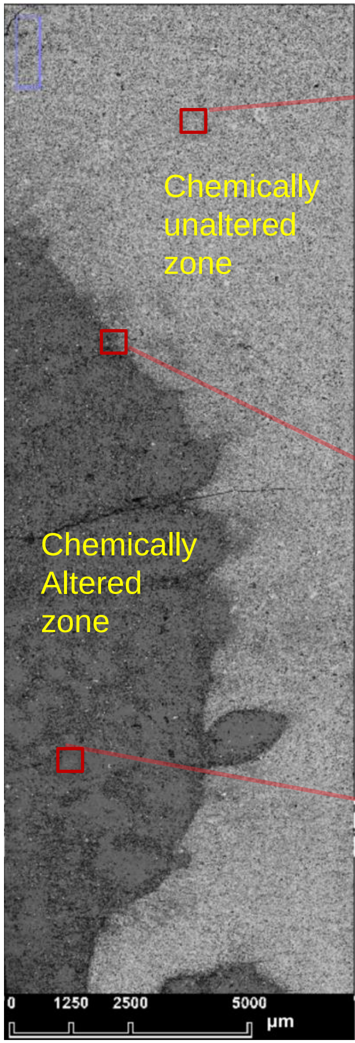


Plasma FIB-SEM

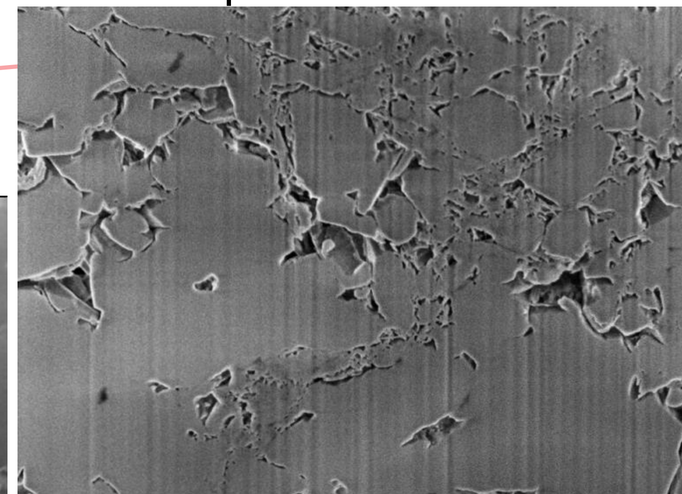
- Altered sample
- Wide-cut (2 mm wide)
- 2.1 mm x 0.89 mm @ 1µm res.
- 299 image slices with 100 nm thickness

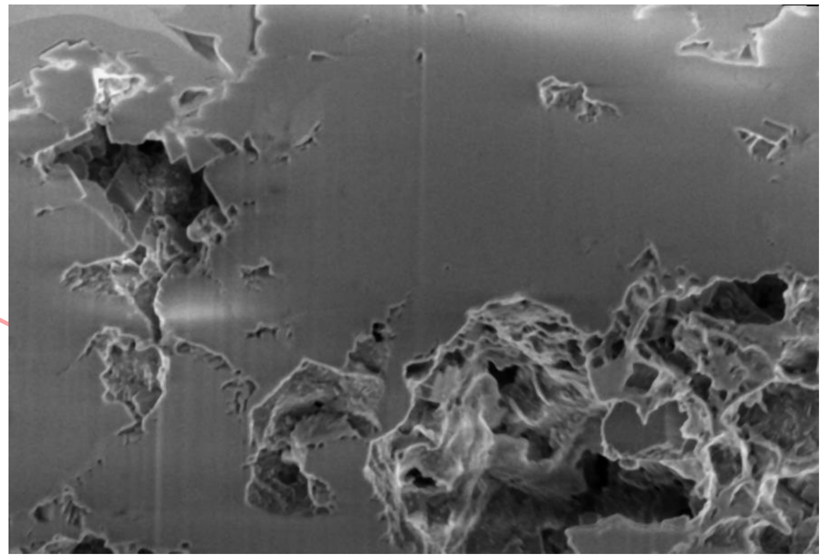


FIB-SEM images



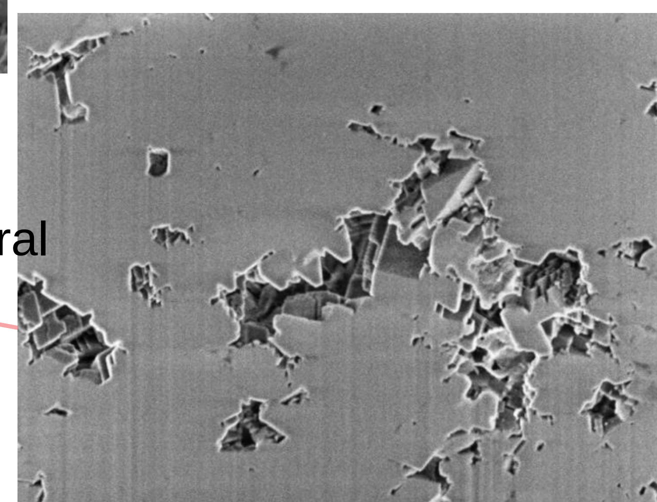
Compaction dominant





Transition (width=~8μm, 10 nm res.)

Rhombohedral



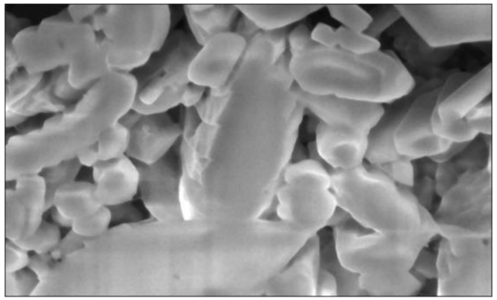
Dissolution, precipitation, and compaction

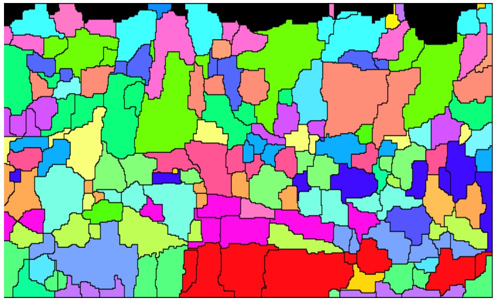
Morphological Watershed Segmentation

- Each image in 3-D image stack shows both foreground and background through pores
  - Typical filter sets fail to distinguish solid from pores
  - Watershed segmentation is promising, but often results in over-segmentation
  - Morphological segmentation through smooth filters improve segmentation
   (Morphological segmentation implemented in Fiji)

Key parameters

- Radius (r), tolerance (t), and connectivity (c) are the parameters for this segmentation.
- The radius value is used in creating the gradient of the image.
- Tolerance: the intensity for the search of the regional minima. Increasing tolerance decreases the number of segments.
- Connectivity: voxel connectivity (6,26). 6 produces more rounded segments.
- Main problem of watershed segmentation is over segmentation due to the presence of false minima.





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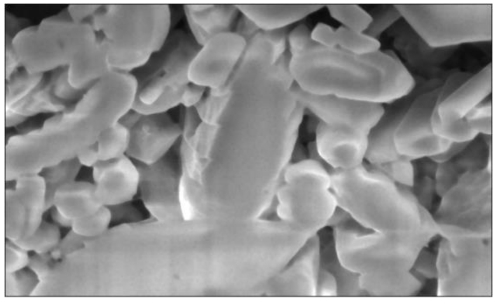
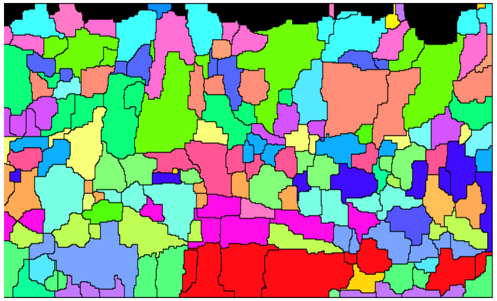
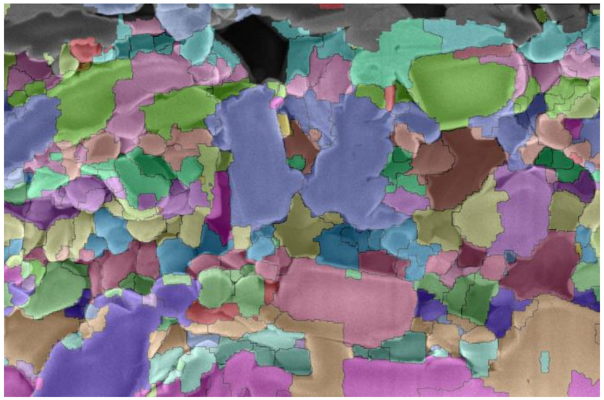


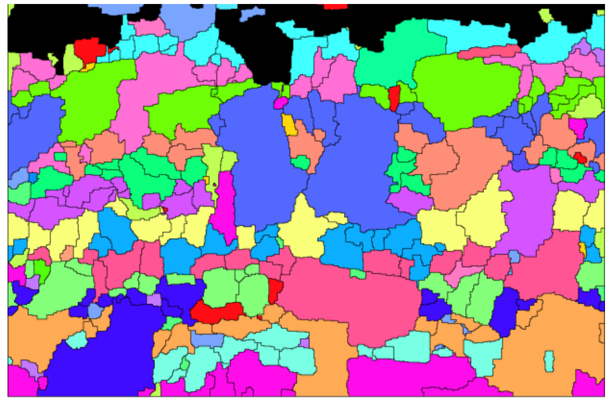


Morphological Watershed Segmentation

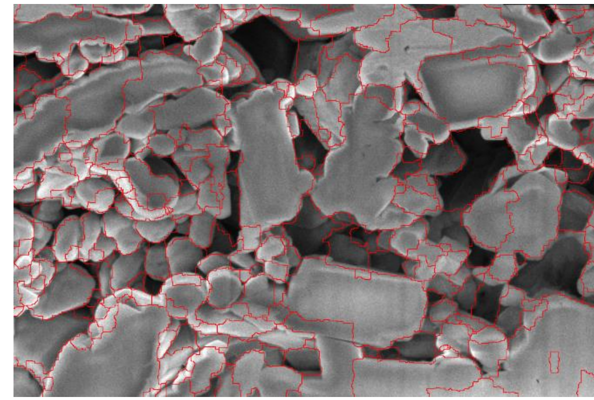
Parameters: r=7; t=12; c=26;



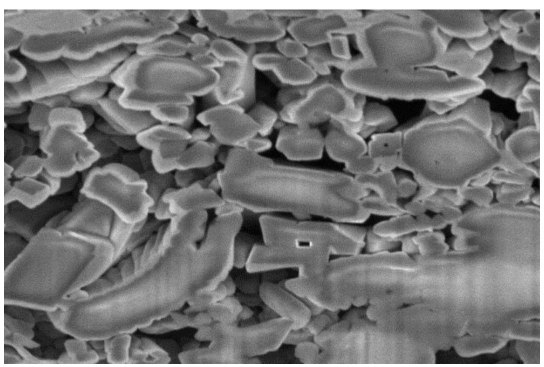
Overlaid Basins



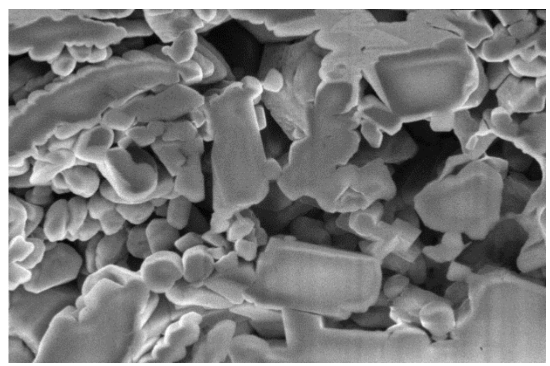
Catchment Basins



Overlaid Dams



Original Image
Dimensions:
1024x884x1000 pixels

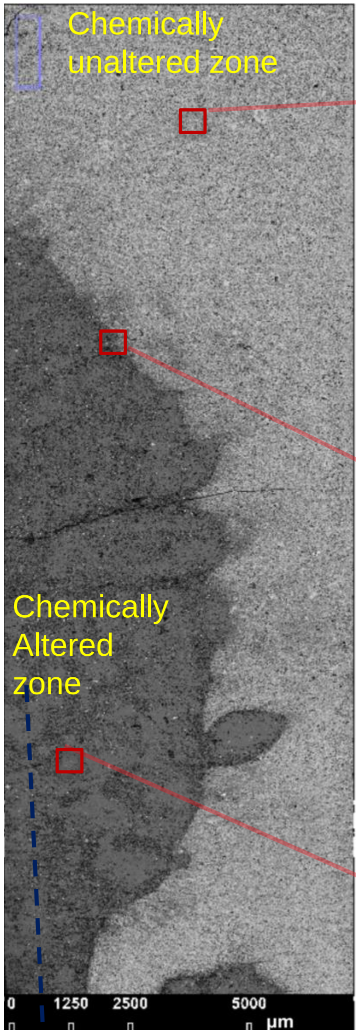


After alignment and 3D Median Filter
Dimensions: 993x654x700 pixels
(Image used in Segmentation)

Binary Image
(Black: solid and white: Pore)

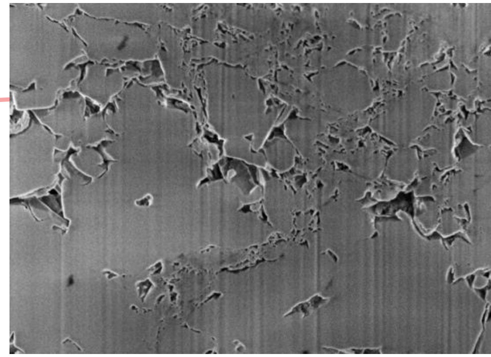
FIB-SEM images

Porosity < 10%

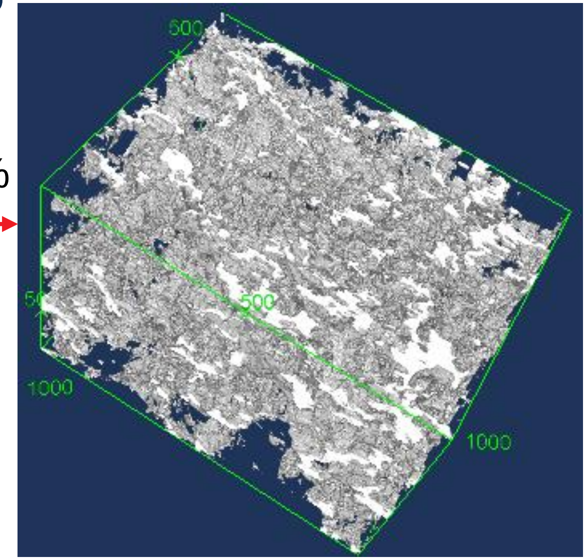


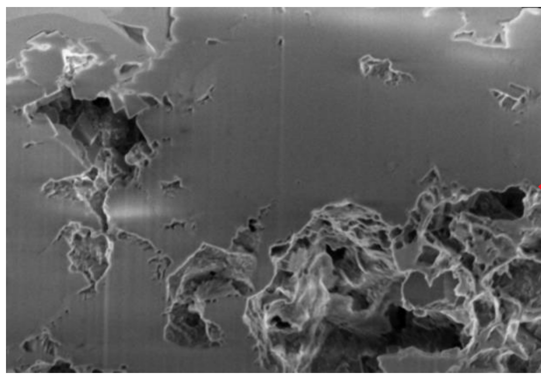
(BSE at 1 um res.)

Porosity = ~42%

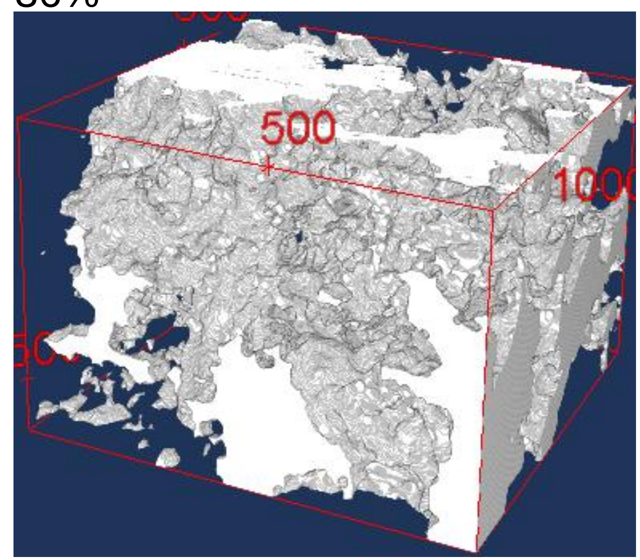


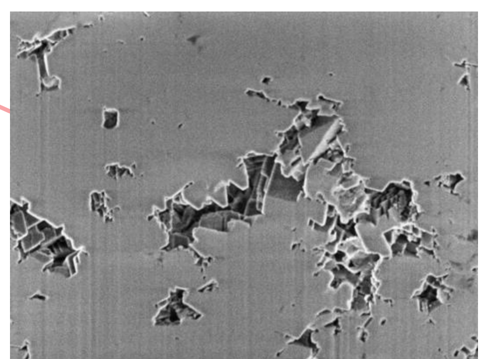
Porosity = 12-13%





Porosity = ~36%





Porosity = 7.7%

Summary

- ► Recent advances in multiscale imaging capabilities provide rich 3D data (e.g., FIB-SEM, EDS, BES, MicroCT) to account for chemo-mechanical processes in a core flooding test
- ► Coupled chemo-mechanical processes are localized, depending on micro-hydrodynamics and reaction fronts
- ► Chemically altered zone shows very heterogeneous pore distribution with high porosity, while mechanically compacted zone shows low porosity with less heterogeneous patterns
- ► Pore scale single- and multi-phase flow modeling and reactive transport modeling are being performed to assess mechanistic understanding of chemo-mechanical processes during core-flooding

Acknowledgments

- Supported in part by the Strategic Partnership Project sponsored by International Research Institute of Stavanger, Norway to apply 3D FIB-SEM/EDS for imaging of carbonate rocks
- Thanks for Lisa Lowery (SNL) and Rebekah Carr (SNL) for FIB-SEM imaging and segmentation