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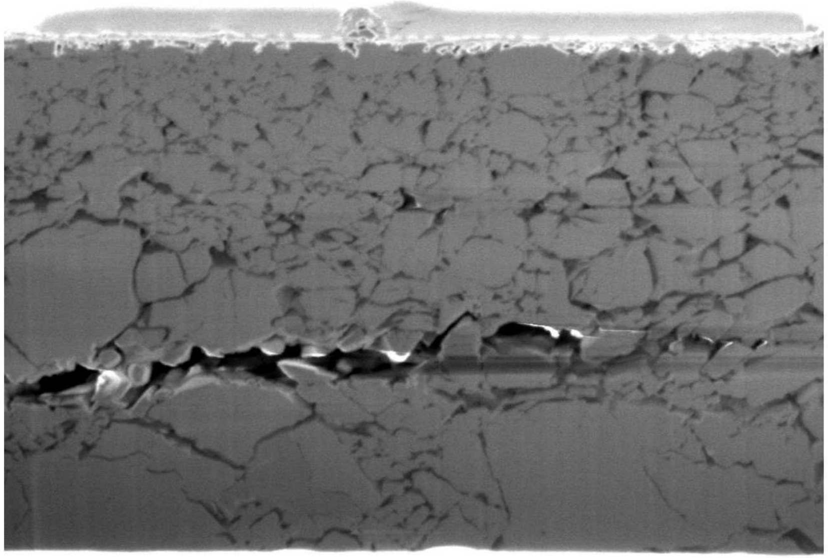
Quantitative analysis of nano-pore carbonate rocks and representative sampling for digital rock physics

Hongkyu Yoon

Thomas Dewers

Geoscience Research & Applications
Sandia National Laboratories
Albuquerque, NM



Exceptional service in the national interest

IOR Norway 2015

April 28-29, 2015

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Acknowledgments

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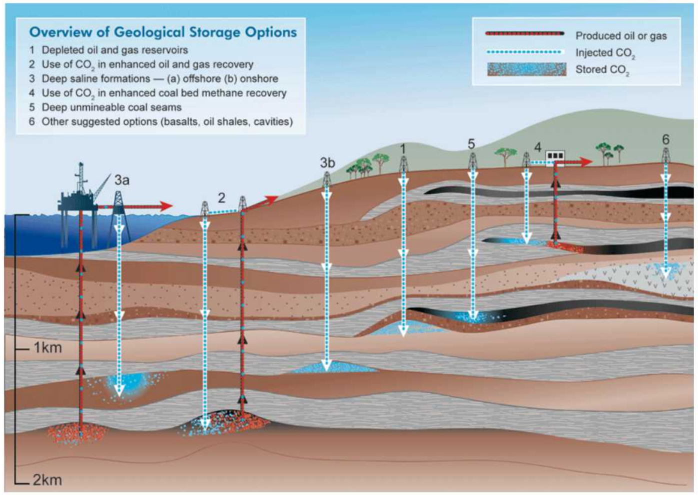
- U.S. Department of Energy Office of Basic Energy Sciences, Division of Chemical Sciences, Geosciences, and Biosciences

Outline

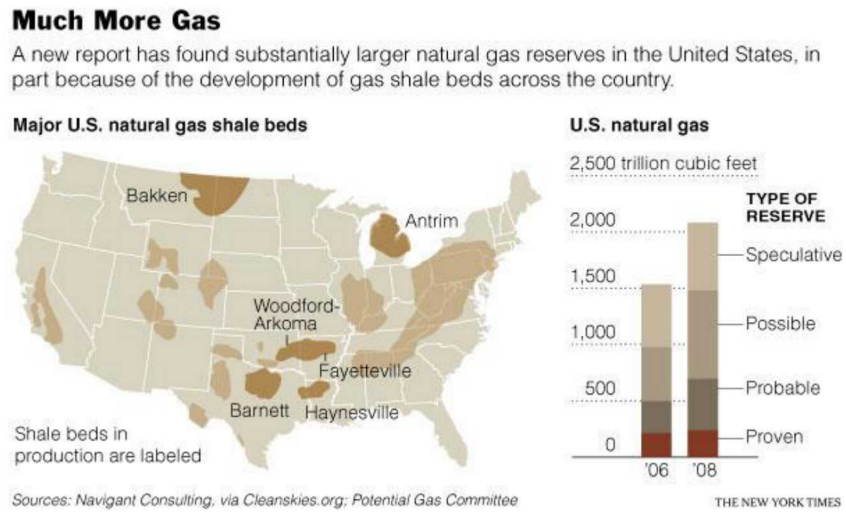
- Motivations
- Multiscale Imaging Characterization
- Digital Rock Physics
- Future works

Why study nano-porous materials

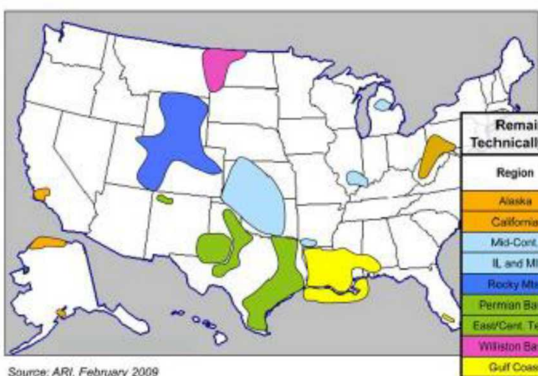
Caprock of subsurface CO_2 storage… IPCC(2005)



… Reservoirs for unconventional resources and EOR….



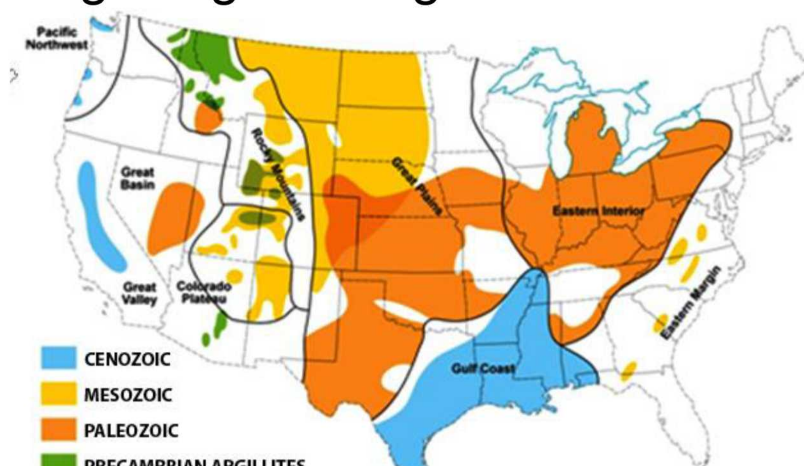
…Enhanced oil recovery … …and for geologic storage of nuclear waste



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"

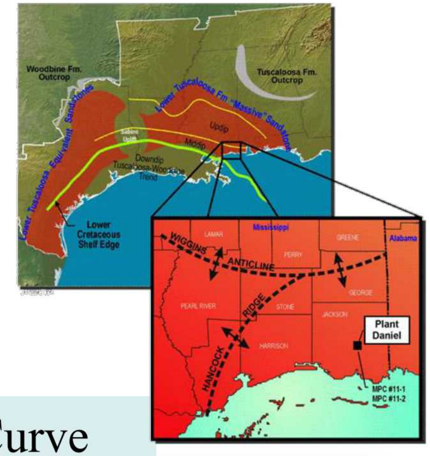


Gonzales and Johnson (1984)

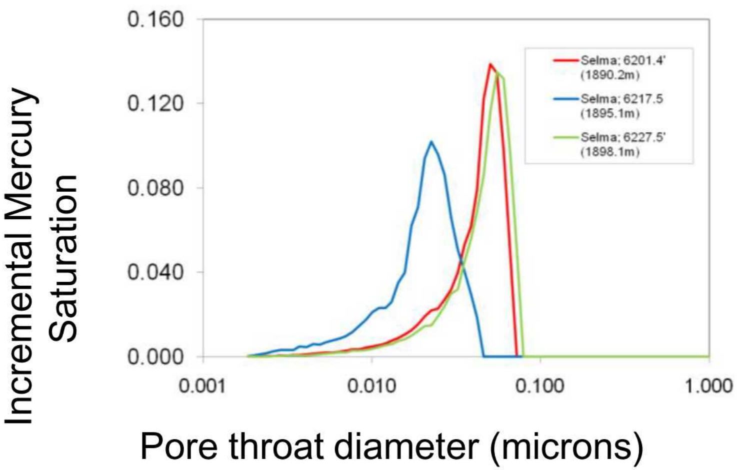
More Motivation…

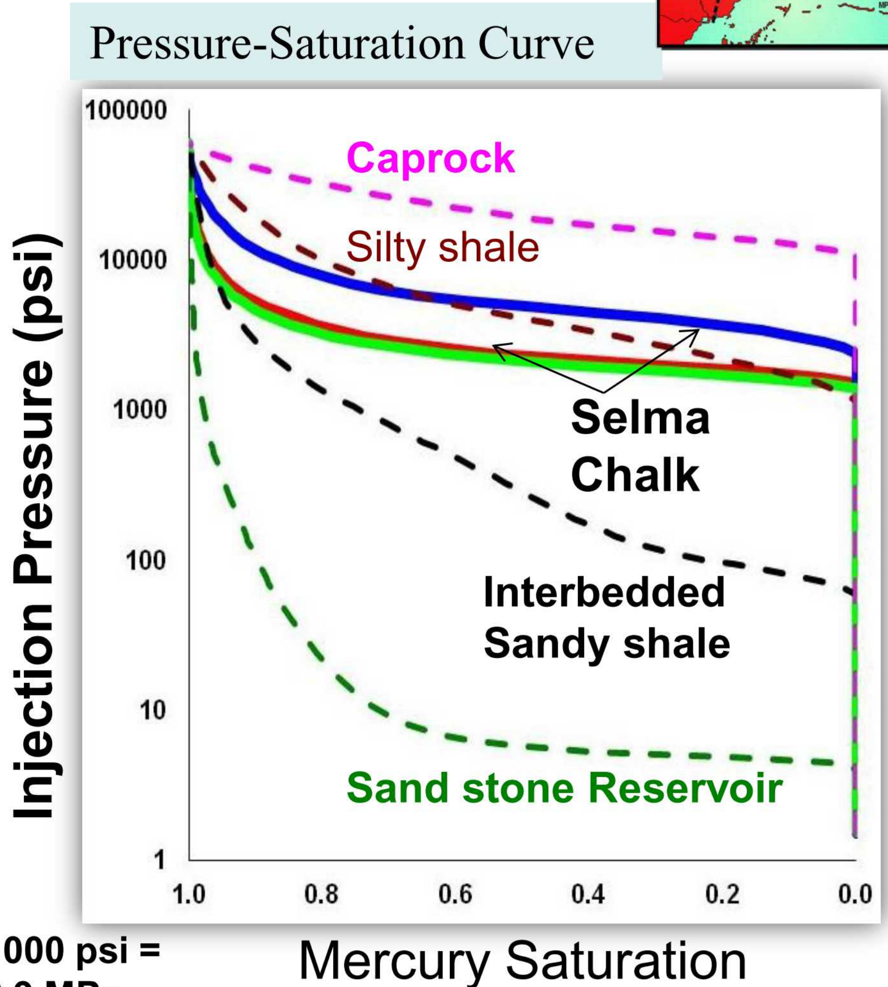
- Plenty of pores at sub-micron scale
  - Recent activities such as enhanced oil recovery, geological CO_2 storage, exploration of unconventional resources highlight the significance of nanopores
- Advances in analytical capabilities with X-ray, electron, and ion beams offer emerging tools for characterizing 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)
- What is appropriate sample volumes for pore scale analysis? Can a small volume of materials for focused ion beam-SEM (FIB-SEM) analysis be representative?
- Digital 3D reconstruction of nano-porous materials over scales

Selma Chalk



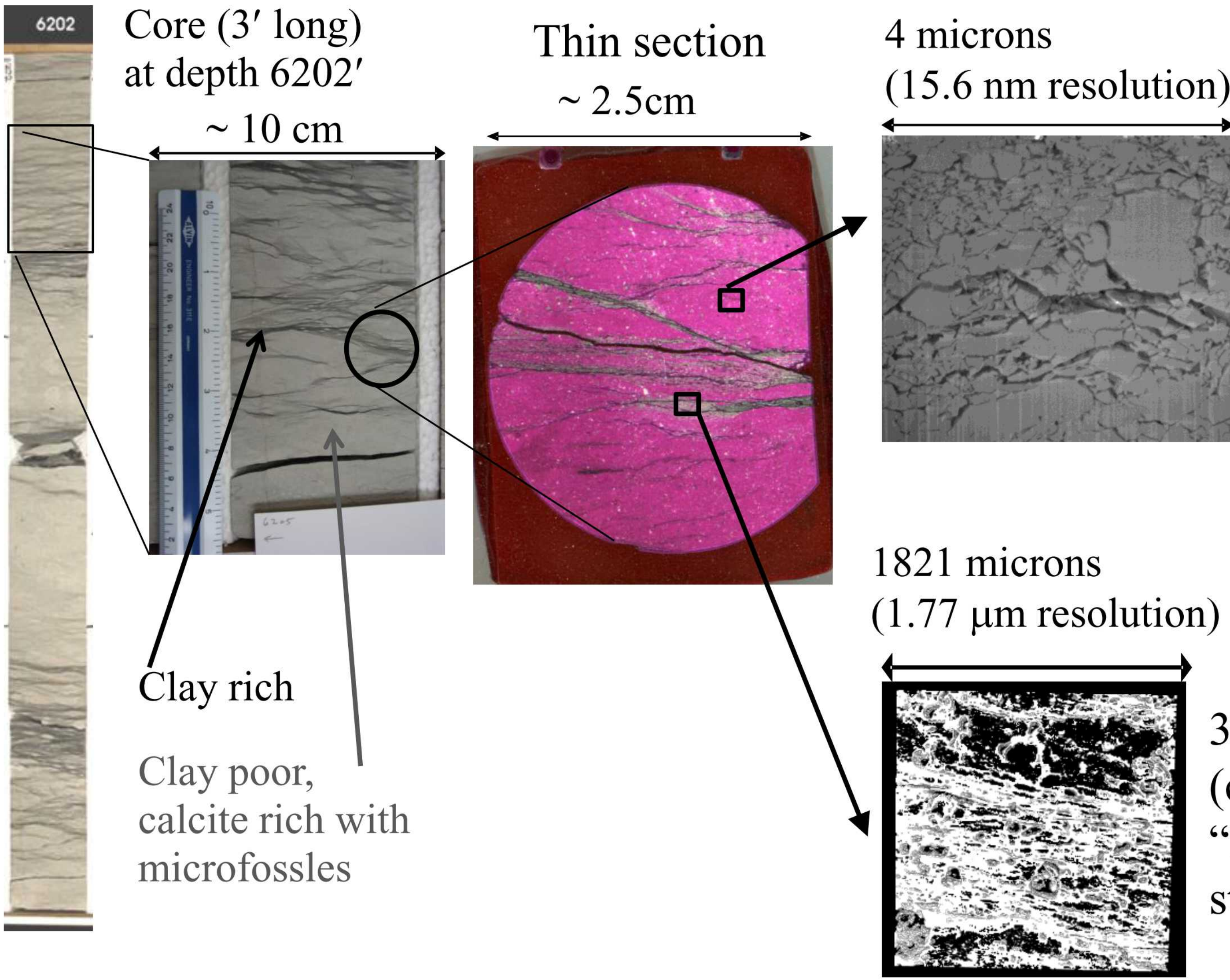
- Secondary “seal” for NETL’s SEACARB Phase II Plant Daniel site for CO_2 injection into Lower Tuscaloosa
- “leaky caprock” to mitigate injection pressure hazard





1000 psi = 6.9 MPa

Chalk Samples across Scales

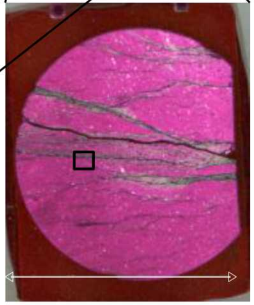


Multi-Scale Imaging of Nano-porous Materials

Characterization of pore structures, compositional distribution, surface properties (fluorescence microscopy, microCT, FIB-SEM, TEM, EDS)

Core

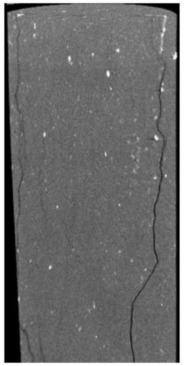
Core (2-5 cm)



~2 cm

Thin Section

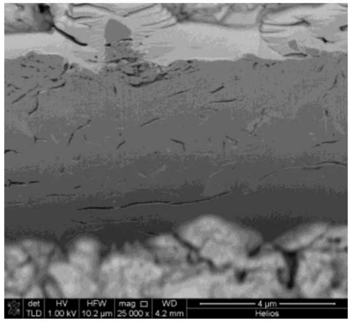
X-ray computed Tomography (CT)



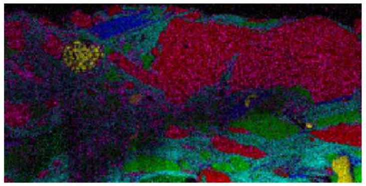
Optical & confocal Microscopy

Focused Ion Beam SEM Analysis

10µm @7 nm



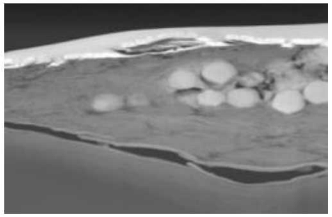
10µm @7 nm

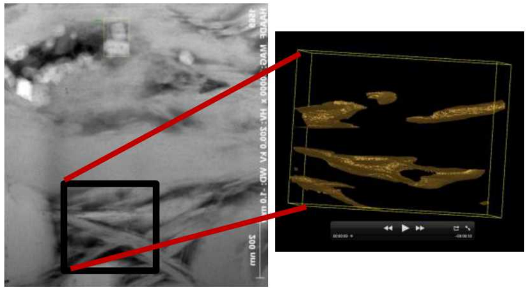


Energy Dispersive Spectroscopy for mineral analysis

Scanning Transmission Electron Microscopy

3 µm (~10 nm res.)



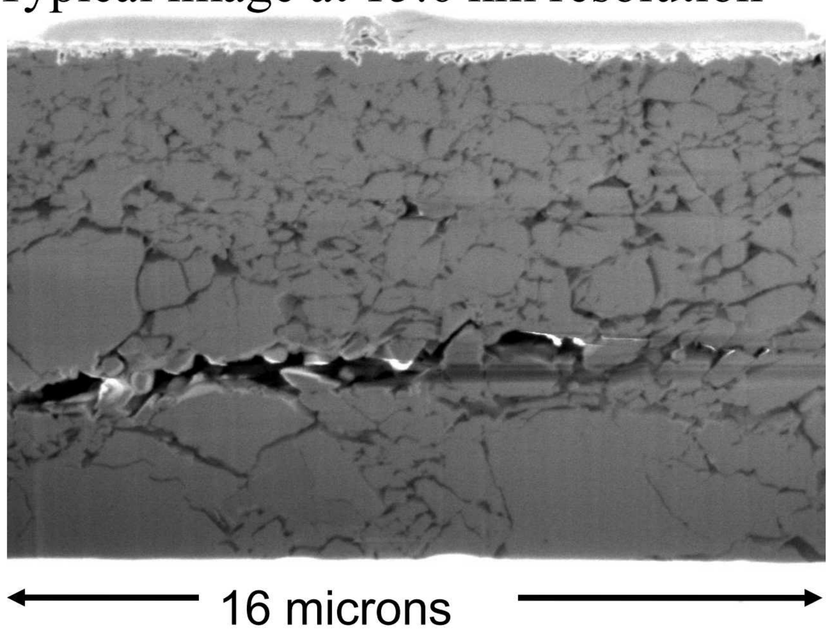


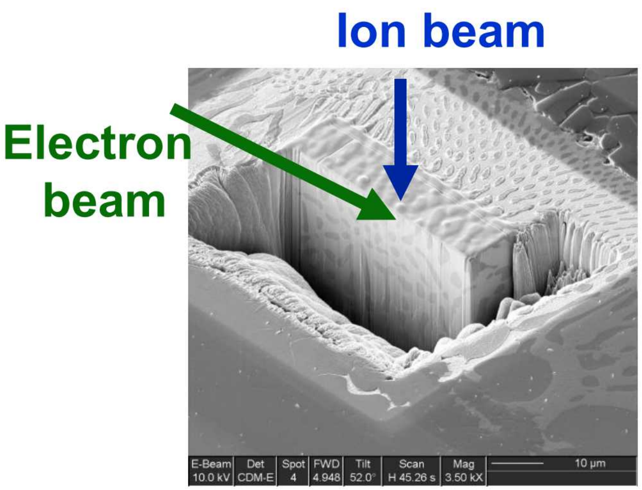
AC-STEM Nano-tomogram at ~1 nm

Dual focused ion beam-SEM (FIB-SEM) imaging

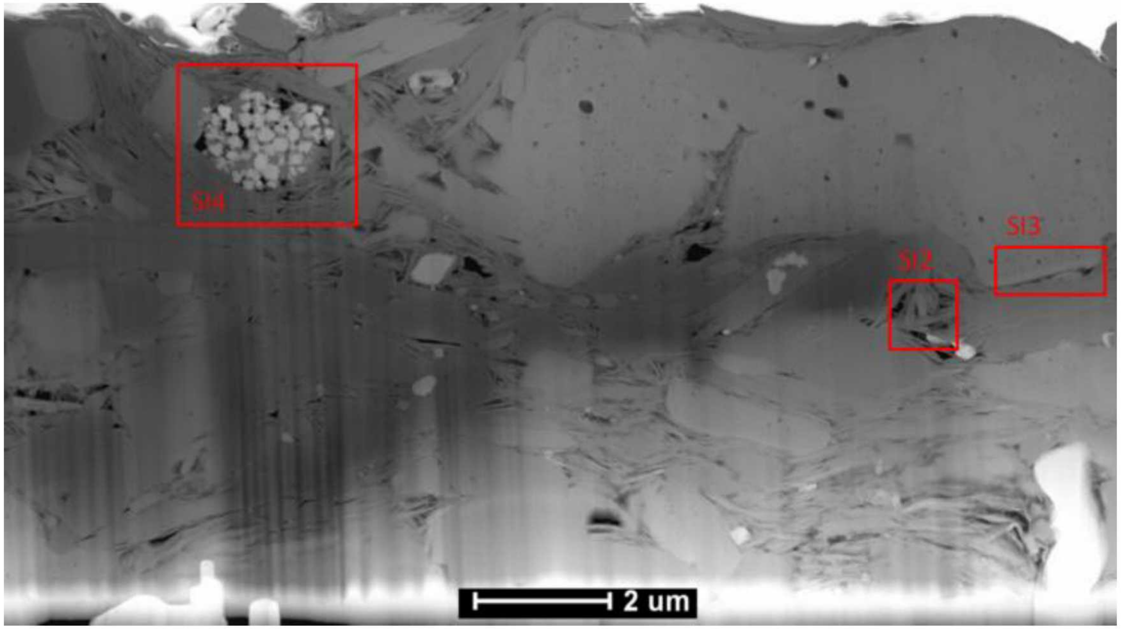
- Focused ion beam for cutting nanometer thin slices
- Scanning & transmission electron microscopy (SEM & TEM) for high resolution imaging (~15 nm to ~ 1 nm)
- Elemental analysis with energy dispersive X-ray spectroscopy

Typical image at 15.6 nm resolution

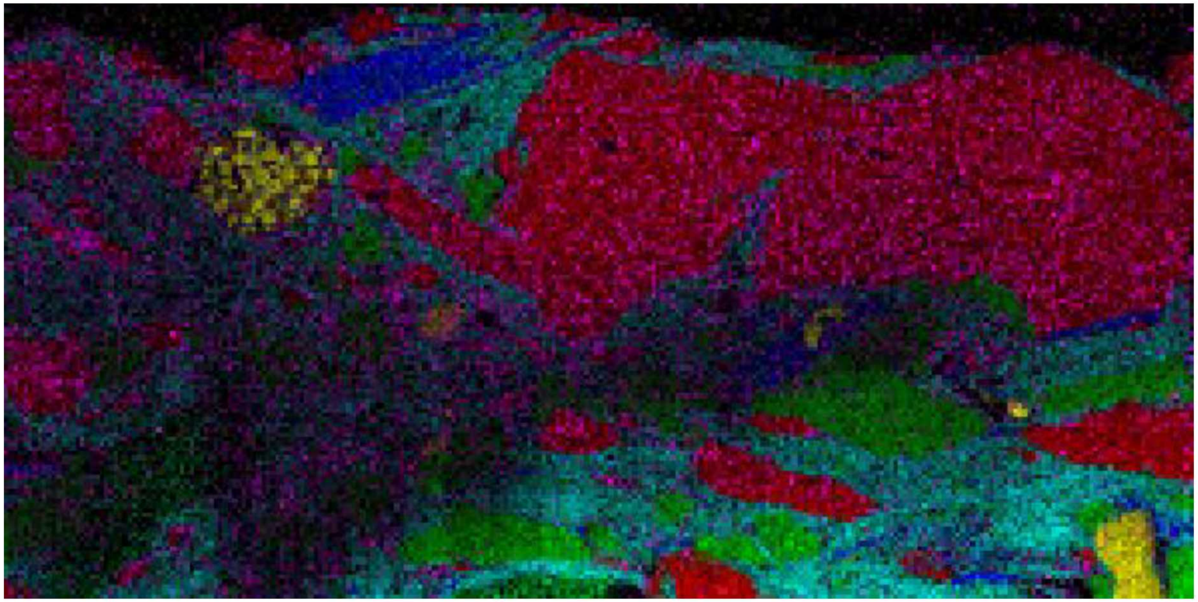




Haynesville Gas Shale



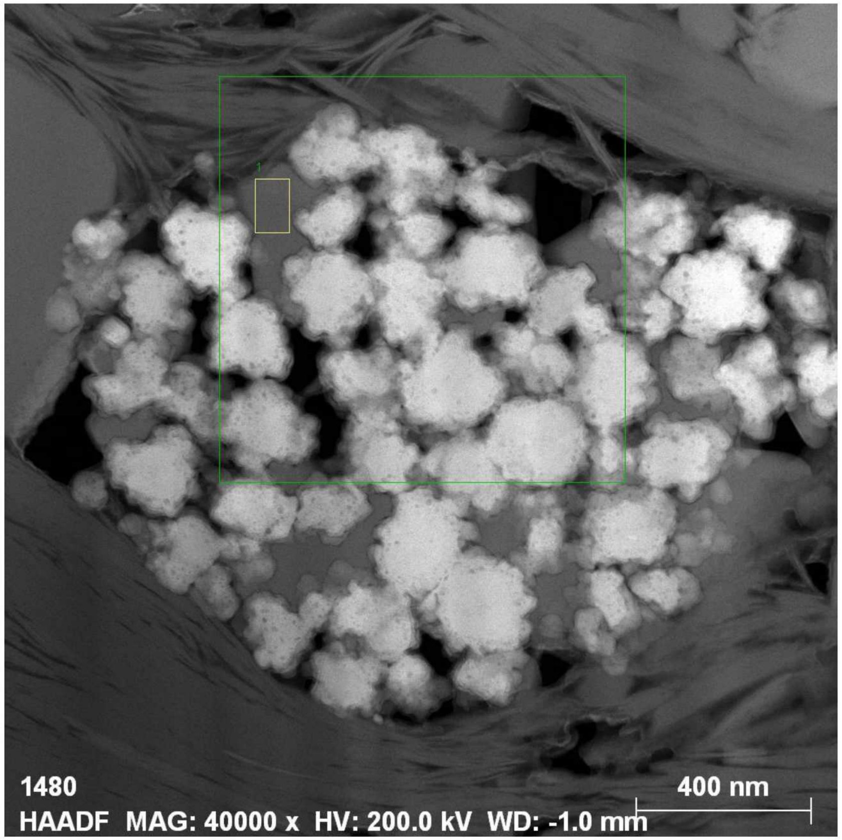
Backscattered Electron Image of the milled section

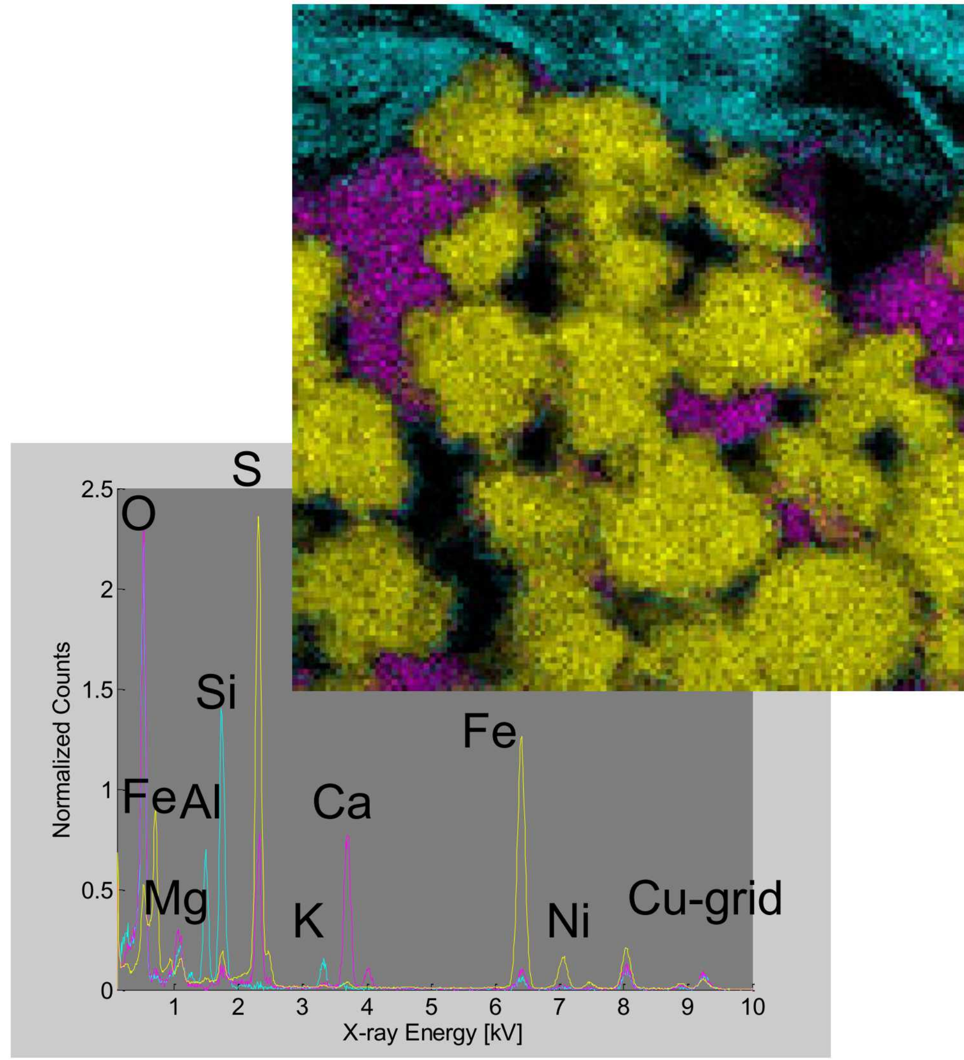


Energy Dispersive X-ray Spectroscopy

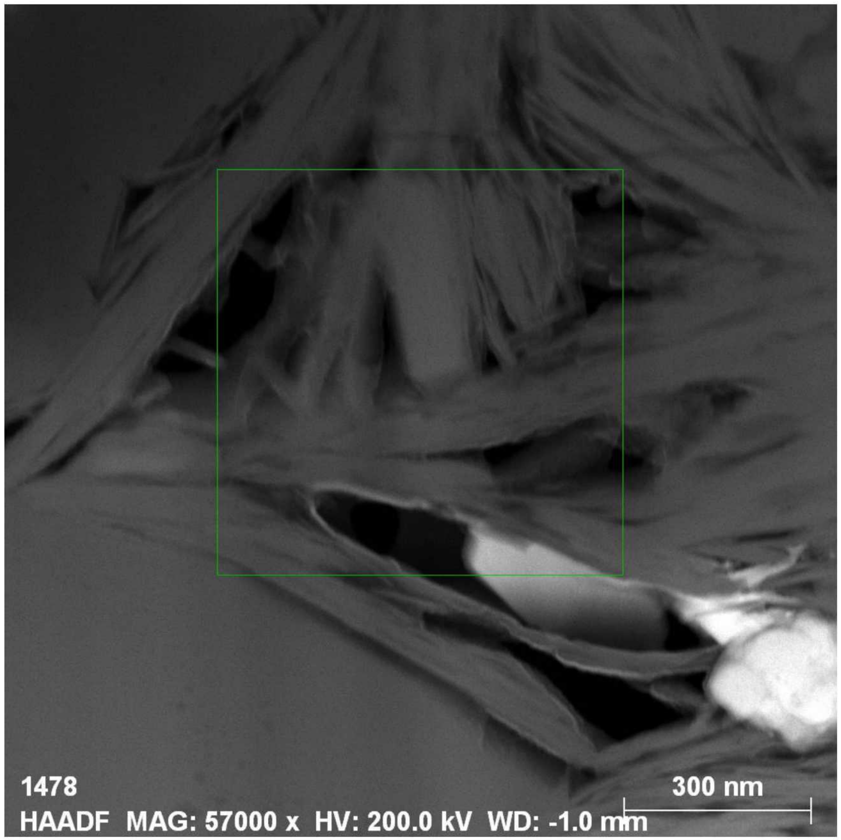
Red = Calcite
Green = Quartz
Blue = Fe-Si-Al-Mg-O
Cyan = Al-Si-K-Mg-O
Magenta = Ca-K-Si-Al-O
Yellow = Combined Fe-S and Ti-O

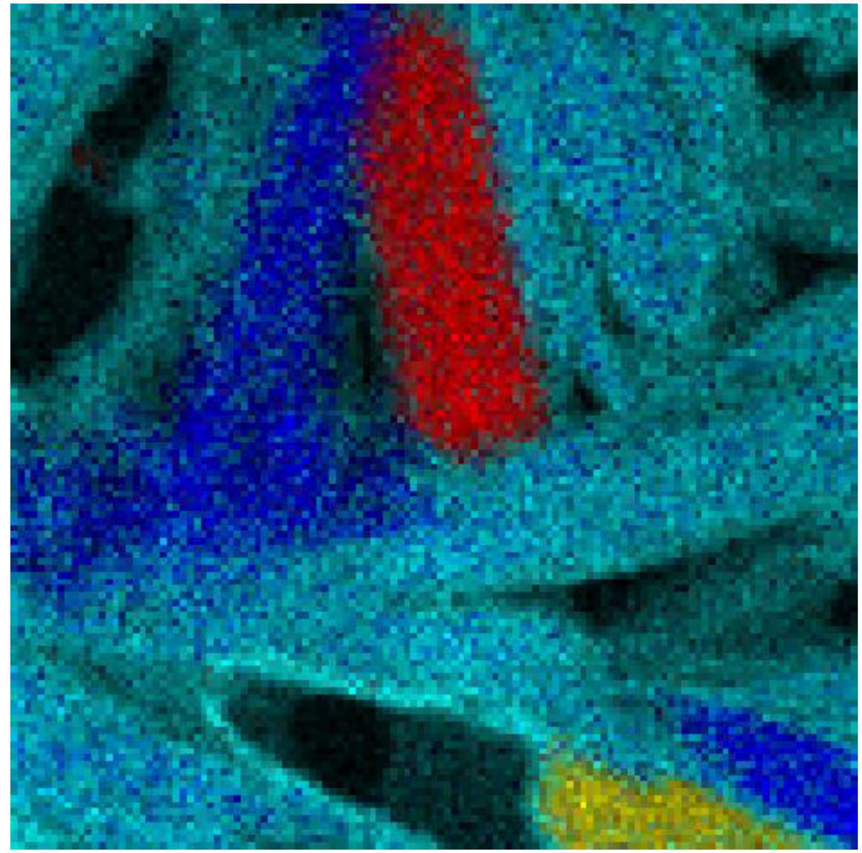
STEM and EDS Analysis





STEM and EDS Analysis





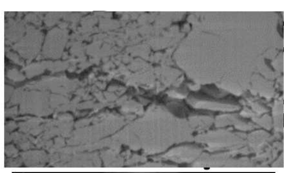
Red = Calcite
Blue = Fe-Si-Al-Mg-K-Ca-O
Cyan = K-Al-Si-O
Yellow = Sr-Ca-Ba-S-O

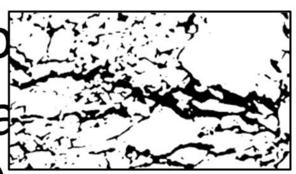
Workflow for Digital Rock Physics

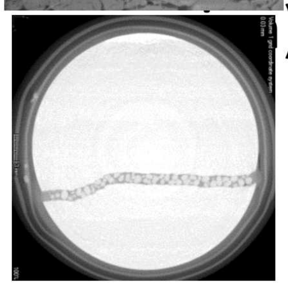
Rock Sample → Multiscale imaging → Image Process → Flow and Transport Properties → Static Effective Elastic Properties → Wave Propagation

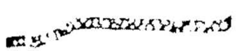
Segmentation Process | **3D Digital Rock Construction** | **Quantitative Analysis**

- Enhance contrasting
- Multiple Filtering
- Thresholding

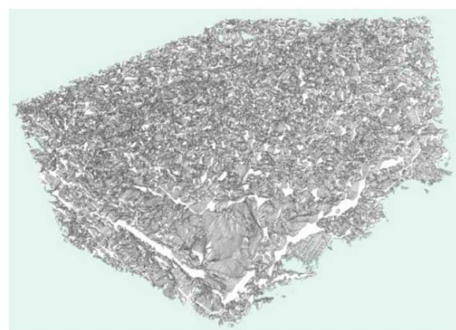


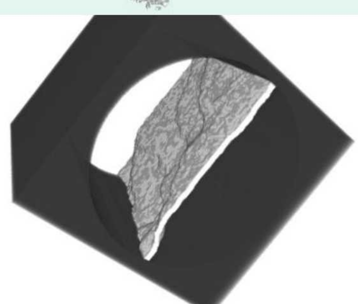


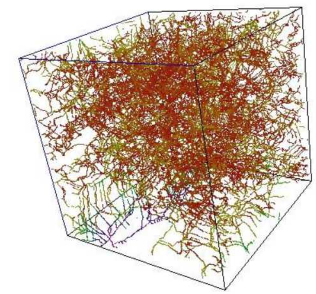




- Binary or ternary pore and fluid distribution construction

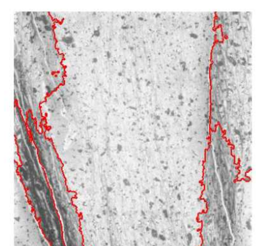


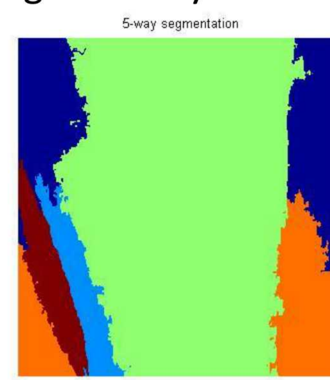




Medial Axis Analysis

Topological Analysis



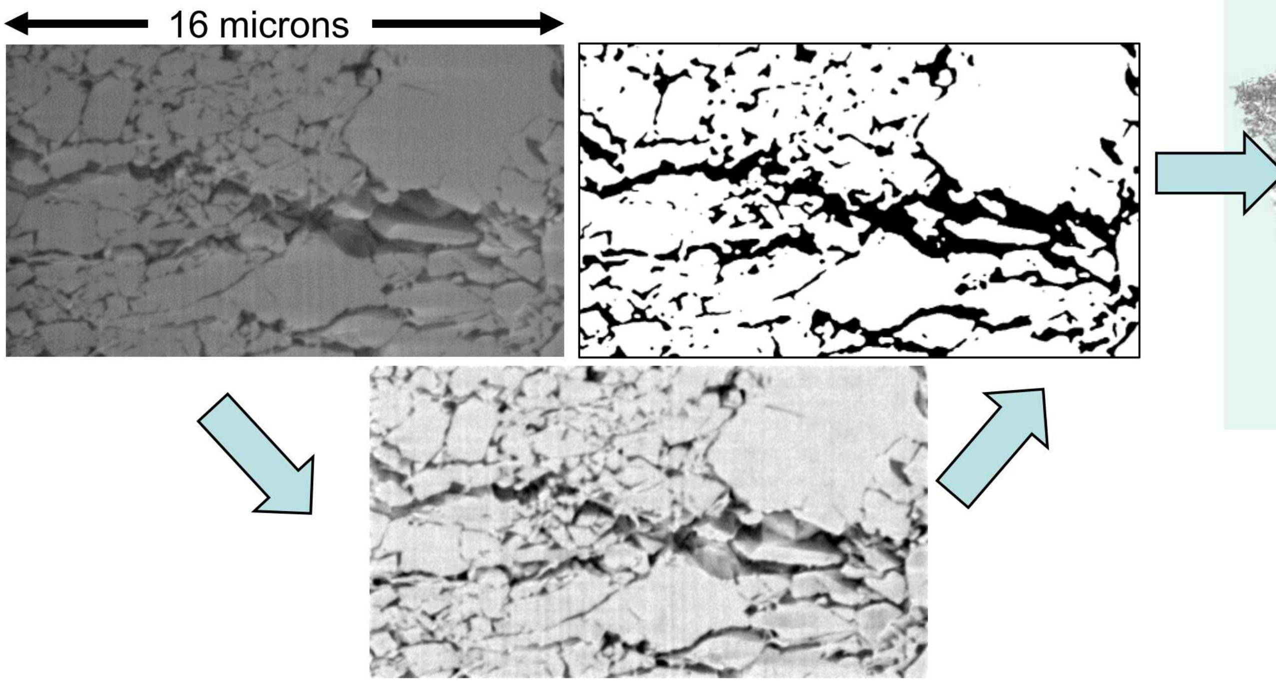


CT images: Courtesy of Mattew Ingraham (SNL)

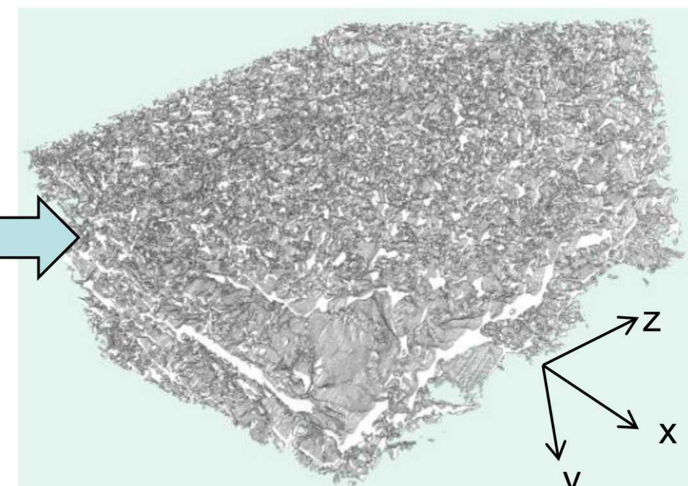
3D Reconstruction of Chalk Sample

Image Analysis:

- Register stack
- Background subtraction & enhance contrast
- Filtering noises (median filter & FFT bandpass filter)
- Segment with thresholding (Adjustment w/ Otsu method)
- Smoothing surfaces with dilation & erosion
- Interpolate in z for cubic voxels and re-segment

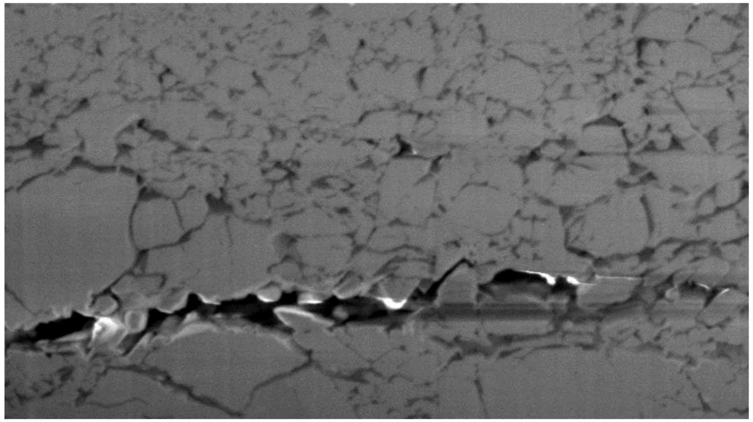


Pore structure (white) in 14.7 (x) x 7.9 (y) x 15.0 (z) micron data set

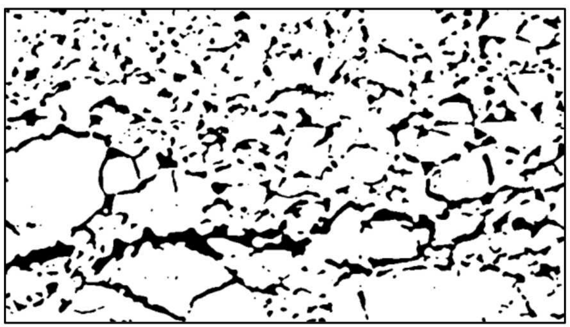


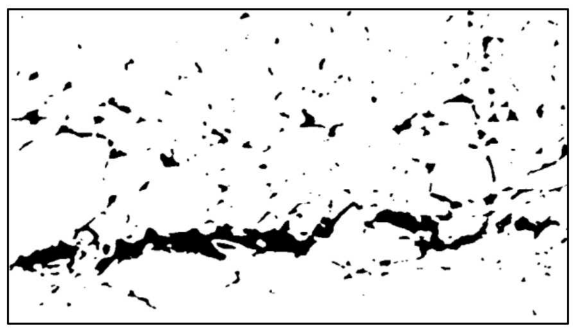
Yoon and Dewers, GRL 2013

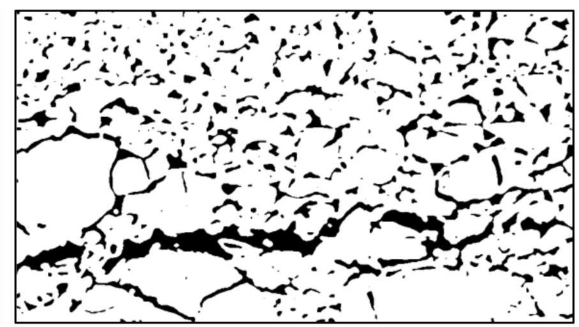
Example of utilizing multiple filters



Raw images have
- Uneven background
- Uneven illumination & horizontal scan lines
- Charging effect (bright white spots)





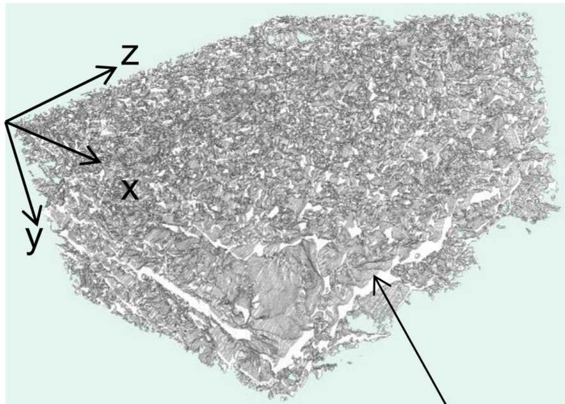


Small pores captured
- Background correction
- FFT bandpass filter
- Median filter
- Threshold (e.g., Otsu method)

Large fractures captured
- Background correction
- Median filter
- Higher threshold value

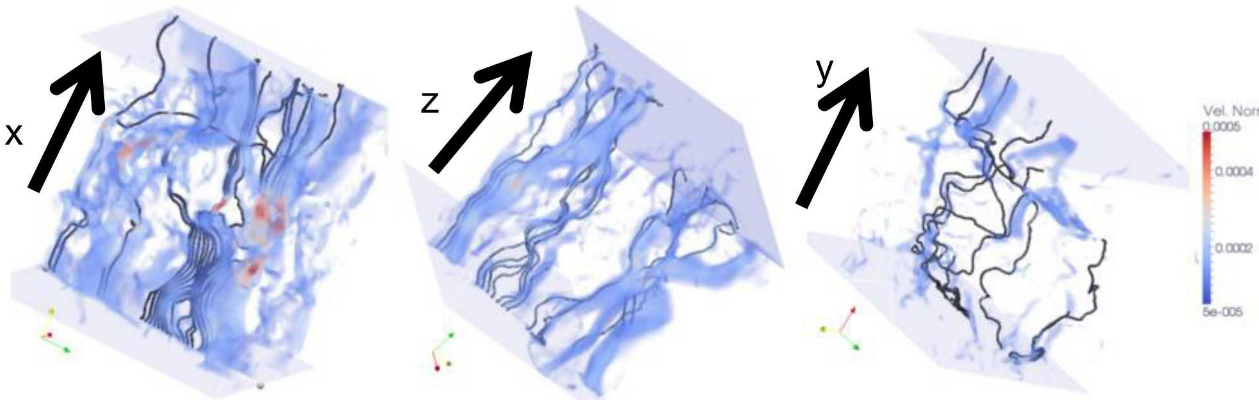
Connectivity recovered
- Combine two binary images
- Dilate
- Erode (twice)

Simulation & Topological Analysis



Highly connected fractures over horizontal plane (x-z)

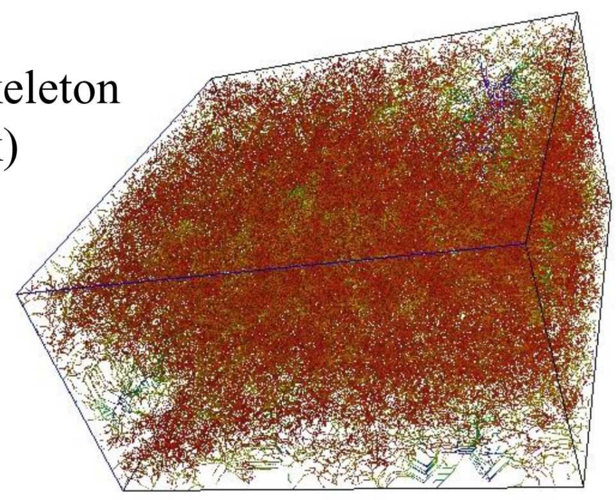
LB simulations

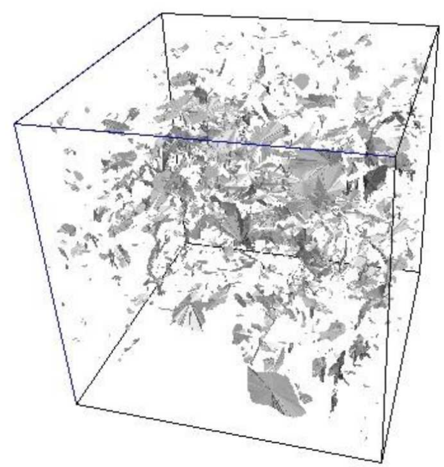


Permeabilities (mD): kx=0.114; ky=0.0064; kz=0.097

3DMA-ROCK (Lindquist et al., 1999)

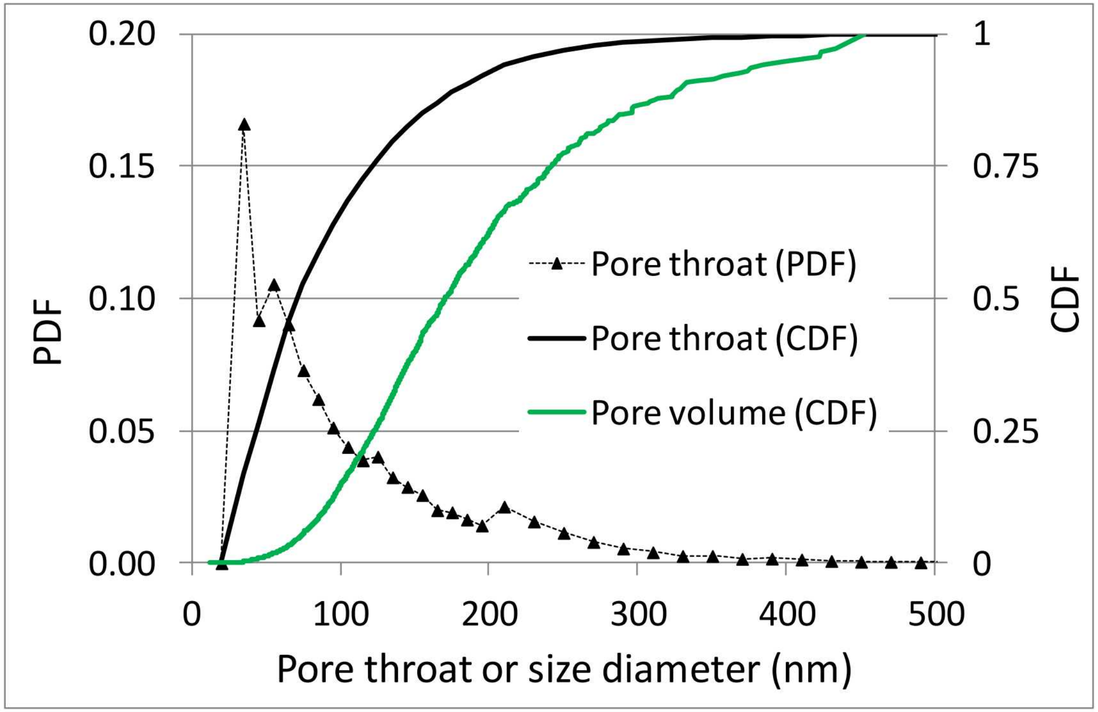
Medial Axis (skeleton of pore network)

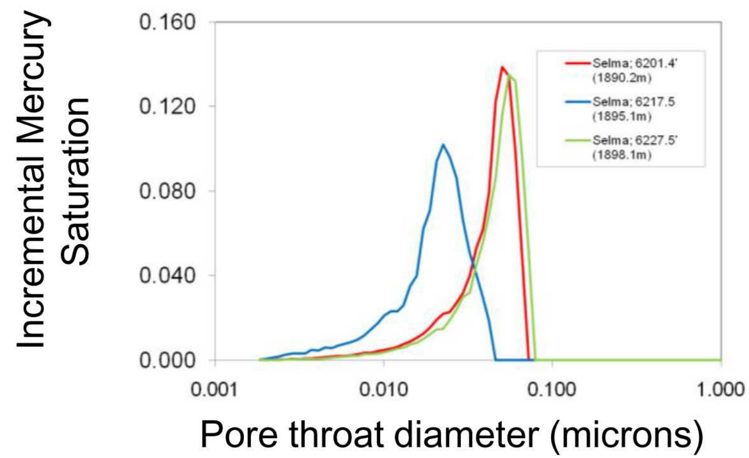




Pore throat visualization

Pore Size Distribution

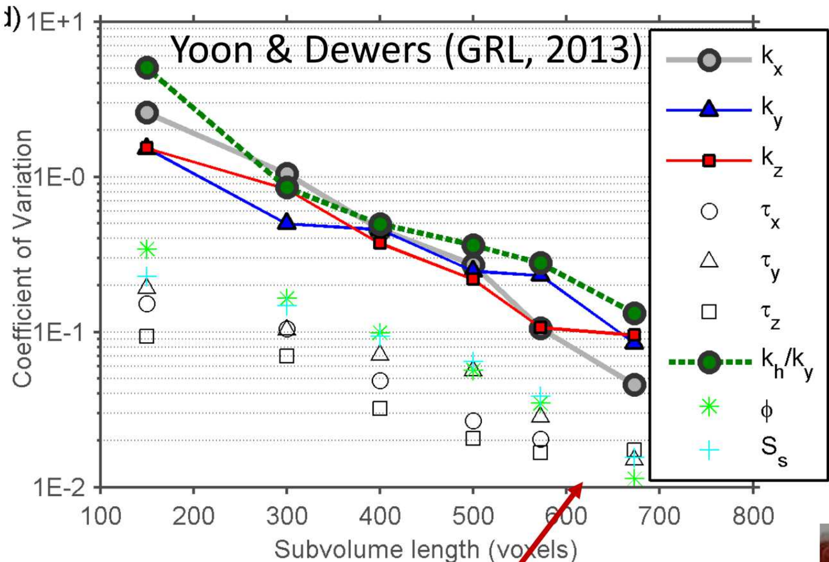




- At the pore size of ~ 80 nm (the decreasing point of kx and kz), cumulative volume fraction is ~ 8% and the cumulative pore throat diameter distribution is ~ 60%
- The decrease of porosity at a function of voxel resolution matches the loss of pore volume fraction well and the decrease of surface area also follows the PDF of pore throat size
- Surface area is strongly affected by the image resolution and is well correlated to the loss of small pores and pore throats, highlighting the significance of nano-pore structures obtained from FIB-SEM analysis

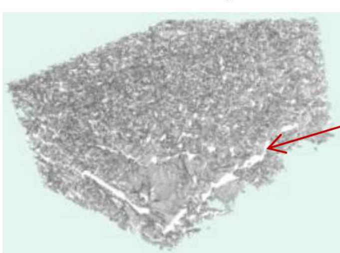
Multi-Scale Reconstruction

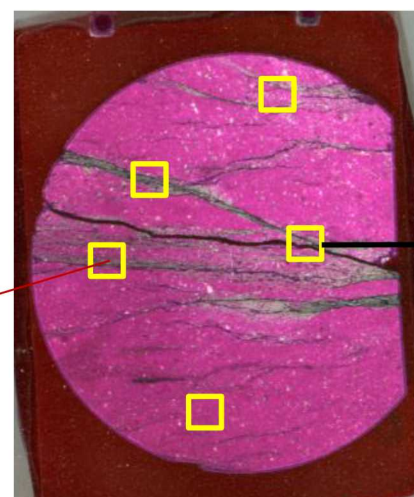
- Reconstruct 3-D pore structures and multi-scale pore networks

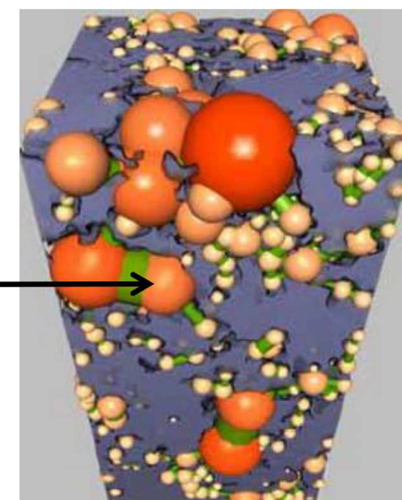


- FIB-SEM samples at ~10 μm
- 4-6 regions can be selected based on principal component analysis of thin section image
- Multi-scale rocks can be reconstructed from nano- to core- scales

FIB-SEM sample volume has a size of statistical elementary volume at ~ 10 μm



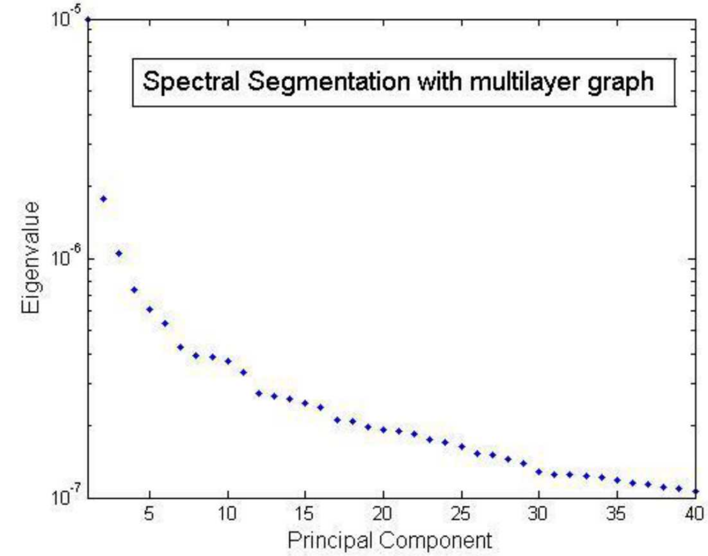




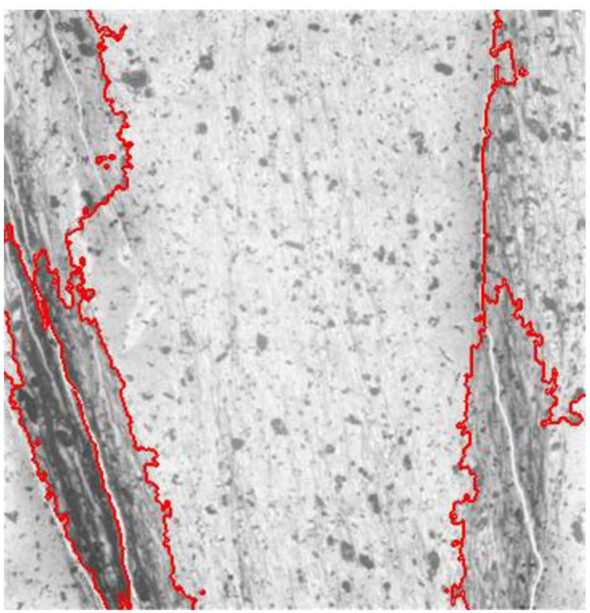
Ghous (2010)

Feature Selection

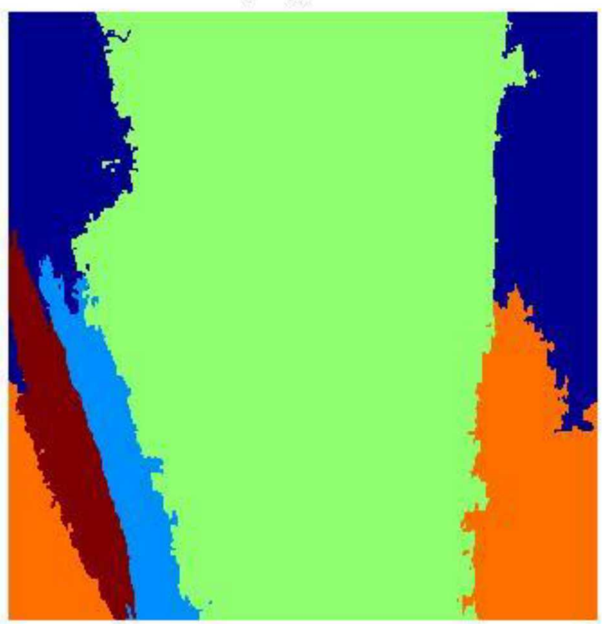
Original image



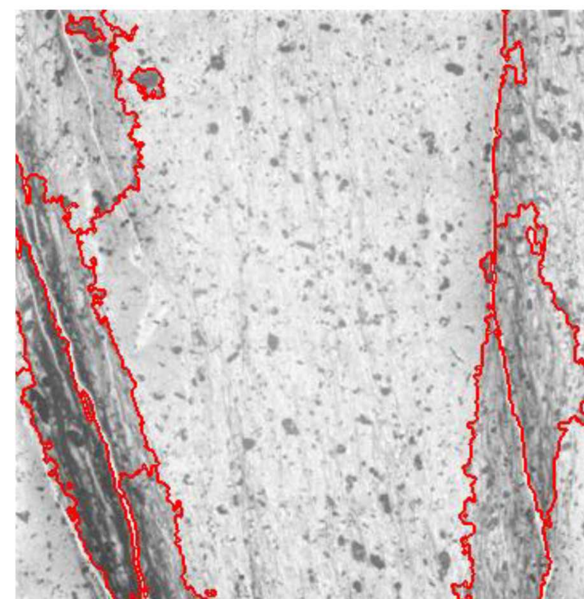
5-way segmentation



5-way segmentation

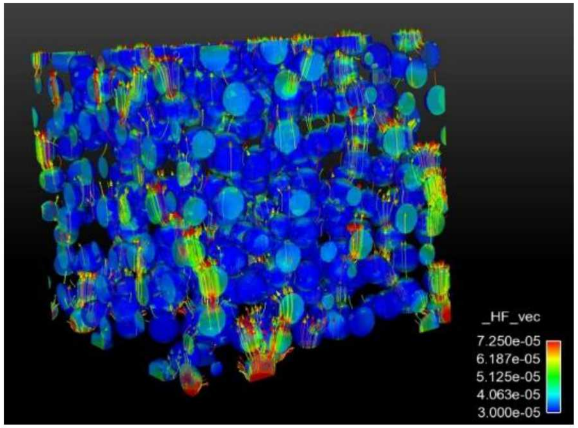


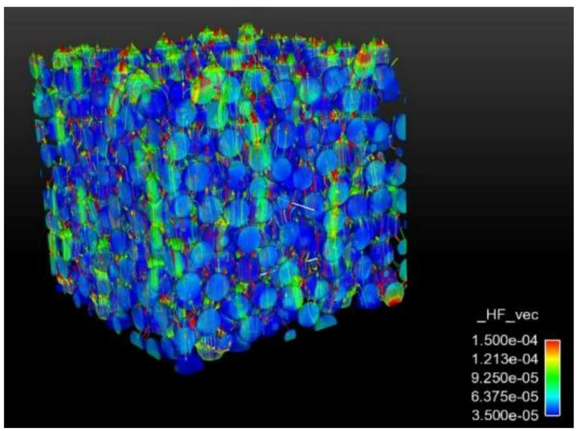
10-way segmentation



Modeling at the Mesoscale

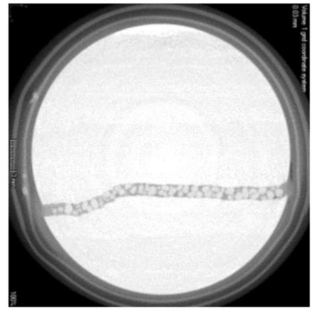
Sierra Mechanics/CDFEM

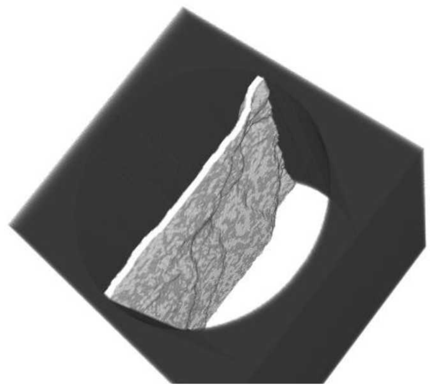


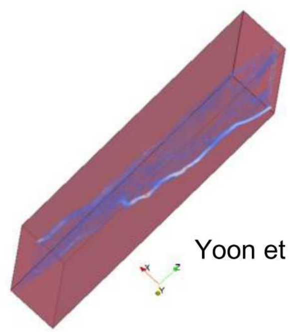


Effective Thermal Conductivity of Particle Dispersions
Jeremy Lechman, SNL

Lattice Boltzmann



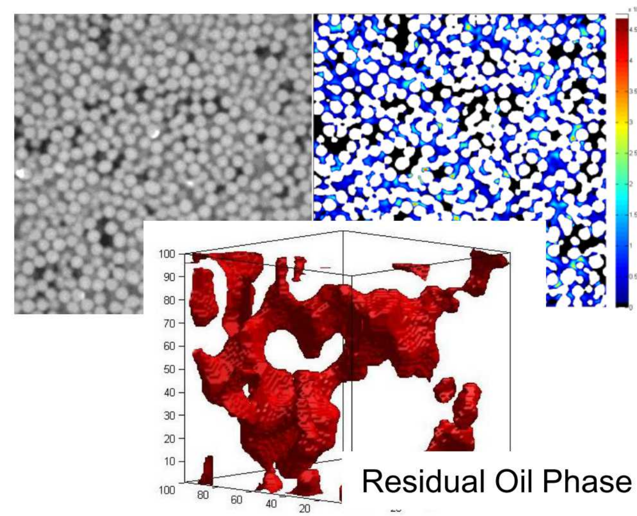


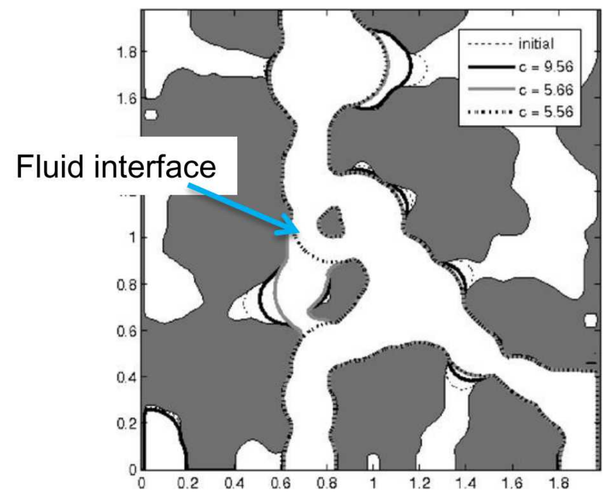


Flow of “frac” fluid in proppant-containing fracture

Yoon et al. SNL

Multiphase Flow with Level Sets





Prodanovic & Bryant 2006

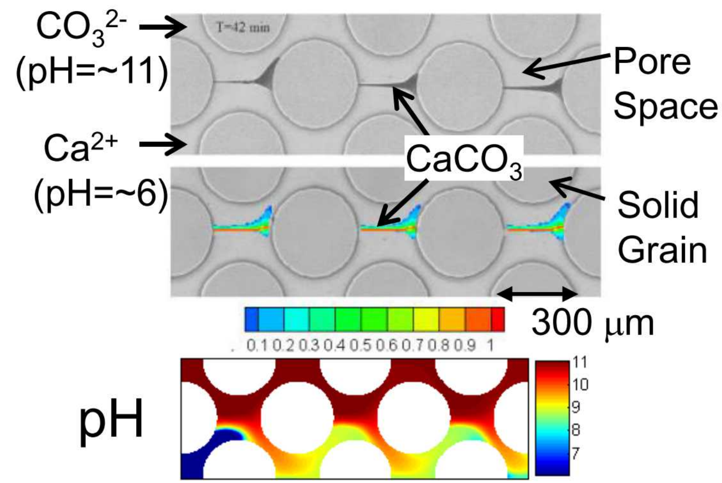
Pore Scale Modeling of Reactive Transport

Research Details

- Simulated transient experimental results of $CaCO_3$ precipitation and dissolution in a microfluidic pore network (shown in Figure)
- Improved understanding of the fundamental physico-chemical processes of $CaCO_3$ precipitation and dissolution at pore scale for coupled reactive transport systems

Applications

- A functional form of reaction rates as a function of system parameters can be developed using pore-scale model
- Results can be used as input for continuum and/or mortar hybrid modeling of more realistic environmental conditions at larger scales



Experimental image (top)
Simulated $CaCO_3$ dist. (middle)
Simulated pH distribution (bottom)

Yoon et al. (2012)

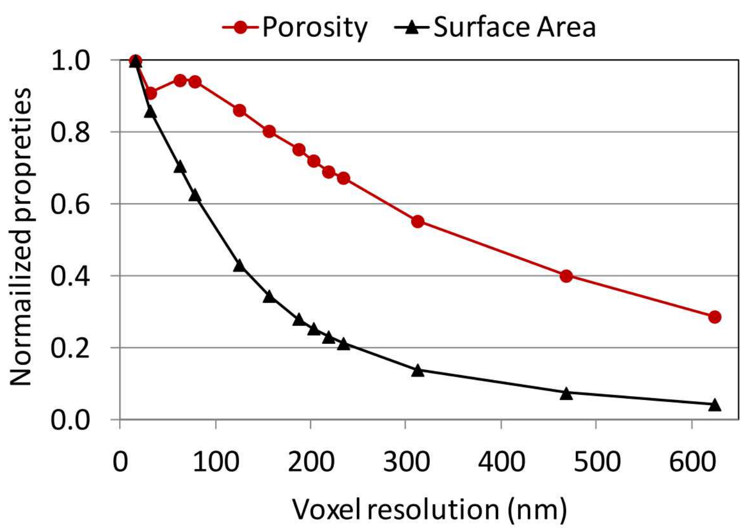
Conclusions

- A significant improvement of multi-scale imaging capabilities with sub-micron FIB-SEM techniques was achieved to accurately account for micro-structural features
- Permeability and specific surface area can be strongly affected by image resolution, highlighting the importance of features at the sub-micron scale where petrophysical and multiphase flow properties in carbonate rocks and mudstones are dependent upon complex 3-D pore structure
- For multiscale reconstruction, segmented 3-D FIB-SEM data at the SREV scale can be directly mapped to a lower resolution 2-D thin section images or 3-D micro-CT data
- Textural properties at the SREV scale can then be used to reconstruct multi-scale pore structure at the scale of practical interest using stochastic methods (e.g., multiple-point simulation) or dual-scale pore network models

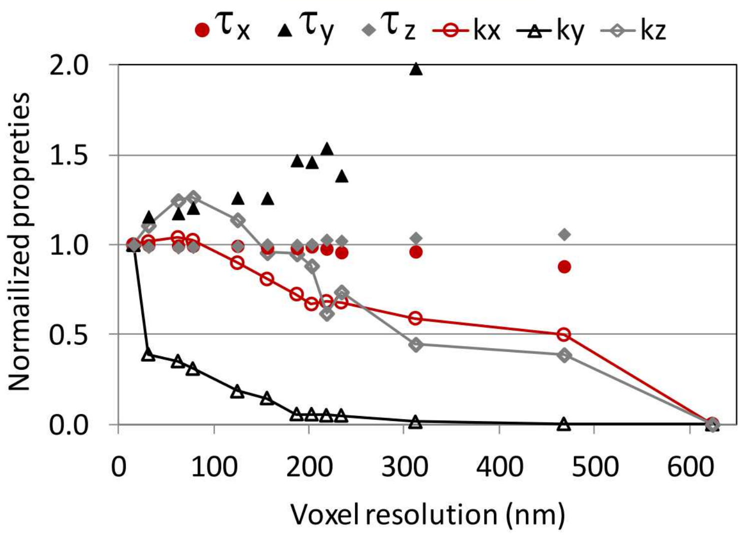
Back up

Impact of Voxel Resolution

Two points statistics

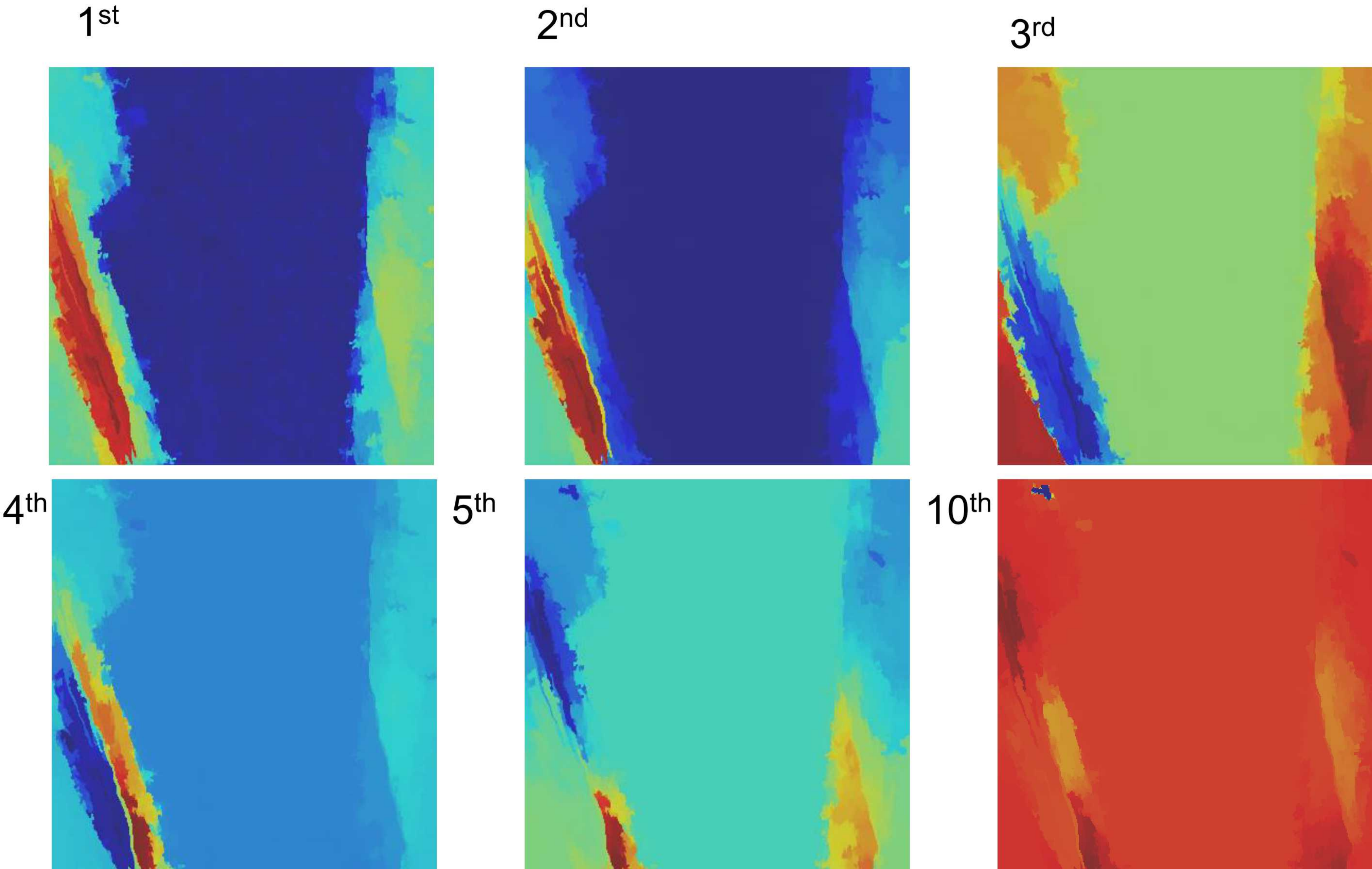


LB results



- The 15.6 nm data was averaged at 31.2 to 624 nm scales
- Porosity decreases at > ~80 nm resolution
- Specific surface area decreases exponentially with increasing resolution
- kx and kz change gradually due to a microfracture, while ky changes sharply
- Tortuosity in x&z directions does not change much, but z increases sharply as nano-cracks are lost at higher resolutions
- Permeability is a better indicator than tortuosity to determine the SREV scale

Five smallest eigenvectors and 10th smallest one



Background: Mercury Injection Porosimetry (MIP)

- Widely used to characterize pore size distributions (psd's) and capillary pressure functions
- Assumes non-wetting fluid (contact angle θ > 90 deg.) intrudes only under pressure
- Derived PSD's use Washburn equation ($P = -4\gamma\cos\theta/d$, γ is surface tension) and "bundle of tubes" model
- When applied to rock pore topologies, often biased towards smaller pores in distribution (e.g. "ink bottle effect")