

Pore-Scale Transport Properties of Mudrocks

**Geosciences Research Program in the
Department of Energy's Office of Basic
Energy Sciences**

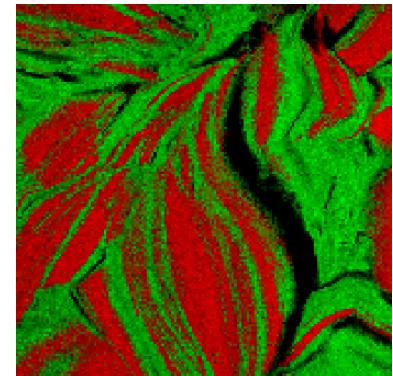
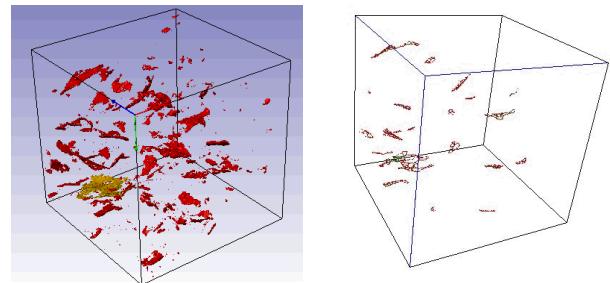
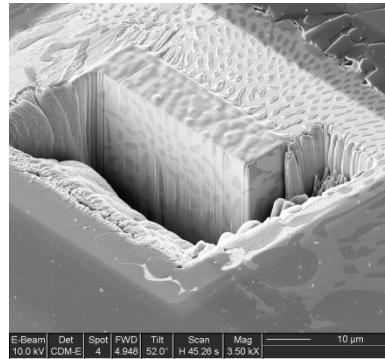
**Research Symposia on Geophysics and Fluid
Flow**

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²Geophysics, Albuquerque, NM

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Energy Basic Energy Sciences/Office of Science**



Outline and Acknowledgements

Pore networks in mudrocks from five depositional settings

- Geology Overview
- FIB/SEM and Pore Network Construction
- Pore Statistics from Imaging and Porosimetry
- CFD Modeling of Flow in Shale Pores
- Shale Nano-Mechanics
- Summary and Conclusions
- Path Forward

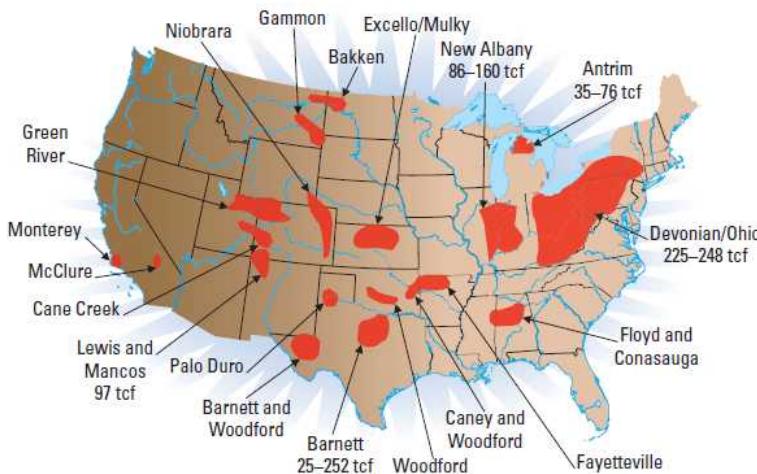
Funding for this study is from Department of Energy Basic Energy Sciences/Office of Science. Thanks also to programs from DOE-NETL's SWP and SEACARB, ARI and The Southern Company, Terra Tek/Schlumberger, OMNI/Weatherford, PoroTek, and to the UGS Shale Gas Project

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Michael Rye, SNL
Scott Cooper and Randy Everett,
SNL
Brian McPherson, UU
Alexis Navarre-Stitchler, CSM

Mudstones are.....

Shale Gas Reservoirs



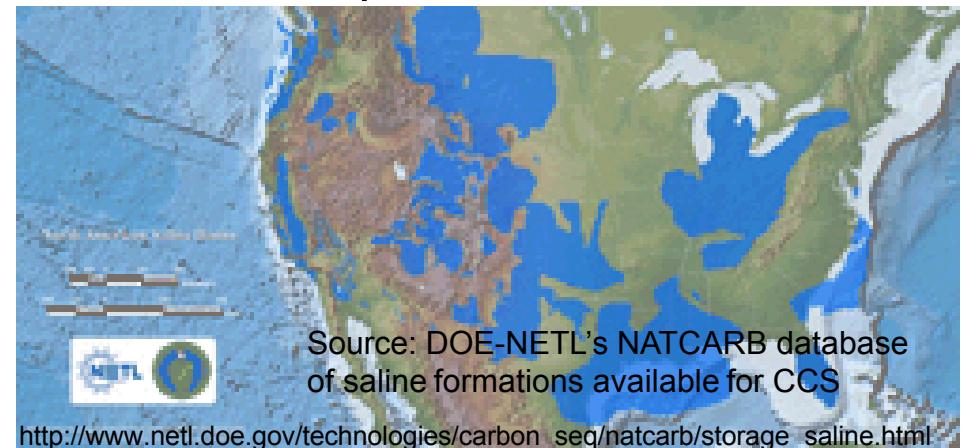
Major shale gas basins in the United States with total resource potential of 500 to 1,000 tcf.

Source: Schlumberger, Shale Gas, 2005

Cook (1999) defines “shale” as a rock with over 50% by wt. clay minerals, and with a continuous network of clay, i.e. clay matrix is load supporting. Other workers use the term “mudrock” with this definition, wherein “shales” are mudrocks with well developed fissility or bedding plane partings.

Schlumberger Oil-Field Glossary defines shale more generally as a “fine-grained, fissile, detrital sedimentary rock formed by consolidation of clay- and silt-sized particles into thin, relatively impermeable layers” (<62 microns)

Seals/Caprocks for CCS

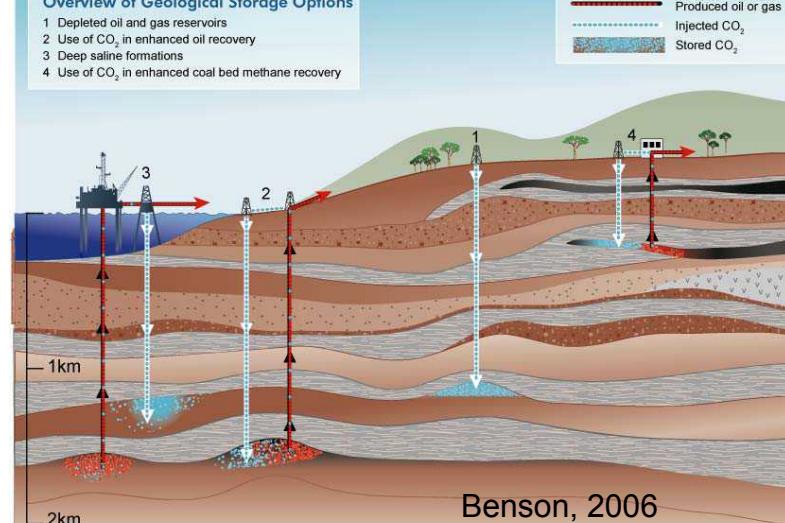


Source: DOE-NETL's NATCARB database of saline formations available for CCS

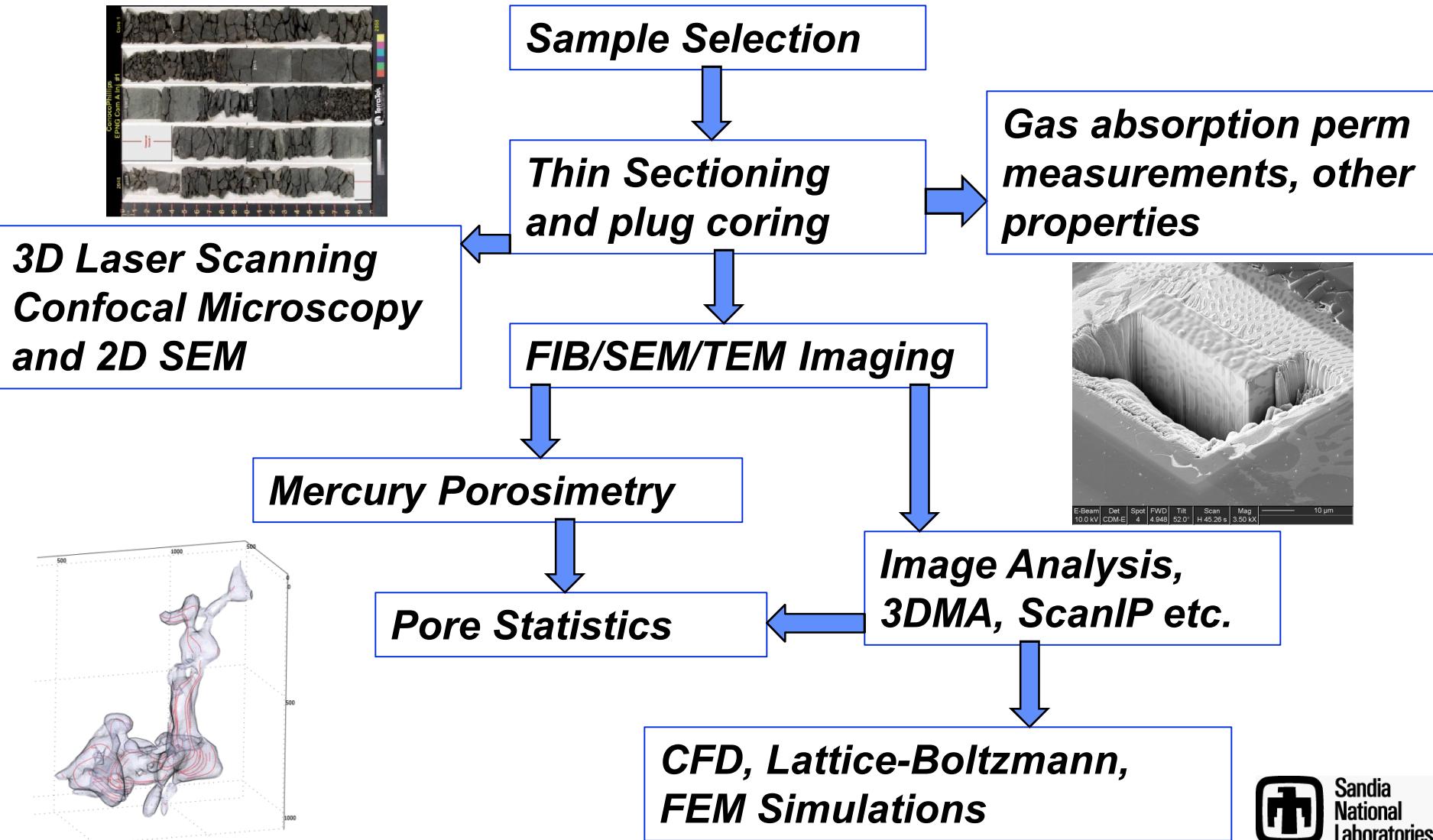
http://www.netl.doe.gov/technologies/carbon_seq/natcarb/storage saline.html

Overview of Geological Storage Options

- 1 Depleted oil and gas reservoirs
- 2 Use of CO₂ in enhanced oil recovery
- 3 Deep saline formations
- 4 Use of CO₂ in enhanced coal bed methane recovery

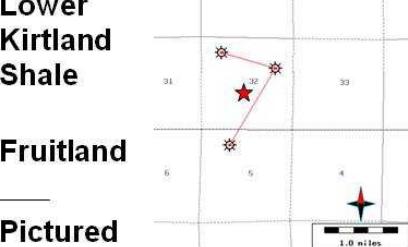
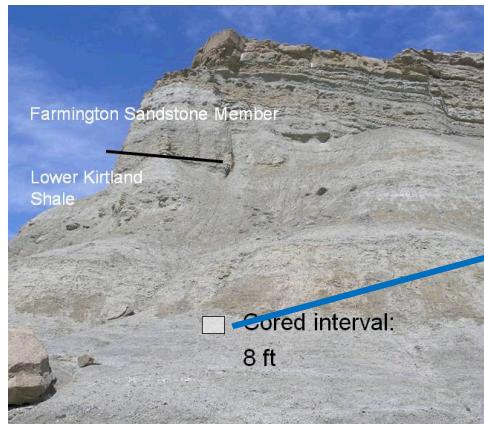
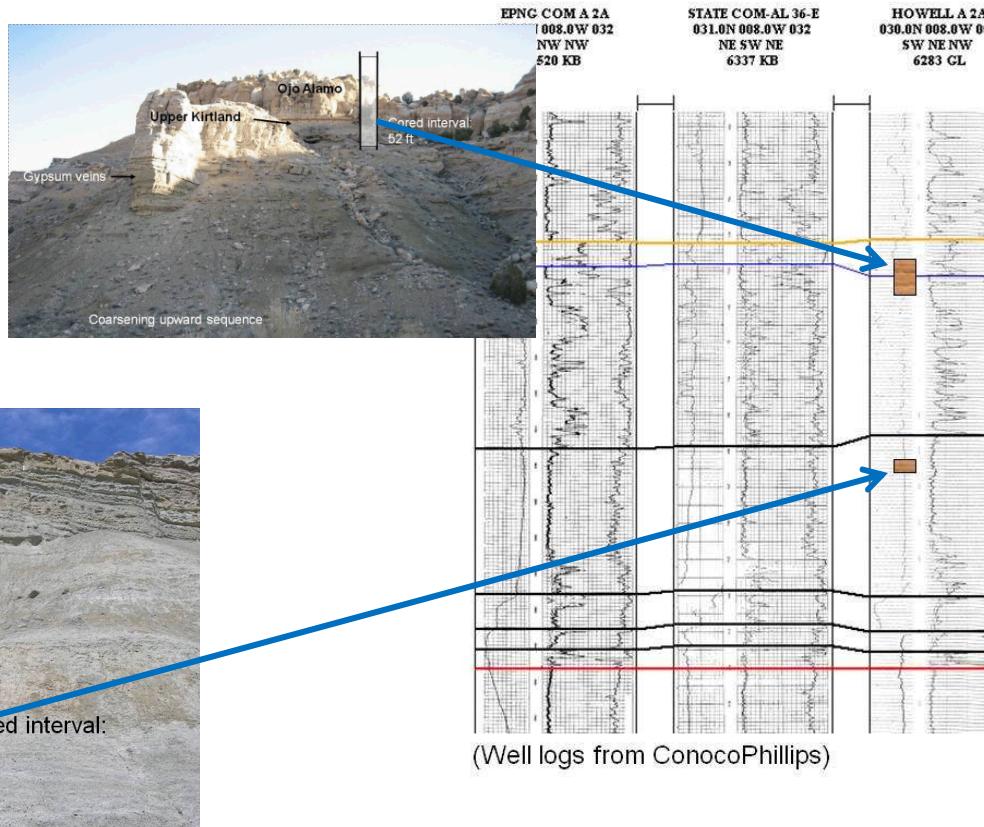
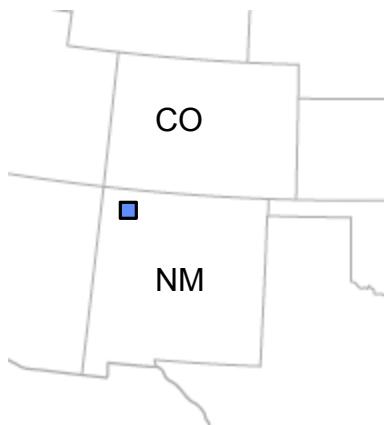


Workflow: Characterization and simulation of pore scale transport properties





Upper and Lower Kirtland Shale, Cretaceous, San Juan Basin, NM



Samples from preserved core, drilled as part of the Pump Canyon Pilot Project, Phase 2 of DOE-NETL's SWP

Gothic Shale, Pennsylvanian, Paradox Basin, Utah

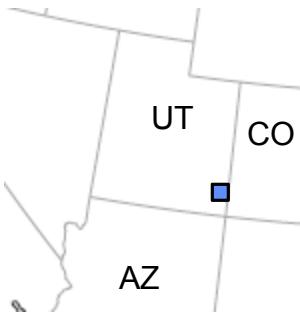


Figure 7-2. Gothic shale in the Pennsylvanian Paradox Formation exposed along the Honaker Trail, San Juan River Canyon, Utah.

Core sampling performed in conjunction with Utah Geological Survey Shale Gas Project and the Phase II Aneth Project of the DOE-NETL SWP

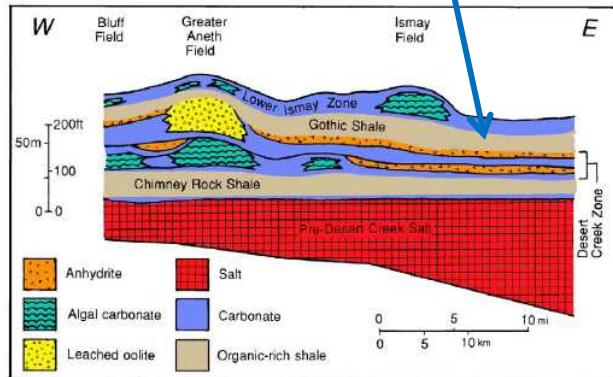


Figure 7-1. Diagrammatic lithofacies east-west cross section across Greater Aneth and Ismay fields, San Juan County, Utah. Modified from Peterson (1992).

Source: Chidsey et al., 2009

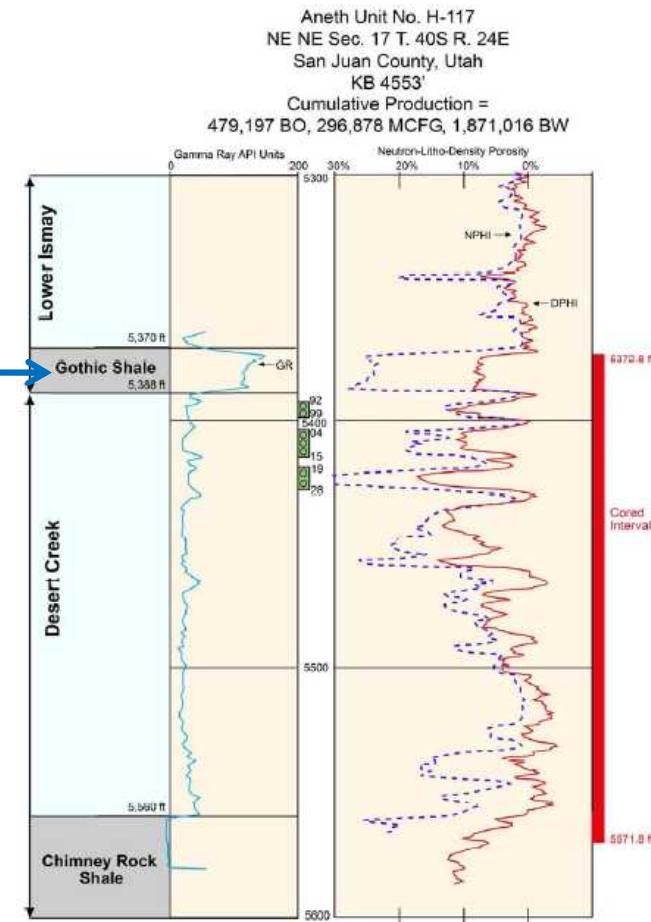


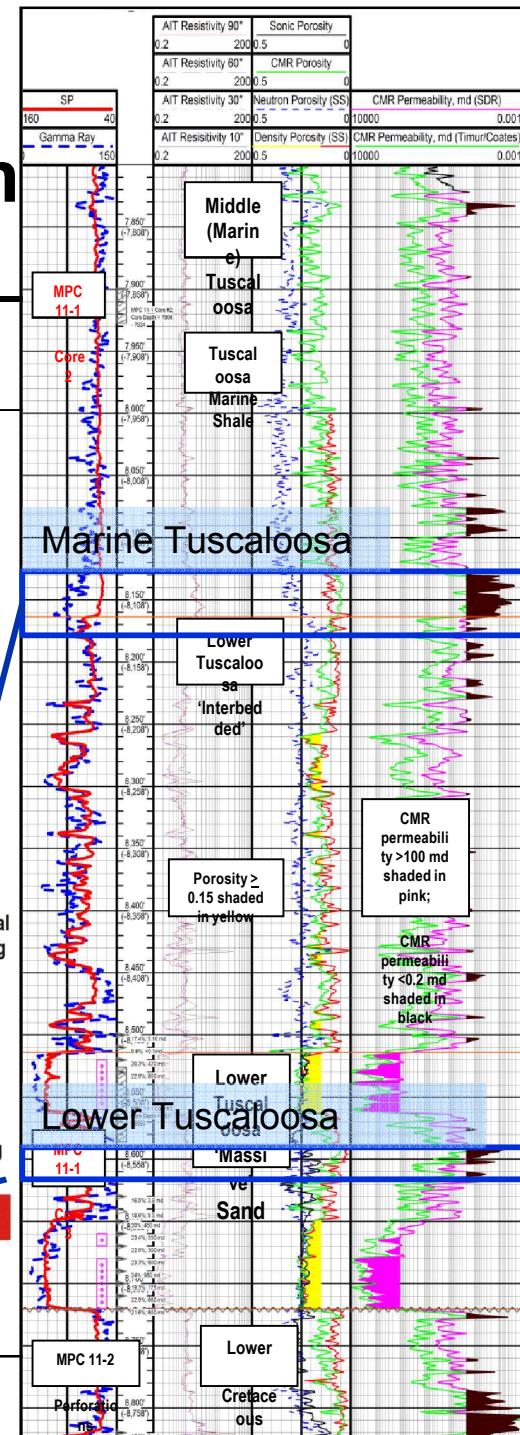
Figure 7-3. Interpreted geophysical log from the Aneth Unit No. H-117 well, NE NE section 17, T. 40 S., R. 24 E., SLBL&M. Cored interval of the Desert Creek zone and Gothic shale shown in red.

Marine and Lower Tuscaloosa Fm Cretaceous, US Gulf Coast



Samples from Mississippi Power Company Well #11-2, DOE- NETL's SEACARB Phase II Demonstration Site

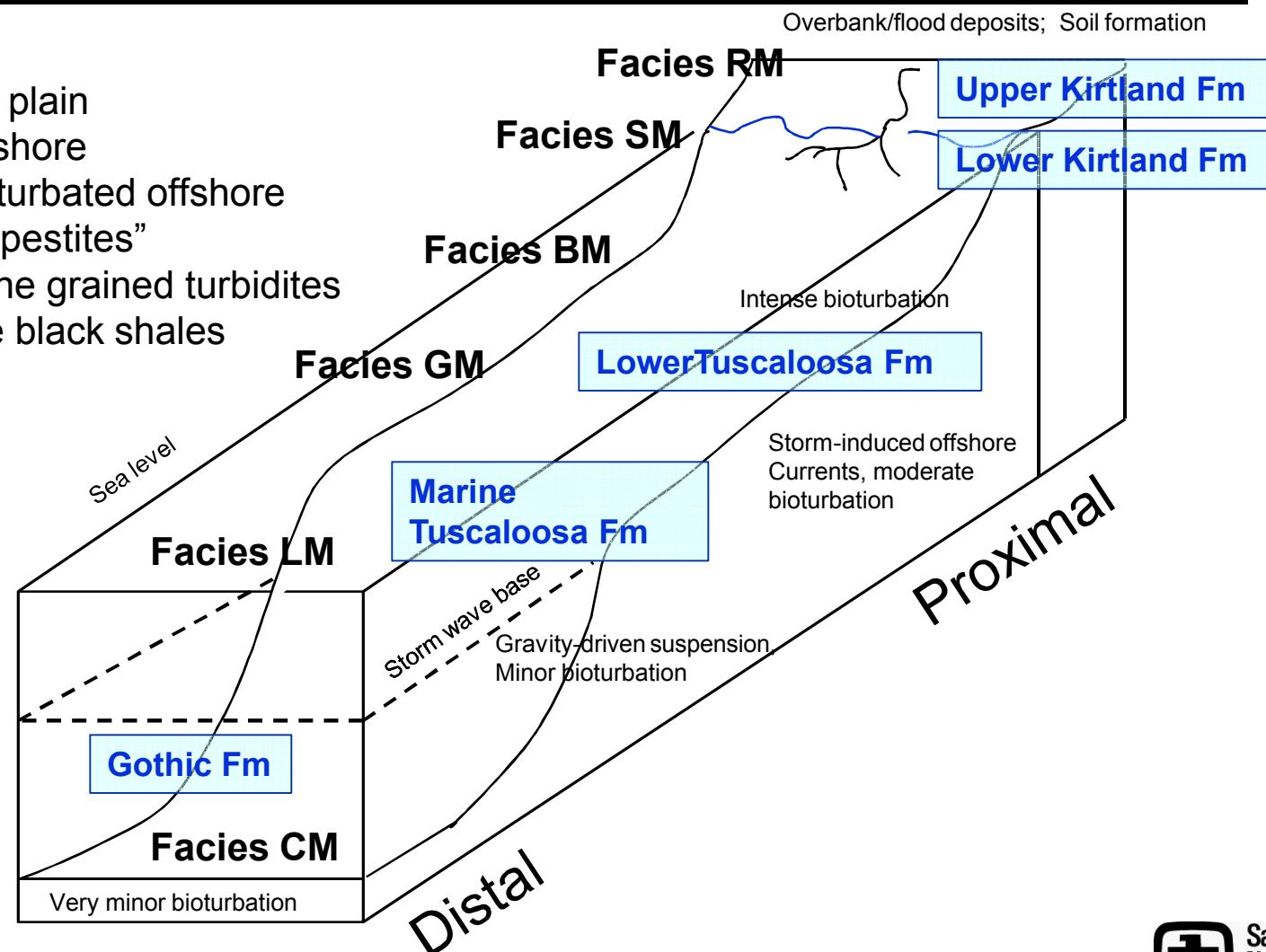
System	Series	Stratigraphic Unit		Stratigraphic Unit	Sub Units	So. Mississippi Potential Reservoirs and Confining Zones
		E. Texas	N. Louisiana			
Tertiary	Plio-cene			Misc. Miocene Units	Citronelle Fm.	Freshwater Aquifers
	Miocene				Graham Ferry Fm.	
	Oligo-cene				Pascagoula Fm.	Freshwater Aquifers
	Eocene				Hattiesburg Fm.	
	Paleo-Cene				Catahoula Fm.	
		Frio	Frio		Frio	Saline Reservoir
		Vicksburg	Vicksburg		Vicksburg	Minor Reservoir
		Jackson	Jackson		Jackson	Saline Reservoir
		Claiborne Grp	Claiborne Grp		Claiborne Grp	Saline Reservoir
		Wilcox Grp	Wilcox Grp		Wilcox Grp	Saline Reservoir
Cretaceous		Midway Shale	Midway Shale	Midway Shale		Confining Unit
		Navarro Fm	Nacotoch	Selma Group	Prairie Bluff	
		Taylor	Ozan/Annona		Ripley	
		Austin	Austin Fm. Eutaw Fm		Demopolis Chalk	
		Eagleford Group	Tuscaloosa Group		Topole SS & Arcadia	
		Woodbine			Mooreville Chalk	
		Buda Limestone	Washita Group	Eutaw Grp	Tombigbee SS	
		Georgetown			Eutaw Fm & Unnamed	Confining Unit
		Edwards/Fredricksburg				Saline Reservoir
		Paluxy	Paluxy	Tuscaloosa Group	Upper Tusc.	Minor Reservoir
					Marine Tusc.	Confining Unit
					Lower "Interbeded" Tusc.	Saline Reservoir
					Massive Sand	



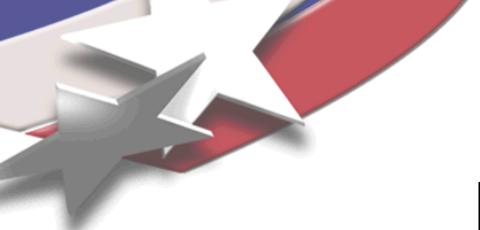
A Word on Mud Depositional Environments & Facies

Facies

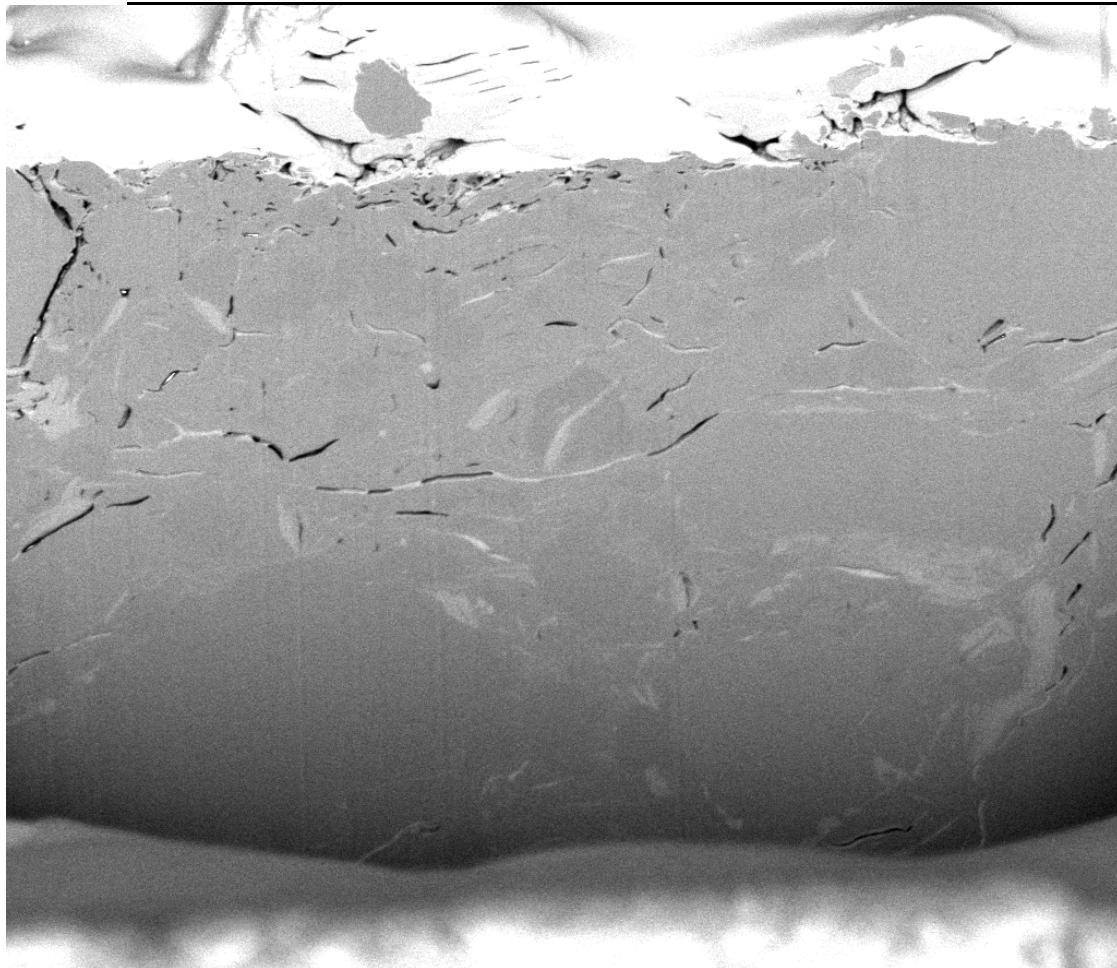
- RM – grey coastal plain
- SM – sandy near-shore
- BM - intensely bioturbated offshore
- GM – graded “tempestites”
- LM – laminated, fine grained turbidites
- CM – deep marine black shales



Simplified from Schieber, 1998

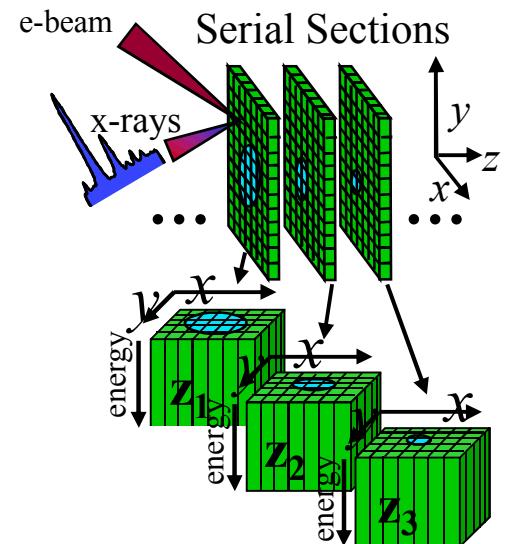
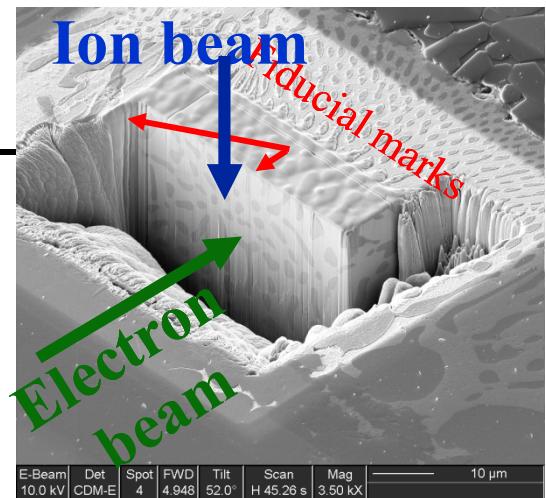


FIB/SEM Animation



1024x884 pixels, 1 pixel = 15.6 nm, 400 slices at 25 nm per slice

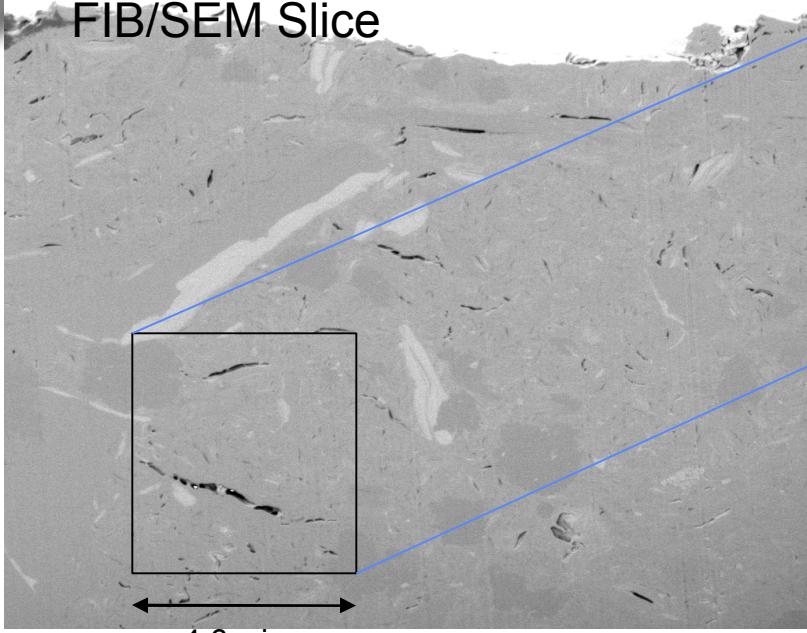
Lower Kirtland Shale, 2692 ft bgs



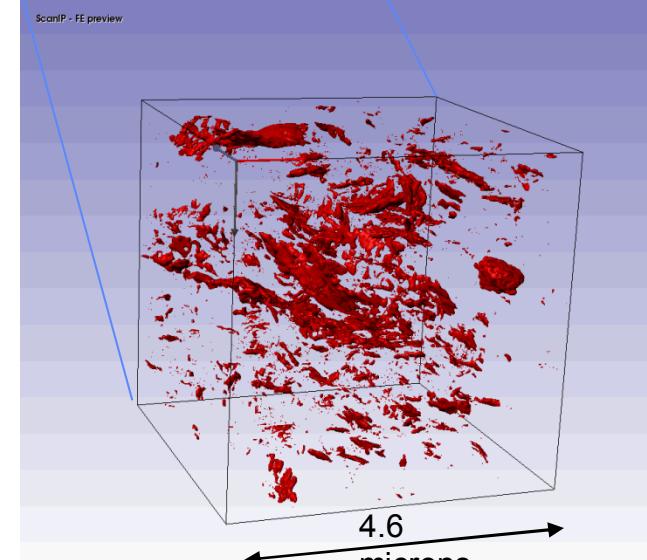
Upper Kirtland Shale @ 2049 ft bgs

1024x884 pixels, 1 pixel = 15.6 nm

FIB/SEM Slice



Registered,
cropped, and
thresholded slice
(299x299 pixels)

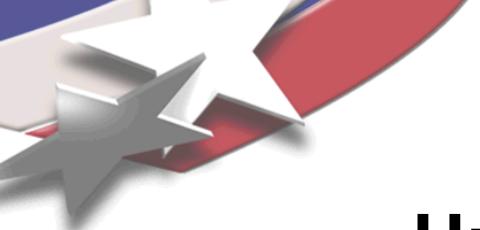


3D reconstructed pores

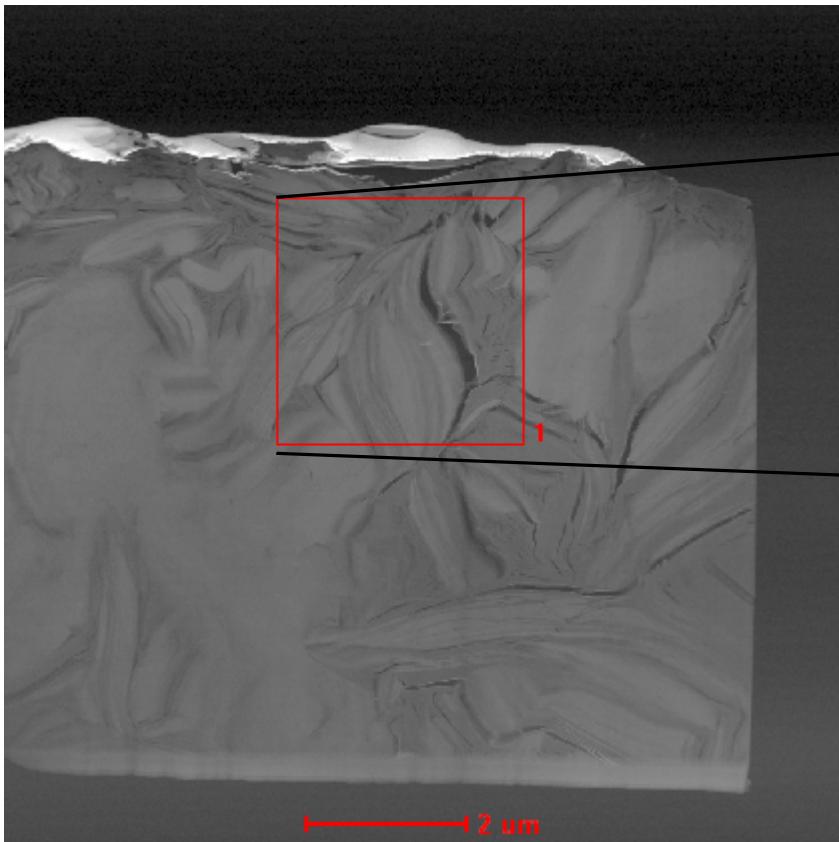
Medial Axes of Pore Networks

Pore throats

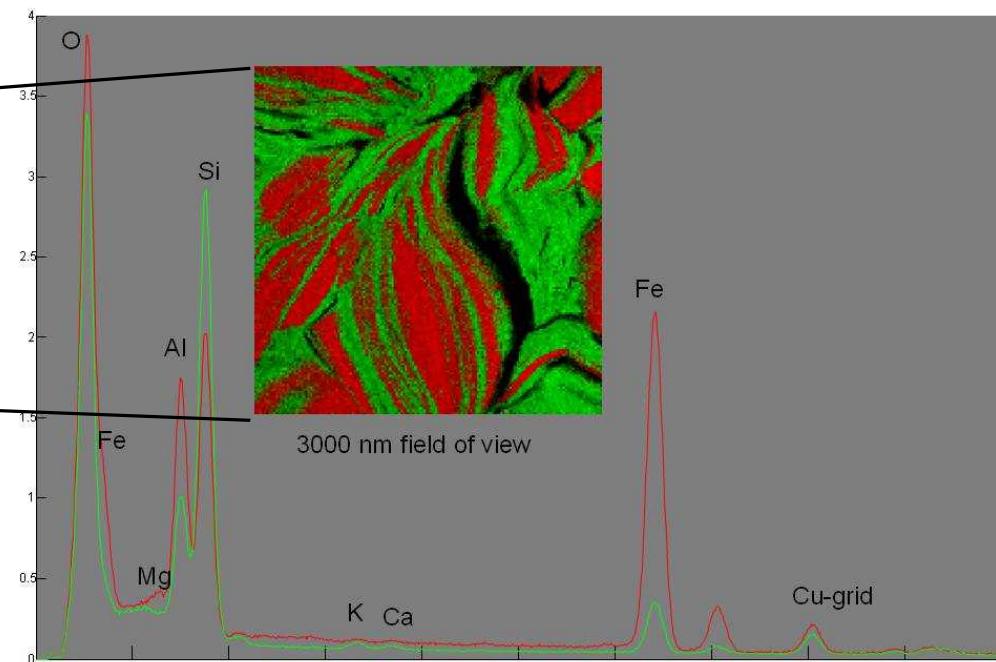
1.04% porosity
37% connected



Upper Kirtland Shale TEM-EDS

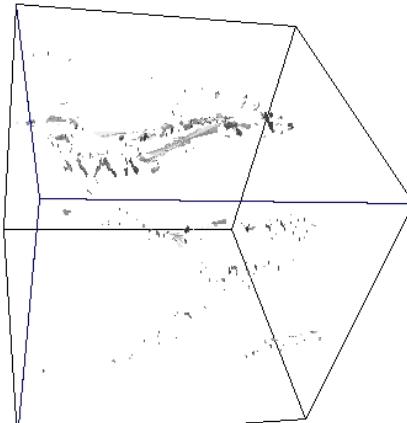
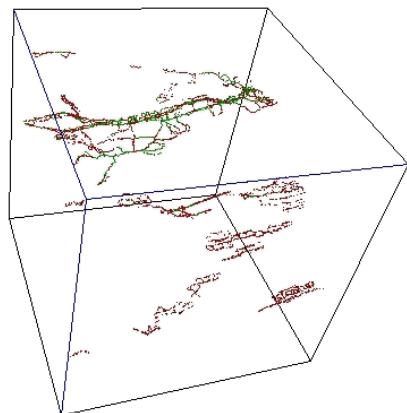
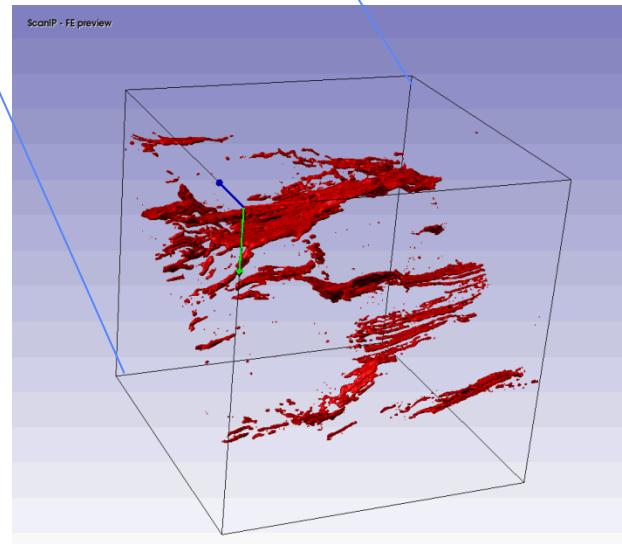
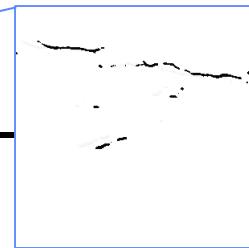
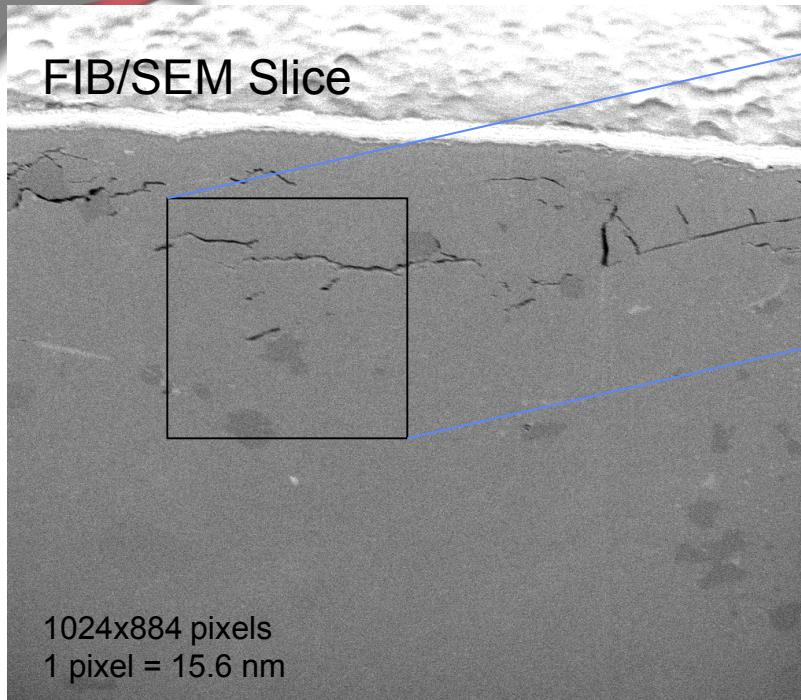


Dewars_Nov2009_2049-7B_SI1_3000nm150pix_1-out-4



Compacted clay floccules
of Scheiber and ???

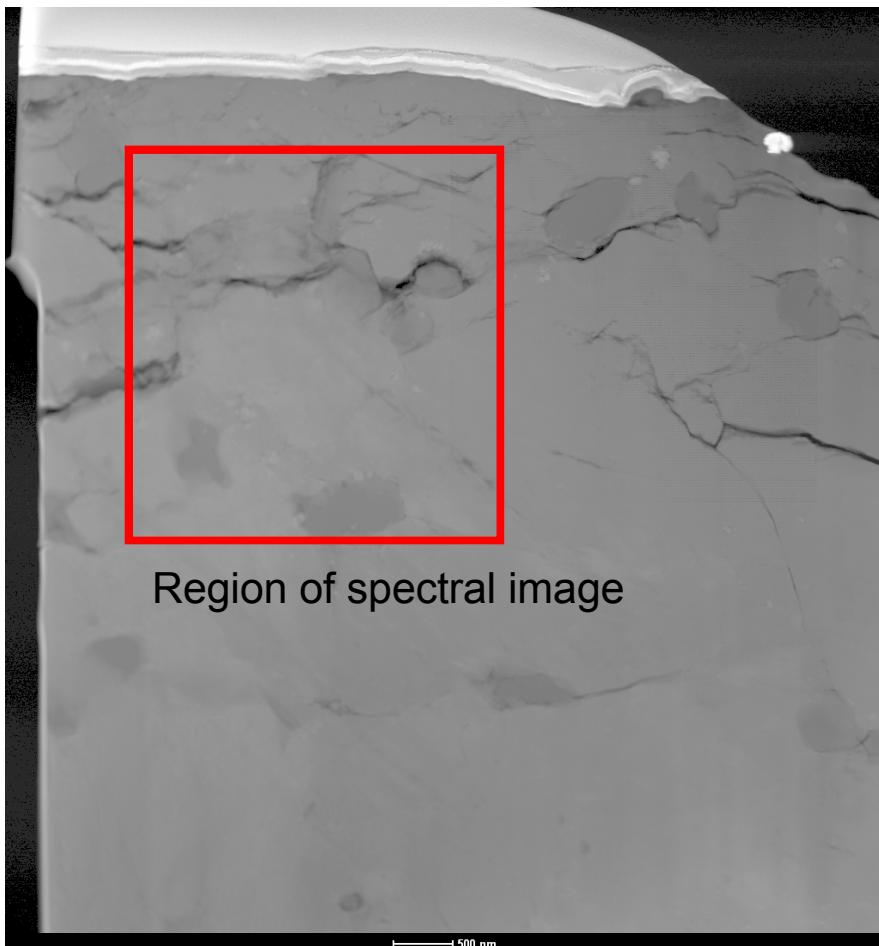
Lower Kirtland Shale @ 2692 ft bgs



Porosity = 0.722%
Connectivity = 28%

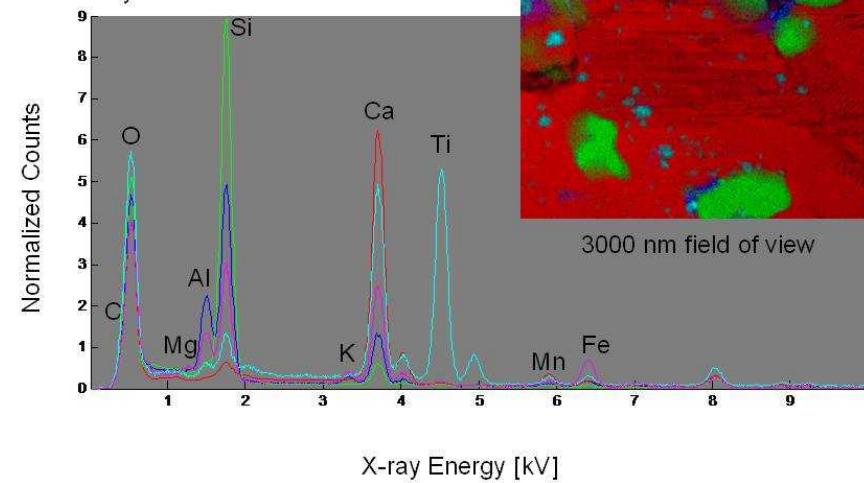


Lower Kirtland Shale @ 2692 ft bgs

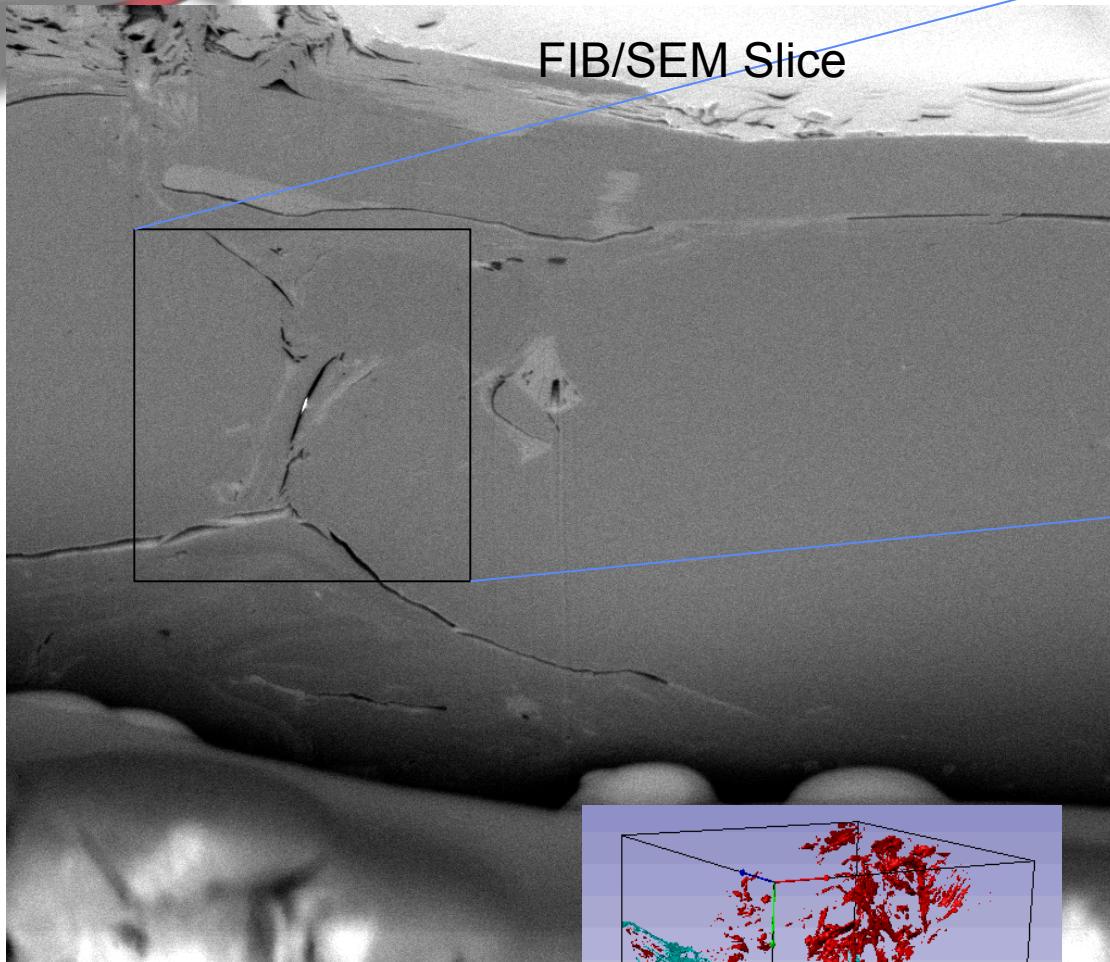


Dewars_Nov2009_2692_9A_SI2_3000nm300pix_1-out-8 (10 nm/pixel)

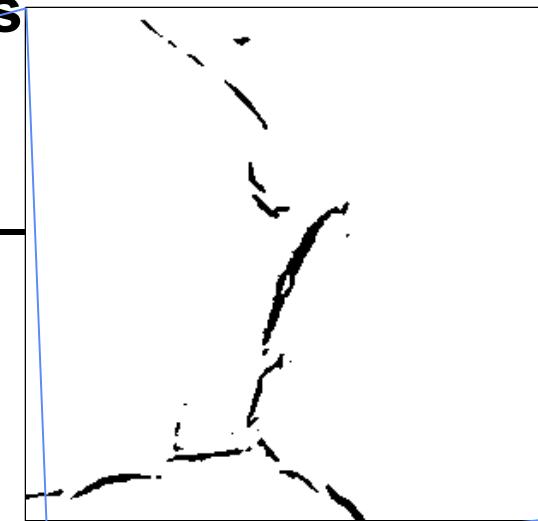
Red = Ca-C-O-Mn-Fe
Green = Si-O
Blue = Si-Al-Ca-K-Fe-Mn-O
Magenta = Si-Al-Mg-Ca-K-Fe-Mn-O
Cyan = Ti-Ca-Si-O



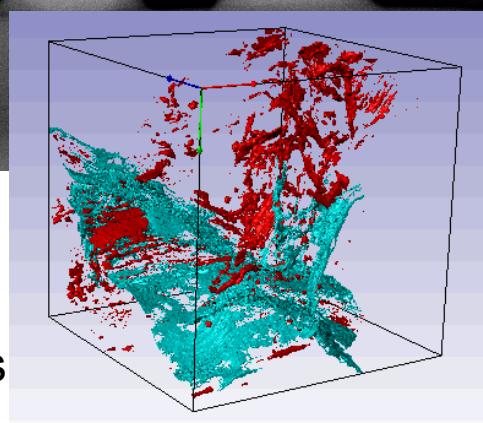
Lower Tuscaloosa, 8590 ft bgs



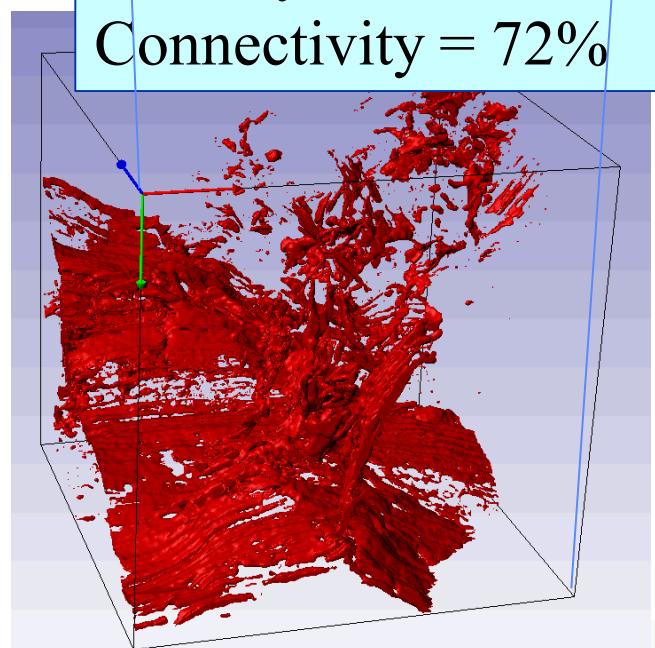
FIB/SEM Slice



Porosity = 2.64%
Connectivity = 72%

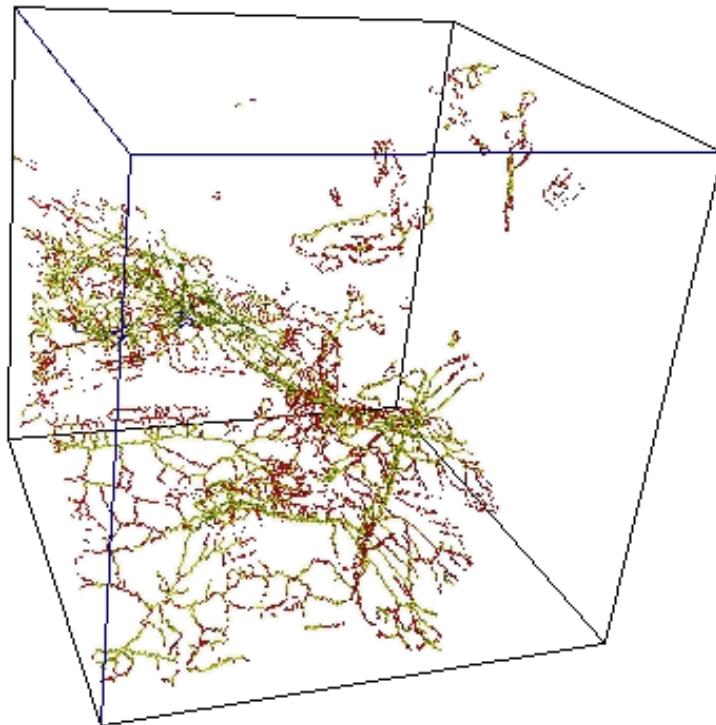


Connected (blue) and
unconnected (red) pores

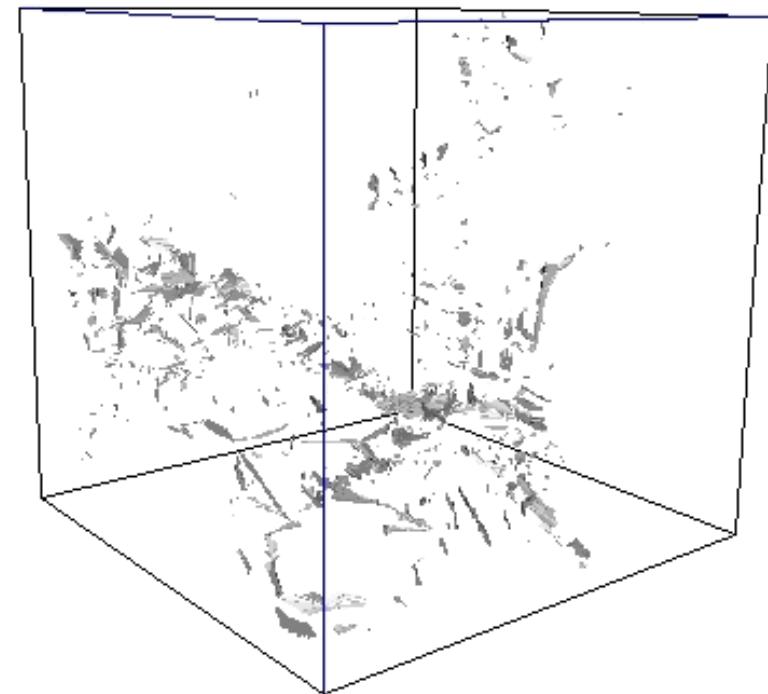




Lower Tuscaloosa, 8590 ft bgs



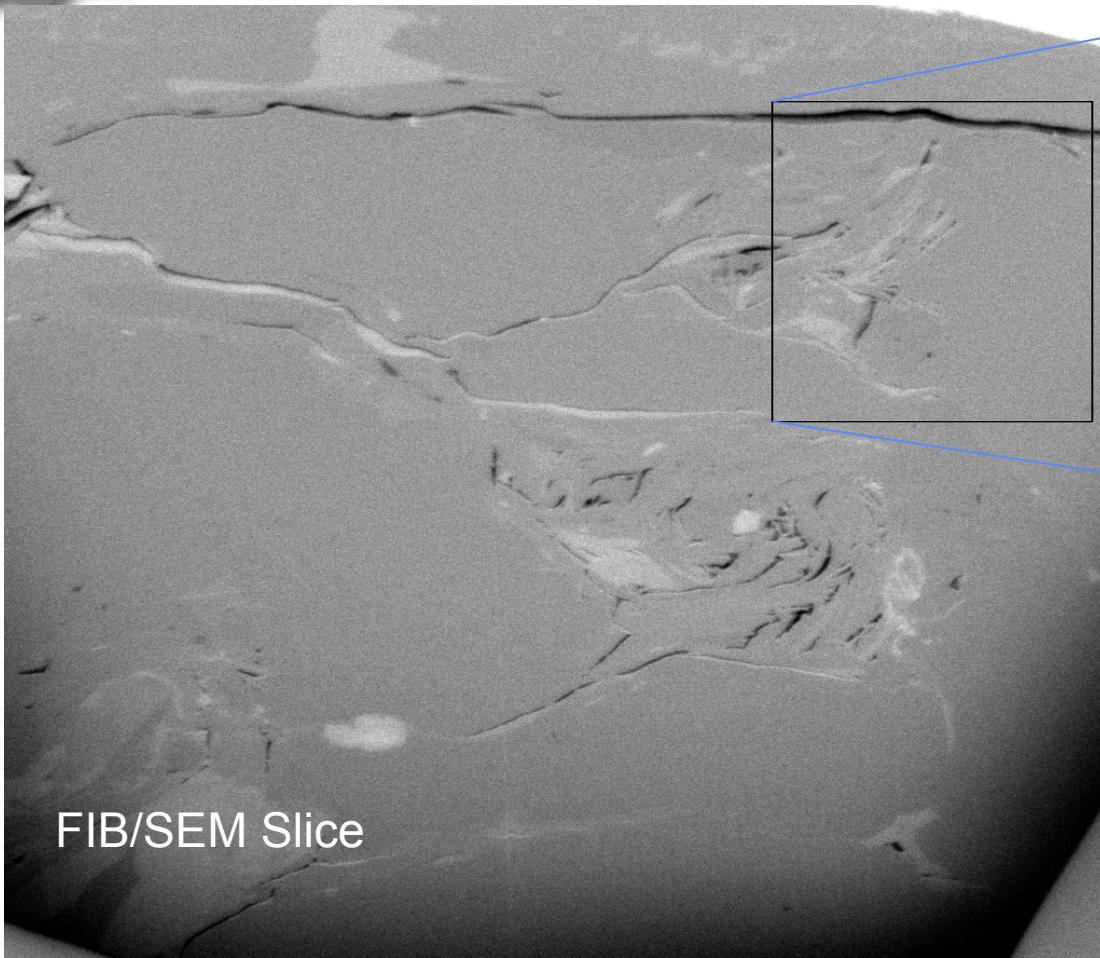
Medial Axes of Pore Networks



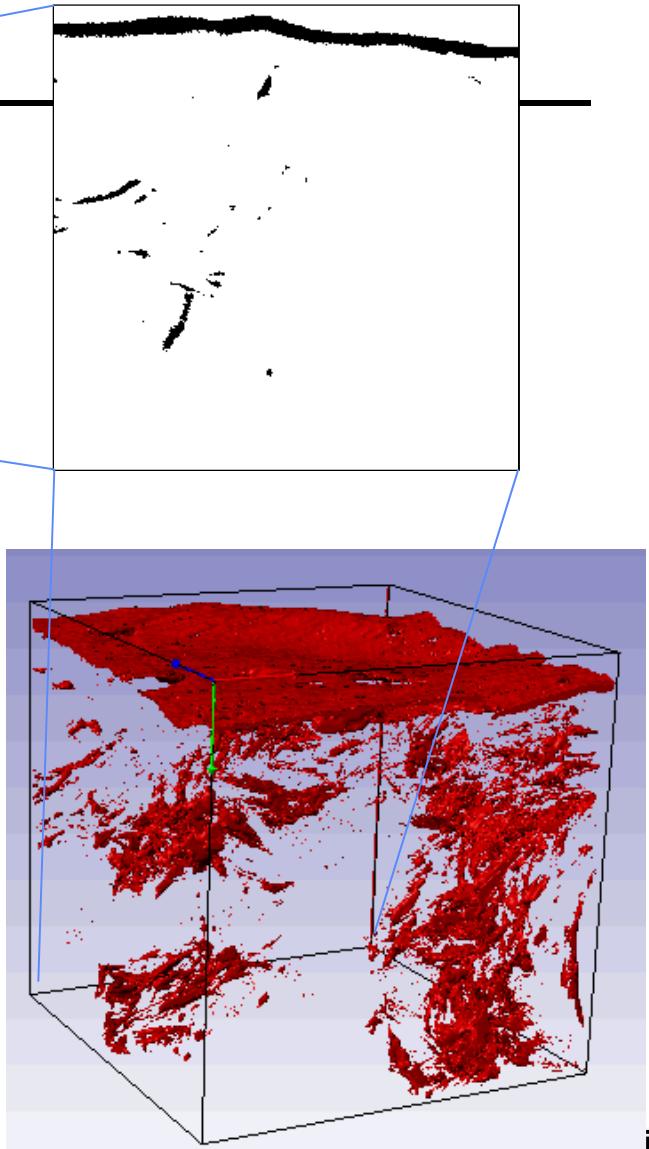
Pore throats



Lower Tuscaloosa @ 8590 ft bgs

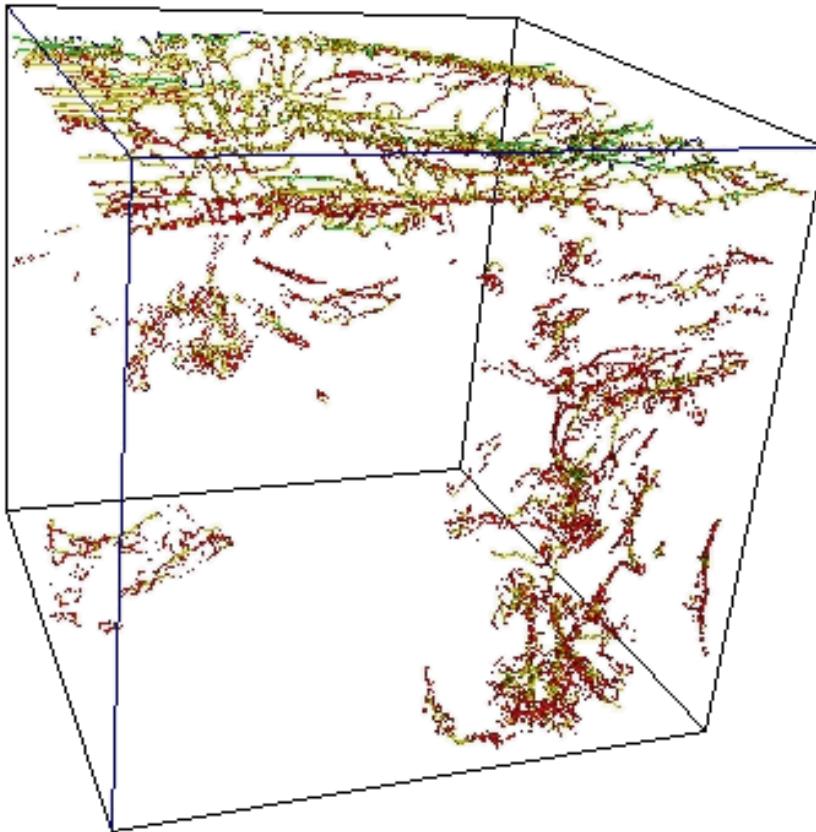


Porosity = 2.93%
Connectivity = 34%

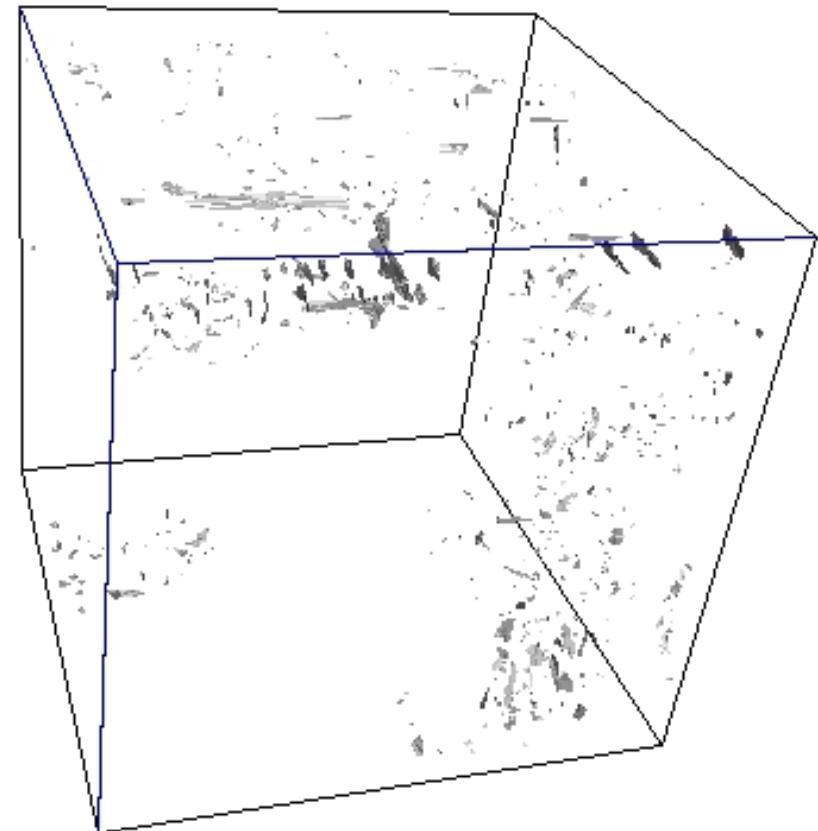




Lower Tuscaloosa @ 8590 ft bgs



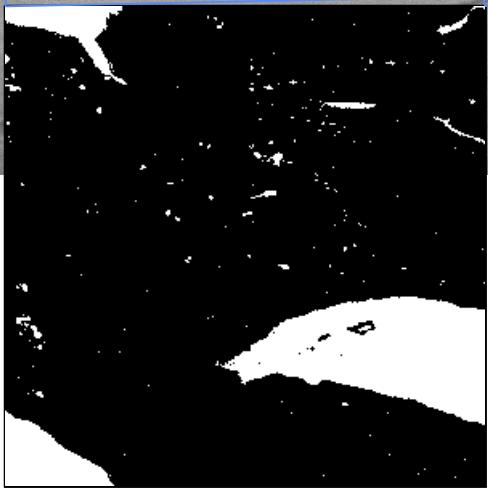
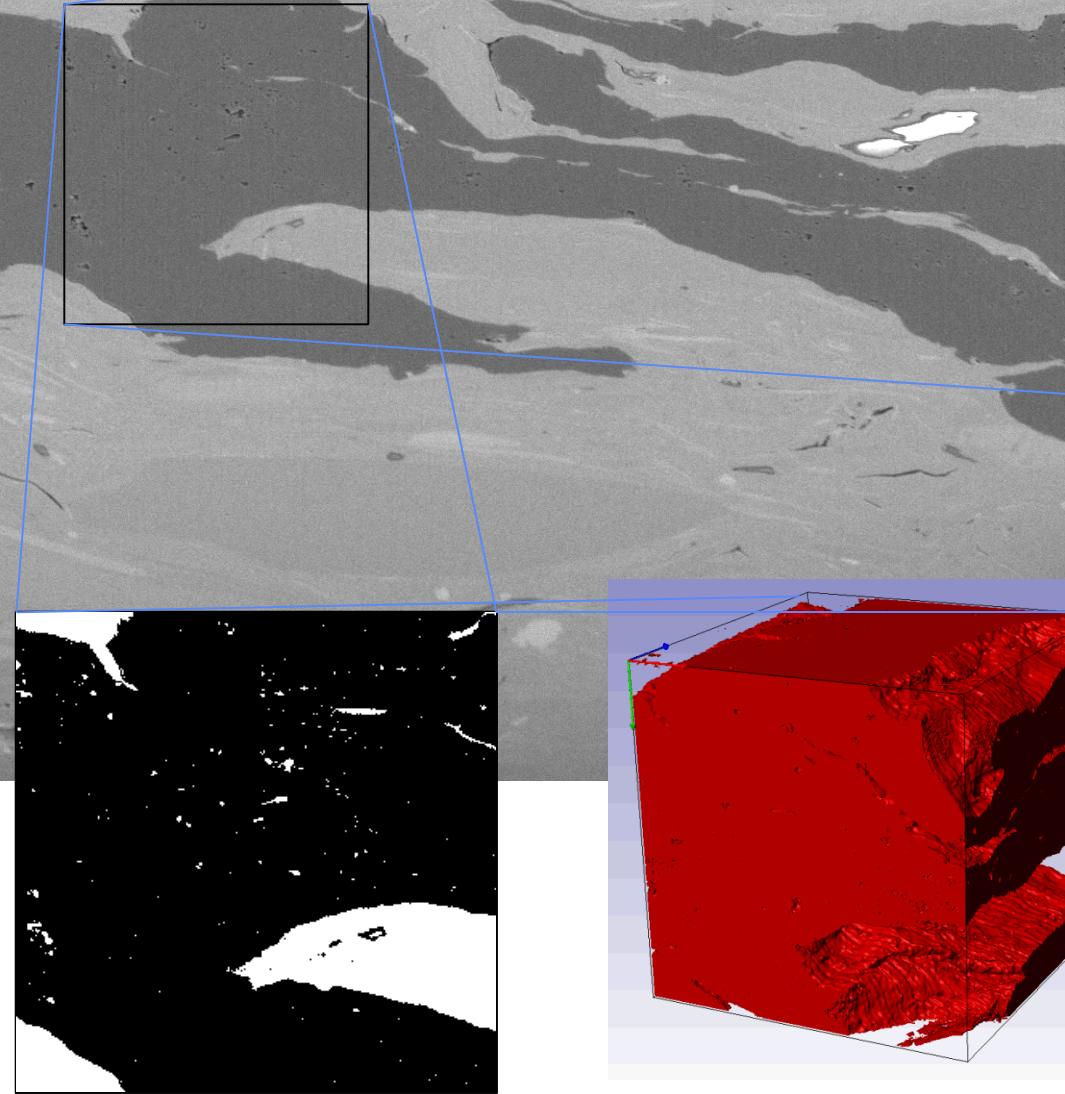
Medial Axes of Pore
Networks



Pore throats

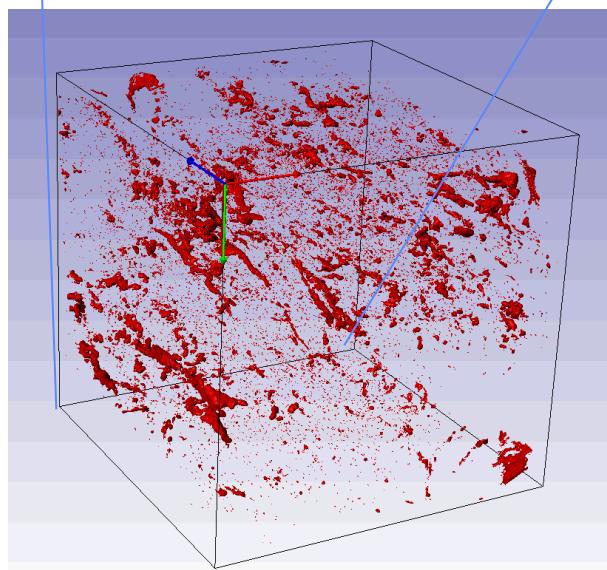
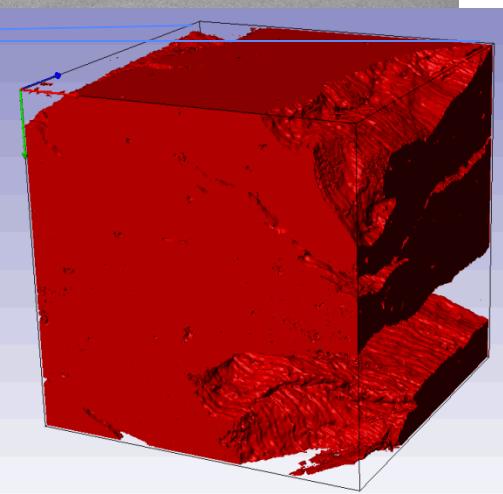
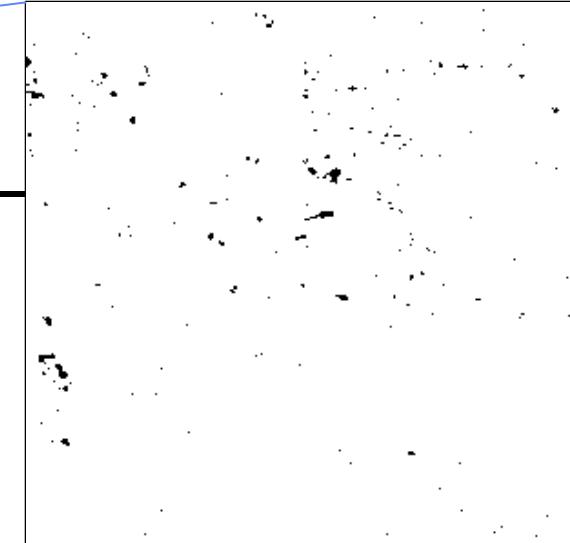
Marine Tuscaloosa @ 7925 ft bgs

FIB/SEM Slice



Organic phase vol. fraction = 85.01%

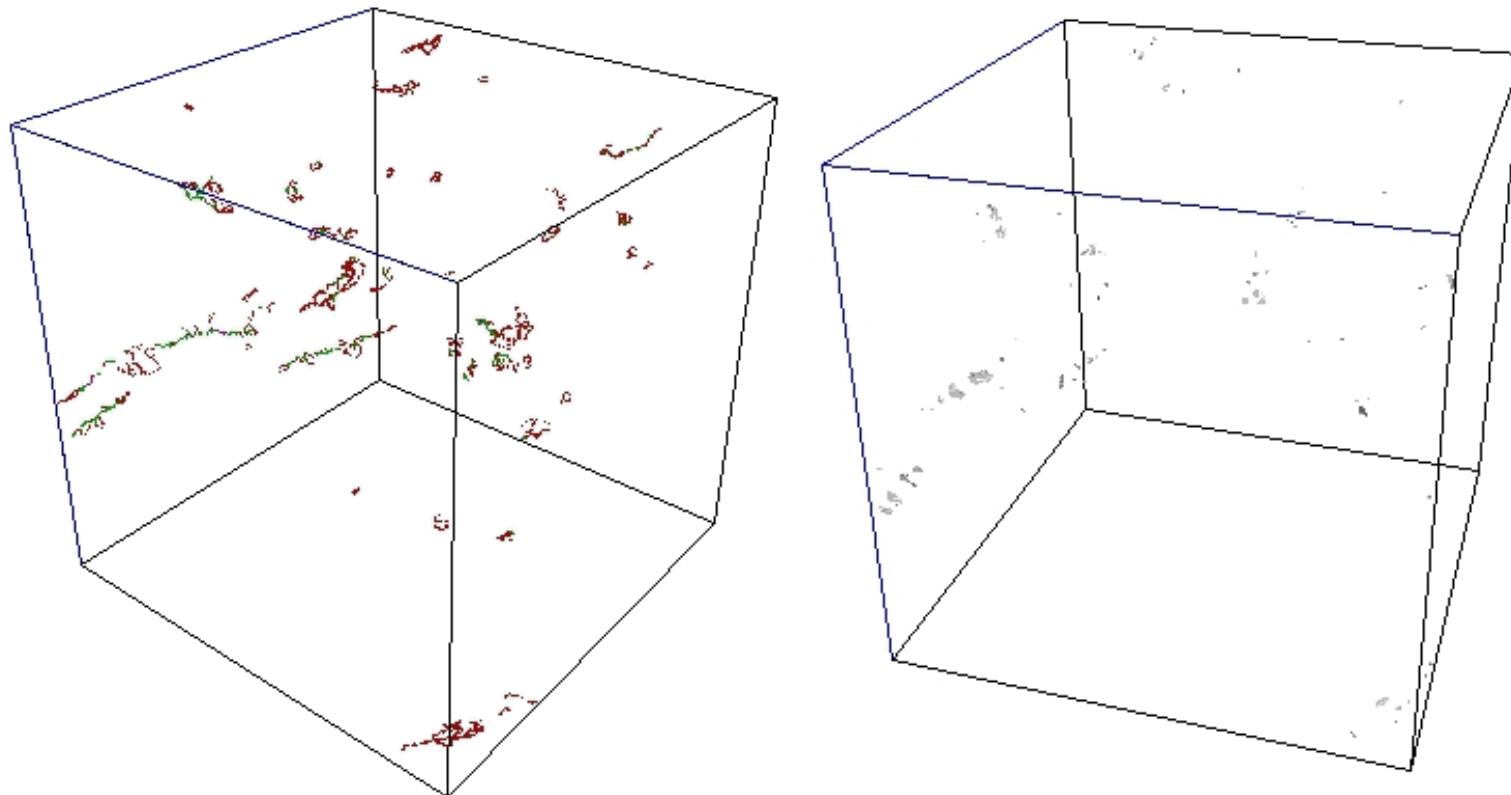
Registered, cropped, and
thresholded slice (299x299 pixels)



Porosity = 0.575
Connectivity = 62%

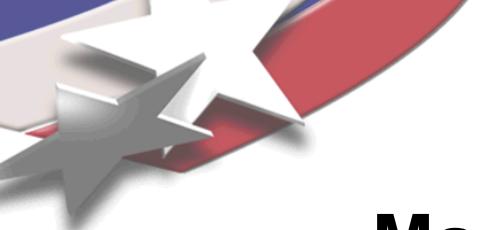


Marine Tuscaloosa @ 7925 ft bgs

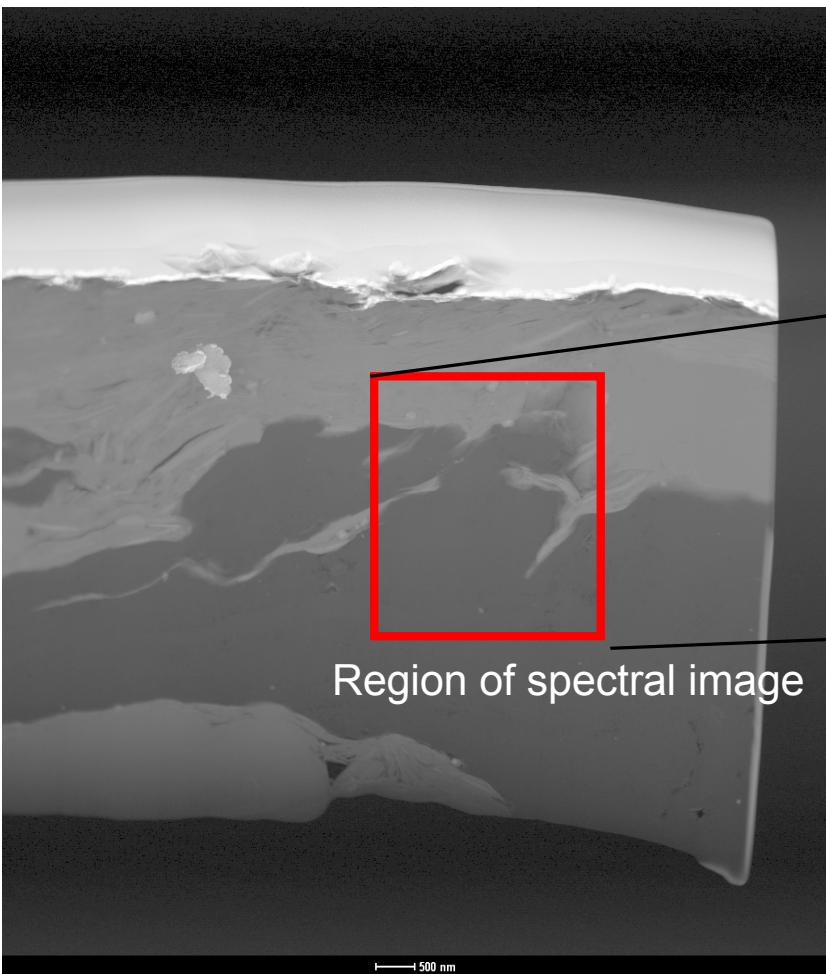


Medial Axes of Pore Networks

Pore throats

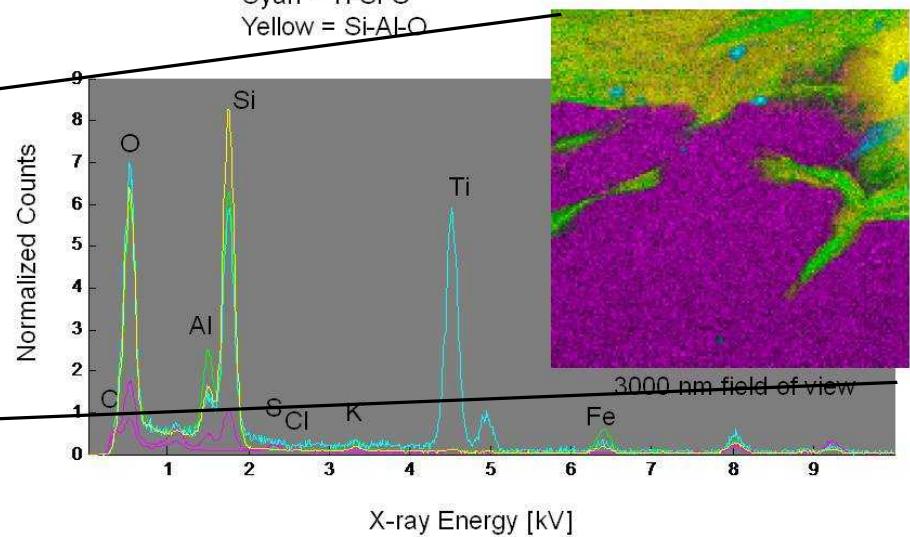


Marine Tuscaloosa @ 7925 ft bgs

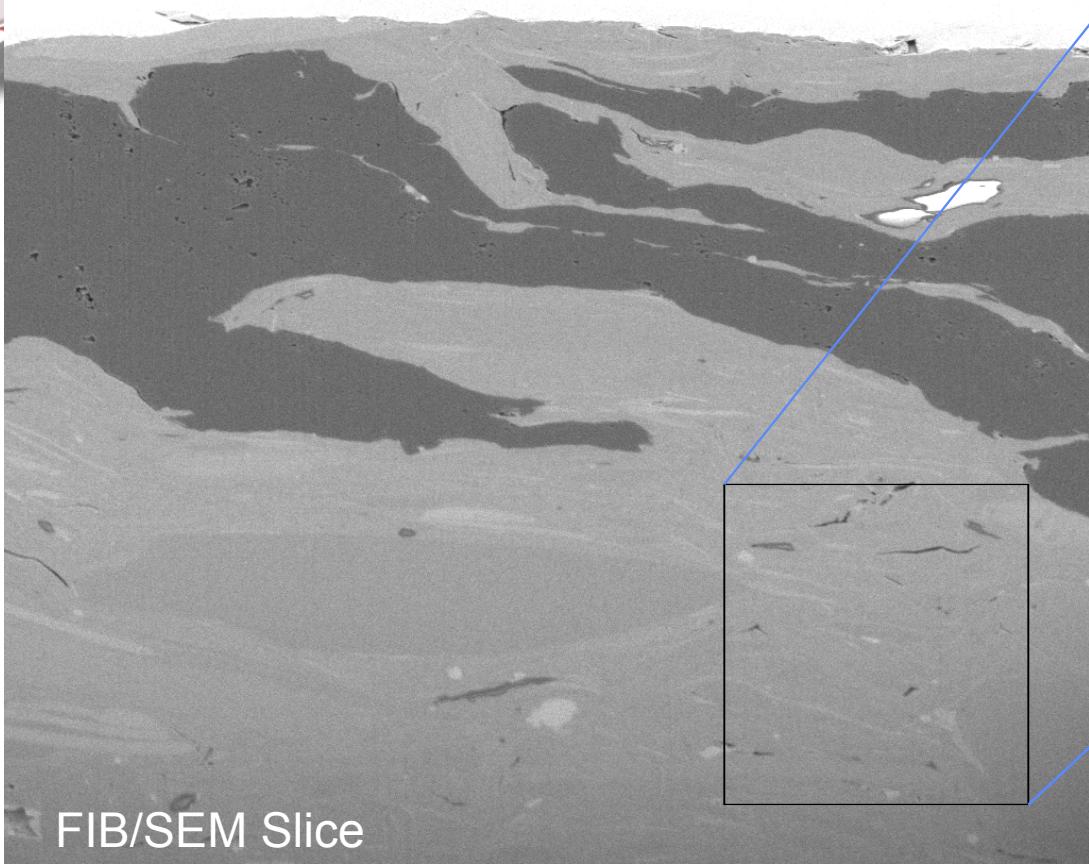


Dewars_Nov2009_7925_5_SI1_3000nm150pix_1-out-5 (20nm/pixel)

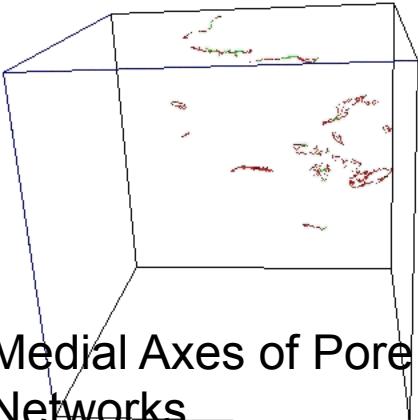
Green = Si-Al-K-Fe-O
Blue = Ca-P-O
Magenta = C-O-S-Cl
Cyan = Ti-Si-O
Yellow = Si-Al-O



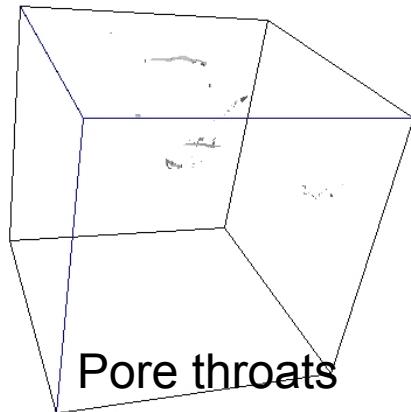
Marine Tuscaloosa @ 7925 ft bgs



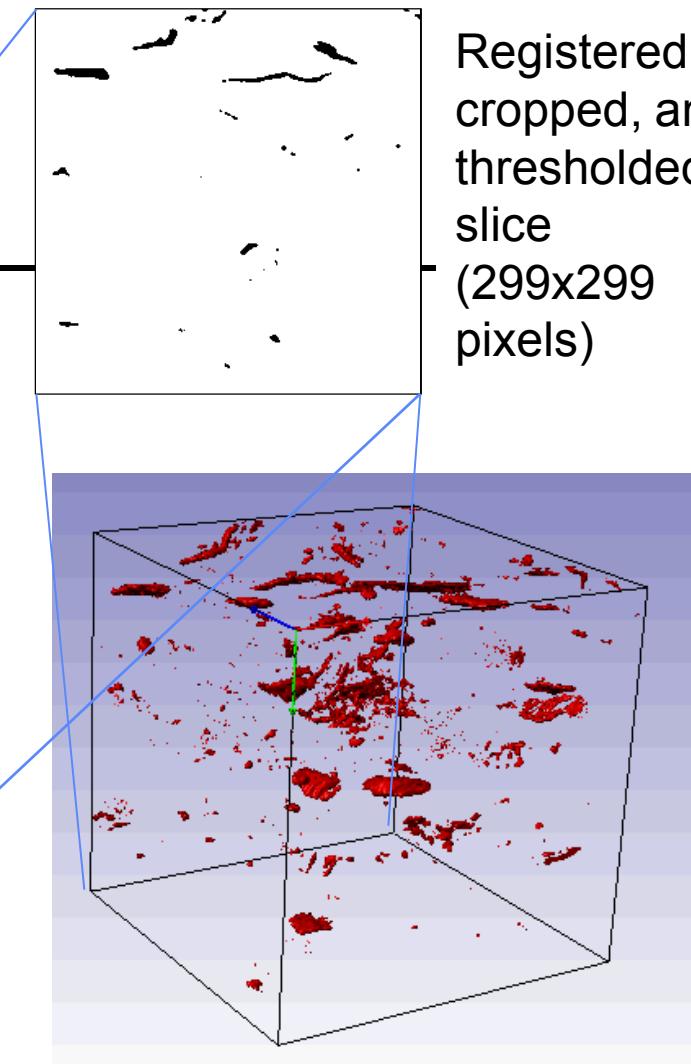
FIB/SEM Slice



Medial Axes of Pore Networks



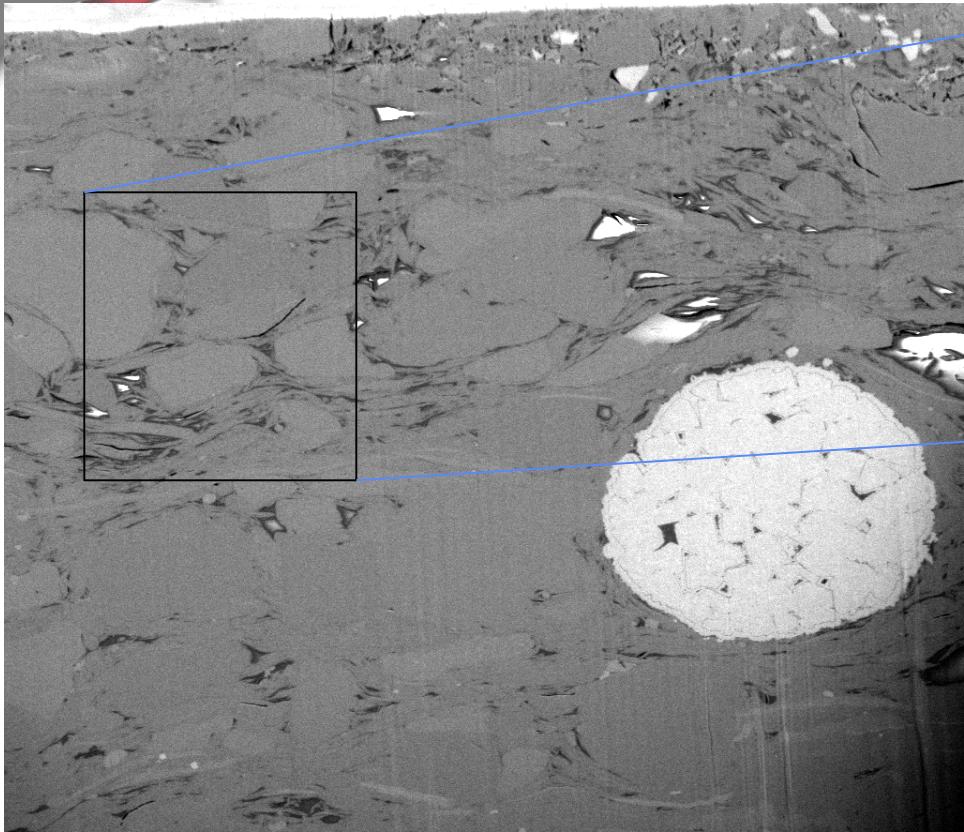
Pore throats



Registered,
cropped, and
thresholded
slice
(299x299
pixels)

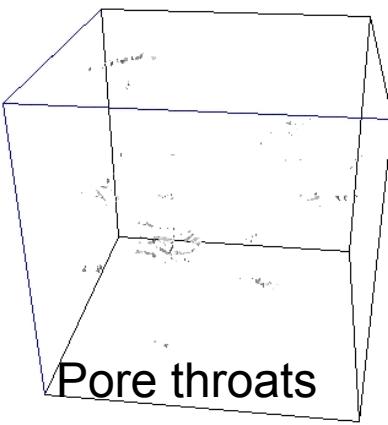
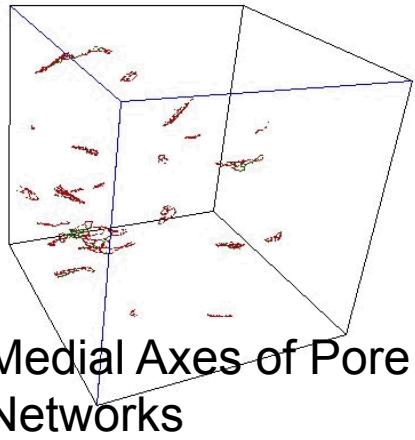
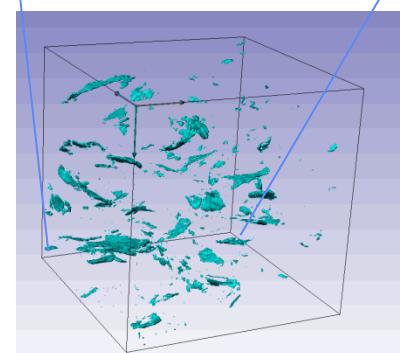
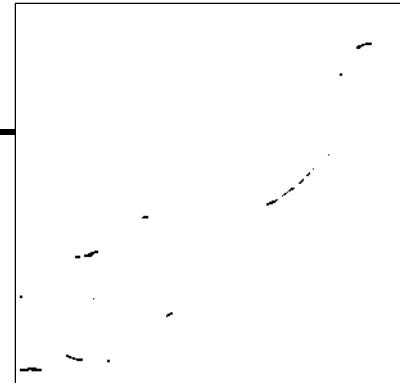
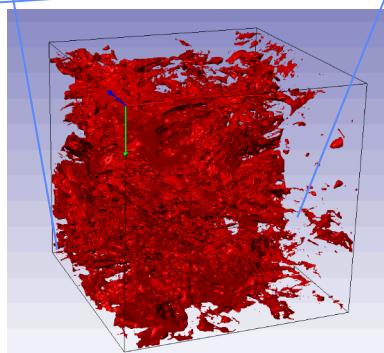
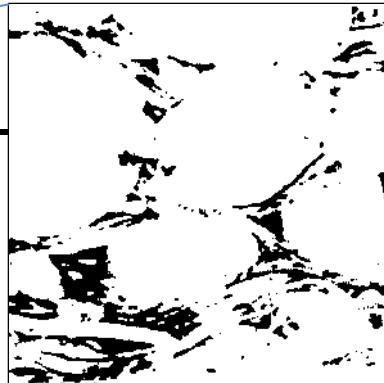
Porosity = 0.47%
Connectivity = 52%

Gothic @ 5390 ft bgs



Organics plus porosity

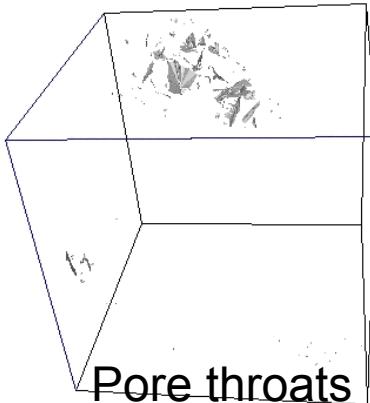
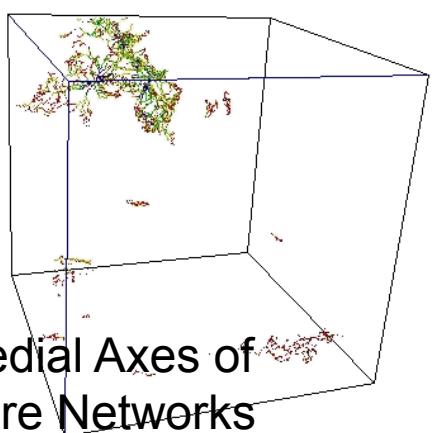
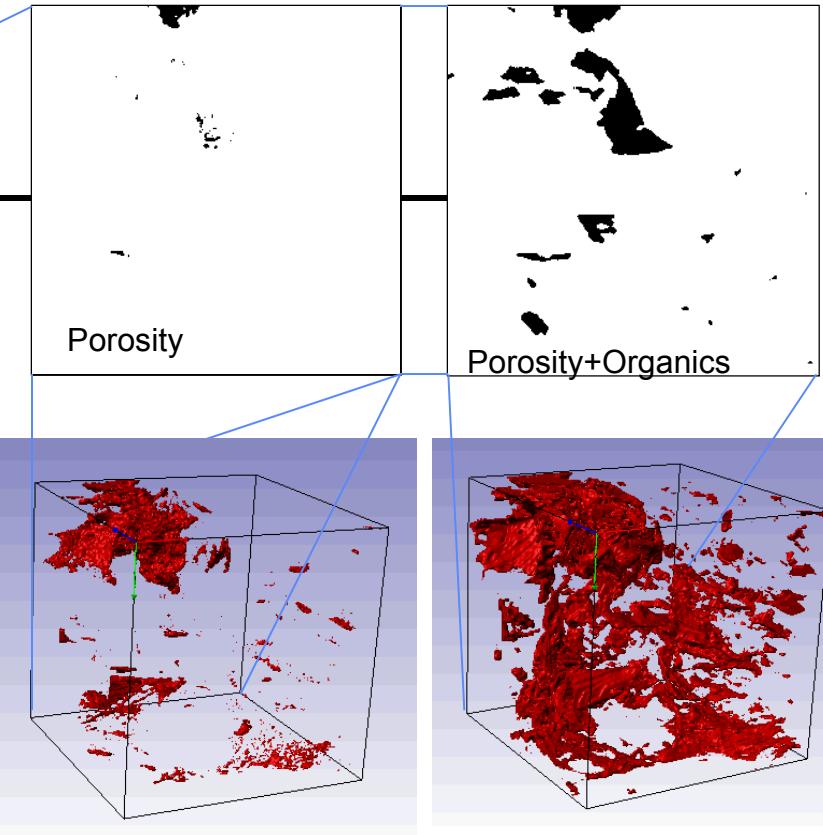
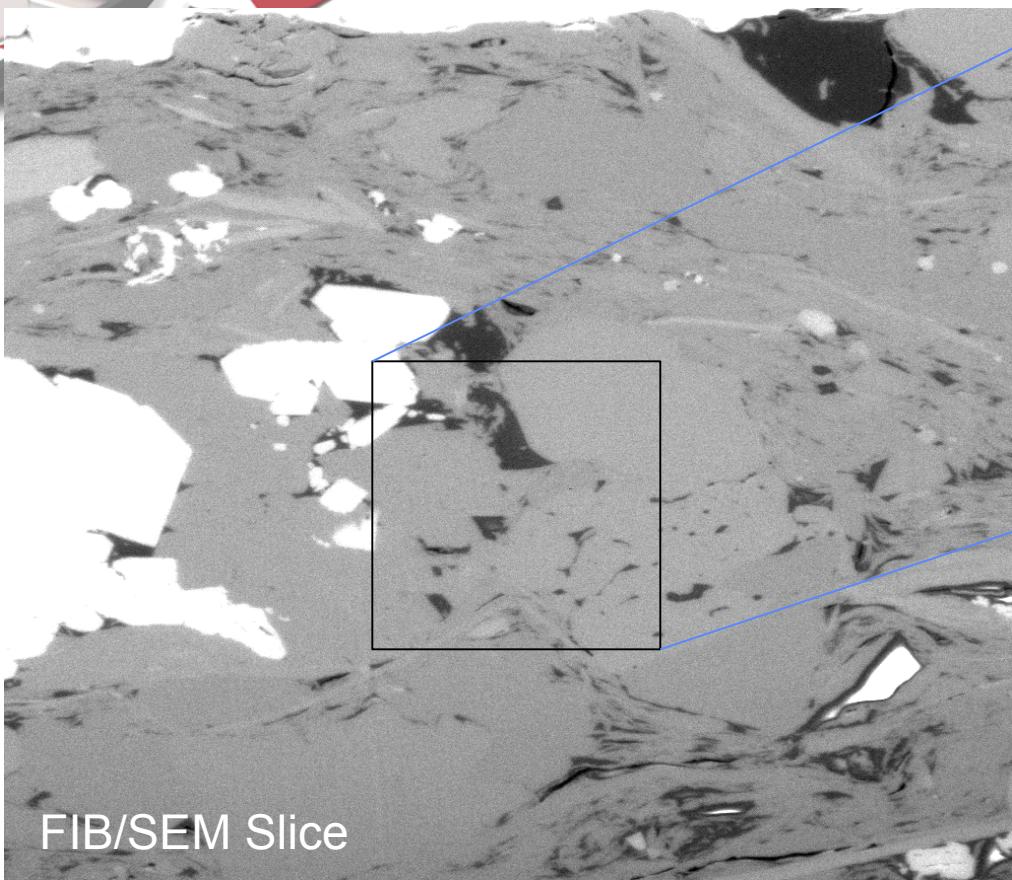
Porosity



15.2% Vol.
Fraction

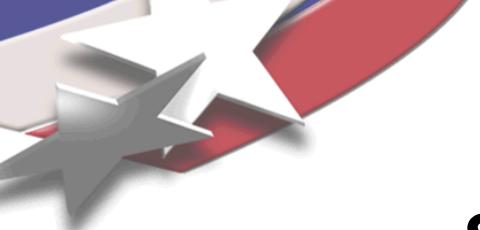
0.42 % Porosity
Connectivity =
44%

Gothic @ 5390 ft bgs



Porosity =
1.33%

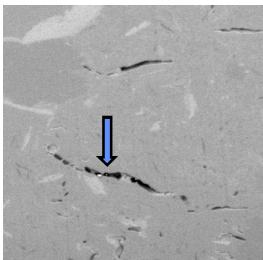
Vol. % =
5.59



Shale “Nano” Pore Networks

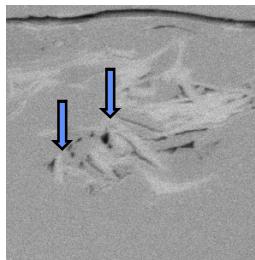
Type I*

Elongated pores between similarly oriented clay sheets¹



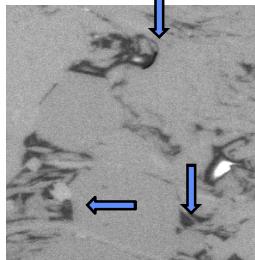
Type II*

Crescent-shaped pores in “saddle reefs” of folded clay sheets



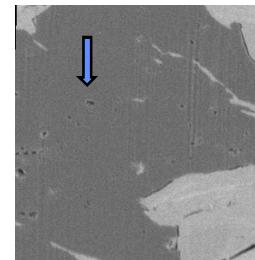
Type III*

“Jagged” pores in compaction shadows around larger clasts



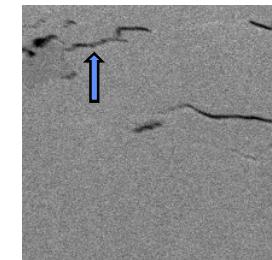
Type IV

Tubular pores in “foamy” organics



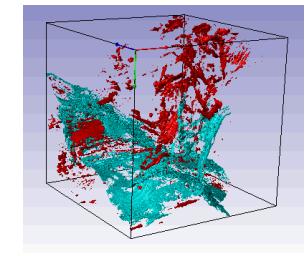
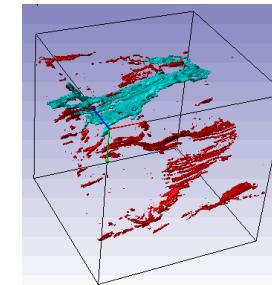
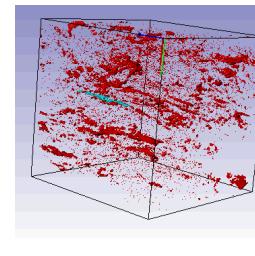
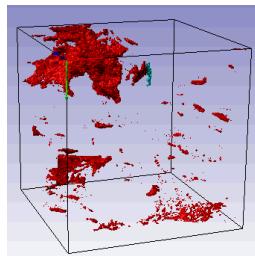
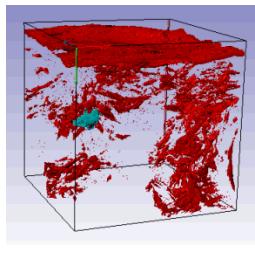
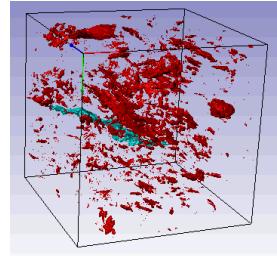
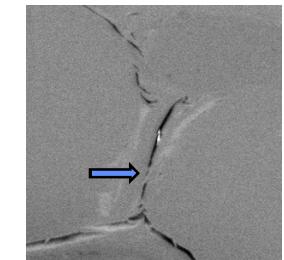
Type V

Diagenetic; clay seams or microstylolitic; grain dissolution, etc.



Type VI

Microfracture related; may or may not be induced



← **Remnant Pore Types** →

← **Secondary Pore Types** →

*As proposed by Desbois et al., 2009

Pore Statistics: Lower Tuscaloosa

Mercury Porosimetry: $D = 2.86$

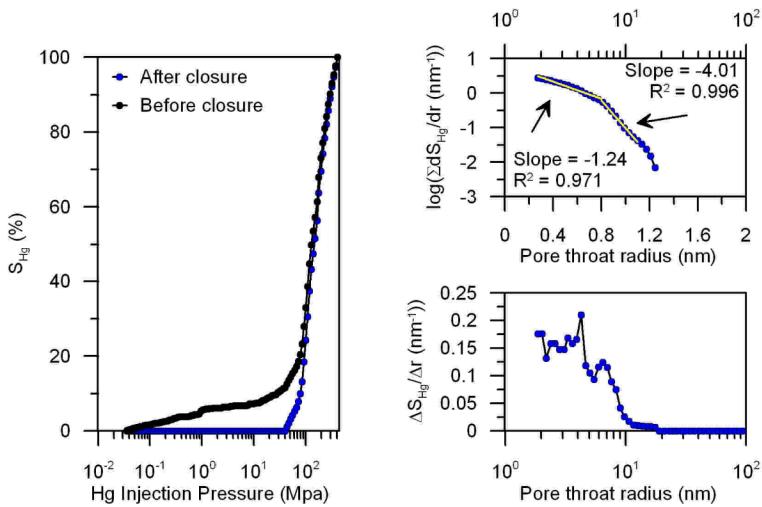
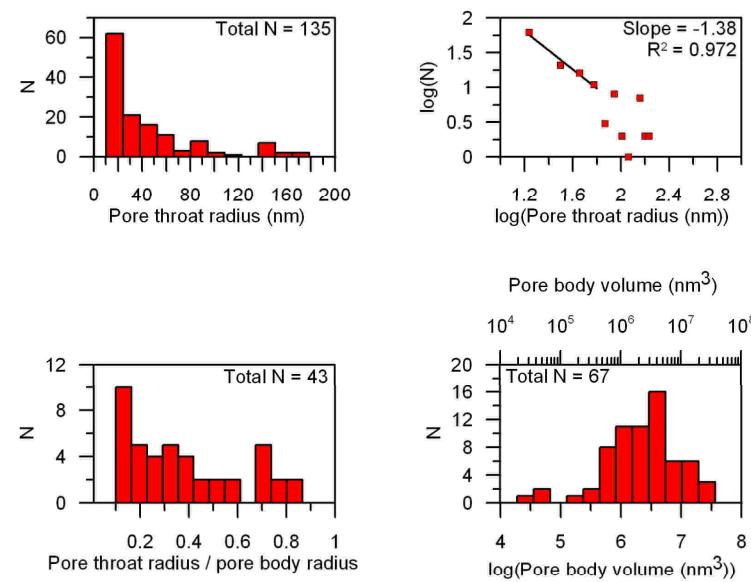


Image-based pore distributions: $D = 2.54$



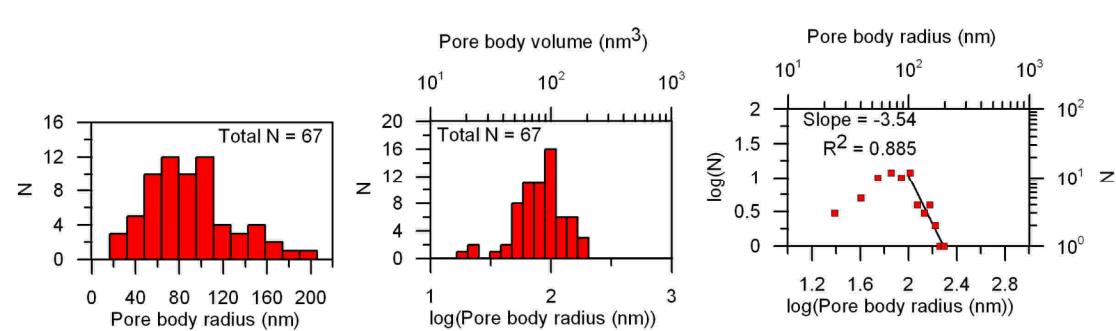
Number Distribution

$$\log f(r) = - (D+1) \log r_{\text{pore volume}}$$

Mercury Intrusion Volume

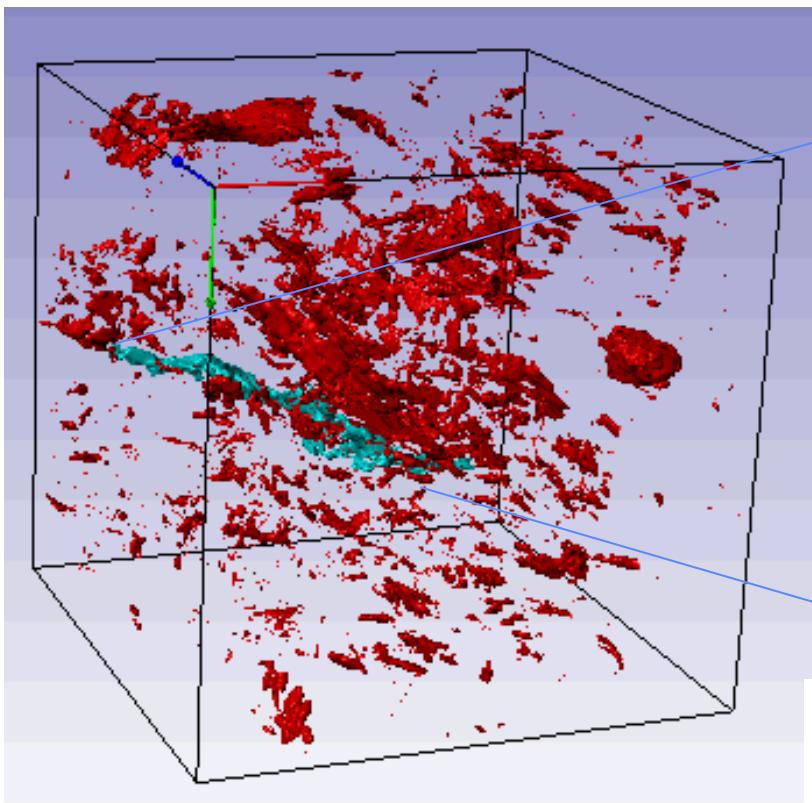
$$\log V_{Hg}(r) = (3-D) \log r_{\text{pore throat}}$$

Bartoli et al., 1990; Han et al., 2006

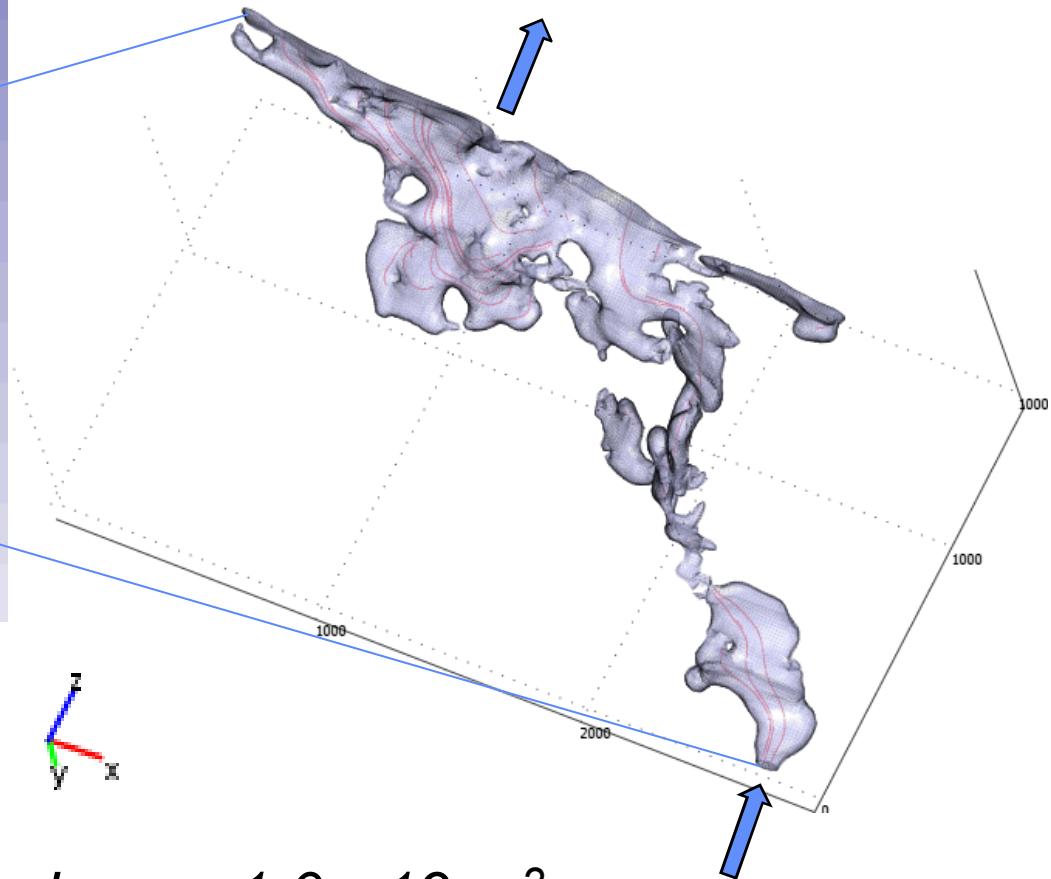


CFD Simulations

Type I Pore; Upper Kirtland Shale @ 2049 ft bgs



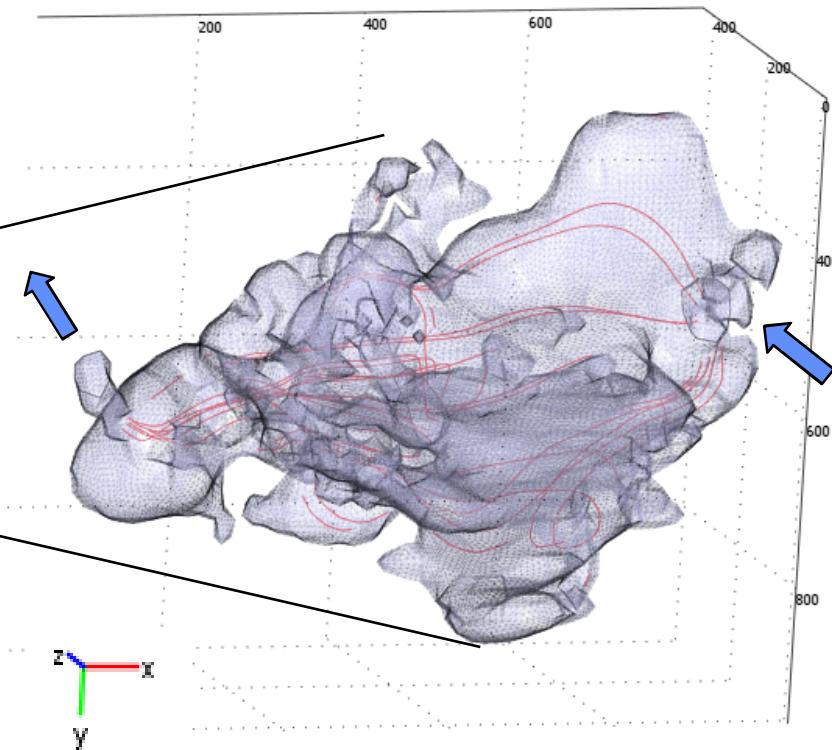
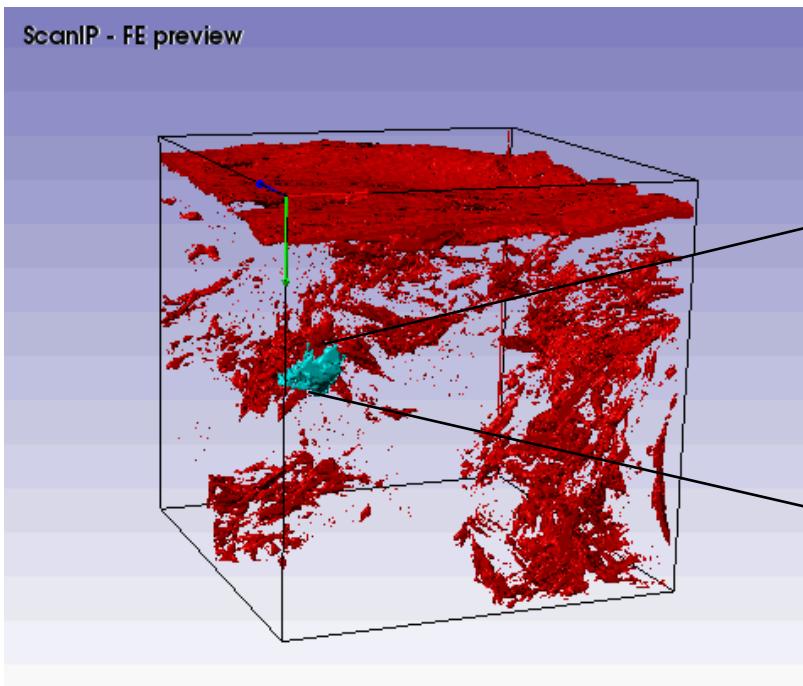
Upper Kirtland Klinkenberg-corrected permeability = 8.3×10^{-20} from 5 measurements from 2048 – 2067 feet bgs



$$k_{pore} = 1.0 \times 10^{-19} \text{ m}^2$$
$$k_{meas} = 7.2 \times 10^{-20} \text{ m}^2$$

CFD Simulations

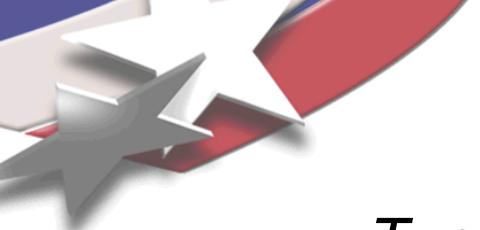
Type II Pore: Lower Tuscaloosa @ 8590 ft bgs



Lower Tuscaloosa Klinkenberg-corrected permeability = $2.8\text{e-}17\text{ m}^2$
from 3 measurements from 8584 to 8590 feet bgs

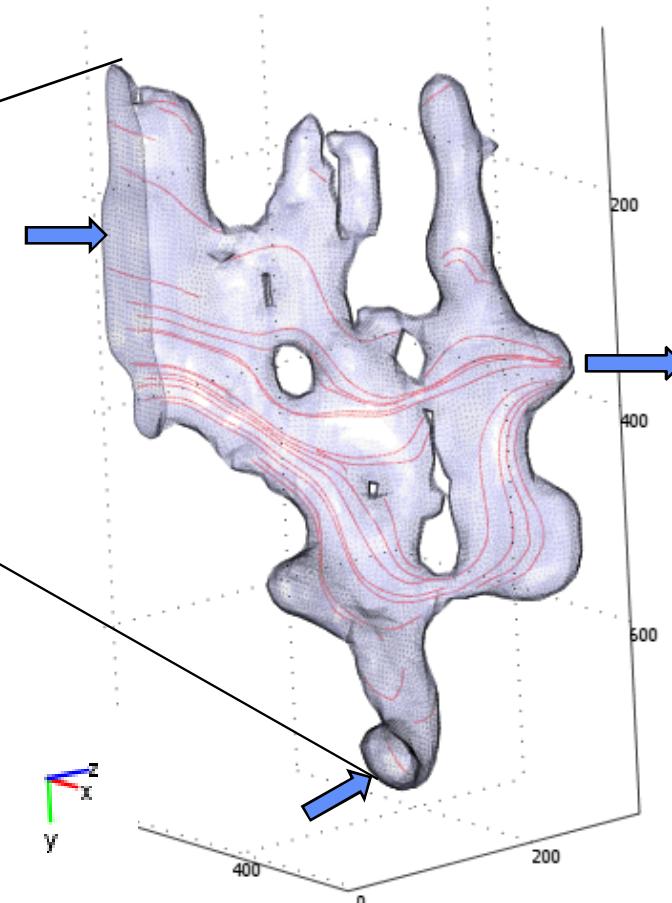
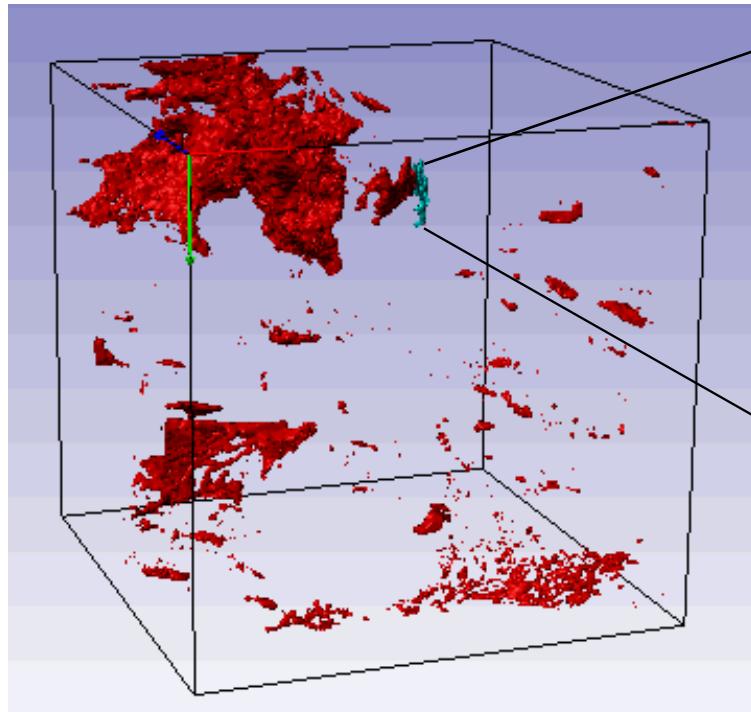
$$k_{pore} = 8.7\text{e-}17\text{ m}^2$$

$$k_{meas} = 6.8\text{e-}17\text{ m}^2$$



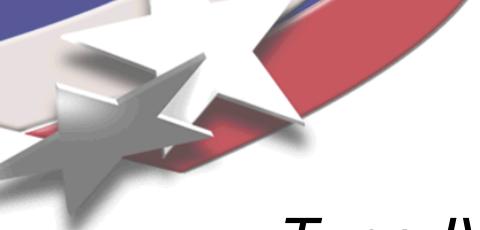
CFD Simulations

Type III Pore: Gothic Shale @ 5390 ft bgs

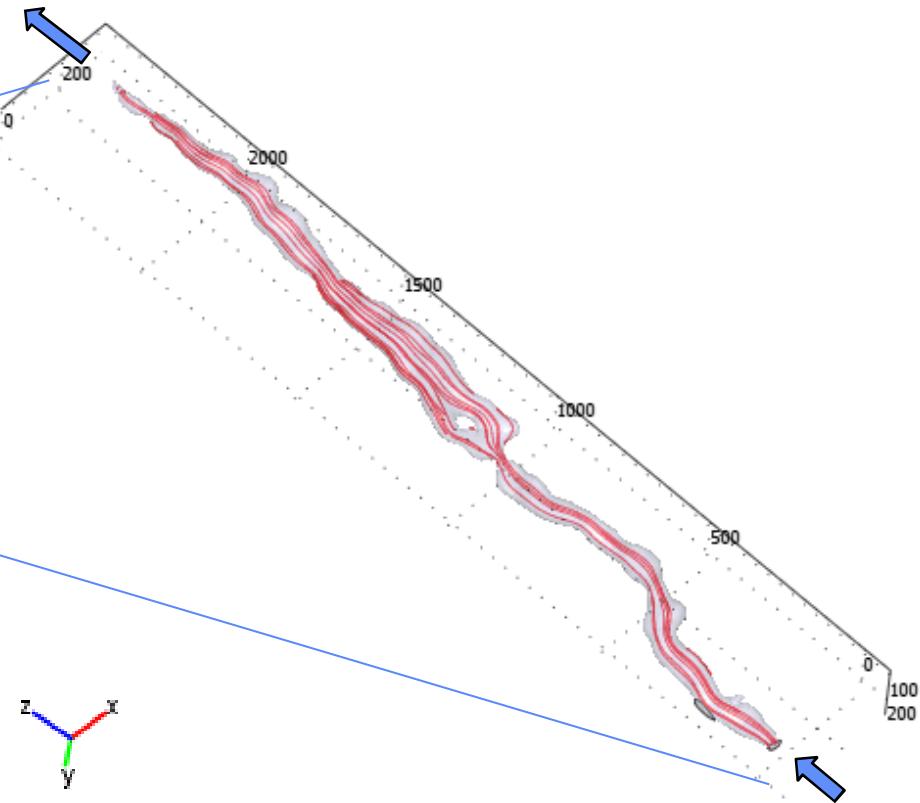
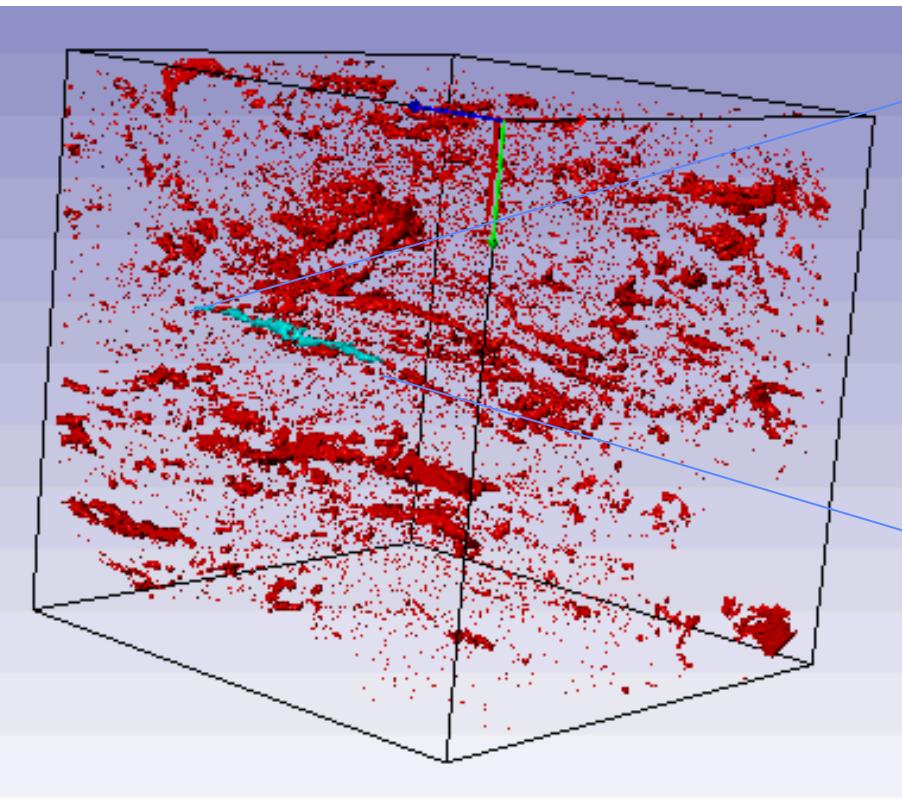


Gothic Klinkenberg-corrected
permeability = $1.4\text{e-}19$ from 4
measurements from 5379 to
5391 feet bgs

$$k_{pore} = 2.1\text{e-}17 \text{ m}^2$$
$$k_{meas} = 1.3\text{e-}19 \text{ m}^2$$



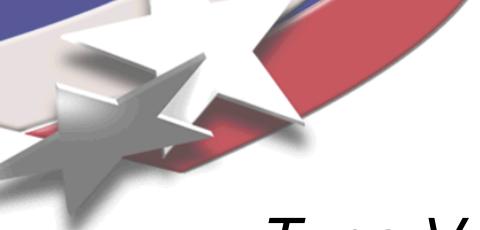
Type IV Pore: Marine Tuscaloosa @ 7925 ft bgs



Marine Tuscaloosa horizontal
Klinkenberg-corrected permeability =
1.5e-18 m² from 6 measurements
from 7818 to 7934 feet bsg

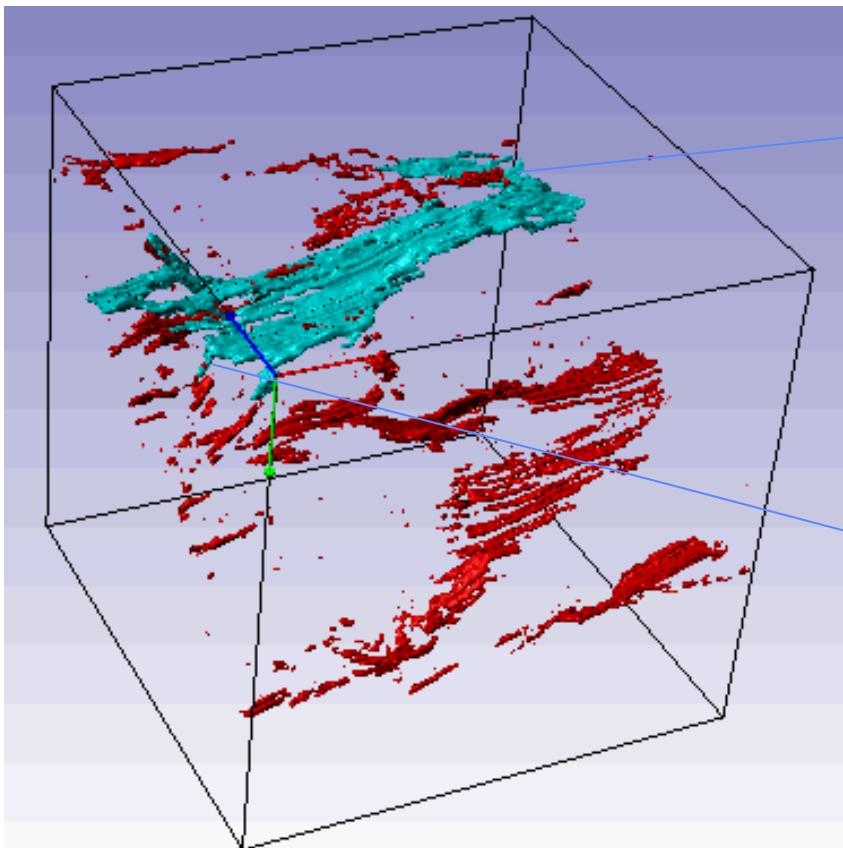
$$k_{pore} = 4.5 \times 10^{-18} \text{ m}^2$$

$$k_{meas} = 9.9 \times 10^{-20} \text{ m}^2$$

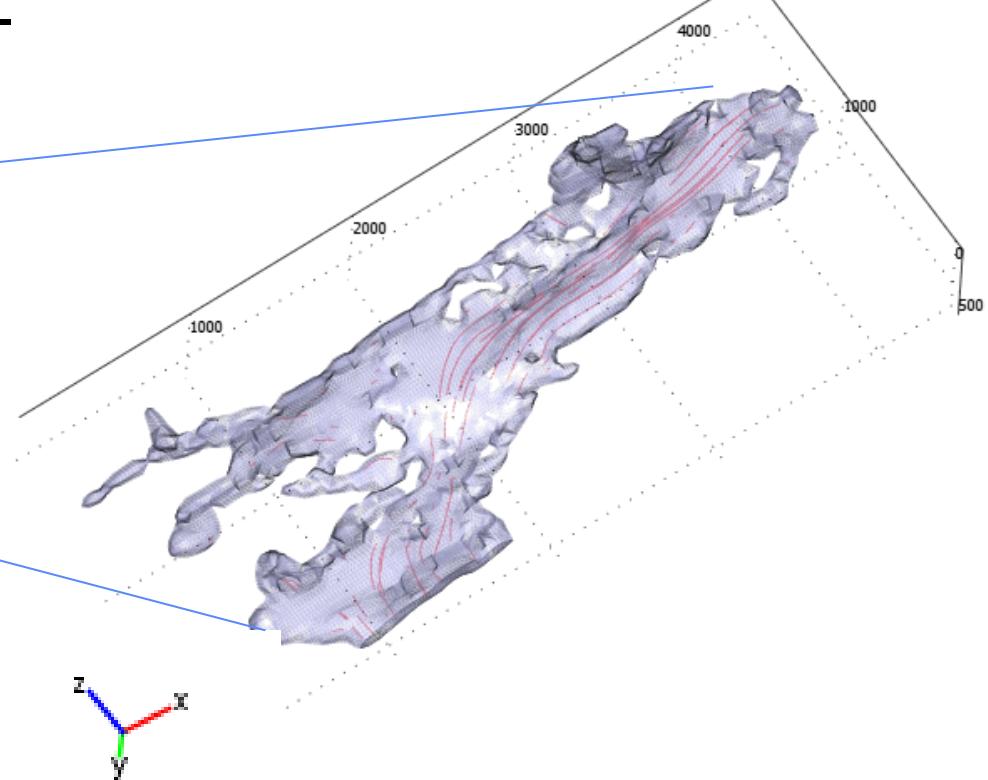


CFD Simulations

Type V Pore: Lower Kirtland Shale @ 2692 ft bgs



Lower Kirtland horizontal
Klinkenberg-corrected permeability =
 $8.1 \text{e-}20 \text{ m}^2$ from 2 measurements
from 2692 to 2697 feet bgs



$$k_{pore} = 4.5 \text{e-}18 \text{ m}^2$$

$$k_{meas} = 7.9 \text{e-}20 \text{ m}^2$$

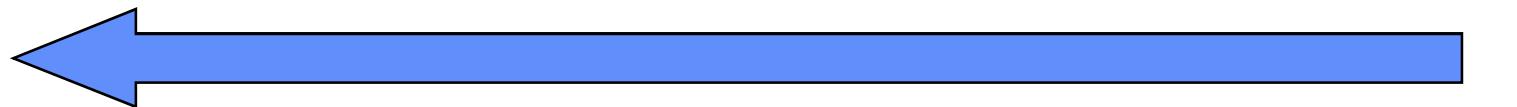


Summary: Sealing Types

Distal

Proximal

Depositional Setting



Marine
Tuscaloosa

Gothic

Sealing Efficiency

Lower
Tuscaloosa

Lower
Kirtland

Upper
Kirtland

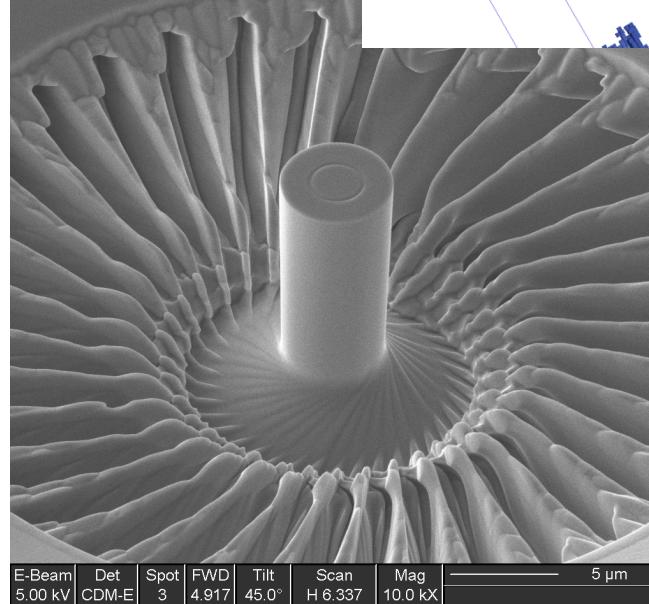
*Depth,
Time/Temperature*

Burial History

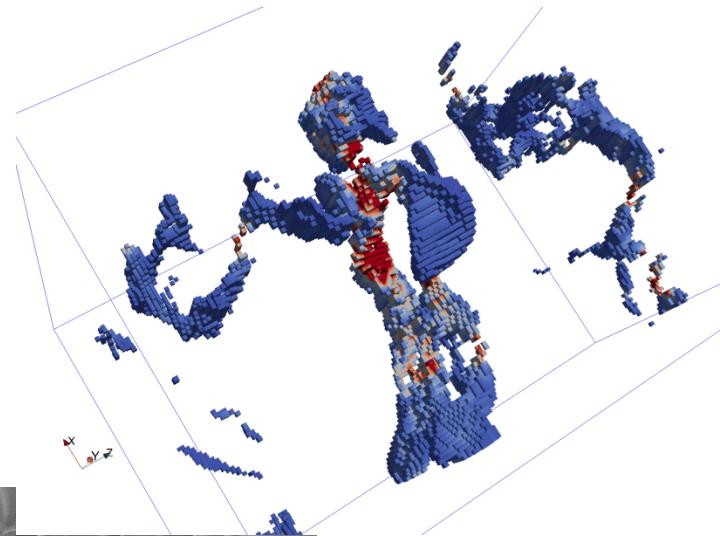
Path Forward

- **SANS (with Alexis Navarre-Stitchler) at ORNL**
- **3DTEM (with Paul Kotula)**
- **Acoustic Emissions/Mancos Shale (with David Holcomb)**
- **Cryo-FIB, FIB/e-SEM, eSEM with loading frame, nano-indenter**
- **Confocal work with re-hydration/swelling**
- **Membrane Efficiency**

Lattice-Boltzmann simulation of flow through type 3 pore network



E-Beam 5.00 kV	Det CDM-E	Spot 3	FWD 4.917	Tilt 45.0°	Scan H 6.337	Mag 10.0 kX	5 μ m
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5 micron diameter by 10 micron long ion milled column of Gothic Shale



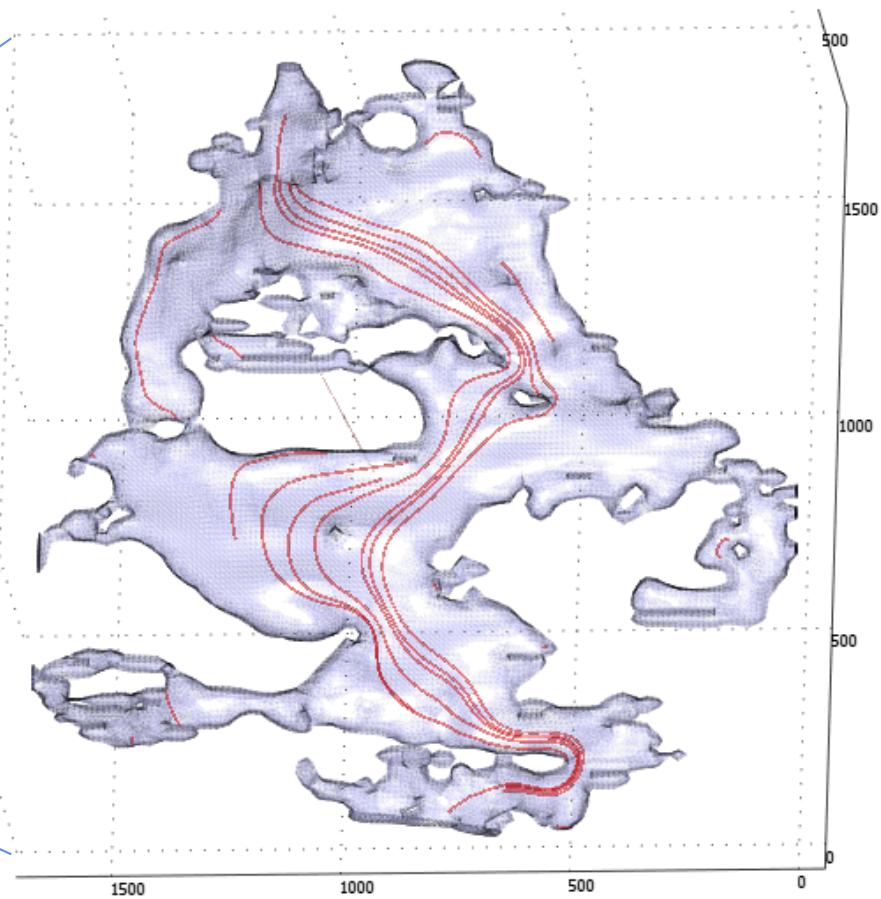
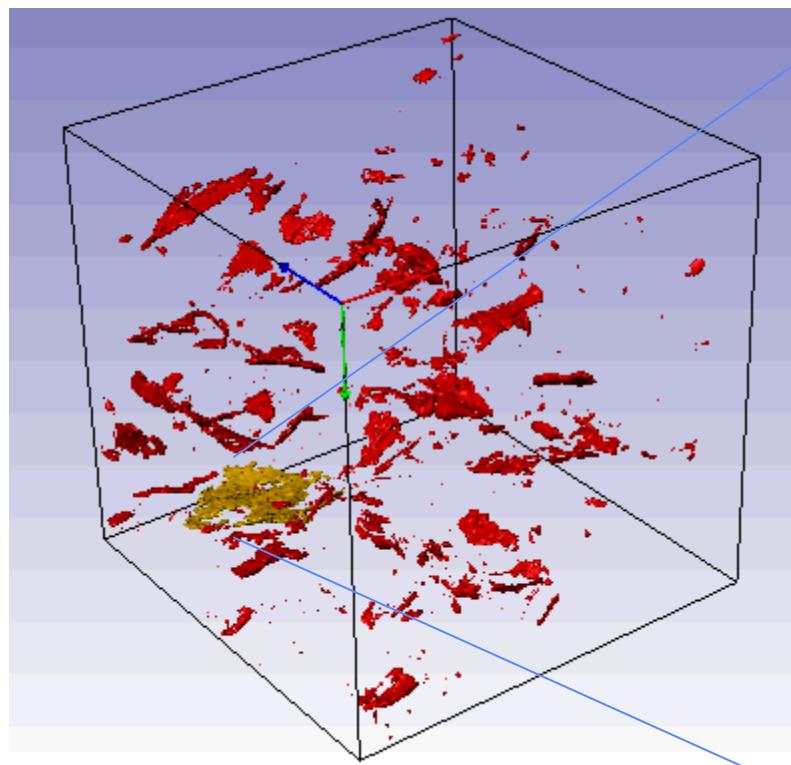
Summary: Shale Transport Properties

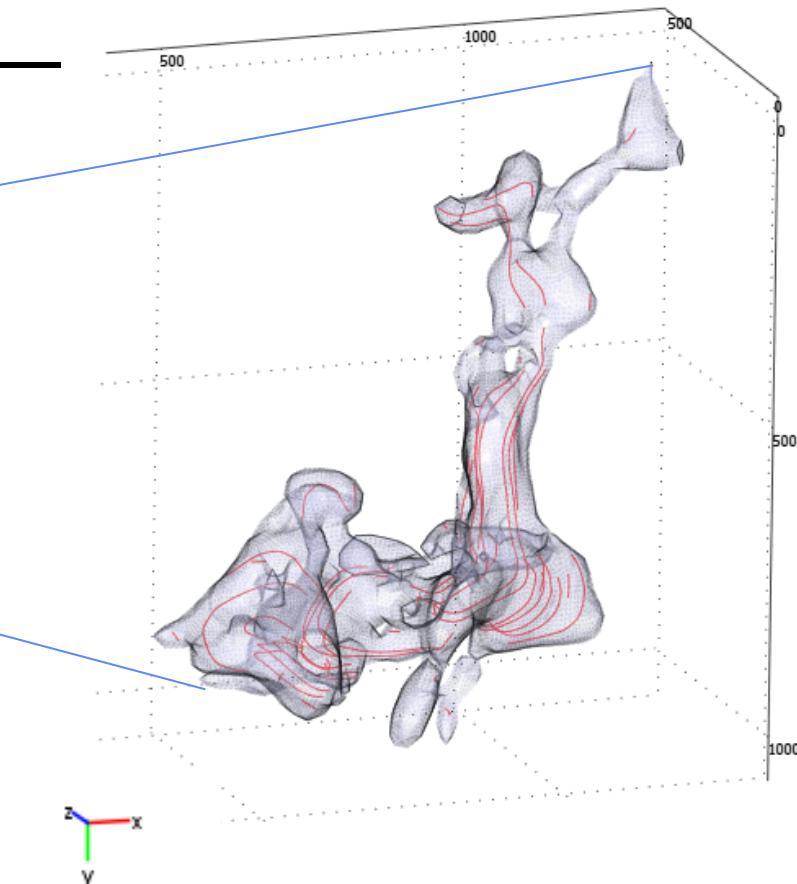
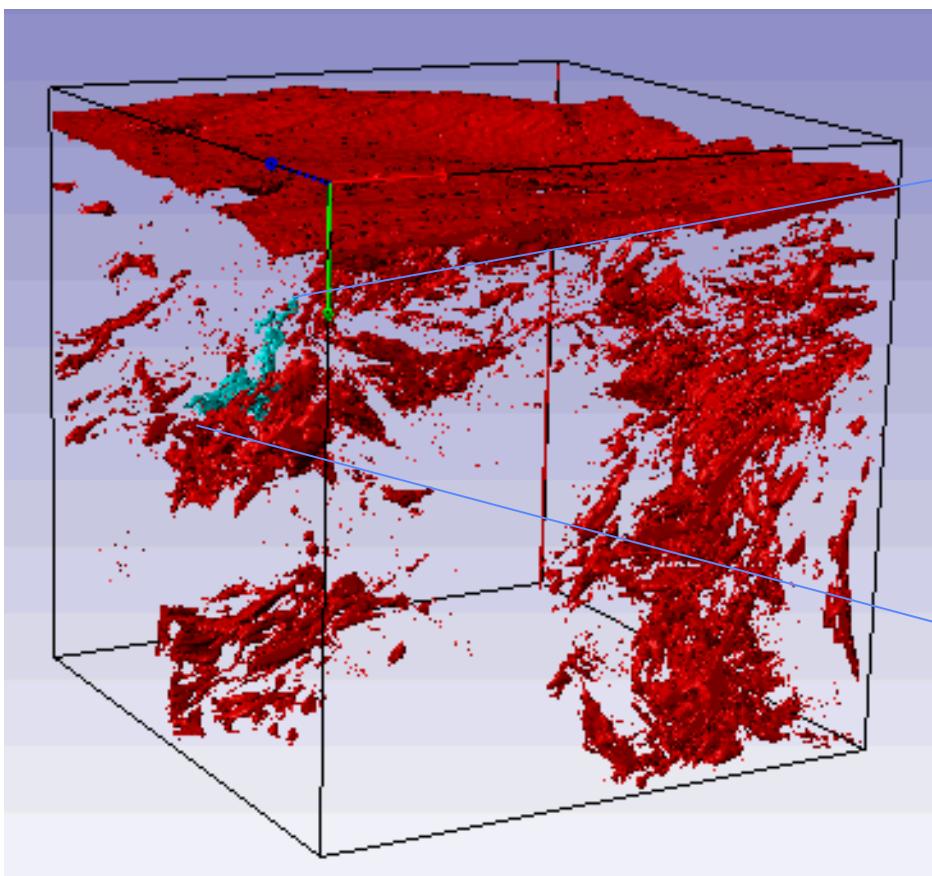
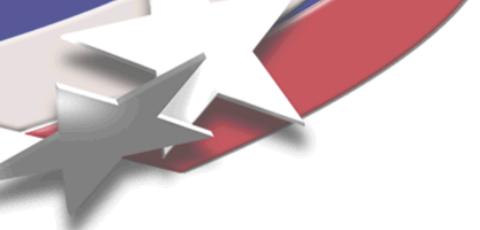
- **What can we know?** By examining a variety of shale types from distinct depositional environments and burial history, we can infer generalizations about pore types and connectivity, and effect of pore topology on single and multiphase transport and sealing behavior.
- **How can we know it/study it/model it?** Combination of old and new technology (i.e. MICP with FIB/SEM, NANS) allows fundamental assessment of pore types and influence on flow behavior.
- **What do the fundamental observations mean to broader problems?** Can justify application of certain model methodology in making predictions on e.g. shale gas, CCS, waste disposal PA, etc.



Extra slides

5390-8-A





Shale Macro Pore Types

- Fracture and fracture-fill porosity
- Pedogenic
- Diagenetic, associated with concretions/nodules

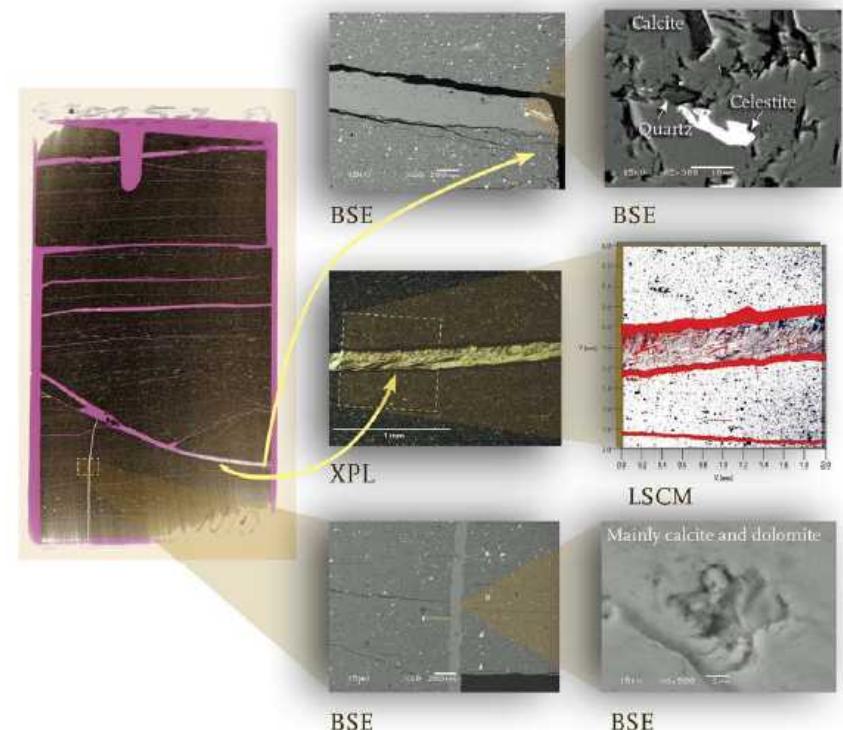


Figure 7-25. Additional views of the inclined shear fracture zone from 5392.30 to 5392.5 feet (core shown on figure 7-23) displayed on various BSE, XPL, and LSCM images at different magnifications derived from the thin section shown on the left.

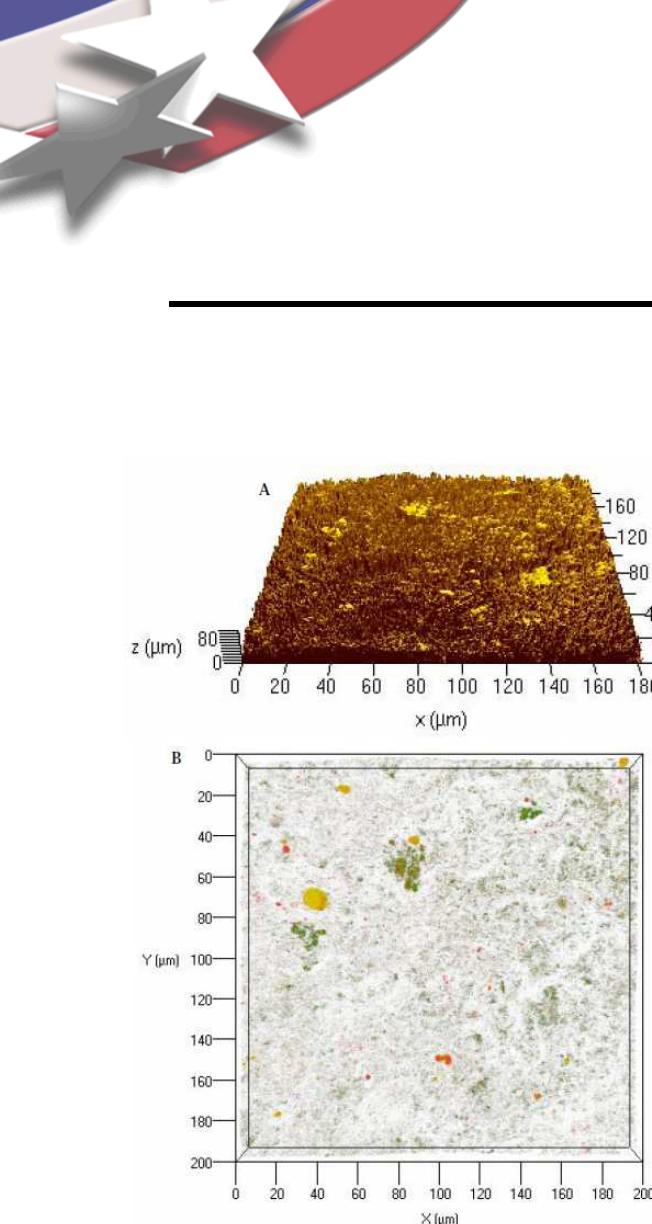


Figure 7-12. Low magnification of corresponding LSCM images of a bedding Gothic shale from 5380.0 feet. A – Image showing topography in an oblique Image shows green, yellow-orange, and red fluorescing material.

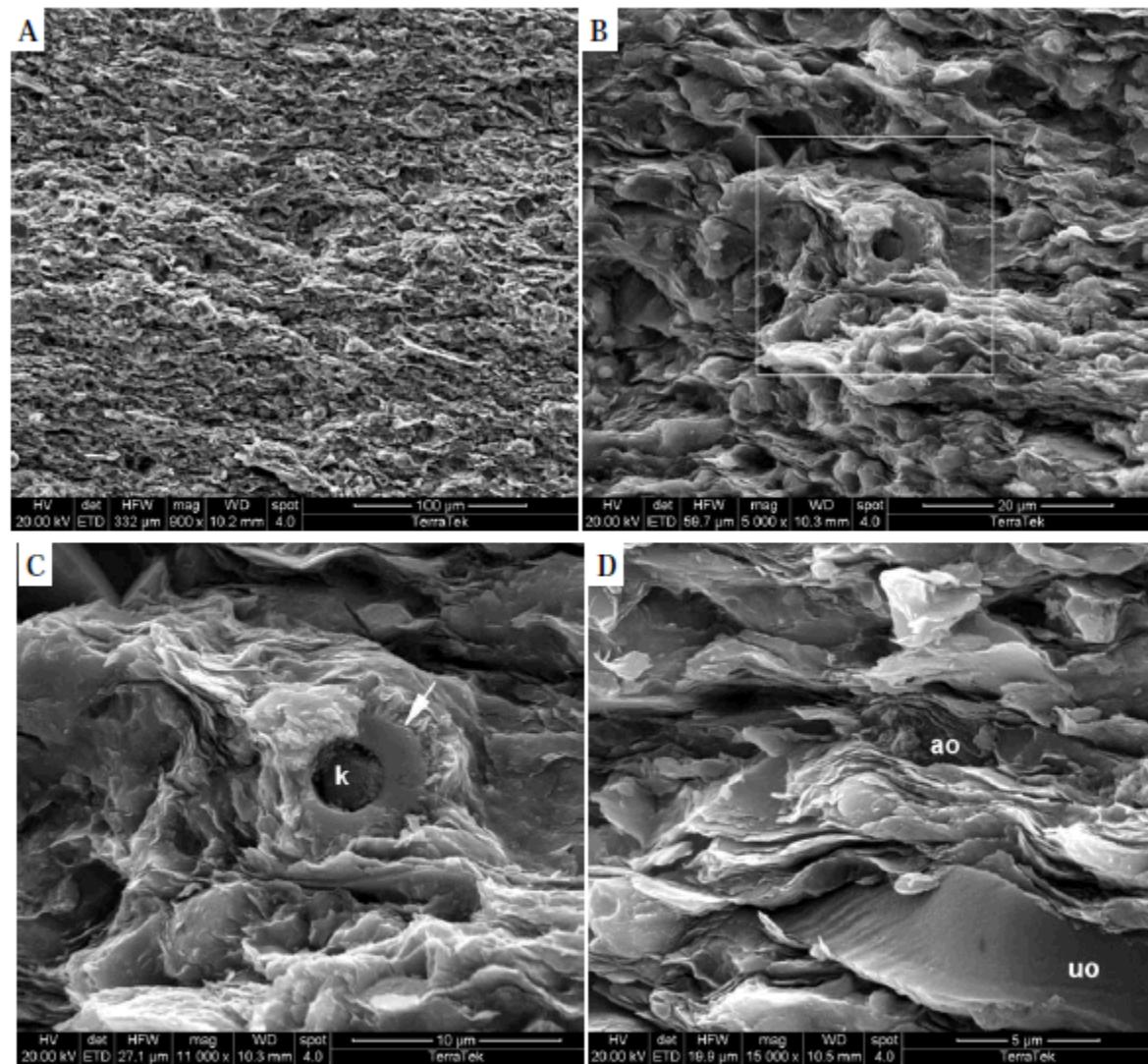


Figure 7-17. Scanning electron images of argillaceous mudstone in Gothic shale from 5382.8 feet. A – Overview of argillaceous mudstone highlighting distinct grain orientation. B – Closer view of mudstone matrix showing wavy parting planes between clay packets. The tube at center (box) is enlarged in the next image (7-17C). C – Detail of pyrite tube (arrow) shown in the previous image. The form represents a replaced microfossil, and is lined with scruffy kerogen residue (k). Note the flakey matrix clays, likely illite and/or mixed layer illite-smectite. D – Matrix detail showing unaltered and altered carbonaceous material. The smooth particle at lower right (uo) represents a discrete carbonaceous grain with little alteration. At top center, a particle representing a different class of organics, embedded