

Impact of Multiscale Heterogeneity on Mechanical Behavior of Mancos Shale



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Sandia
National
Laboratories

*Exceptional
service
in the
national
interest*



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Acknowledgments

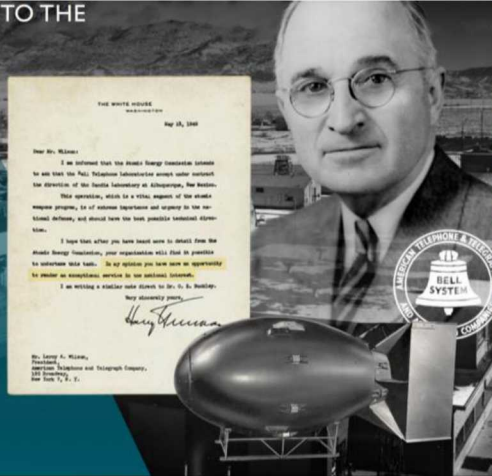
- Laboratory Directed Research and Development program at Sandia National Laboratories
- U.S. Department of Energy Office of Basic Energy Sciences, Division of Chemical Sciences, Geosciences, and Biosciences
- Thomas Dewers, Mathew Ingraham, Bill Mook (SNL)
- Peter Mozley, Oh-Tark Kwon (NMT)

SANDIA NATIONAL LABORATORIES OVERVIEW

SANDIA'S HISTORY IS TRACED TO THE MANHATTAN PROJECT

...In my opinion you have here an opportunity to render an exceptional service in the national interest.

- July 1945
Los Alamos creates Z Division
- Nonnuclear component engineering
- November 1, 1949
Sandia Laboratory established
- AT&T: 1949–1993
- Martin Marietta: 1993–1995
- Lockheed Martin: 1995–2017
- Honeywell: 2017–present



SANDIA HAS FACILITIES ACROSS THE NATION

Main sites

- Albuquerque, New Mexico
- Livermore, California



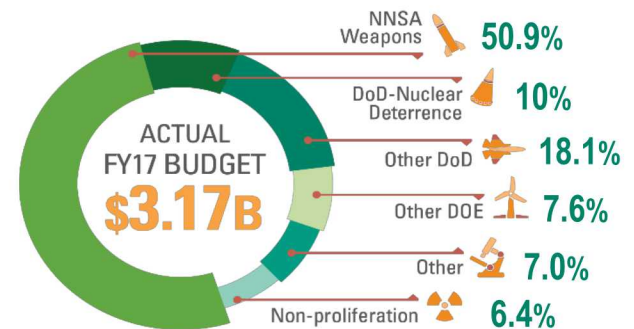
Activity locations

- Kauai, Hawaii
- Waste Isolation Pilot Plant, Carlsbad, New Mexico
- Pantex Plant, Amarillo, Texas
- Tonopah, Nevada

SANDIA ADDRESSES NATIONAL SECURITY CHALLENGES



SANDIA'S BUDGET and WORKFORCE



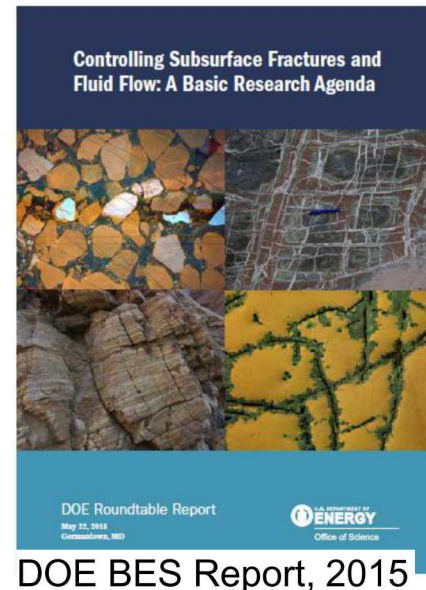
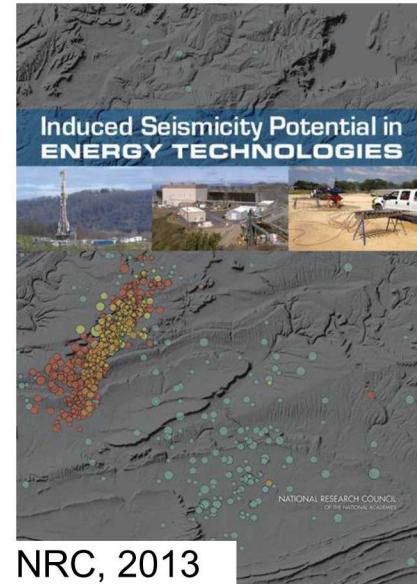
12,256
EMPLOYEES

10,940
New Mexico

1,316
California

Subsurface Energy Activities

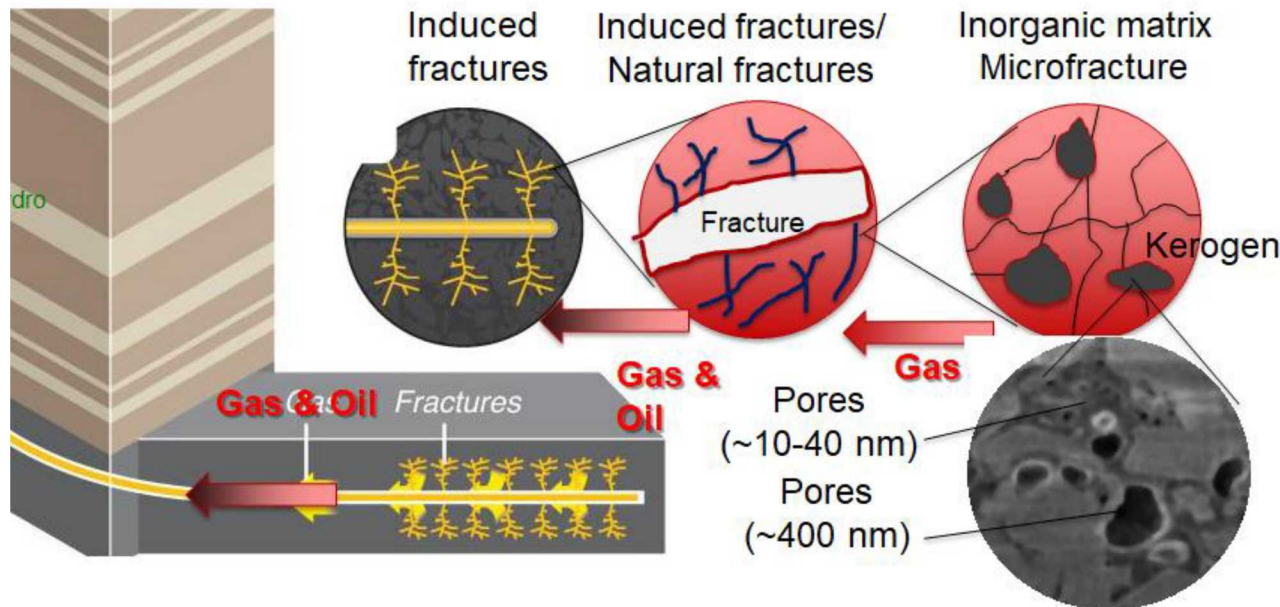
- Subsurface energy technologies typically involve the change of fluid flow, stress, thermal, chemical (aka THMC) status in fractured and porous media
 - **Unconventional resources recovery**
 - Disposal of wastewater and nuclear waste
 - Subsurface carbon and compressed gas storage
- To improve subsurface energy activities and reduce adverse risks (e.g., induced seismicity and environmental impact), current understanding of (thermo-, chemo-)poro-mechanics needs to be improved
- Mesoscale analysis – **linking discrete and complex pore-scale behavior to continuum (macroscale) reservoir response** – is key, yet remains elusive as a result of the extreme heterogeneity and resulting scale dependence.



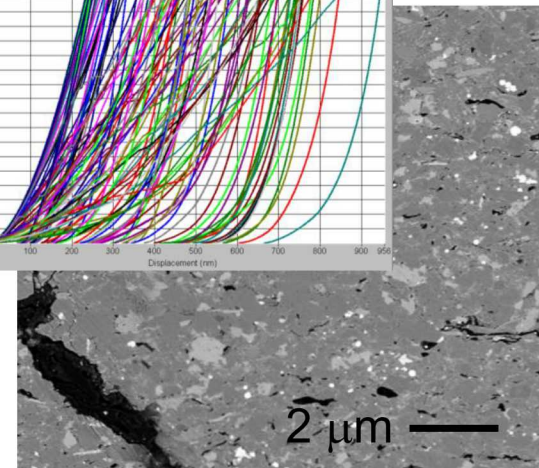
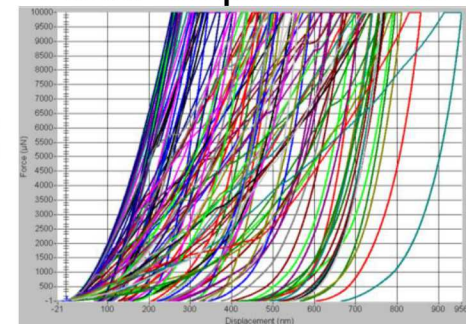
DOE BES Report, 2015

Multiscale Heterogeneity in Compositions, Pore structure, and Mechanical Properties

- Understand how heterogeneity, pores, cracks, flaws etc. contribute to shale poromechanics over scales and provide physical basis for core-scale measured deformational and transport constitutive behavior
- Develop novel techniques and workflow for a linked imaging, experimental, and modeling-based advancement of shale poromechanics



Clay-rich area,
64 nanoindentations
Load-displacement



Integrated Approach

- **40 cm diameter core of Mancos Shale**

- Interlaminated fine mud, medium/ coarse mud, and very fine sand
- 1-3 mm laminae
- Parallel lamina, wavy lenticular lamina, ripple forms, and bioturbation

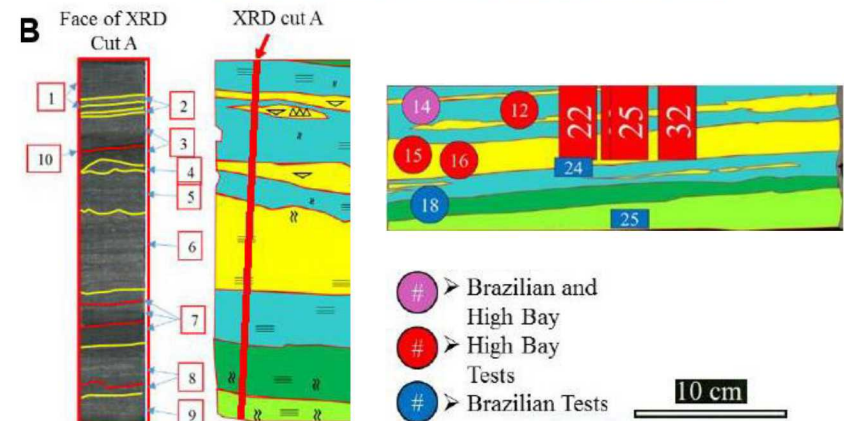
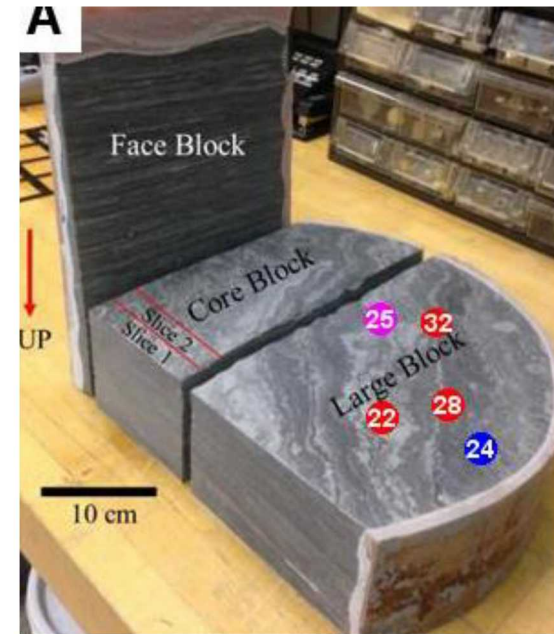
- **Mineralogical and textural characterization**

- Macroscopic
- Optical petrography/microscopy
- Micro-CT
- XRD/X-ray Microprobe
- Small Angle Neutron Scattering
- Focused Ion Beam-SEM
- Back-Scattered Electron Microscopy
- MAPS Mineralogy

- **Mechanical Experiments**

- Uni-/Tri-axial compression (1x2")
- Brazilian Test (1x0.5")
- Nanoindentation

- **Computational modeling**



Yoon et al. (AAPG, Memoir 2019, in press)
Dewers et al. (AGU, Monograph, 2019)

Integrated Approach

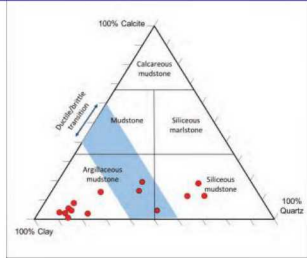
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Multiscale characterization of physical, chemical, and mechanical heterogeneity of nano-porous geomaterials

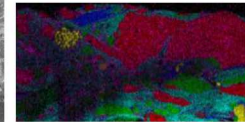
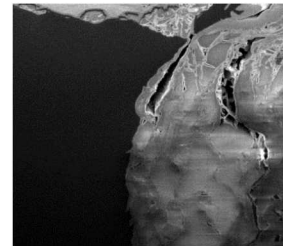
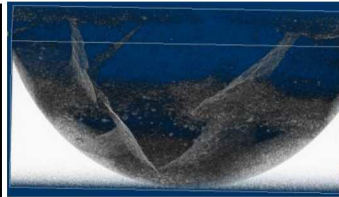
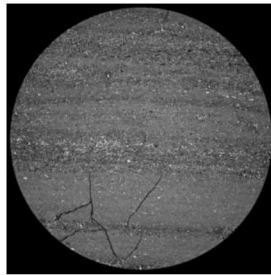
Macroscopic and microscopic lithofacies (optical petrography)

Optical and Confocal Microscopy

Focused-Ion Beam & Broad-Ion Beam for milling



3D multiscale microCT
X-ray probe and QEMSCAN for mineralogy

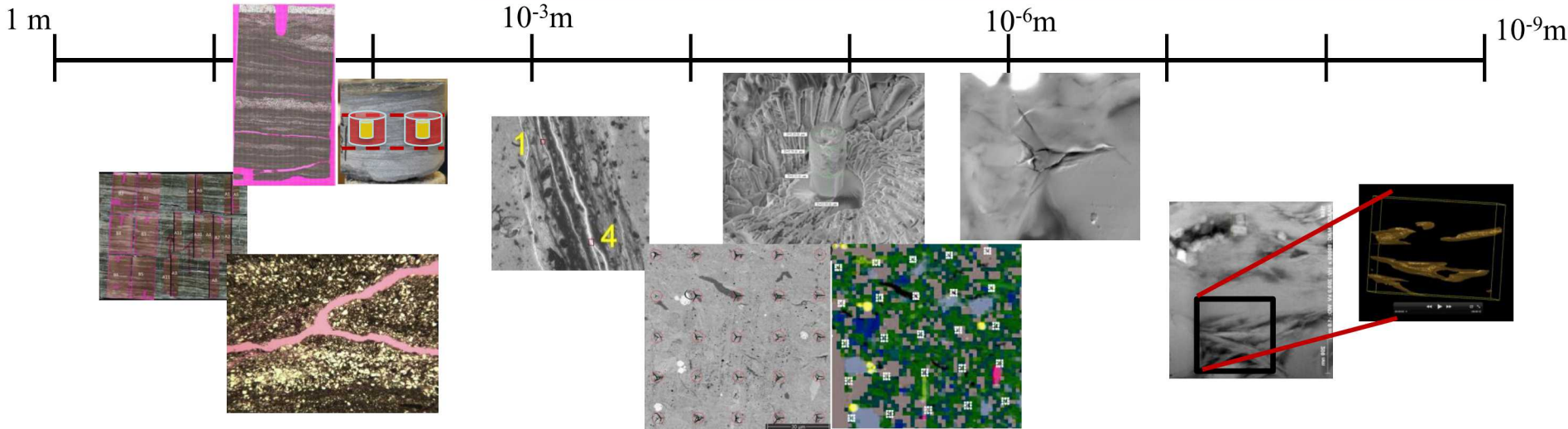


SEM, EDS

mSEM, Maps Mineralogy

Electron Microscopy

(Ultra) Small Angle Neutron Scattering



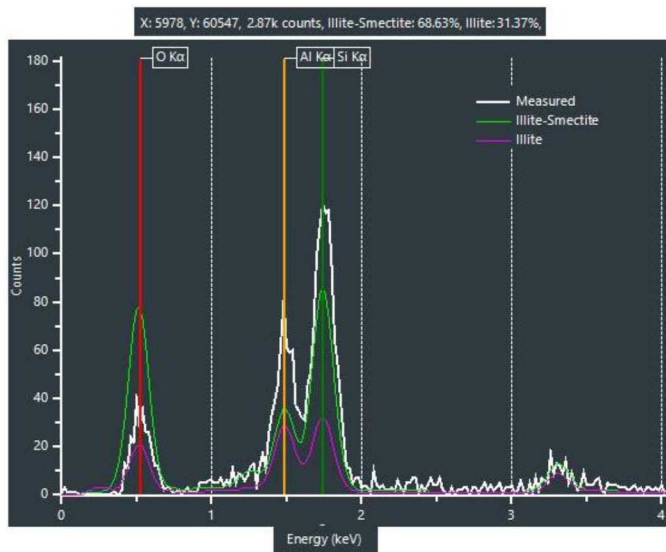
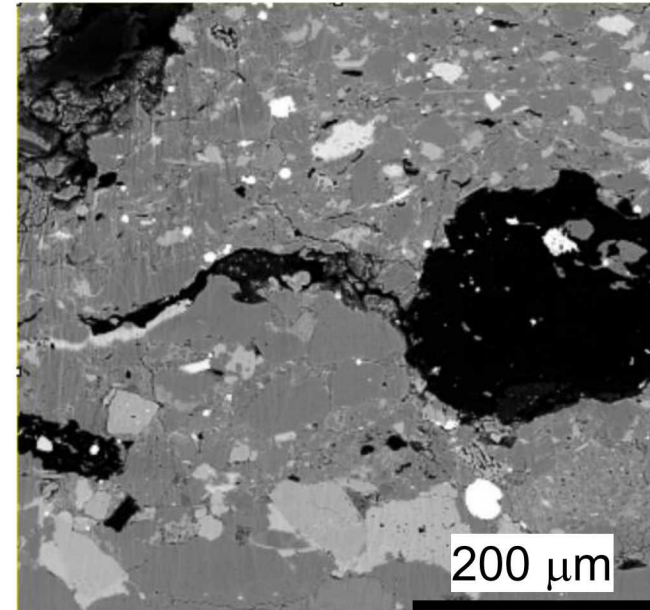
MAPS Mineralogy

- **SEM-based automated mineralogical measurement, analysis, data integration**

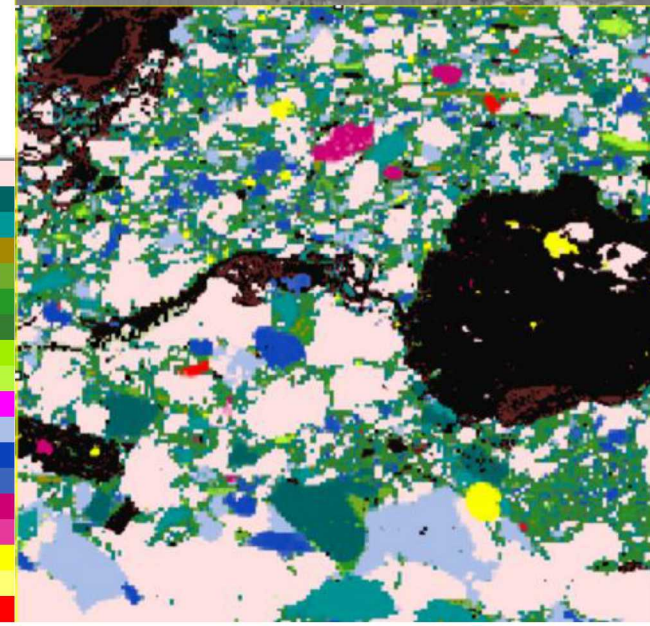
- Collection, overlay and re-registration of multiple images from different modalities
- SEM, SEM-EDS, optical, CL, EBSD
- QEMSCAN measurement algorithms

- **Mineral identification**

- Spectral matching
- Each pixel – single/multiple minerals
- Elemental substitutions
- Simultaneous mineral element and count maps

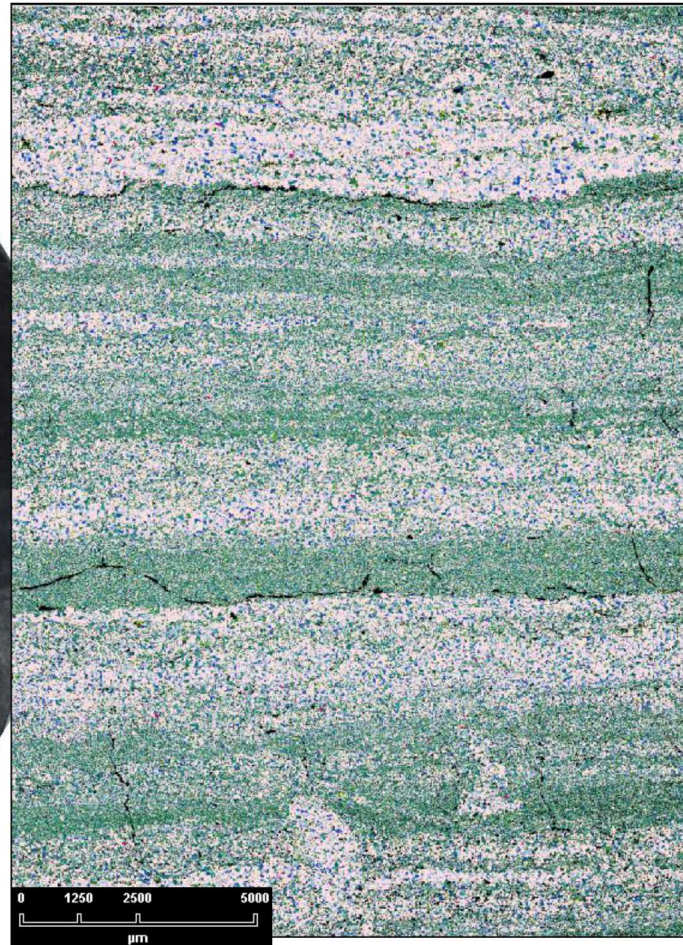
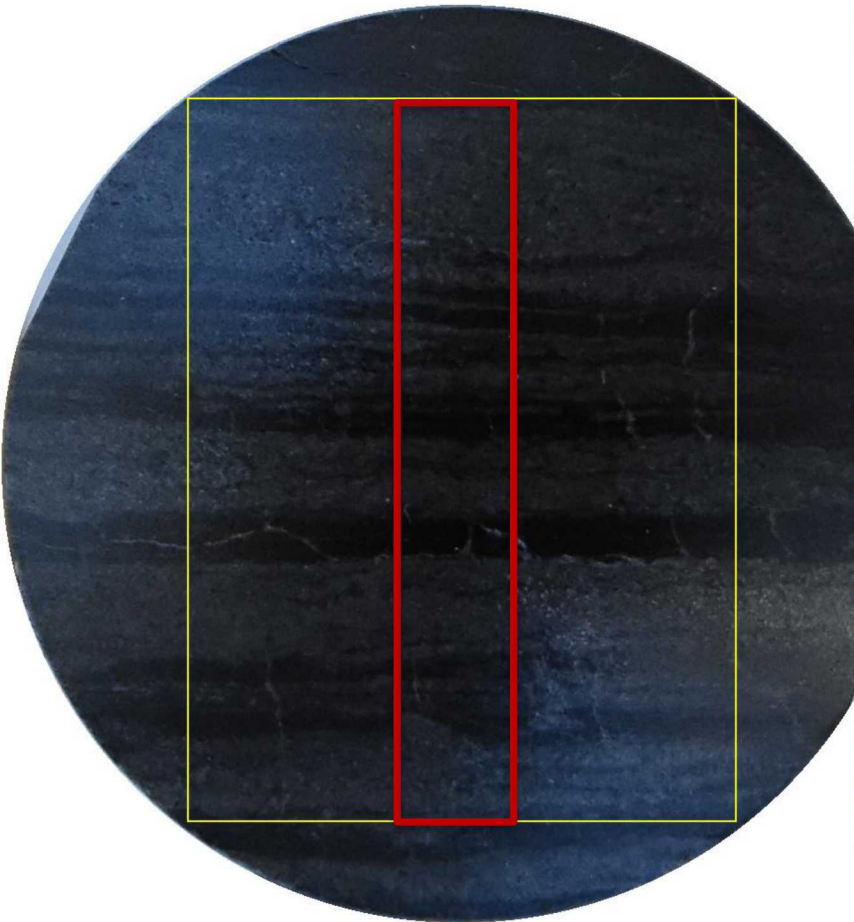


☒	Quartz (Silica)
☒	K-feldspar
☒	Albite
☒	Muscovite
☒	Kaolinite (Halloysite, Dickite)
☒	Illite
☒	Illite-Smectite
☒	Clinocllore
☒	Chamosite
☒	Zircon
☒	Calcite (Aragonite)
☒	Dolomite
☒	Ankerite
☒	Apatite (F)
☒	Apatite (Cl)
☒	Pyrite
☒	Sphalerite
☒	Rutile/Anatase/Brookite



Mineralogy Mapping

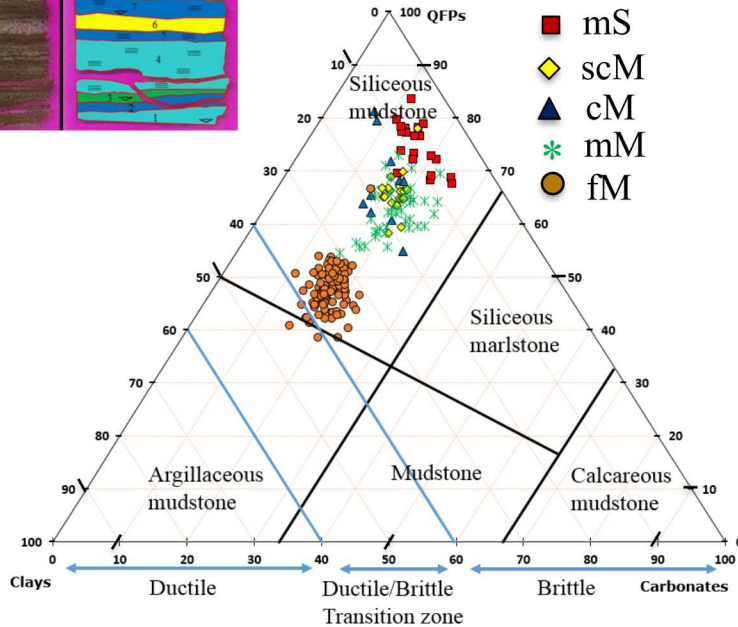
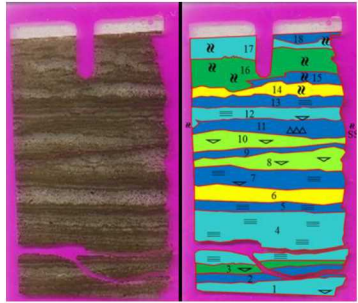
Ion-milling polished Mancos
(1 inch diameter)



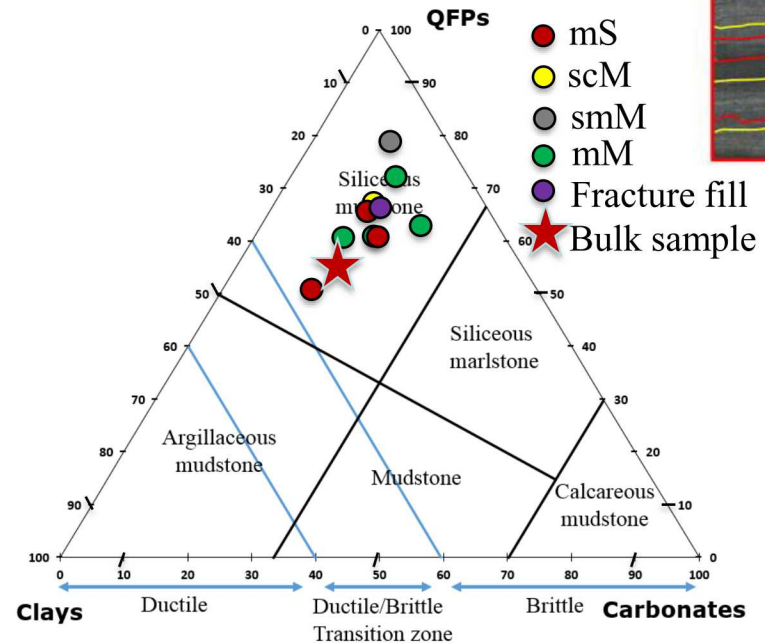
☒	Quartz (Silica)
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☒	Clinochlore
☒	Chamosite
☒	Zircon
☒	Calcite (Aragonite)
☒	Dolomite
☒	Ankerite
☒	Apatite (F)
☒	Apatite (Cl)
☒	Pyrite
☒	Sphalerite
☒	Rutile/Anatase/Brookite

Yellow Box (1.45 x 1.98 cm): BSE @ $1\mu\text{m}$ & MAPS @ $10\mu\text{m}$
Red box (0.18 x 1.98 cm): BSE @ $0.2\mu\text{m}$ & MAPS @ $2\mu\text{m}$

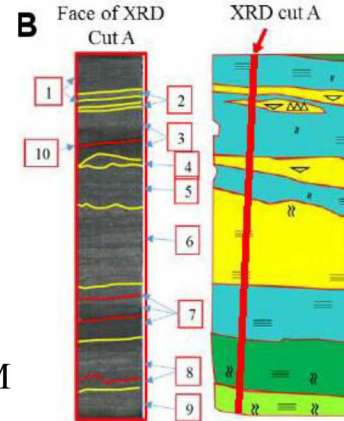
Mineralogy Mapping: Scale/methods dependent



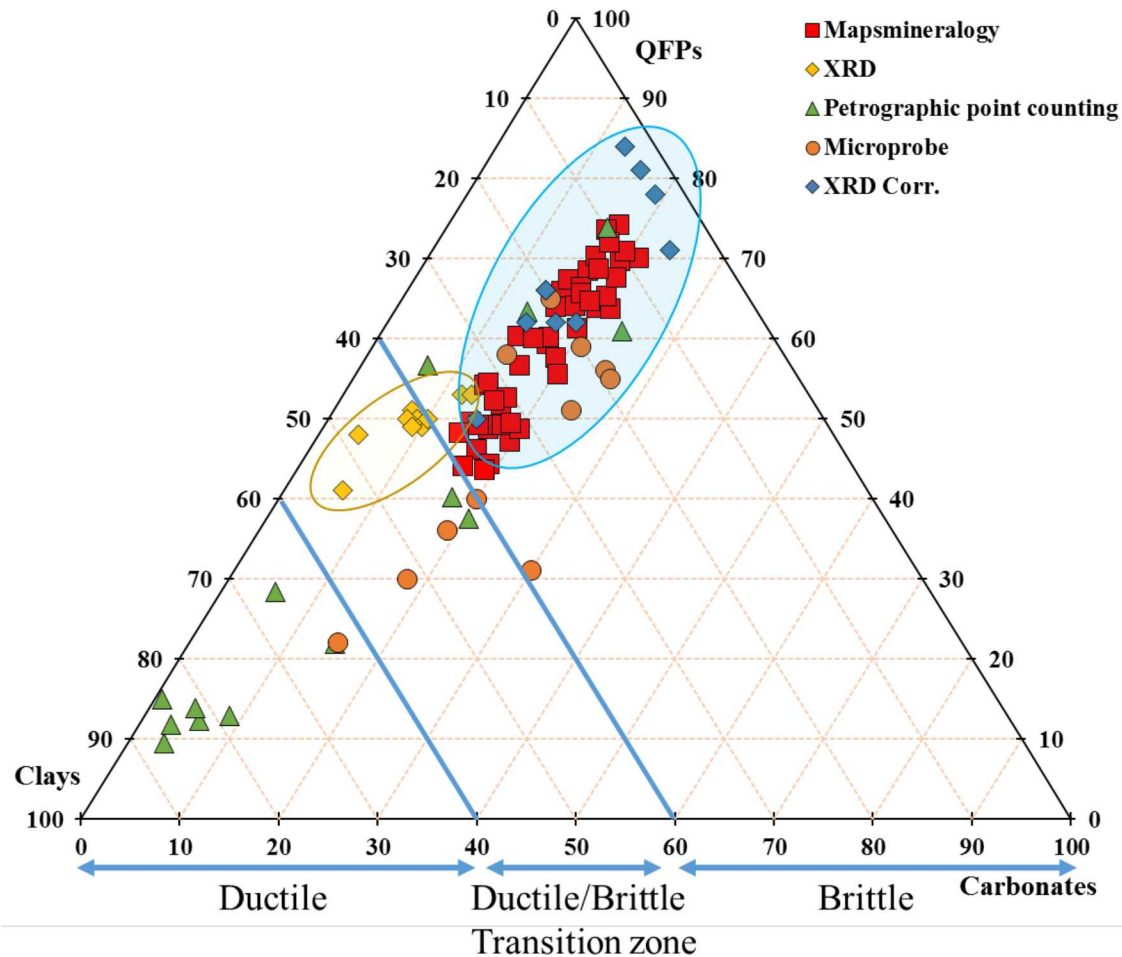
MAPS
(2 and 10 microns)



XRD
(10+1 samples)



Mineralogy Mapping: Scale/methods dependent



Influence of geological attributes on mechanical properties

Micro-lithofacies

BSE Mineralogy Mechanical

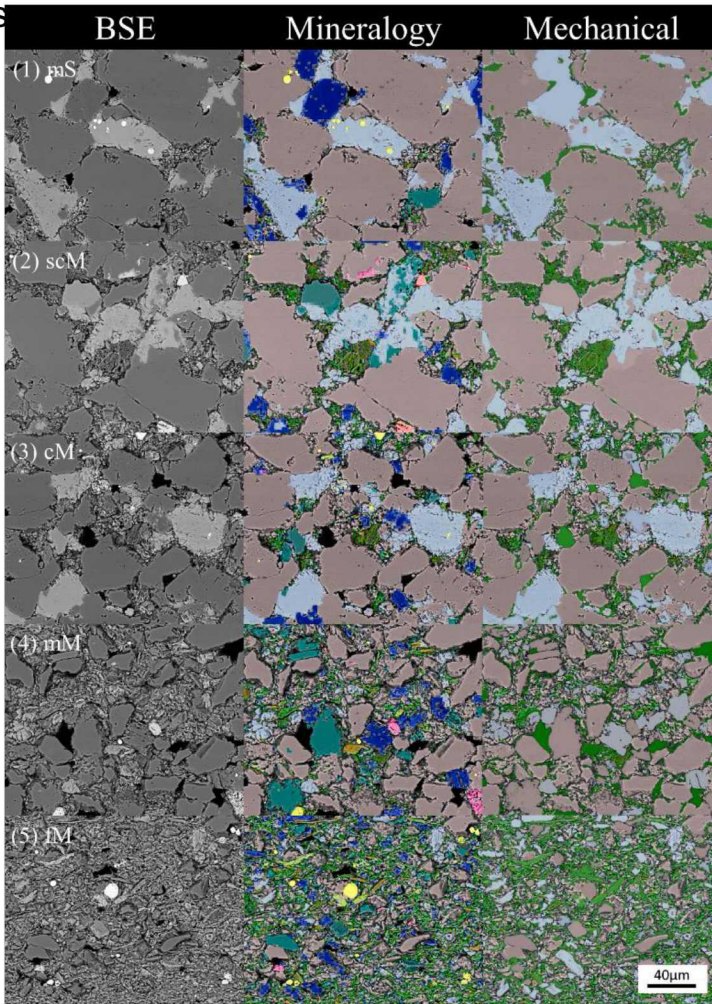
mS

scM

cM

mM

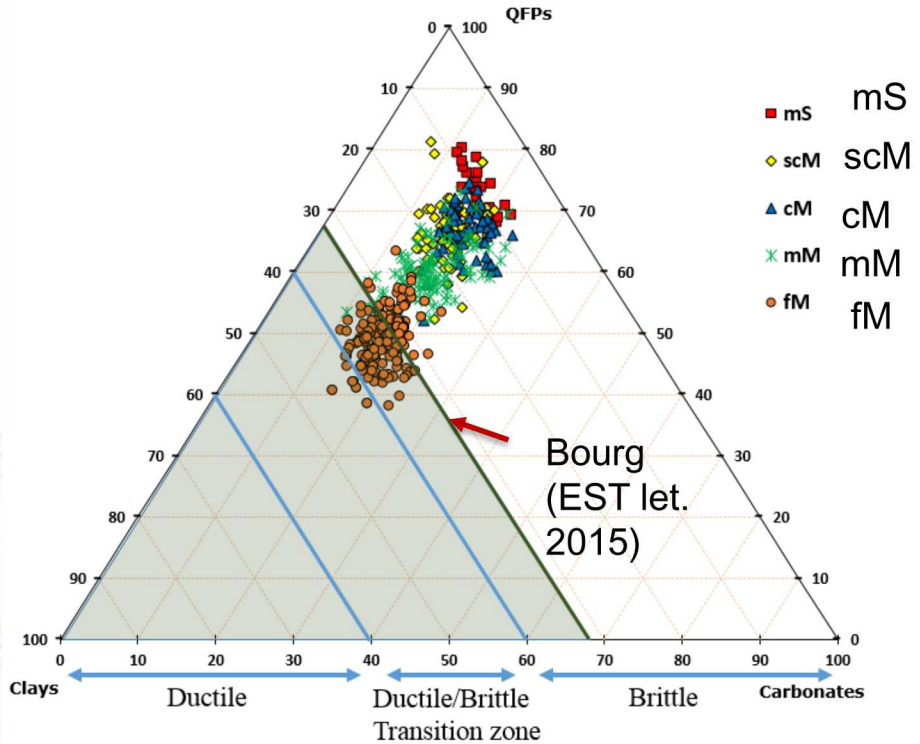
fM



Quartz	Illite	Calcite	Pyrite	Qua+Fel+Pyr+Apa+Zir
K-Feldspar	Ill-Smectite	Dolomite	Sphalerite	Cal+Dol+Ank
Albite	Clinocllore	Ankerite	Rutile	Mus+Kao+Ill+Org+Etc.
Muscovite	Chamosite	Apatite(F)	Unclassified	
Kaolinite	Zircon	Apatite(Cl)	Org./Por.	

Mineral Assemblages

Grain supported
Transition zone
Matrix supported



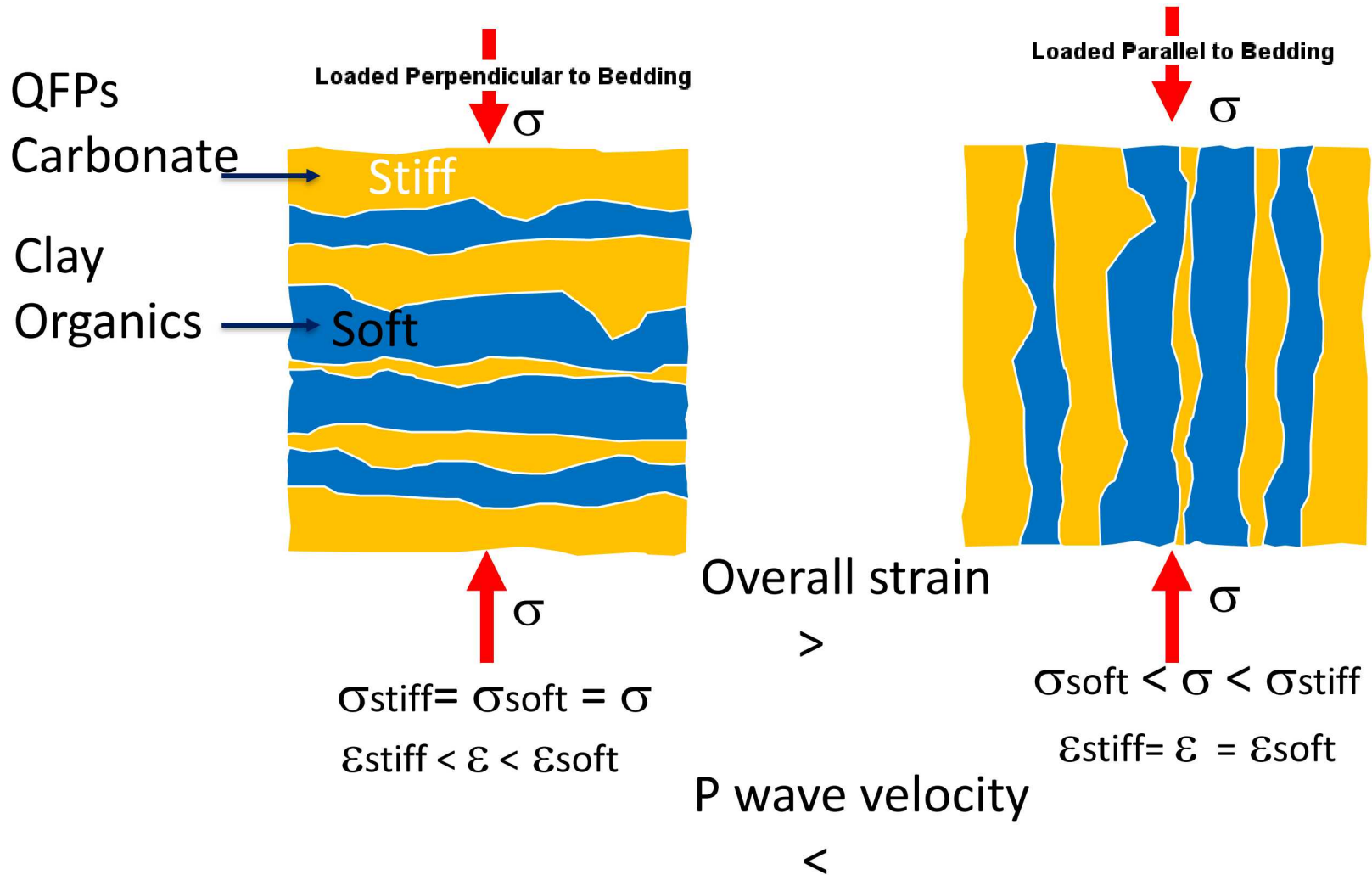
Ulmer-Scholle et al. (2014)

Kwon (@NMT, MS work, 2018)

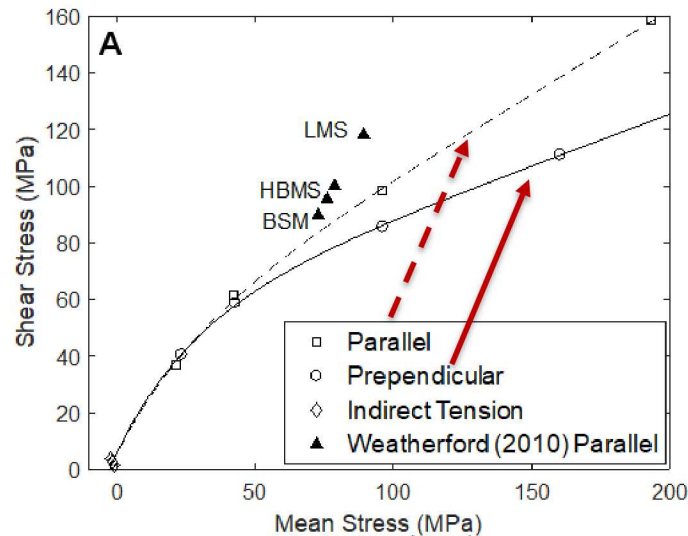
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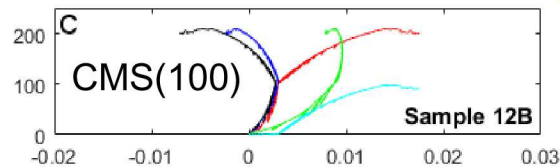
Conceptual Model of Anisotropic Layered System



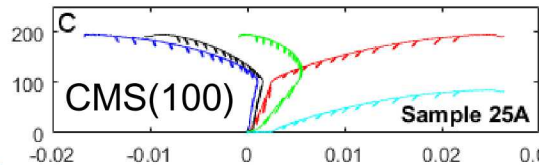
Axisymmetric Compression Testing



Loaded Parallel to Bedding



Loaded Perpendicular to Bedding

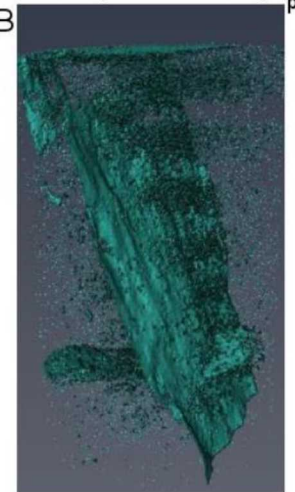


— Stress-Ax Strain — Stress-Vol Strain
— Stress-Lat Strain1 — Stress-Lat Strain2
— Shear Stress-Ax Strain

Loaded perpendicular to bedding



Loaded parallel to bedding



Segmented
MicroCT images

Confined (MPa)			Axial Stress	Mean Stress	Shear Stress	Fracture Angle (γ)
15B	CMS (50)	Parallel	113.00	42.20	61.31	13.5
28A	CMS (50)	Perpendicular	110.64	42.80	58.75	15.5
12B	CMS (100)	Parallel	209.59	96.09	98.30	23.5
25A	CMS (100)	Perpendicular	195.07	96.20	85.62	30
14B	CMS (200)	Parallel	376.37	193.42	158.44	27.5
22A	CMS (160)	Perpendicular	288.64	160.31	111.14	35.5

CMS: Constant Mean Stress

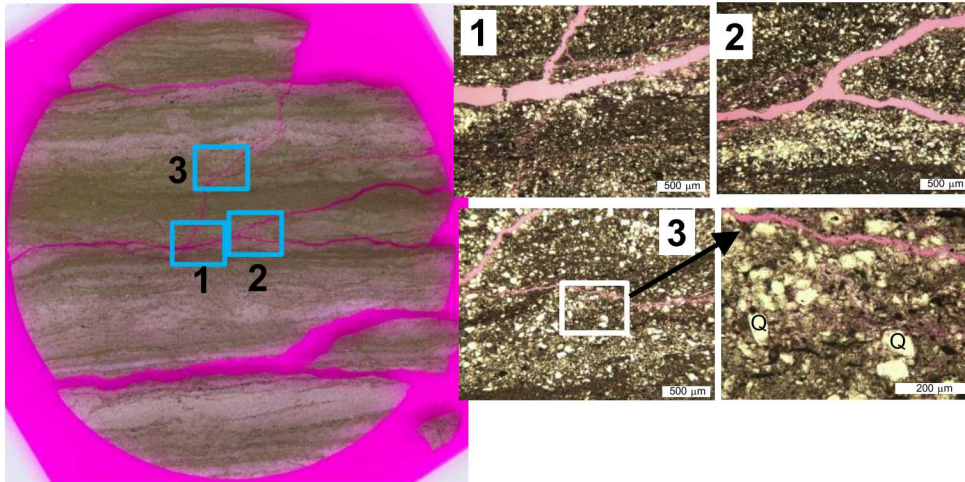
Yoon et al. (AAPG, Memoir 2019, in press)

Axisymmetric Compression Testing

Horizontal slice
through the central
part of sample

One primary &
two secondary
fractures

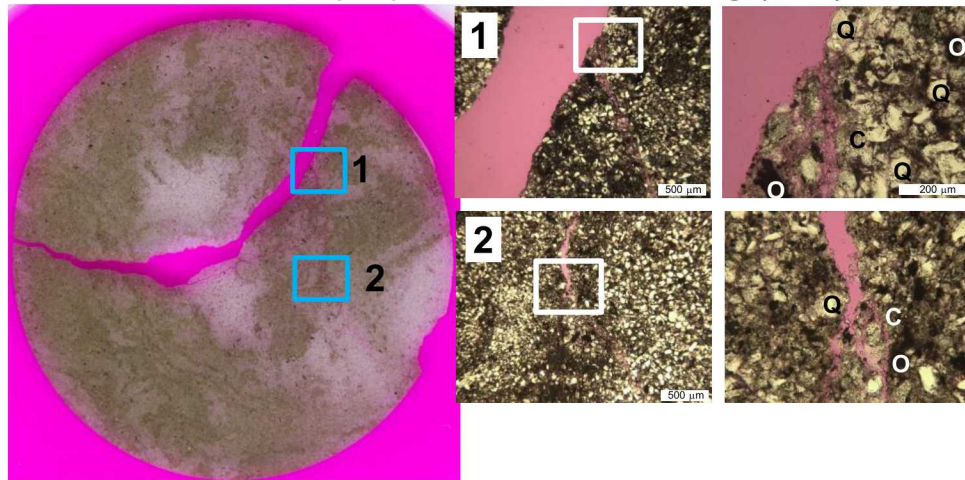
A. Loaded parallel to bedding (15B)



Fracture intersection &
bifurcation with small
aperture microfractures

Microfracture
propagation in between
quartz grains

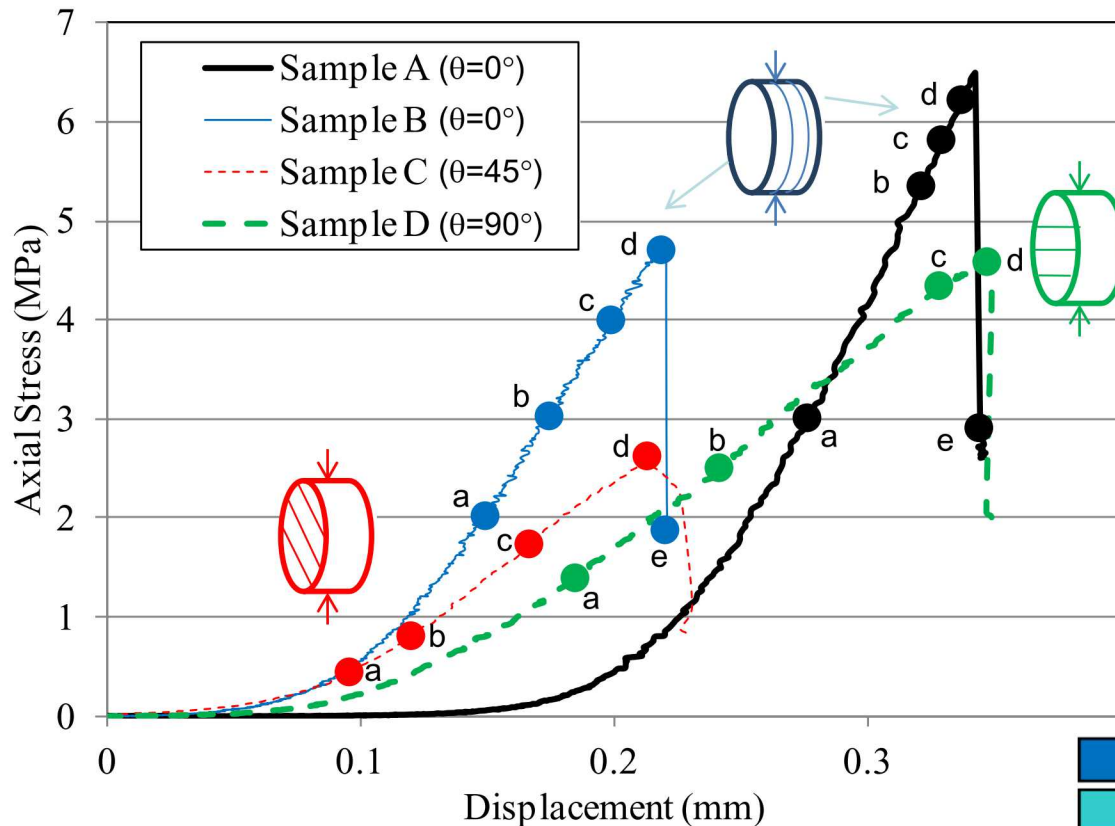
B. Loaded perpendicular to bedding (28A)



Microfracture patterns
in between quartz
grains

Curved main
fracture

Indirect Tension Results



$$\sigma_t = \frac{2P}{\pi Dt}$$

P: Loading

D: Diameter

t: thickness

- fine Mud (fM)
- medium Mud (mM)
- course Mud (sM)
- sandy fine Mud (sfm)
- sandy medium Mud (smM)
- sandy course Mud (scM)
- muddy Sand (mS)

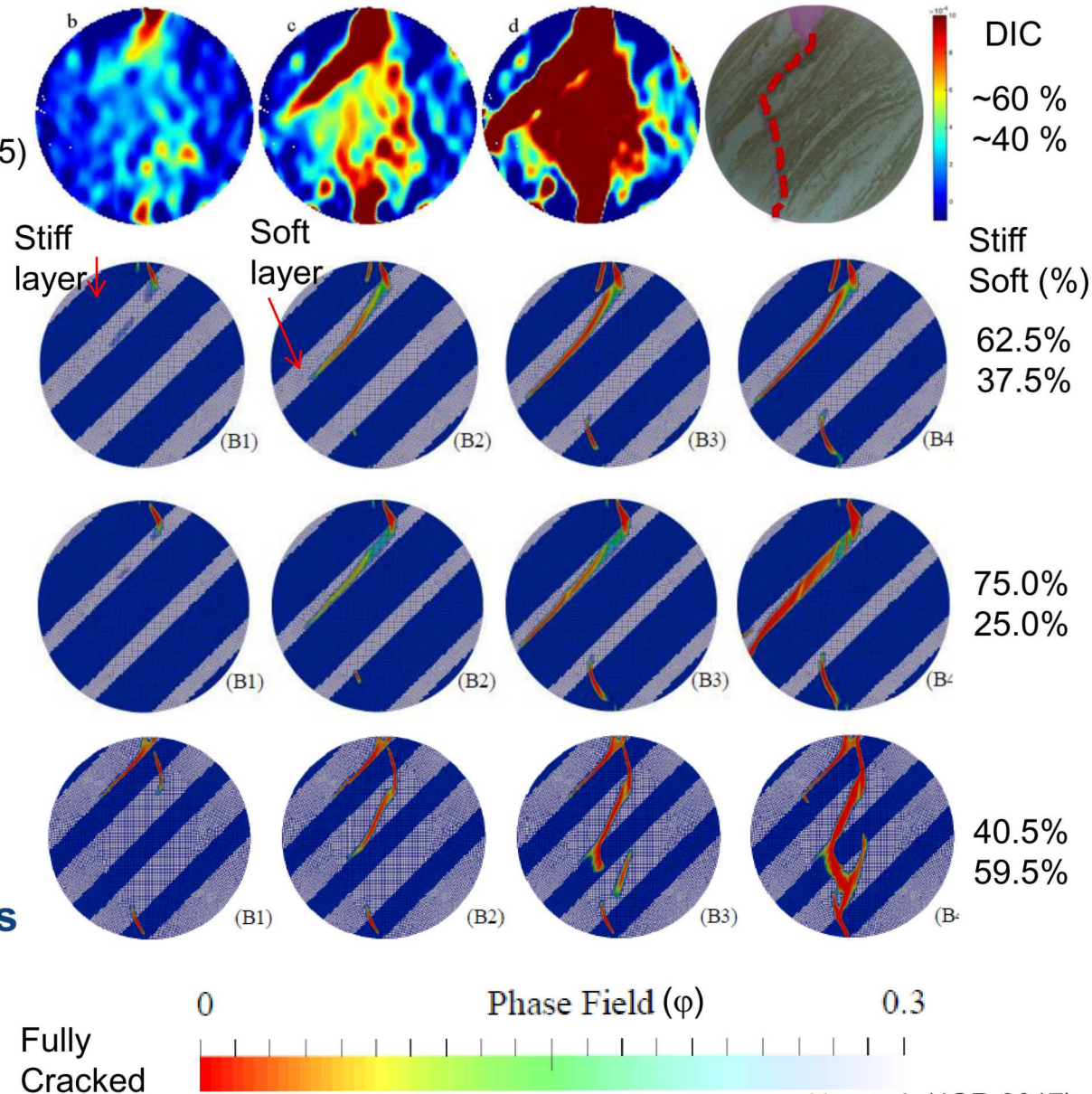
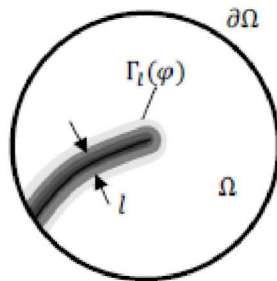
- bioturbation
- possible bioturbation
- planner laminated
- ripple laminated
- lenticular laminated



Numerical Simulations of Brittle Fracturing

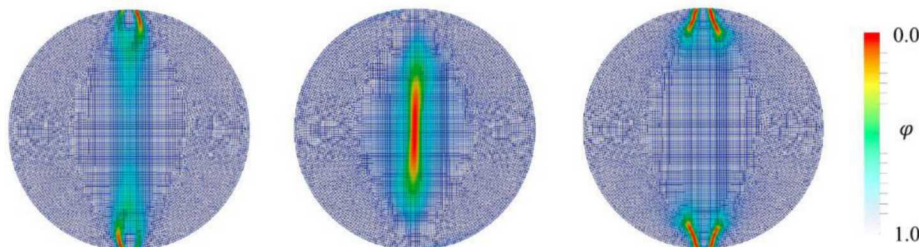
- Phase field model for crack representation (Heister et al, 2015)
- Shale is modeled as two-constituent brittle materials with stiff and soft layers:
 - Young's Modulus
 - (Pore pressure)
 - (Chemo-mechanical coupling)

Crack phase field (ϕ)



Effective Properties of Heterogeneous Materials

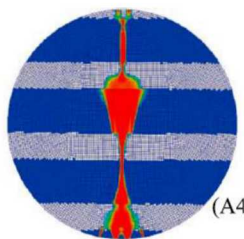
- Transversely isotropic effective medium for elastic parameters (Berryman, 1998)
 - Spatial homogenization procedure leads to much simpler crack patterns than those from the layered isotropic materials
 - Crack paths in the effective medium are less tortuous due to (probably) filtering out mesoscopic information via homogenization
 - Smaller surface area created by the fracture process yields the reduced tortuous crack paths with a diminished amount of energy dissipation (much higher effective fracture toughness)



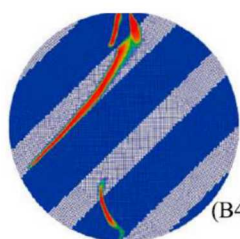
(a) Transversely isotropic (horizontal)

(b) Transversely isotropic (inclined)

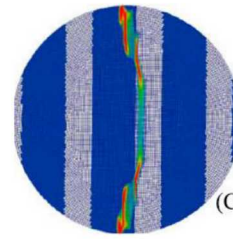
(c) Transversely isotropic (vertical)



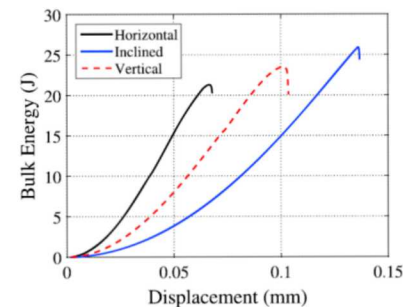
(A4)



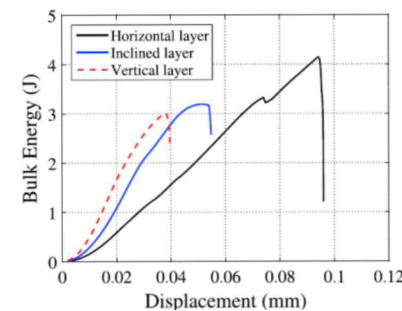
(B4)



(C4)



Transversely Isotropic



Layered Isotropic

Na et al.
(JGR 2017)

Integrated Approach

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NanoIndentation

- **Depth sensing/instrumented indentation**

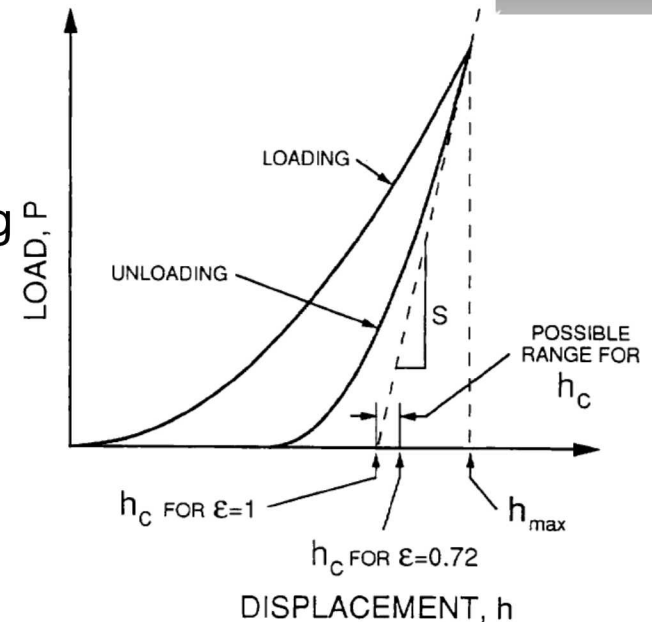
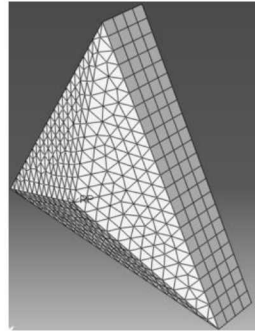
- highly accurate load-displacement record
- Analytical models to determine modulus, hardness and other mechanical properties using the load-displacement data

- **Analytical concept**

- Purely elastic deformation upon initial unloading
- Contact between a rigid indenter and homogeneous isotropic elastic half spacing
- Compliance of the sample and indenter tip – springs in series
- Hardness = load/contact area
- Elastic modulus determined by stiffness (S)

- **Dynamic Modulus Analysis at nm scale**

Berkovich indenter



Oliver & Pharr (1992)

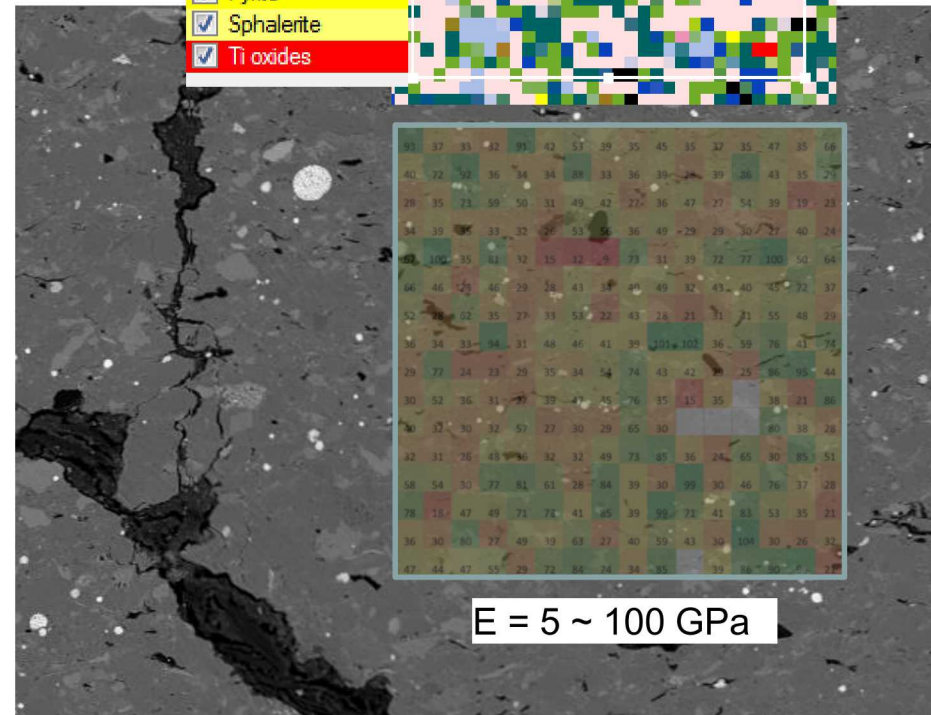
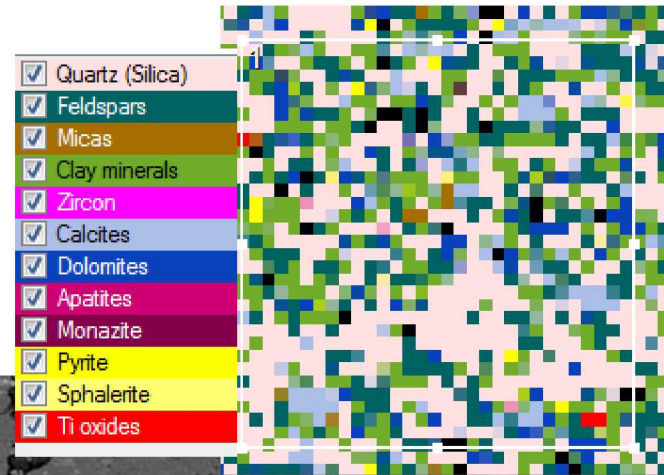
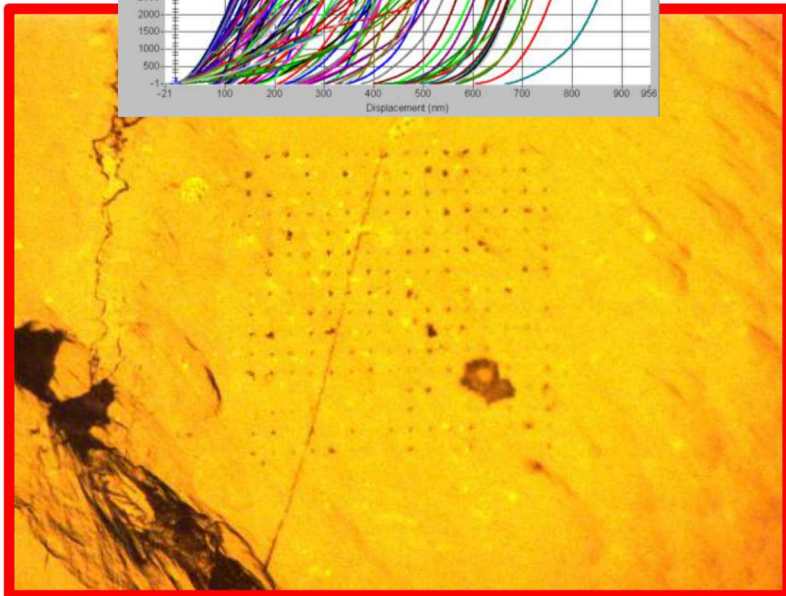
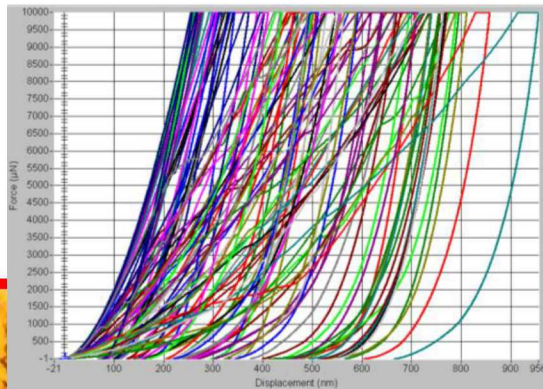
Initial Indentation Results

Indentation array: 16 x 16, 20 μm spacing

[Hysitron TriboIndenter 900]

Indentation strain rate = 0.1 (Oliver et al., 1997)
(current change in displacement/current total disp.)

Maximum load = 10 mN

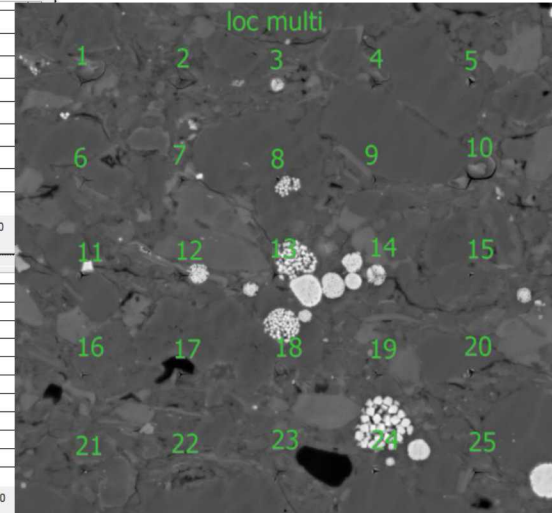
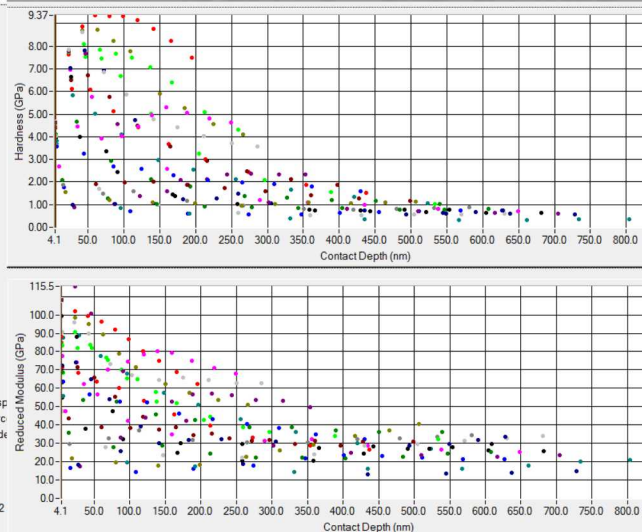
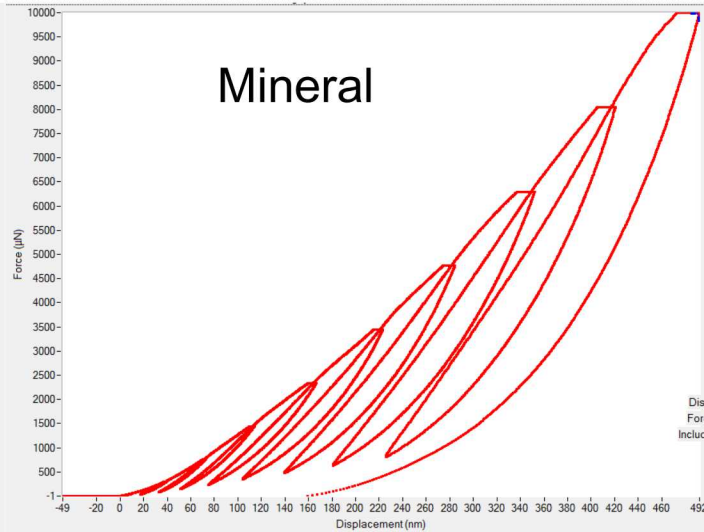


$E = 5 \sim 100 \text{ GPa}$

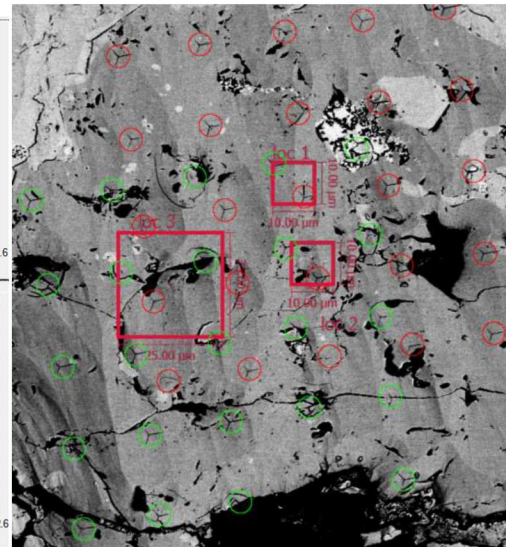
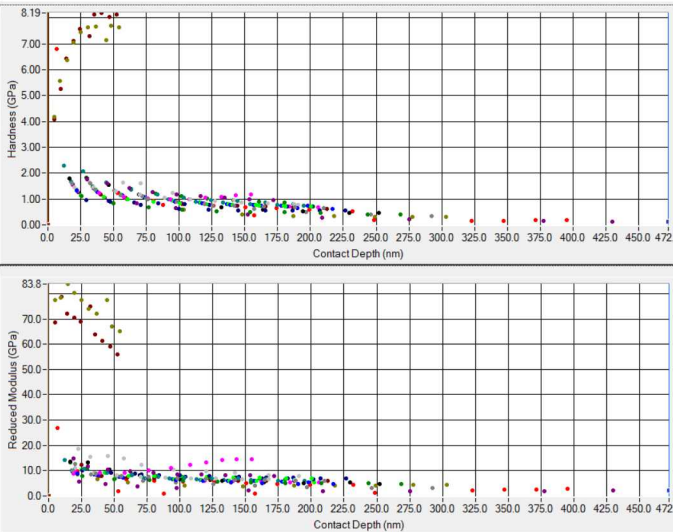
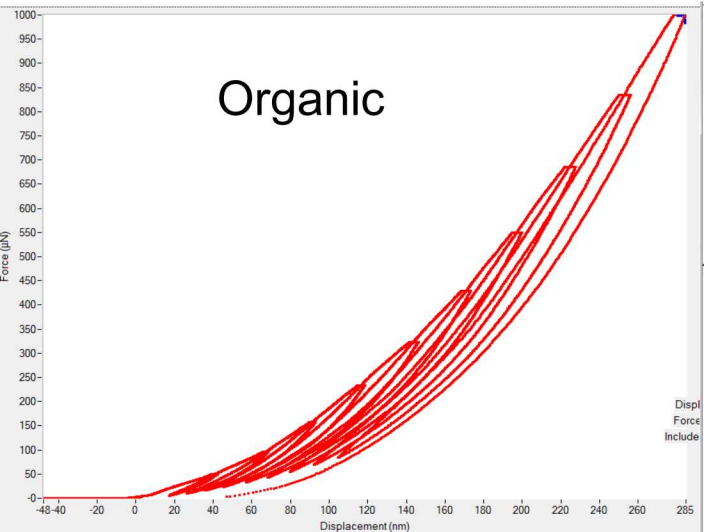
Multiscale Indentation Testing

Loading-unloading cycle

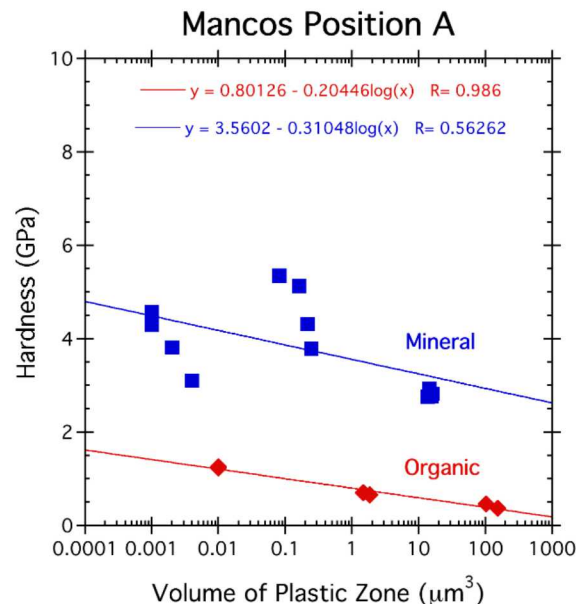
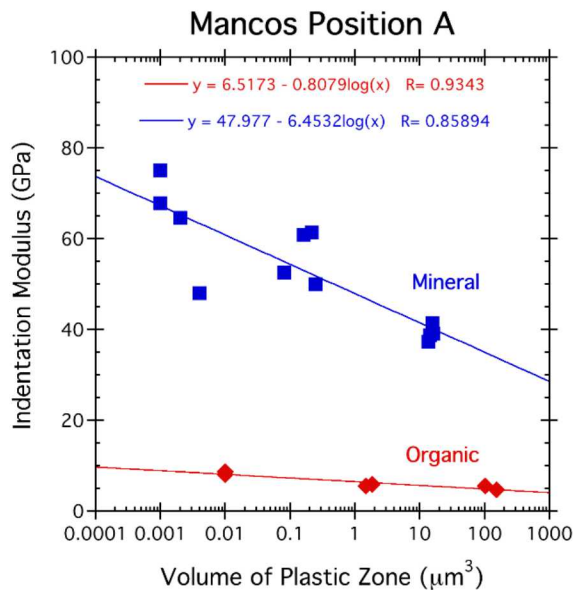
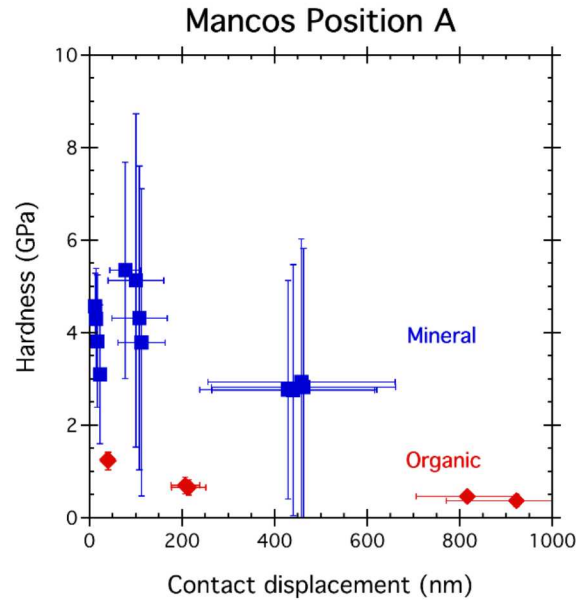
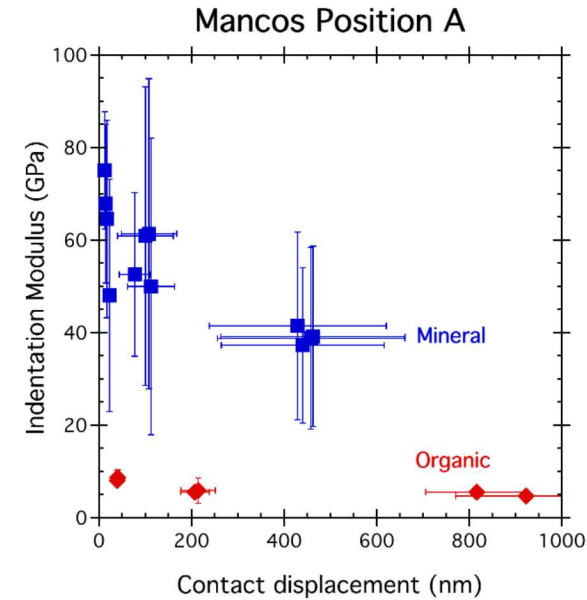
Mineral



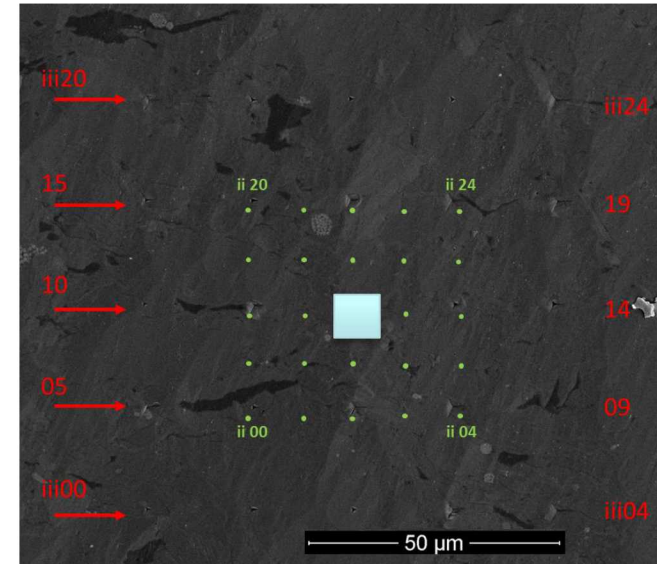
Organic



Multiscale Indentation Testing



Distributed arrays



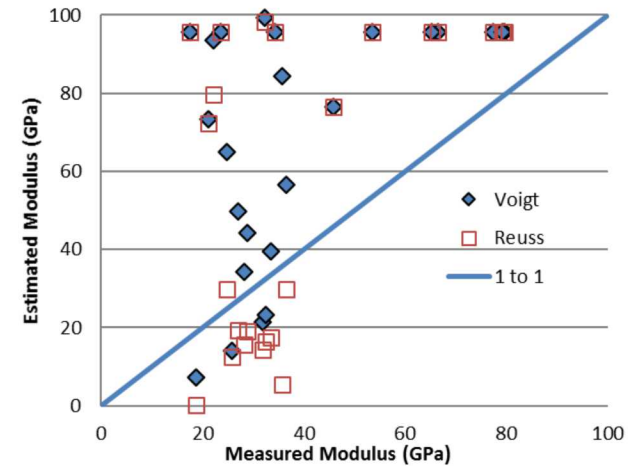
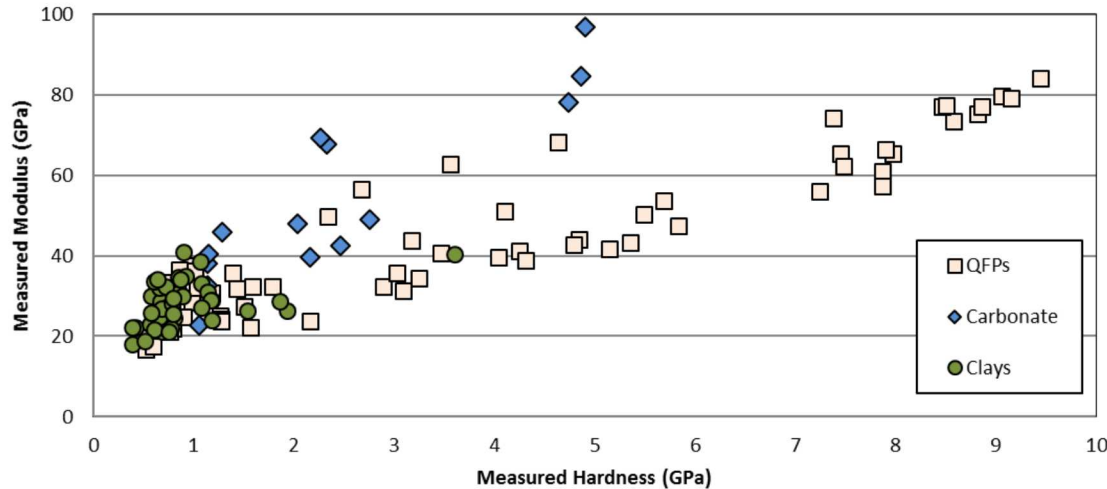
Indentation key	Array dimension	indent spacing	Max Load
i	8x8 μm	2 μm	0.1 mN
ii	40x40 μm	10 μm	1 mN
iii	80x80 μm	20 μm	10 mN

Estimate plastic zone radius

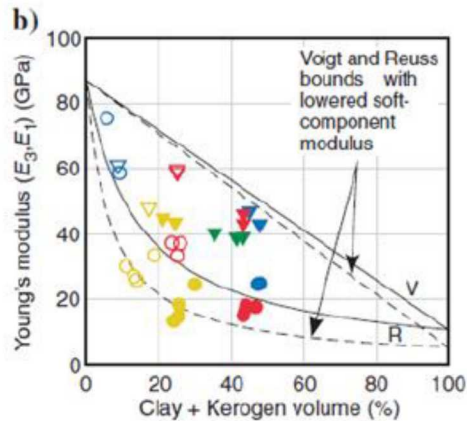
$$\frac{R_p}{\delta_m} = -12.907 \frac{H}{E_r} + 4.5451 \quad \frac{H}{E_r} < 0.35$$

Chen & Bull, Surface & Coatings Technology (2006) 4289–4293.

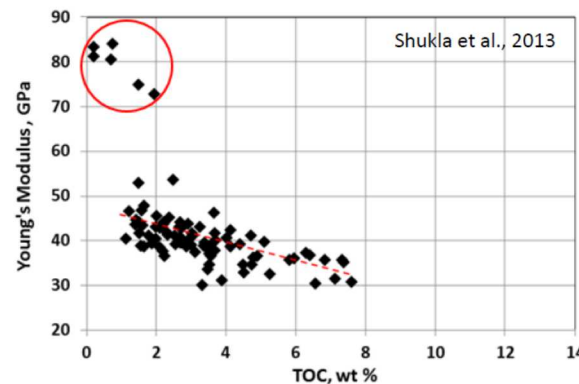
Effect of Mineralogy on Mechanical Properties



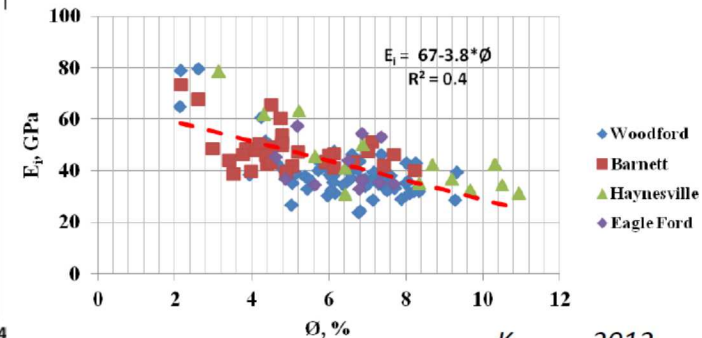
Sone & Zoback (2013)



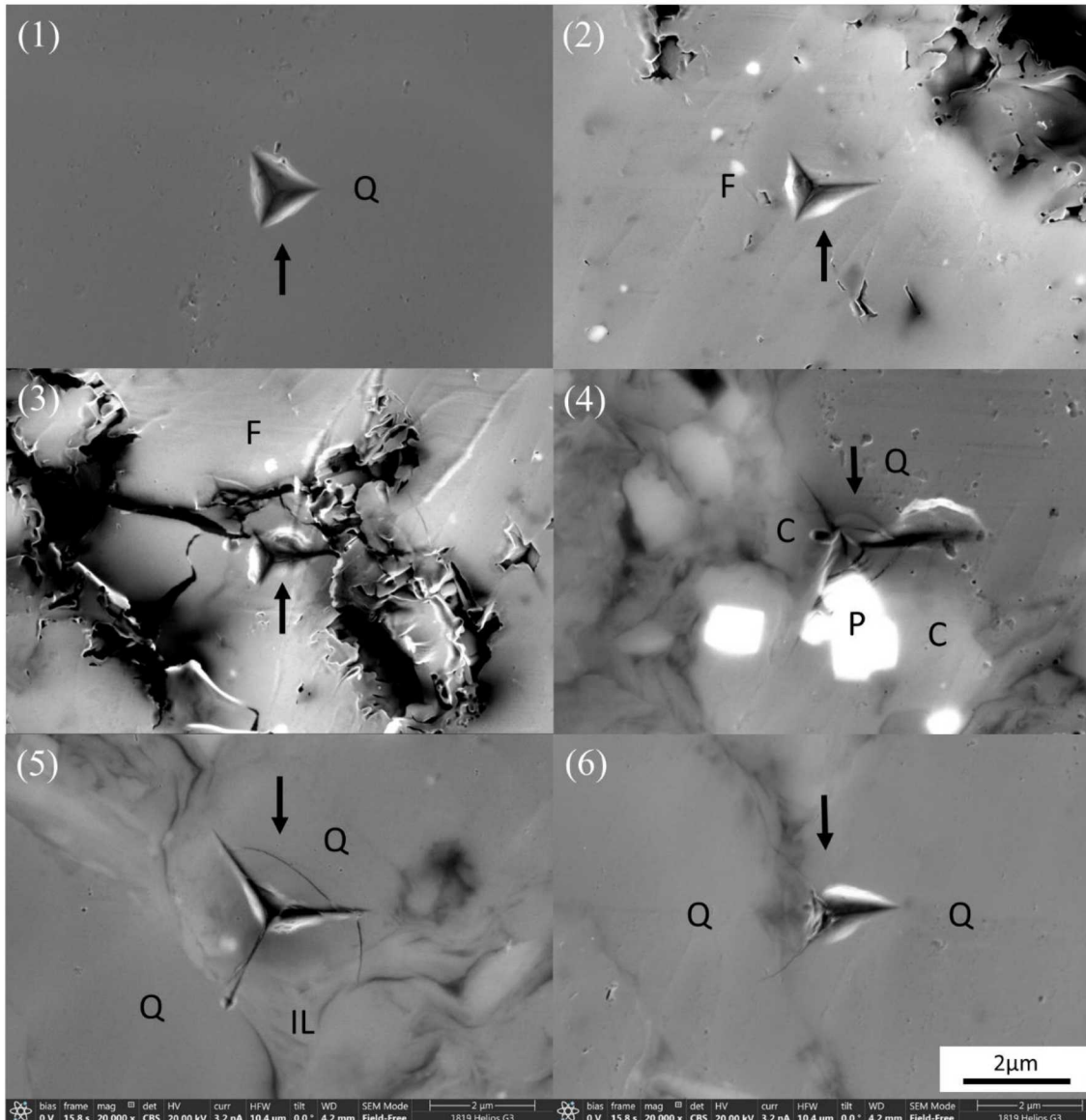
Shulka et al. (2013)



Kumar et al. (2012)



Nanoindentation Impressions

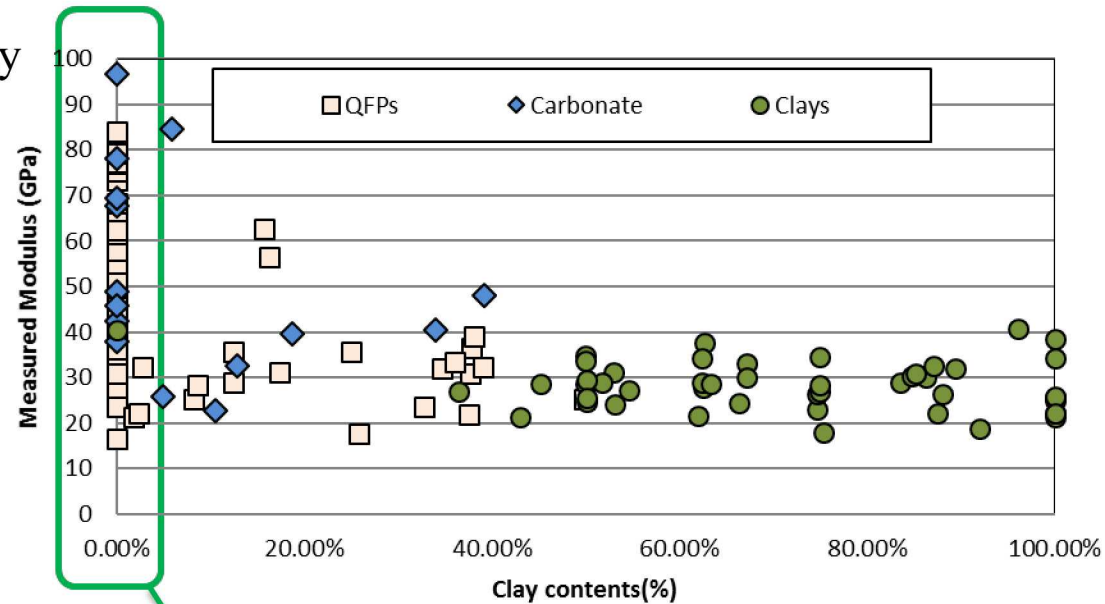


Low-clay percentage samples.
 (1&2): surface of pure quartz and feldspar having higher values of mechanical properties such as elastic modulus and hardness.
 (3): dissolution surface of feldspar
 (4-6): grain-to-grain boundary and edge-of-grain, which have lower mechanical properties values.

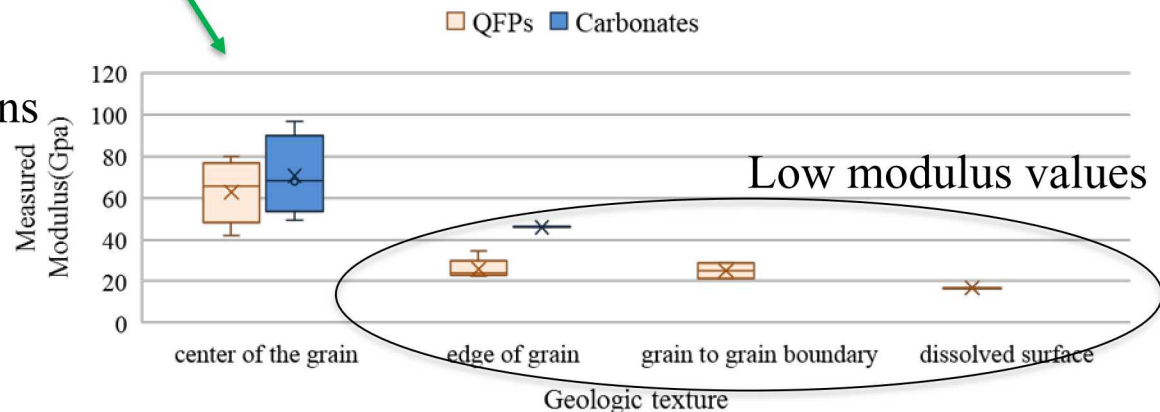
NOTE: Q=quartz, P=pyrite, C=carbonate, F=feldspar, and IL=illite)

Effect of Compositions and textures on Mechanical Properties

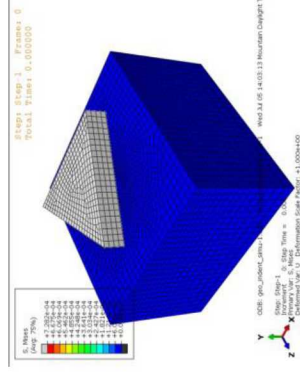
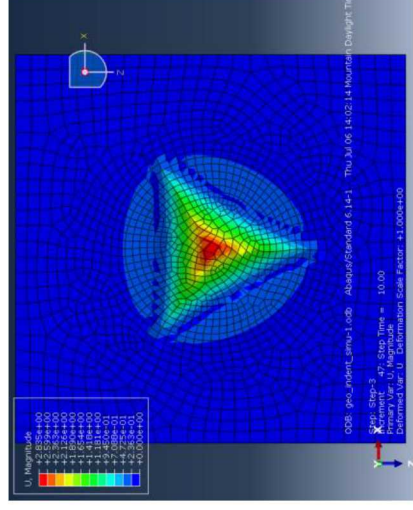
High variability



Analysis of SEM image on indentation impressions

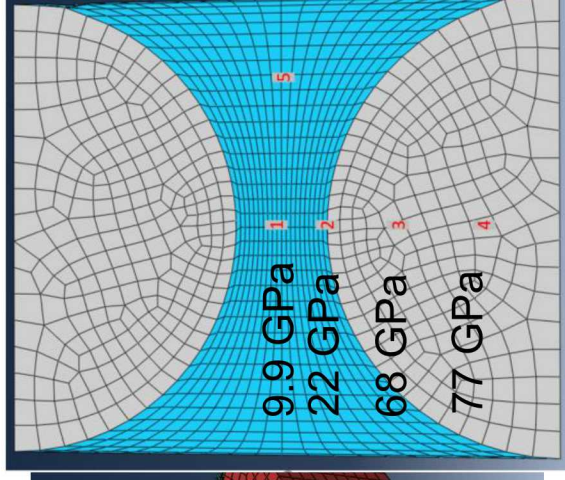
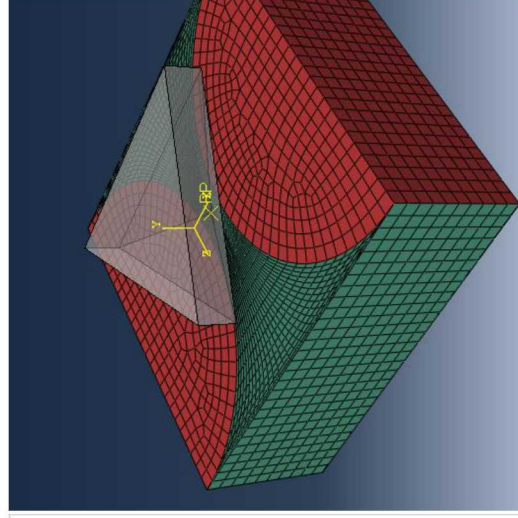
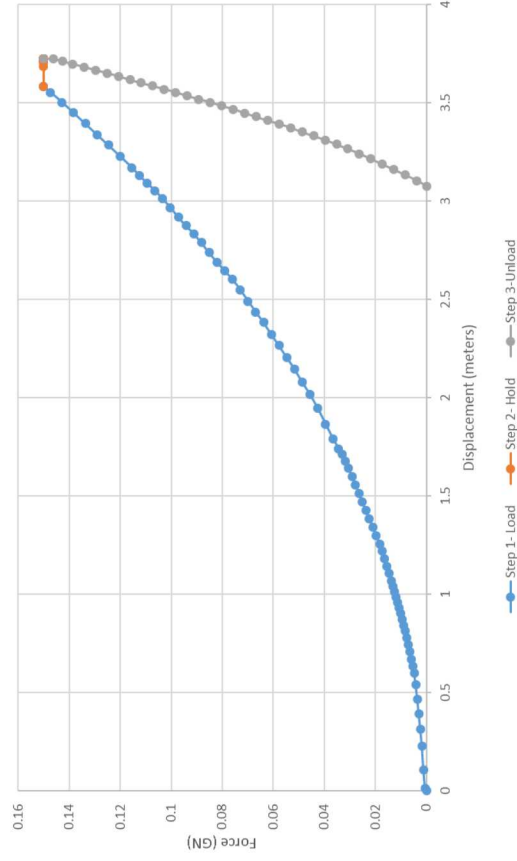


3D Mechanistic Modeling



ABAQUS FEA

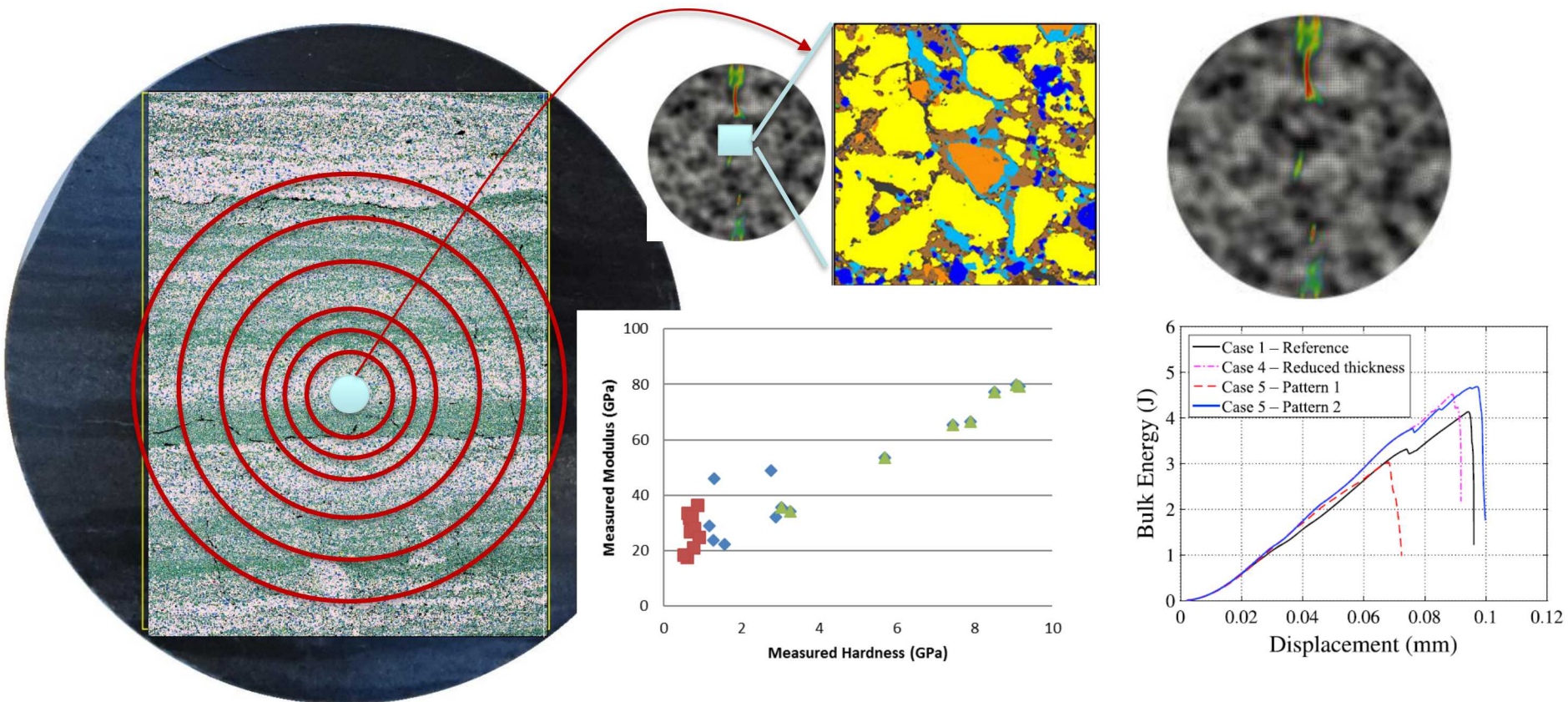
Force vs Displacement



Elastic Modulus

Upscaling for Mechanical Properties

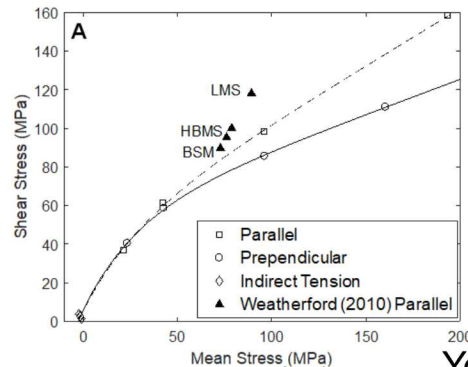
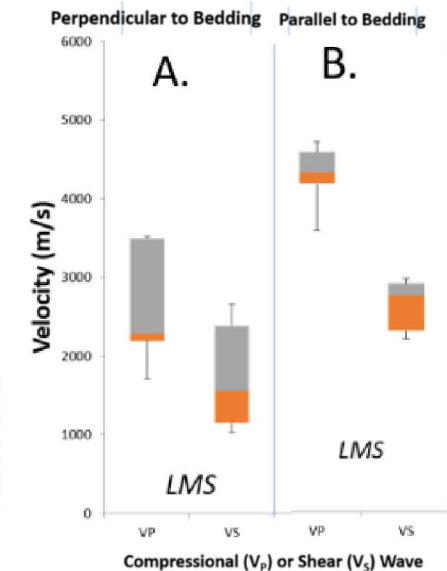
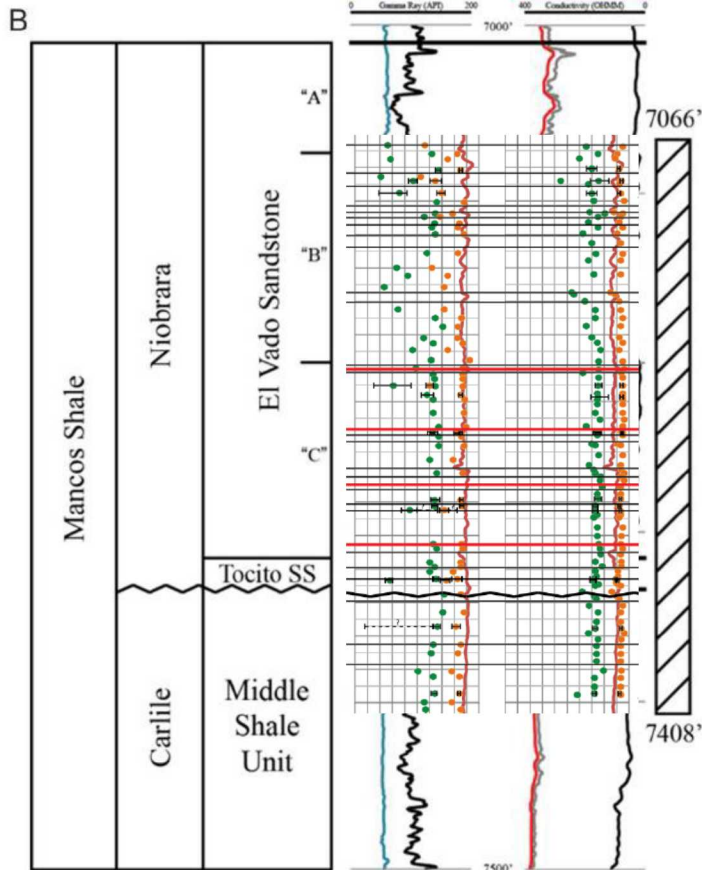
- Phase field modeling for averaging mechanical properties
 - Spatial mineralogical mapping with compositional heterogeneity
 - Development of correlation with nanoindentation results
 - Evaluation of soft cement or multi-mineral regions on mechanical responses with various conditions (e.g., defects, layering, anisotropy)



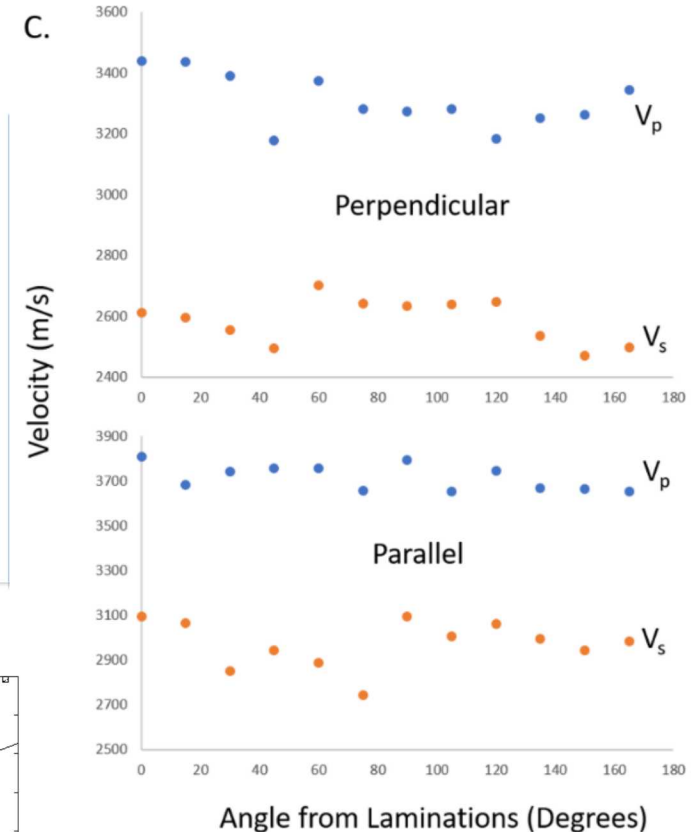
Velocities of Mancos Shale lithofacies



Drilled Core Samples



Lab Block Samples

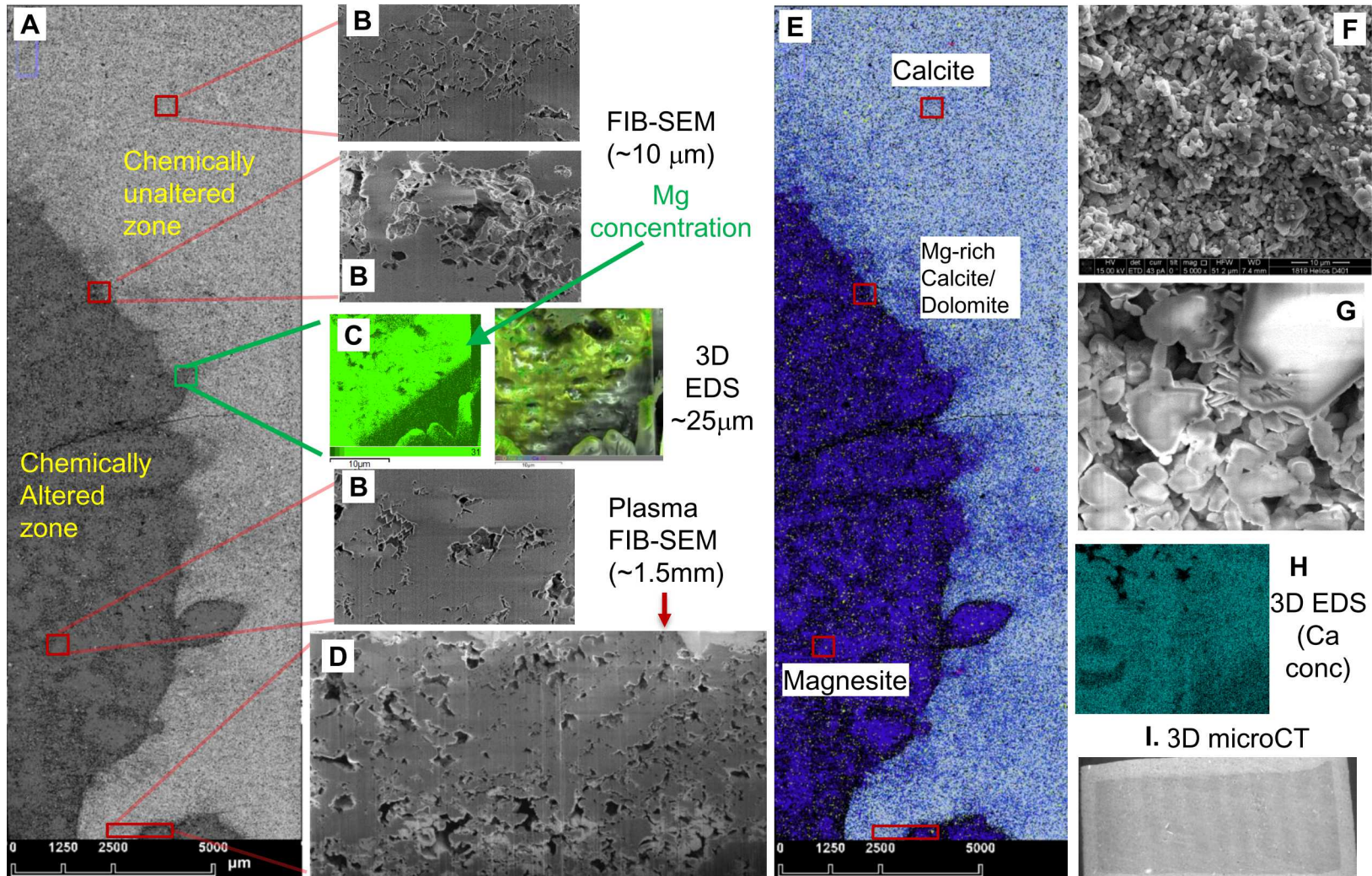


Other Ongoing Works

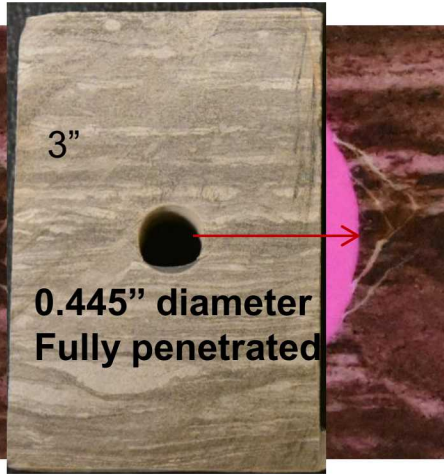
Chemo-mechanical Processes: Chemical Flooding in Nano-porous Chalk

Long-term (512 days) creep testing with chemical flooding (Liege Chalk)

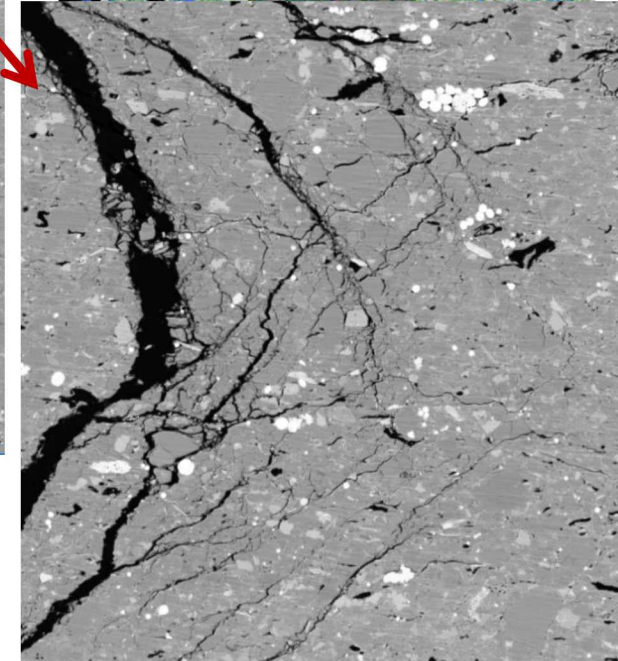
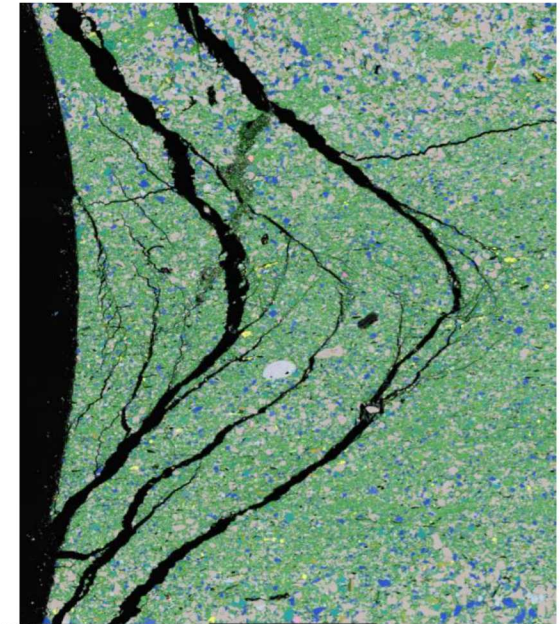
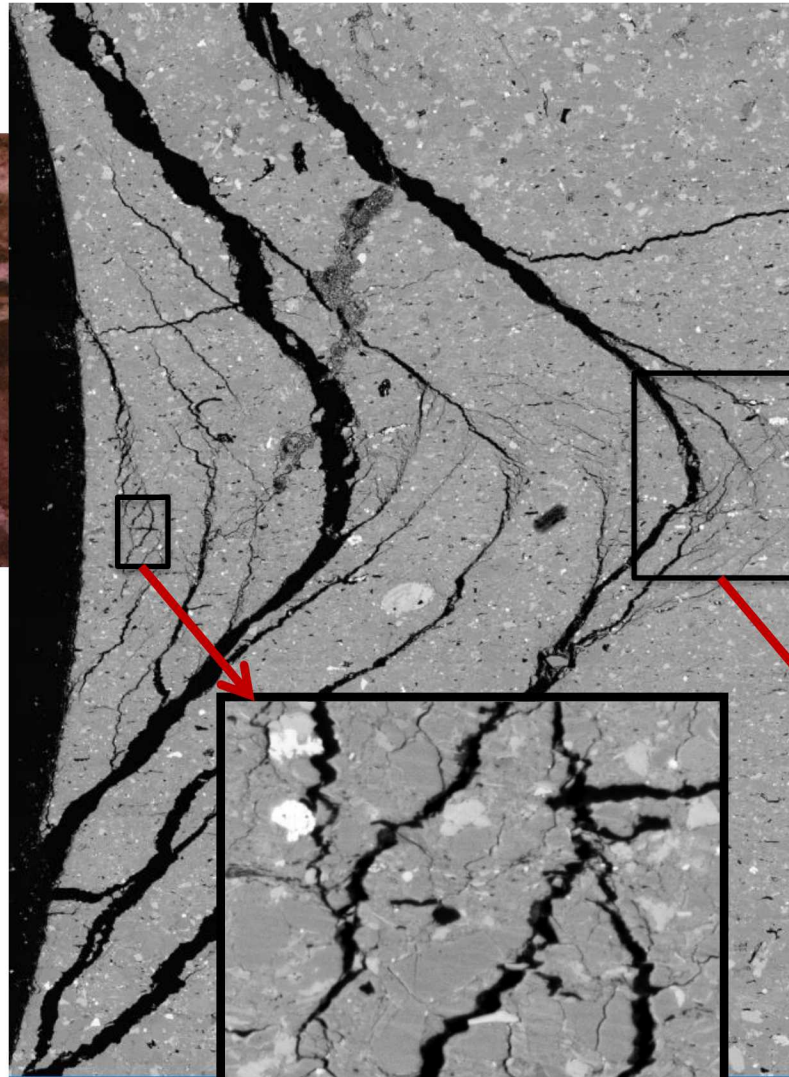
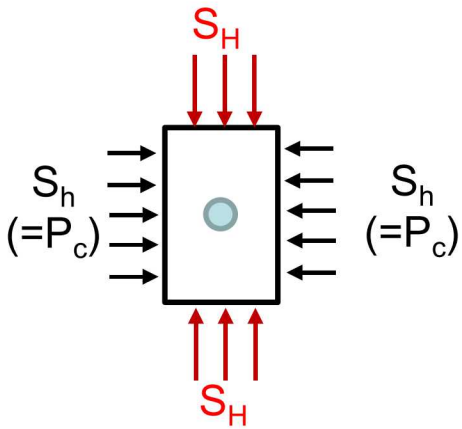
Unflooded sample



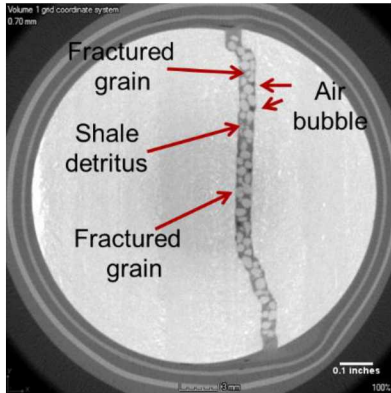
Fracture Patterns: Borehole Breakout Test



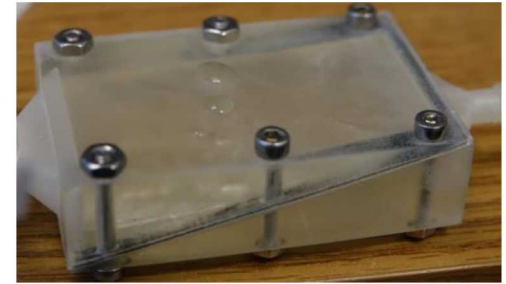
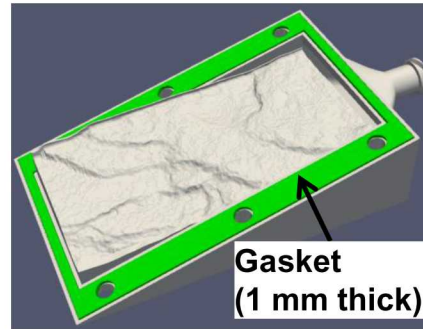
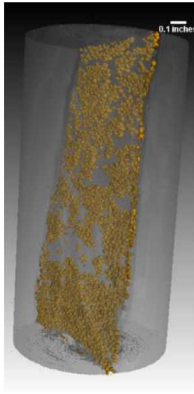
2" diameter



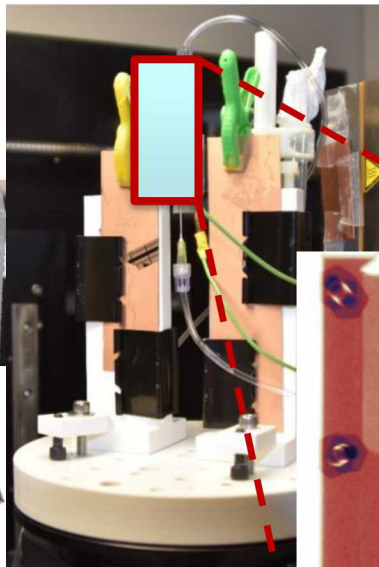
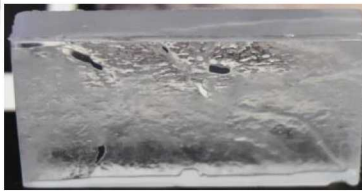
Permeability Control and Proppants' Behavior in Fractures



Micro-CT image with proppants in a fracture



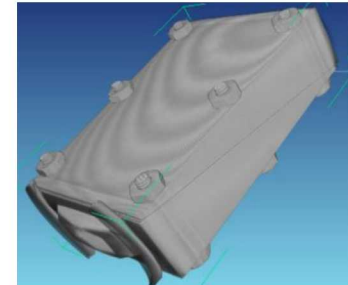
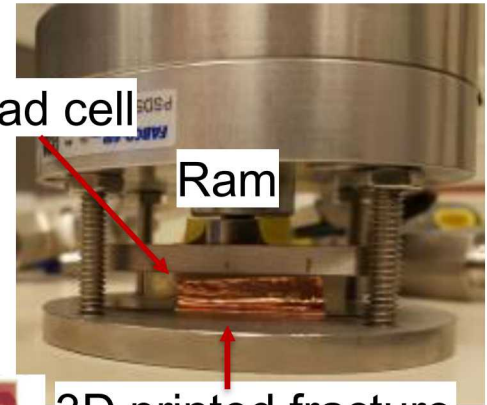
3D printing applications



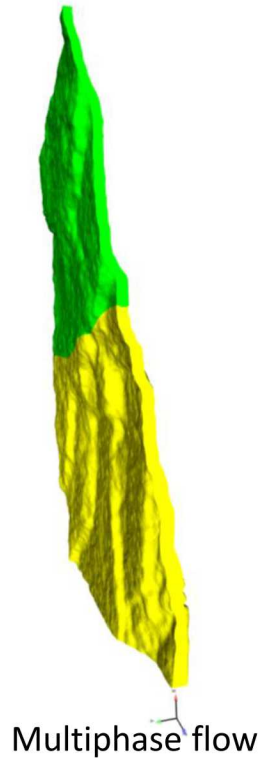
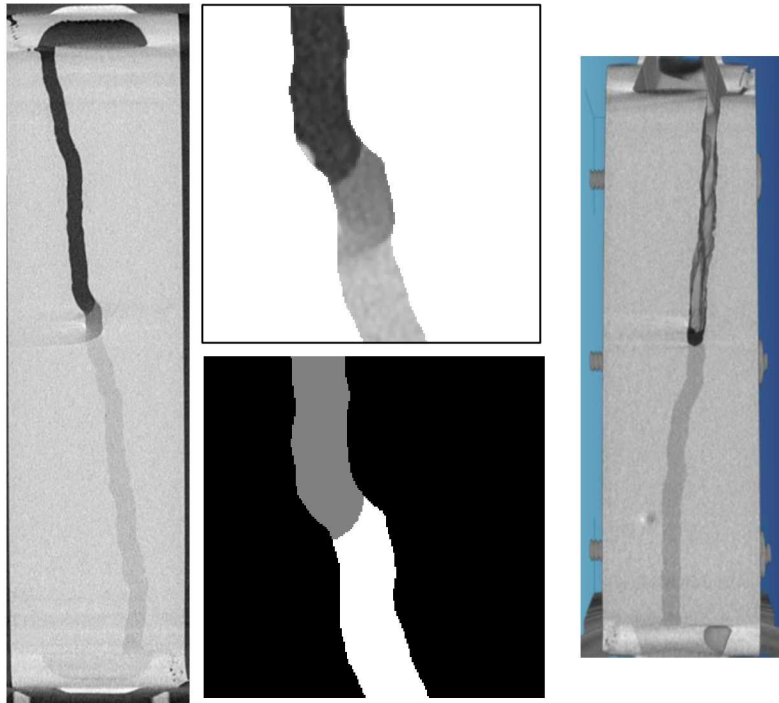
Load cell

Ram

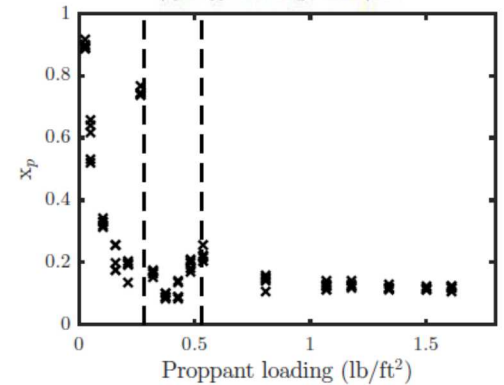
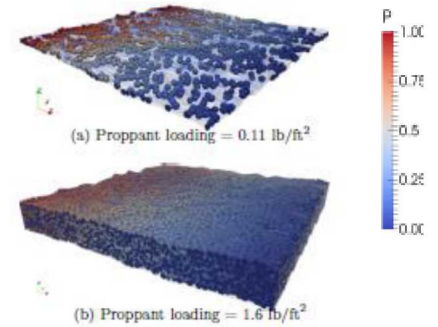
3D printed fracture



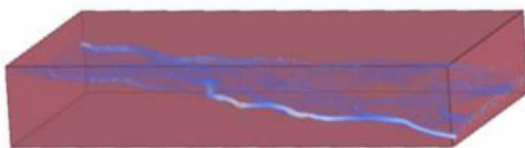
Multiphysics Simulations



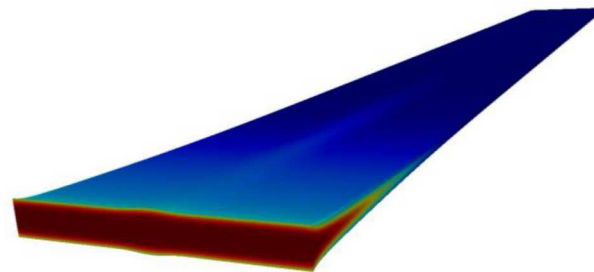
Multiphase flow



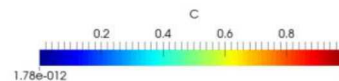
Proppants' behavior



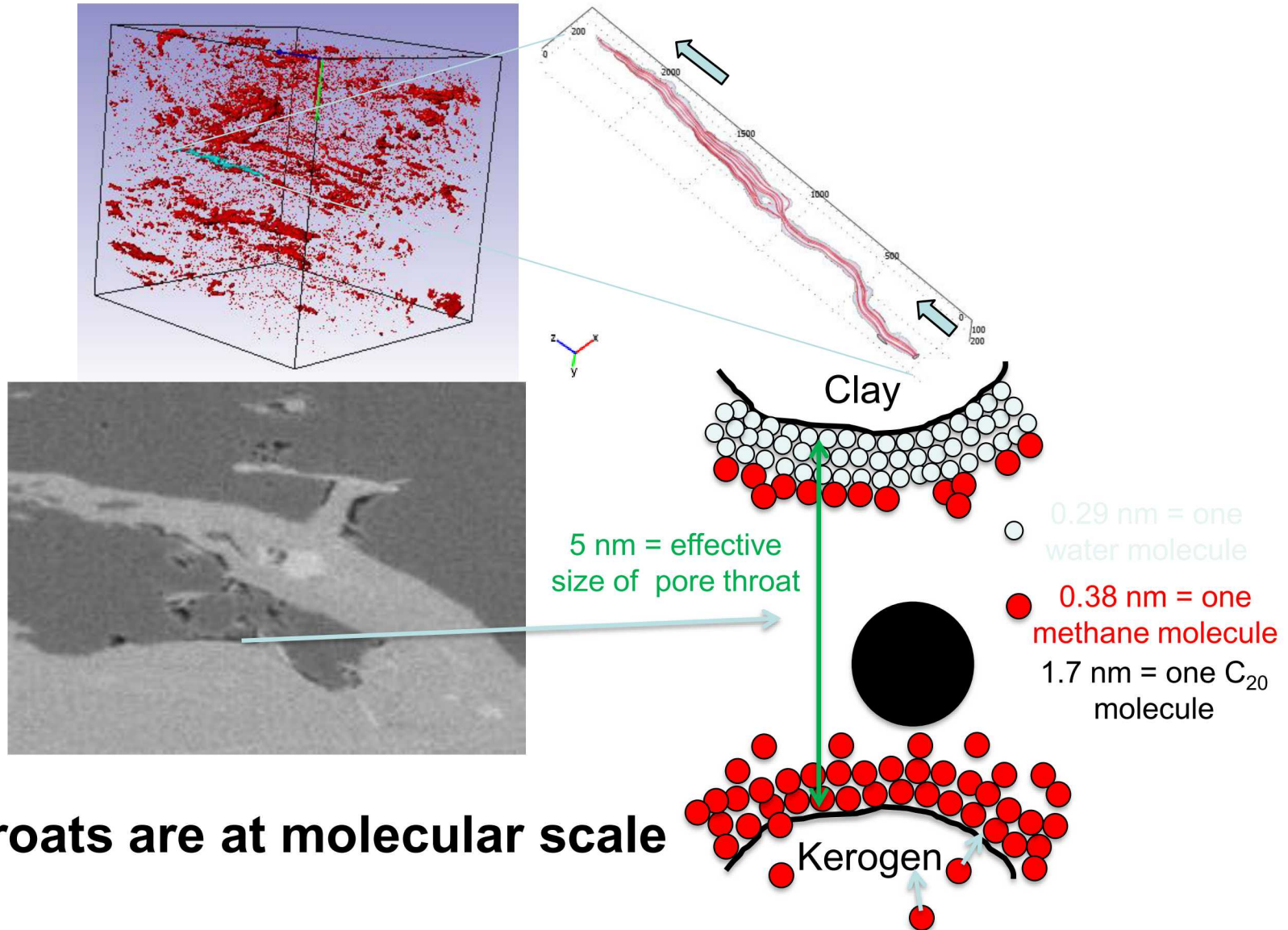
CFD simulation flow field



Reactive Transport



Methane flow in kerogen “tubule”

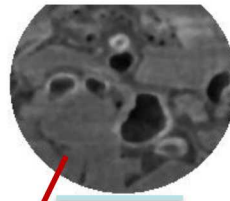


Multiscale network and flows with ROMs + ML

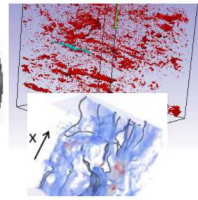
Level 1:

- Nano/micro pore network (PN) & flow model
- Generative adversarial neural networks

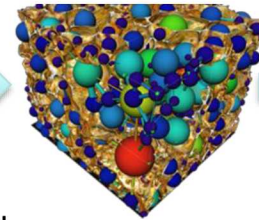
Degree of Freedom (DoF) = 10^9 PN, DoF = 10^3 - 10^4



1 μ m



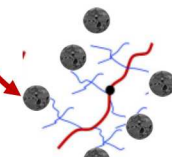
High fidelity model



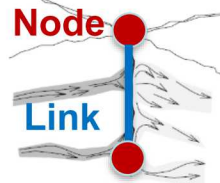
Data-driven model with ROMs (DL for flow sculpting, Srep46368)

Level 2:

- Natural/induced fracture network (FN) & flow model
- Graph network system

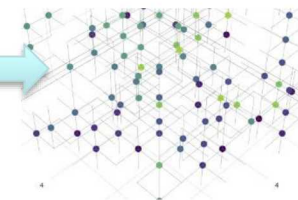


100's m



High fidelity model

Graph network



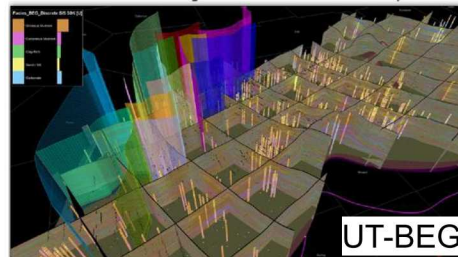
Transfer learning or response function

Data-driven model with graph network model

Level 3:

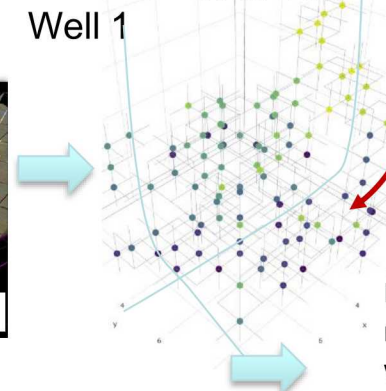
- Well production & operations
- Well systems + graph network under realistic operation conditions

Well systems + Operations



Well 1

Well 100



Multi graph networks

Transfer learning or response function

Data-driven model developed with field data

Summary

- Integrated multiscale imaging and mechanical testing with numerical simulation provides a robust approach to advancing our understanding of shale (poro-) mechanical behaviors
- Texture/mineralogical characterization
 - Recent advances in mineralogical mapping with high resolution imaging over the large area
 - Multiscale mineralogical and geologic features lead to considerable heterogeneity of mechanical properties
- Mechanical tests
 - Macroscopic and microscopic lithofacies have distinctively different mechanical properties
 - Bulk properties/averaging theory may be misleading as they can represent averages of mechanically heterogeneous rock
 - Microscopic heterogeneity of mechanical properties can control the spatial distribution of fractures
 - This heterogeneity should be taken into account for realistic mechanical modeling and can scale up by rigorous theoretical and numerical modeling