Multiscale characteristics of mechanical and mineralogical heterogeneity using nanoindentation and Maps Mineralogy in Mancos Shale

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Exceptional service in the national interest

AGU 2017

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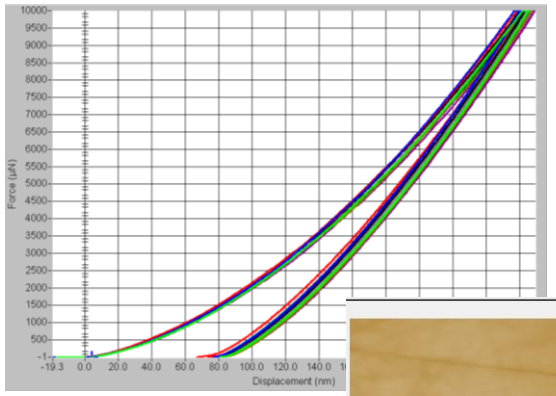
National Nuclear Security Administration

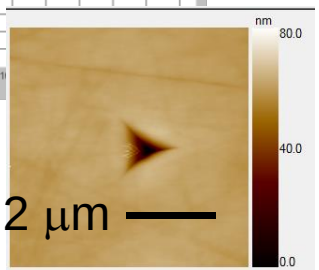
Multiscale Heterogeneity in Compositions, Pore structure, and Mechanical Properties

► 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

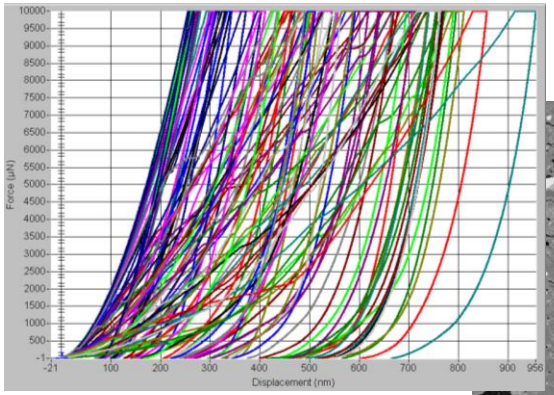
Quartz area, 20 indents
Load-displacement

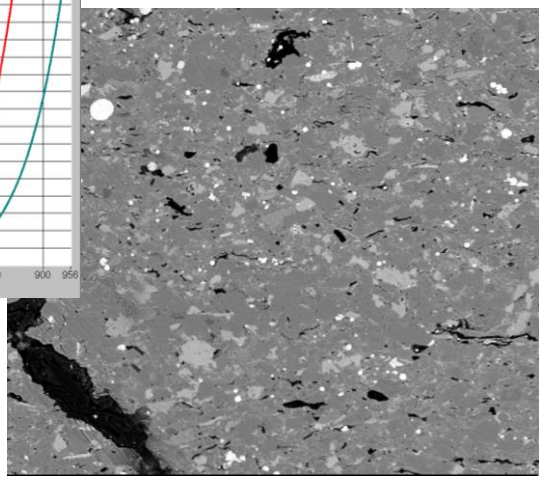




Er (Gpa) =
80.8 ± 1.3

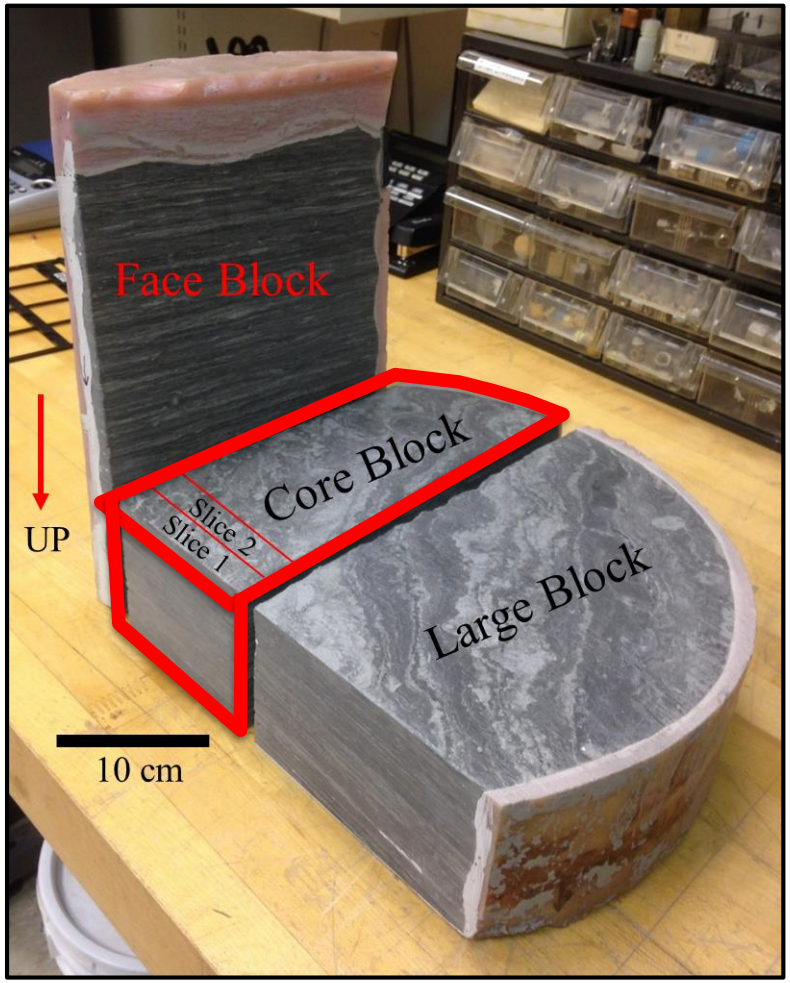
Clay-rich area, 64 indents
Load-displacement





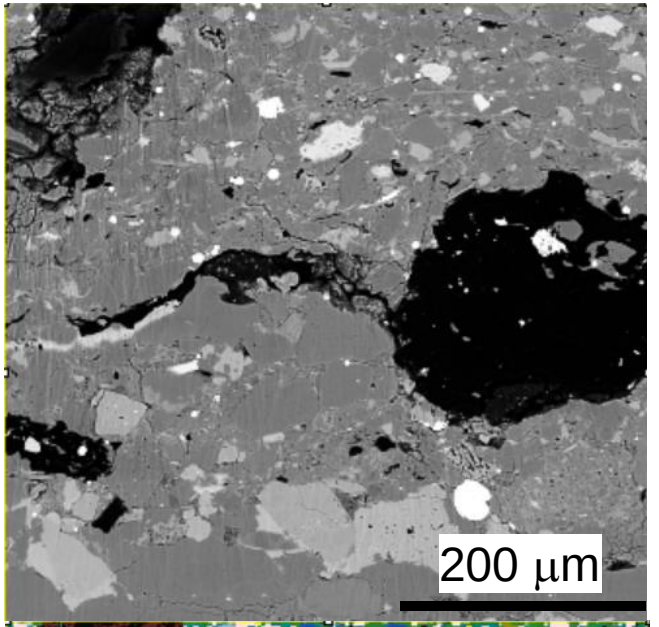
Multiscale Approach

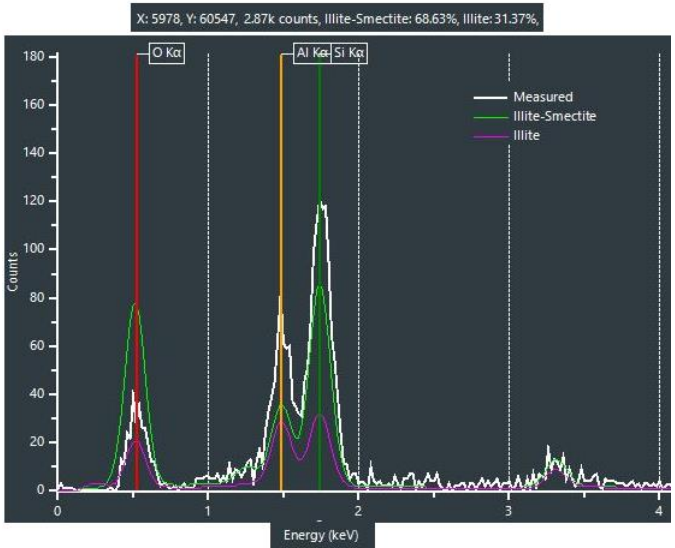
- **40 cm diameter core of Mancos Shale**
  - Interlaminated fine mud, medium/ coarse mud, and very fine sand
  - 1-3 mm laminae
  - Parallel lamina, wavy lenticular lamina, ripple forms, and bioturbation
- **Mineralogical and textural characterization**
  - Macroscopic
  - Optical petrography/microscopy
  - Micro-CT
  - FIB-SEM
  - **BSE**
  - **MAPS Mineralogy**
- **Mechanical tests**
  - Uni-/Tri-axial compression (1x2")
  - Brazilian Test (1x0.5")
  - **Nanoindentation**
- **Computational modeling**

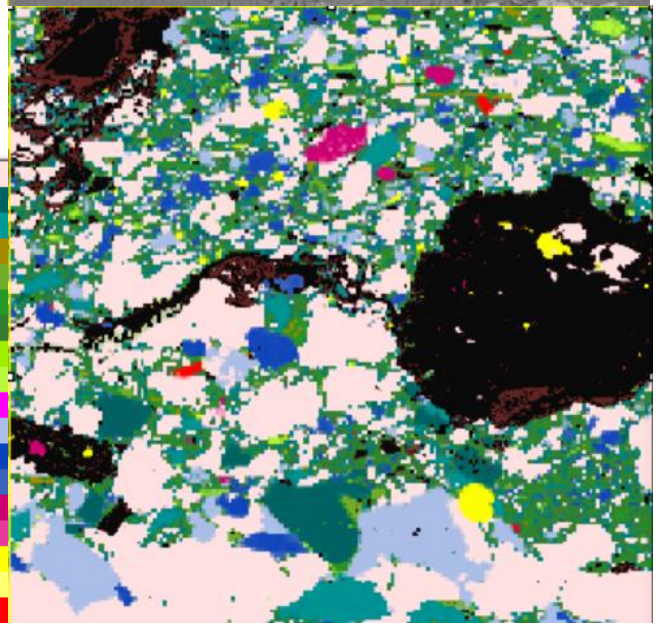


MAPS Mineralogy

- **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

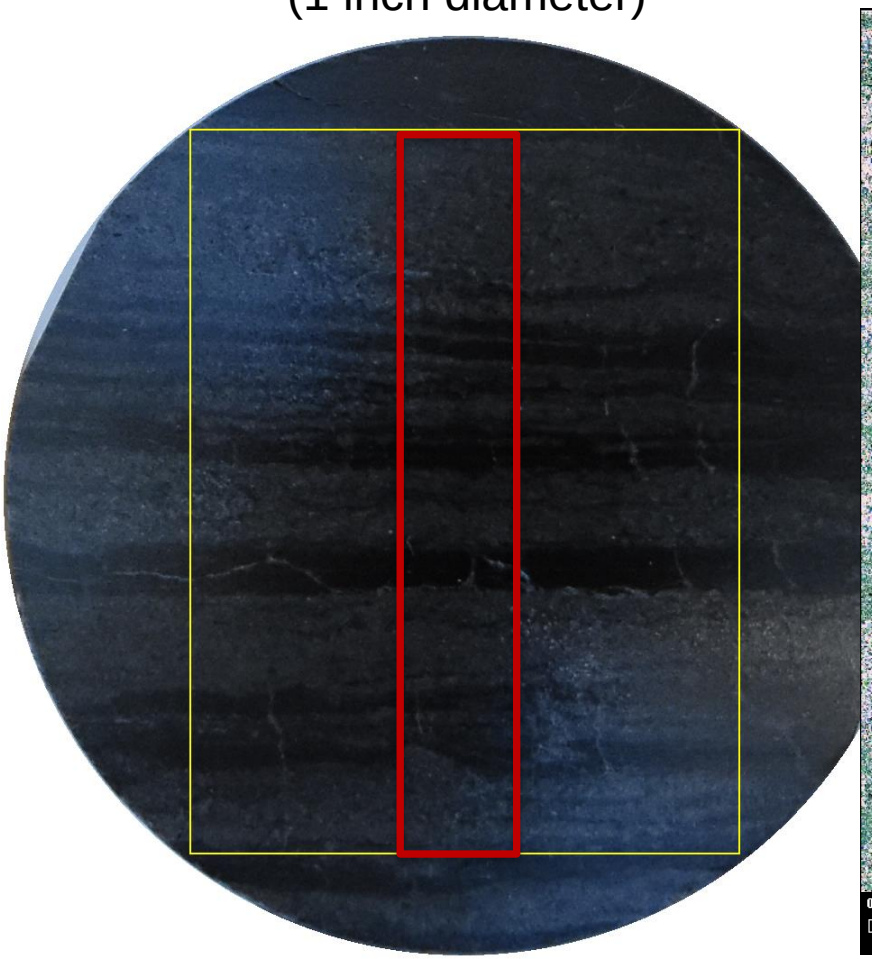


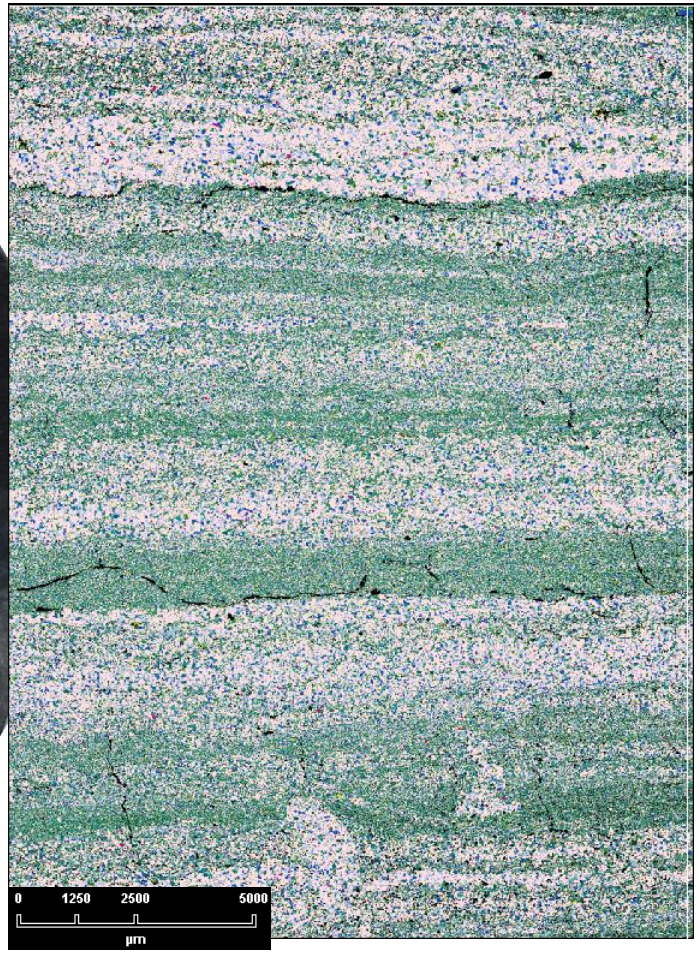




Mineralogy Mapping

Ion-milling polished Mancos
(1 inch diameter)

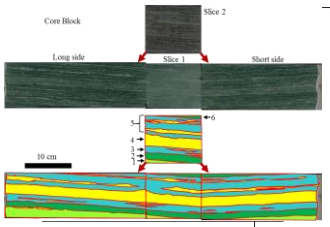


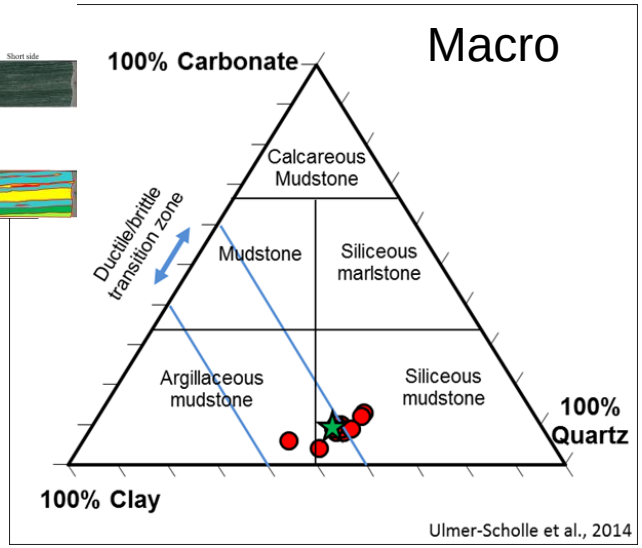


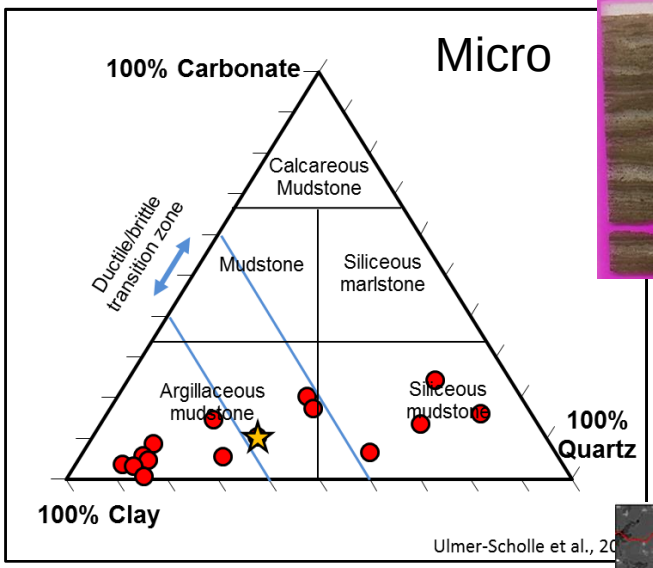
- Quartz (Silica)
- K-feldspar
- Albite
- Muscovite
- Kaolinite (Halloysite, Dickite)
- Illite
- Illite-Smectite
- Clinochlore
- Chamosite
- Zircon
- Calcite (Aragonite)
- Dolomite
- Ankerite
- Apatite (F)
- Apatite (Cl)
- Pyrite
- Sphalerite
- Rutile/Anatase/Brookite

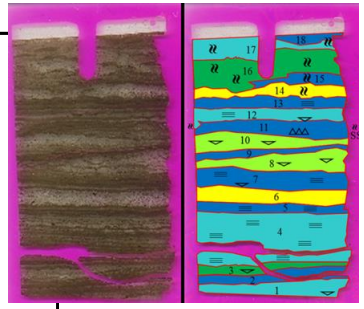
Yellow Box (1.45 x 1.98 cm): BSE @ 1µm & MAPS @ 10µm
Red box (0.18 x 1.98 cm): BSE @ 0.2µm & MAPS @ 2µm

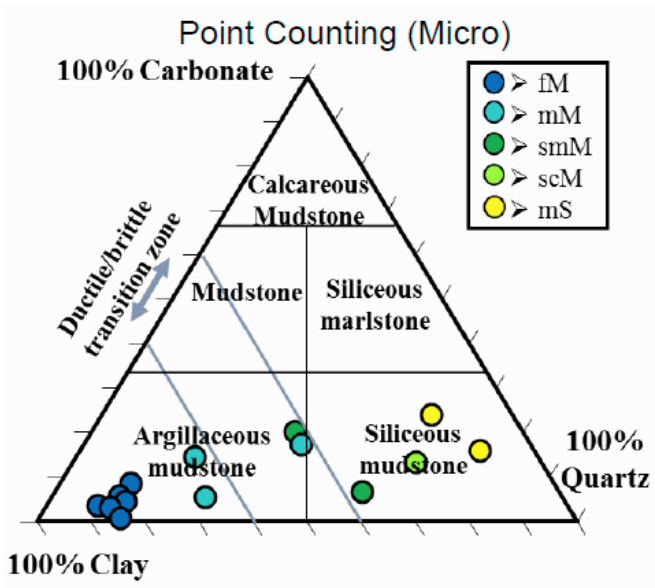
Mineralogy Mapping: Scale/methods dependent

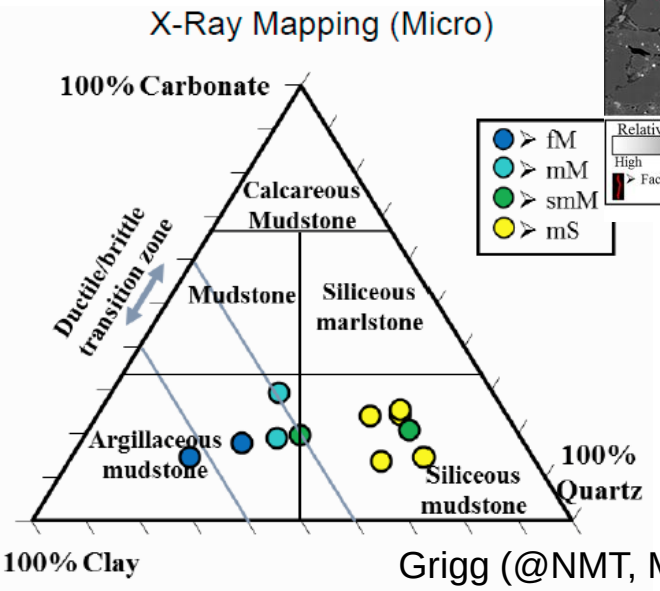


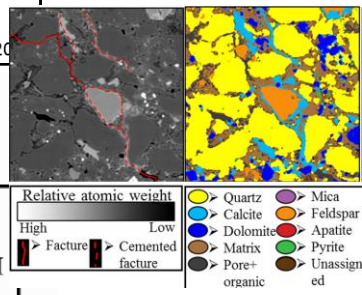






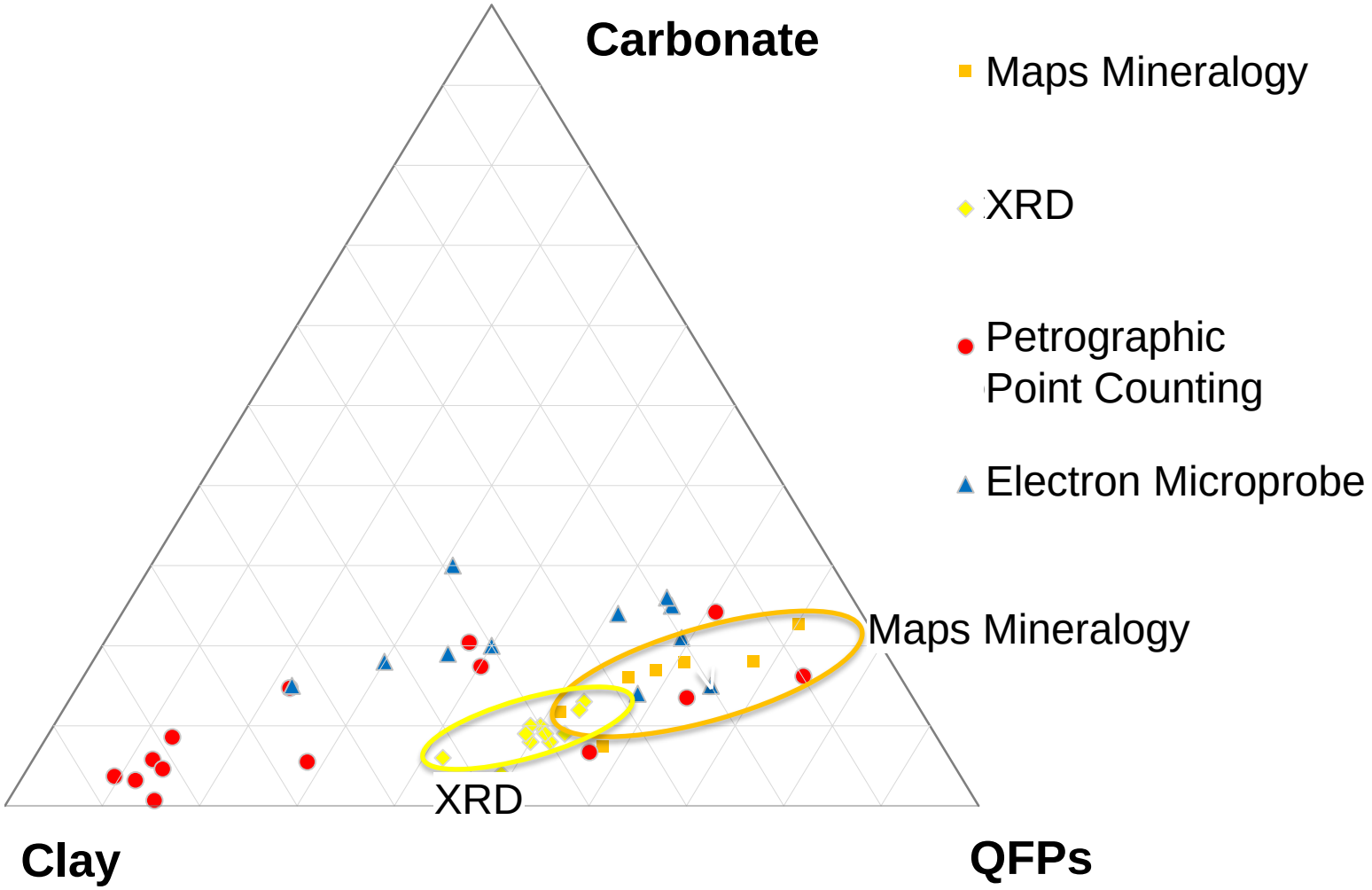




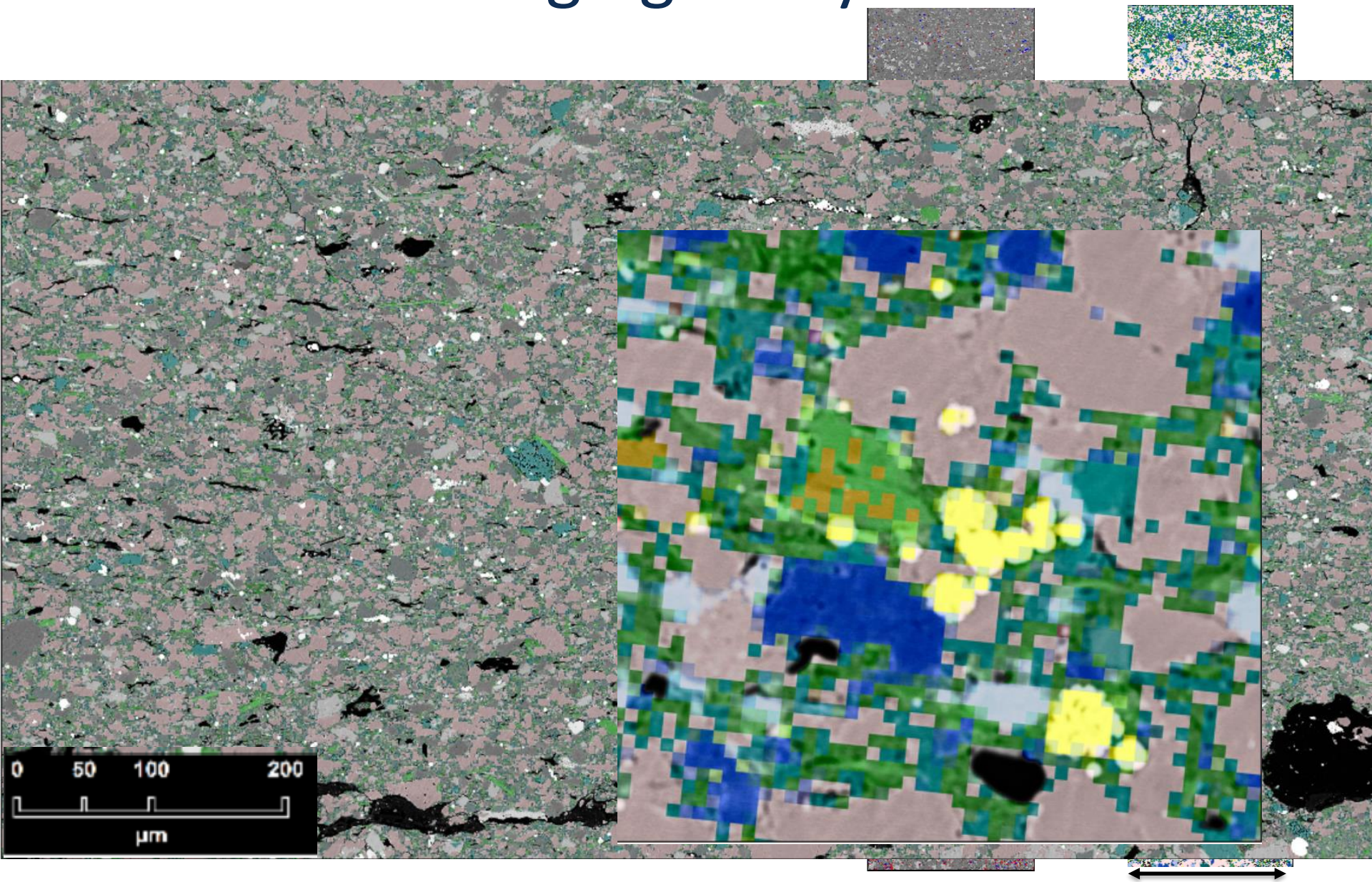


Grigg (@NMT, MS thesis, 2016)

Mineralogy Mapping: Scale/methods dependent

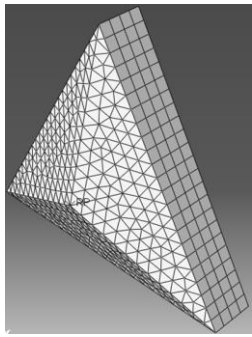


Imaging Analysis

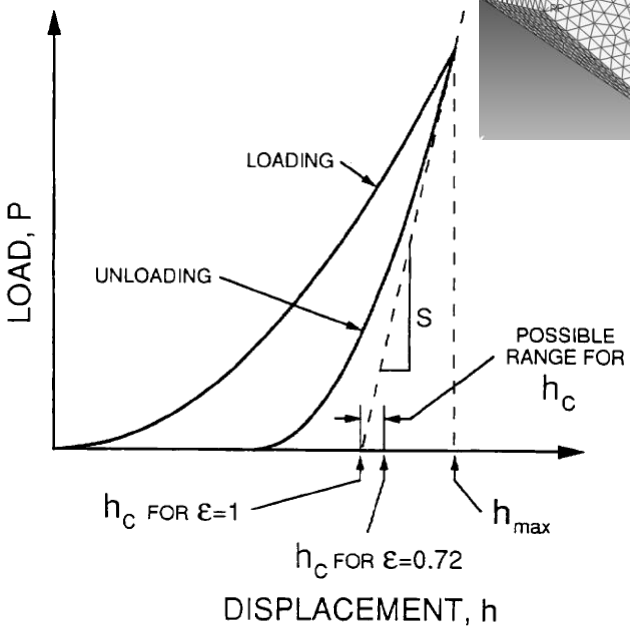


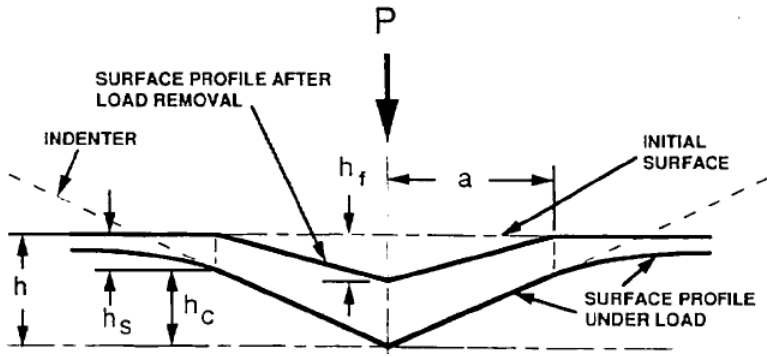
NanoIndentation

- **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)
- **Berkovich indenter**
  - – Sharp and well-defined tip geometry
  - – Well defined plastic deformation into the surfa
  - – good for modulus and hardness measures
- **Dynamic Modulus Analysis at nm scale**



Berkovich indenter

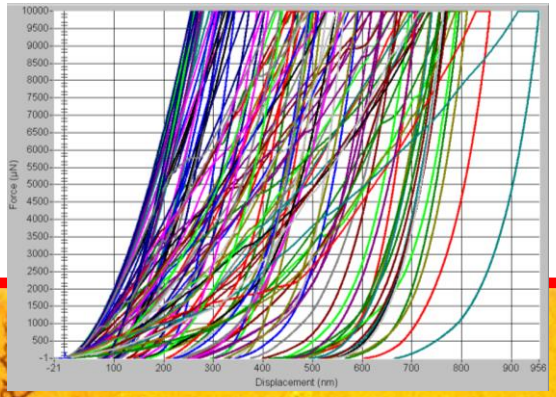


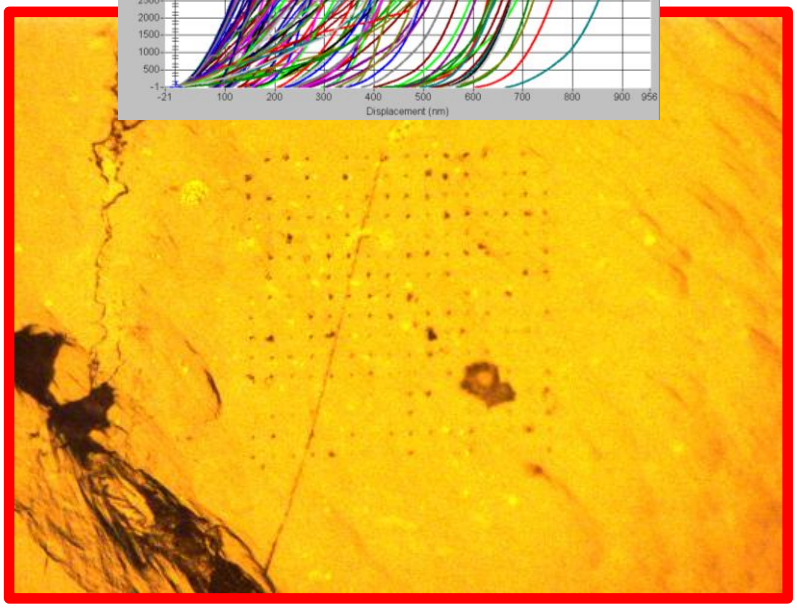


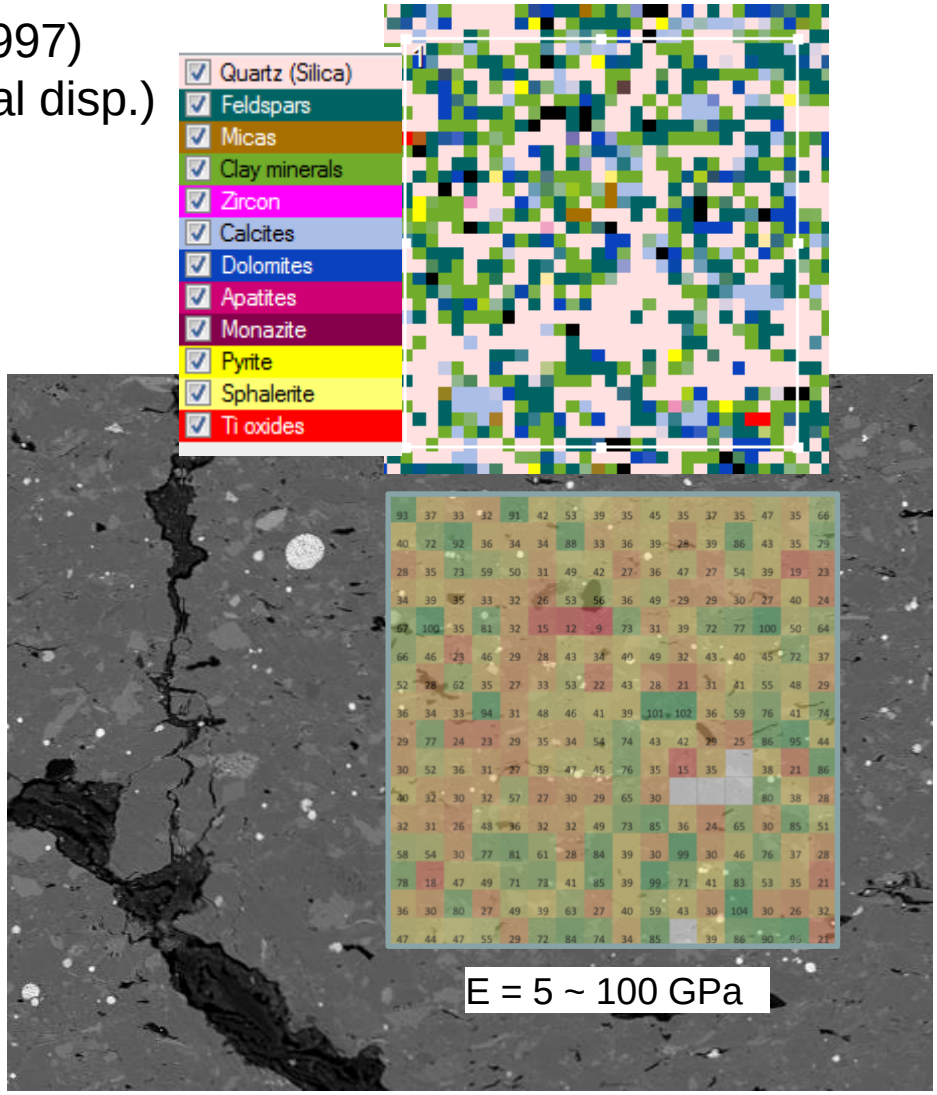
Oliver & Pharr (1992)

Initial Indentation Results

Indentation array: 16 x 16, 20 um spacing
[Hysitron TriboIndenter 900]
Indentation strain rate = 0.1 (Oliver et al., 1997)
(current change in displacement/current total disp.)
Maximum load = 10 mN

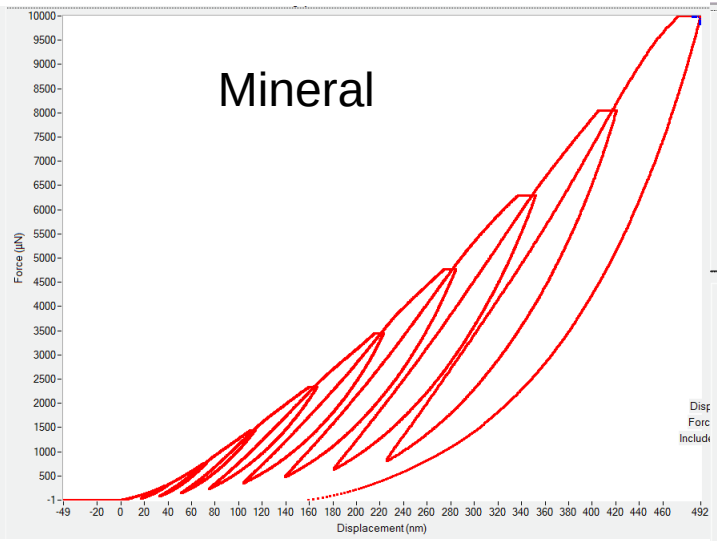


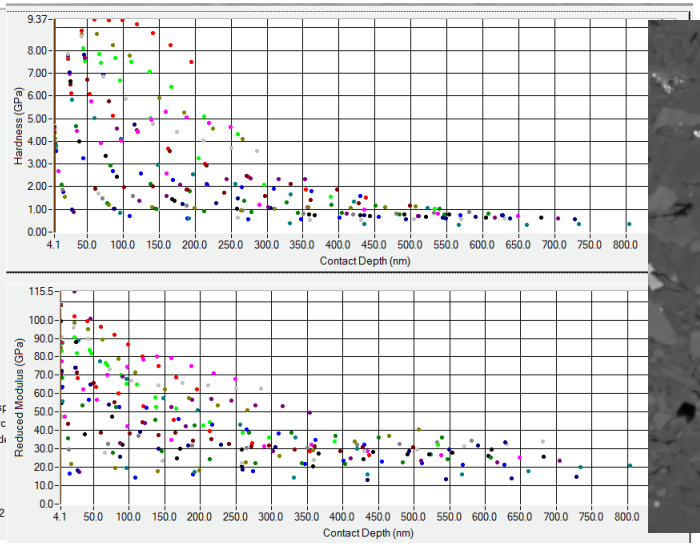


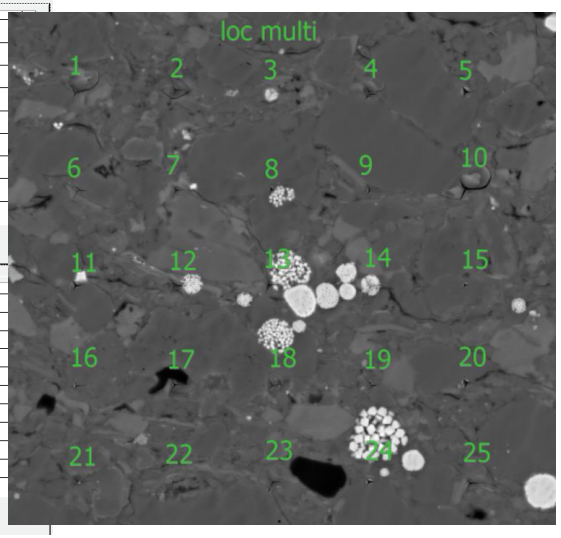


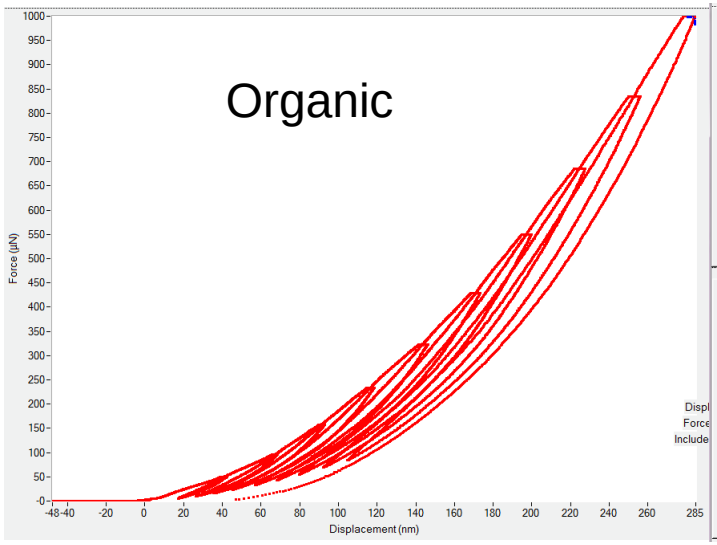
Multiscale Indentation Testing

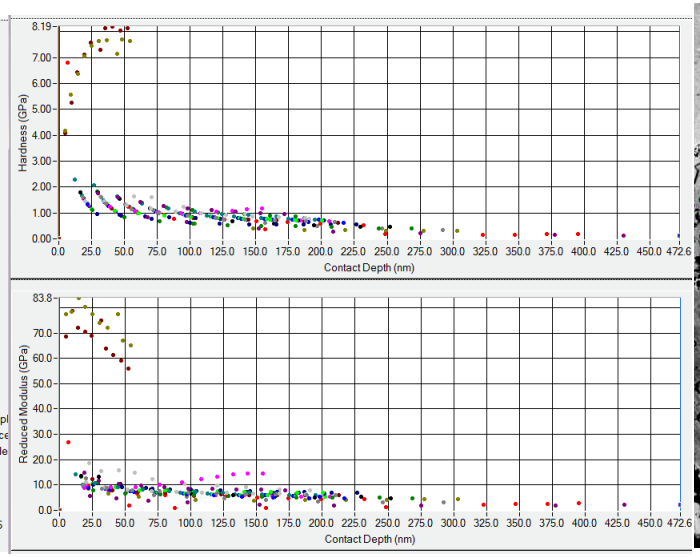
Loading-unloading cycle

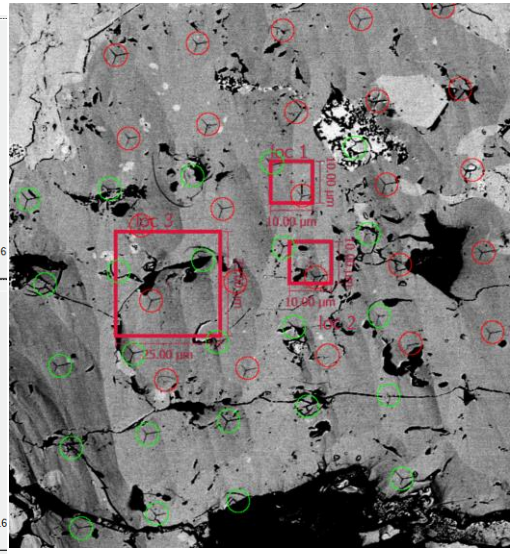




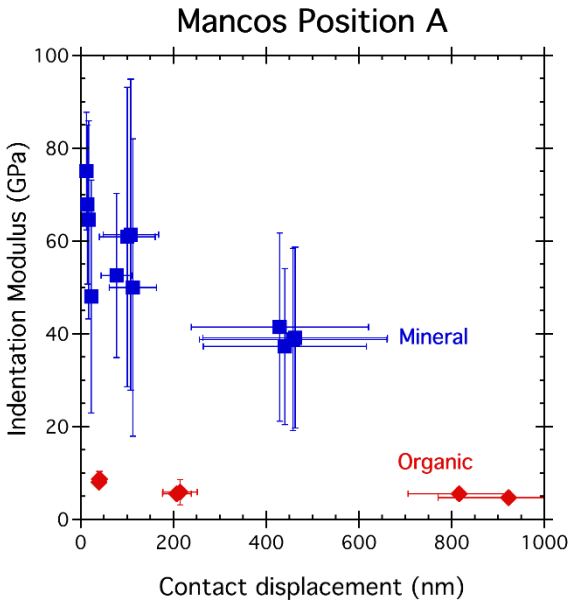


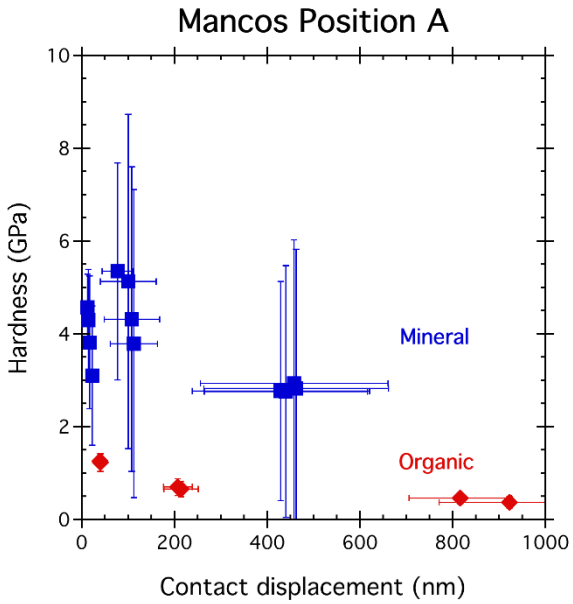


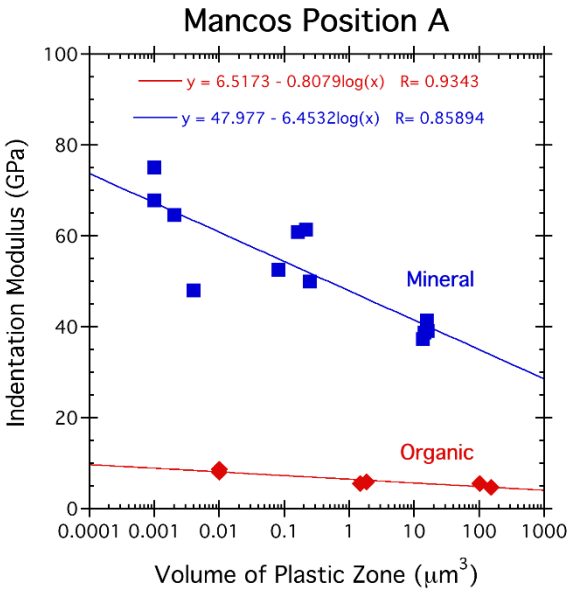


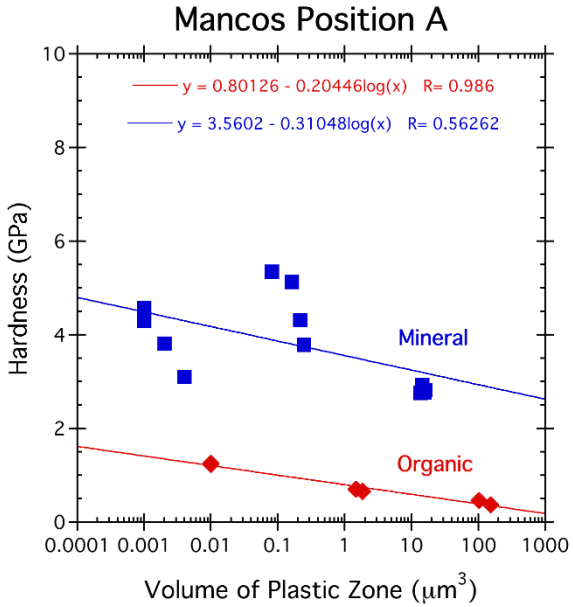


Multiscale Indentation Testing

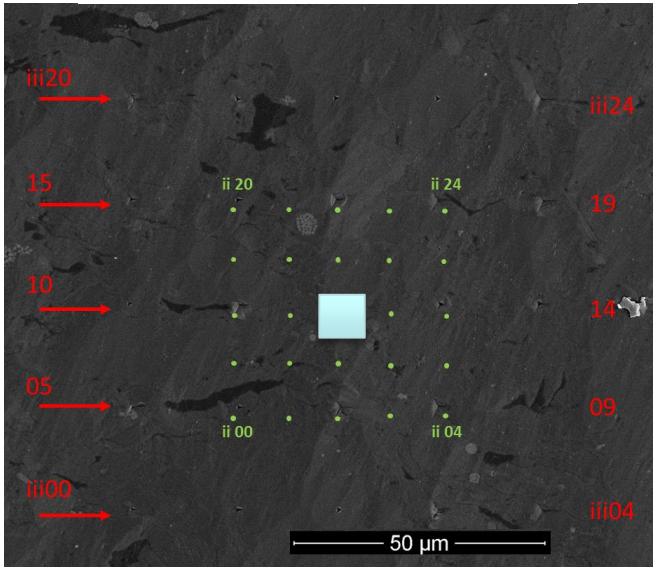








Distributed arrays



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

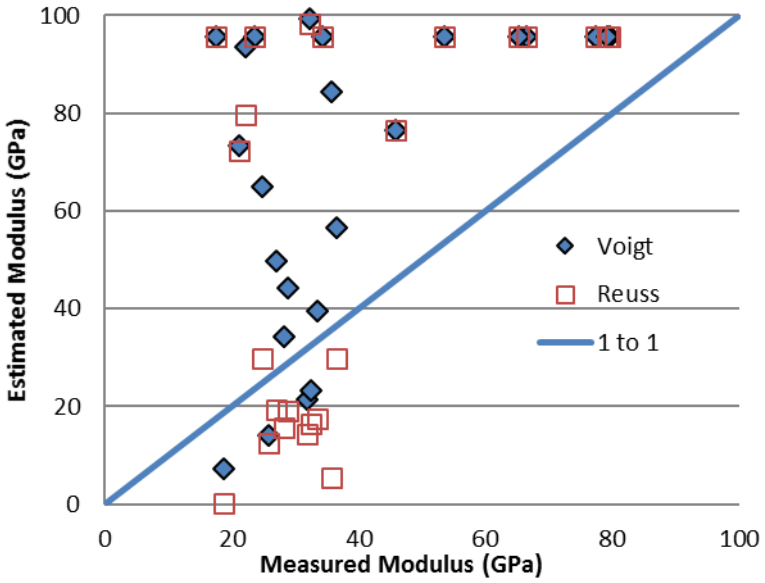
Estimate plastic zone radius

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

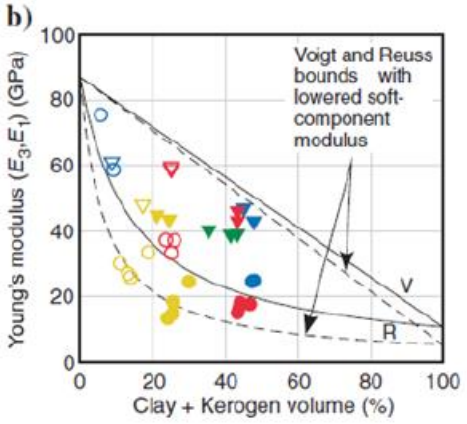
Chen & Bull, Surface & Coatings Technology (2006) 4289–4293.

Estimation of Elastic Modulus

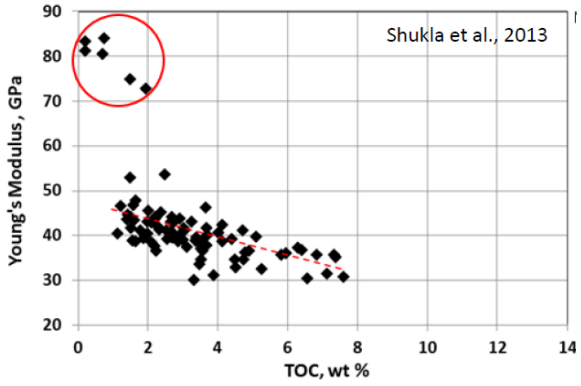
5 x 5 grid indentation (A7)



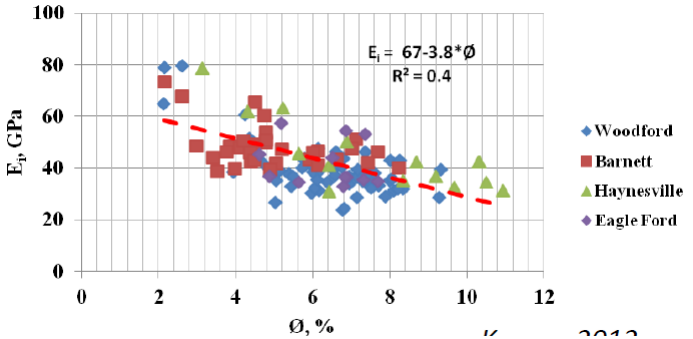
Sone & Zoback (2013)



Shulka et al. (2013)

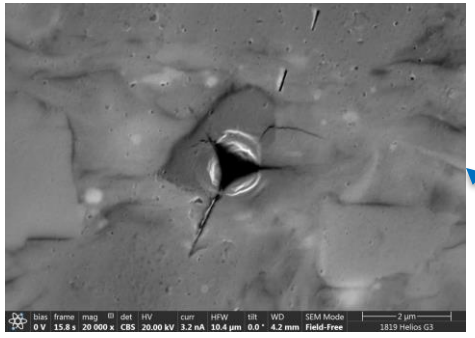


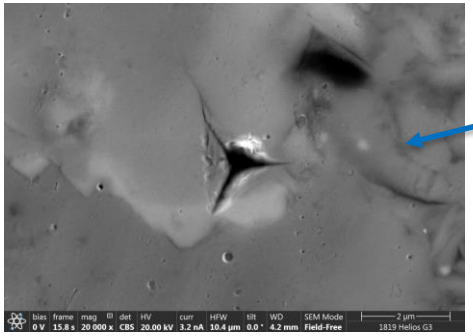
Kumar et al. (2012)

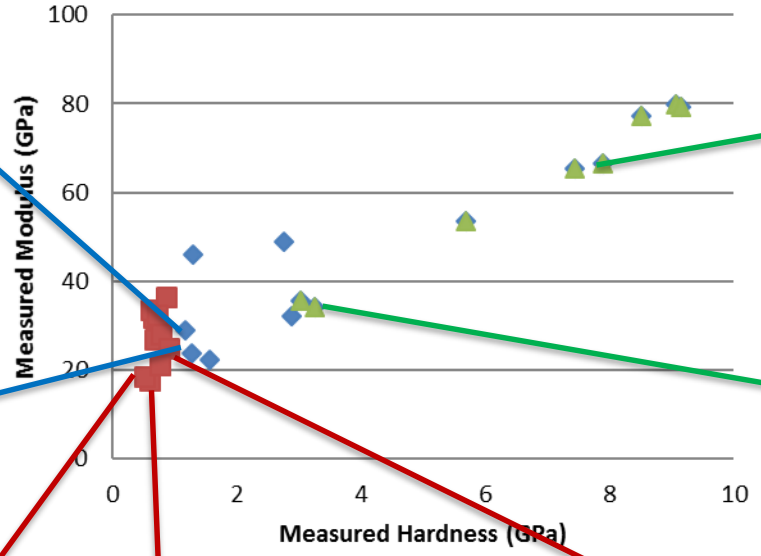


Elastic Modulus-Hardness Relation

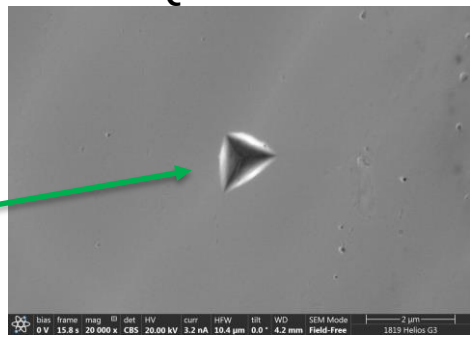
Dolomite

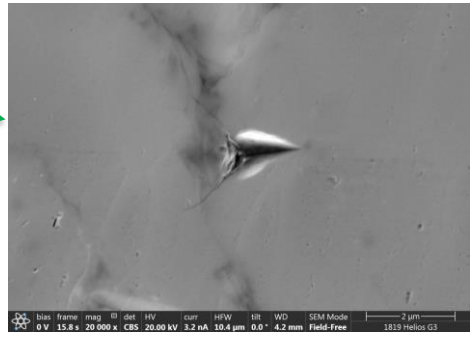




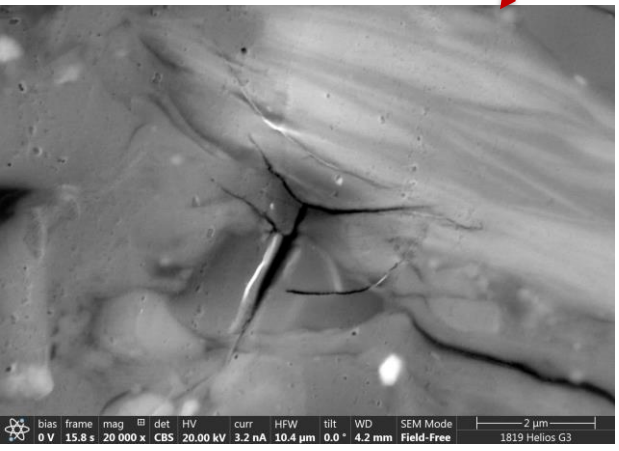


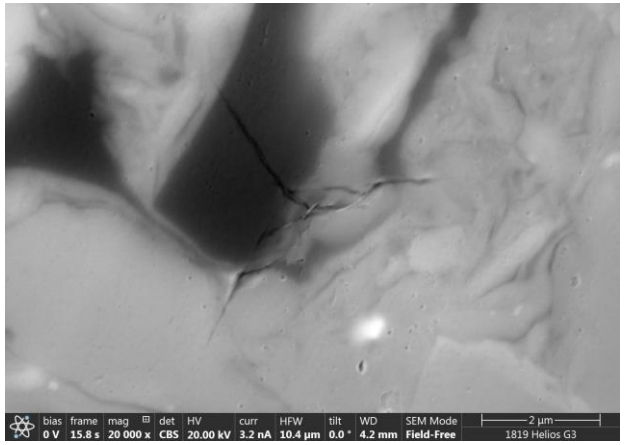
Quartz

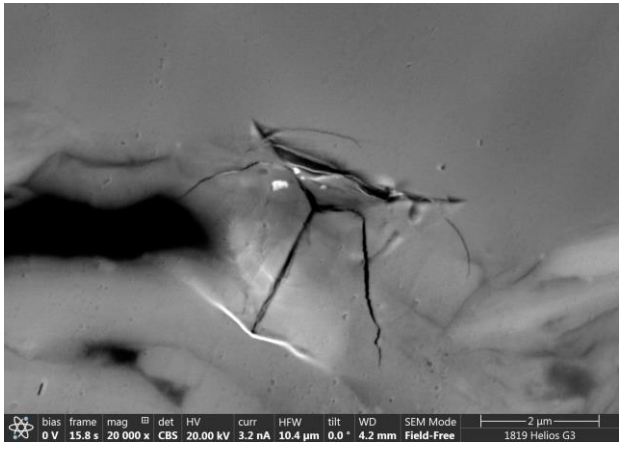




Mixtures

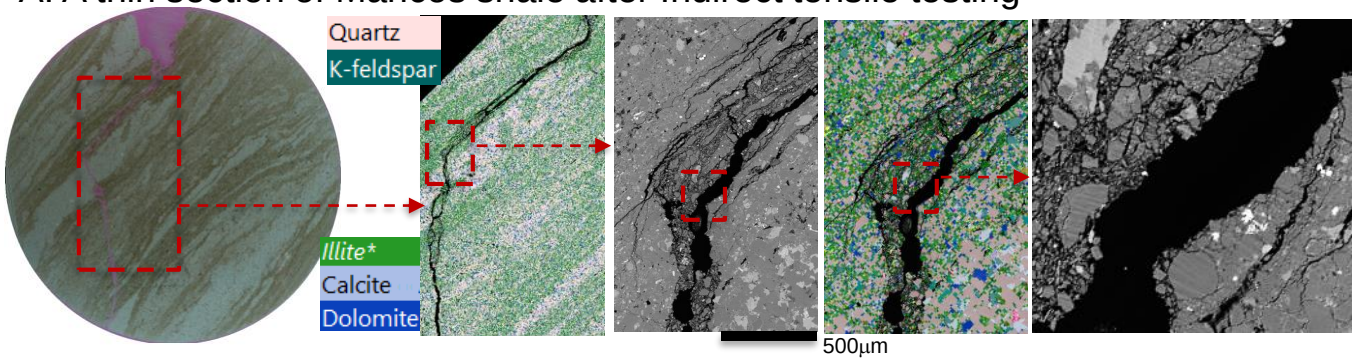




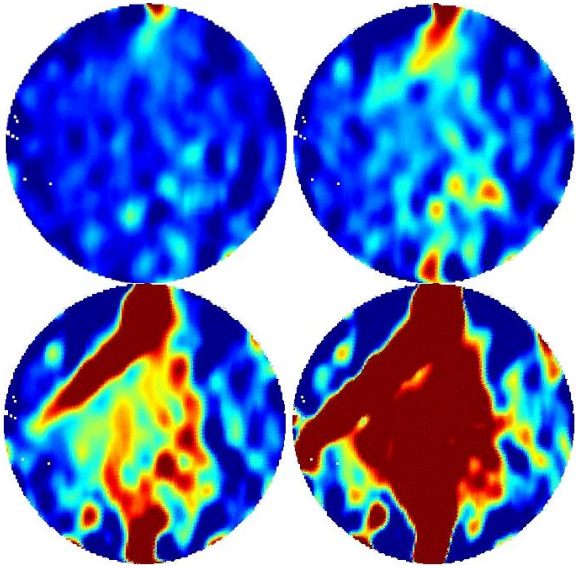


Phase field modeling for crack propagation

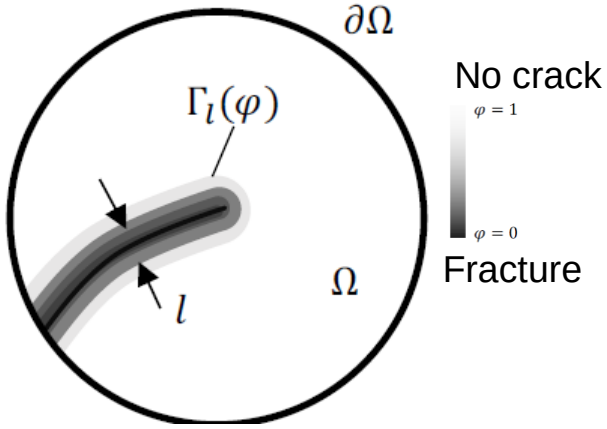
A. A thin section of Mancos shale after Indirect tensile testing



B. Lateral strain based on digital image correlation measurements



C. Phase field modeling results (crack initiation & propagation)

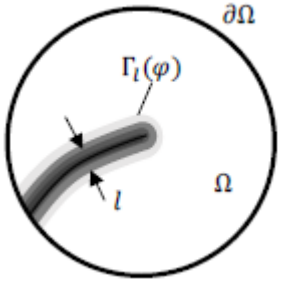


Na et al. (JGR 2017)

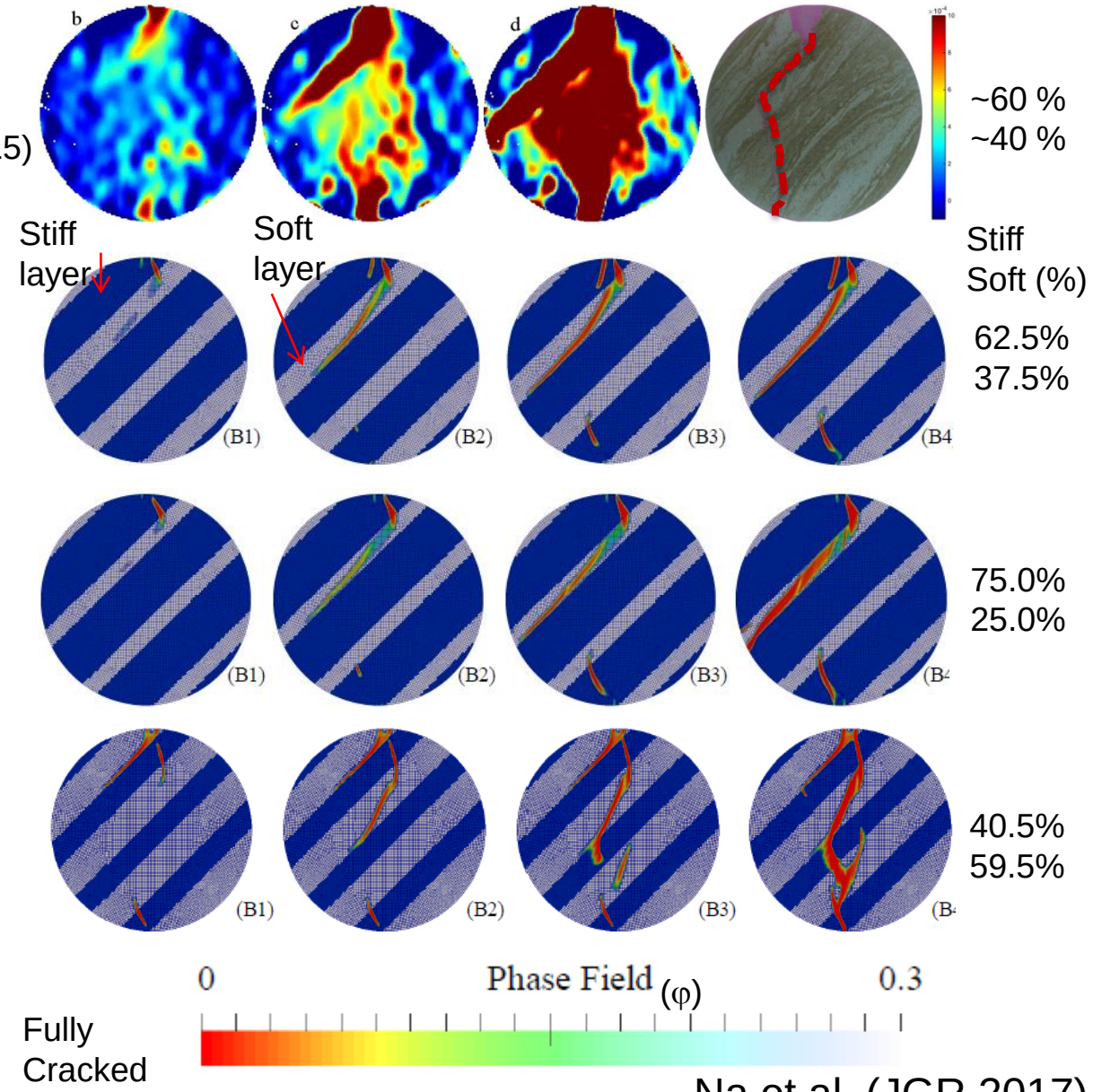
Numerical Simulations of Brittle Cracking

- 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)

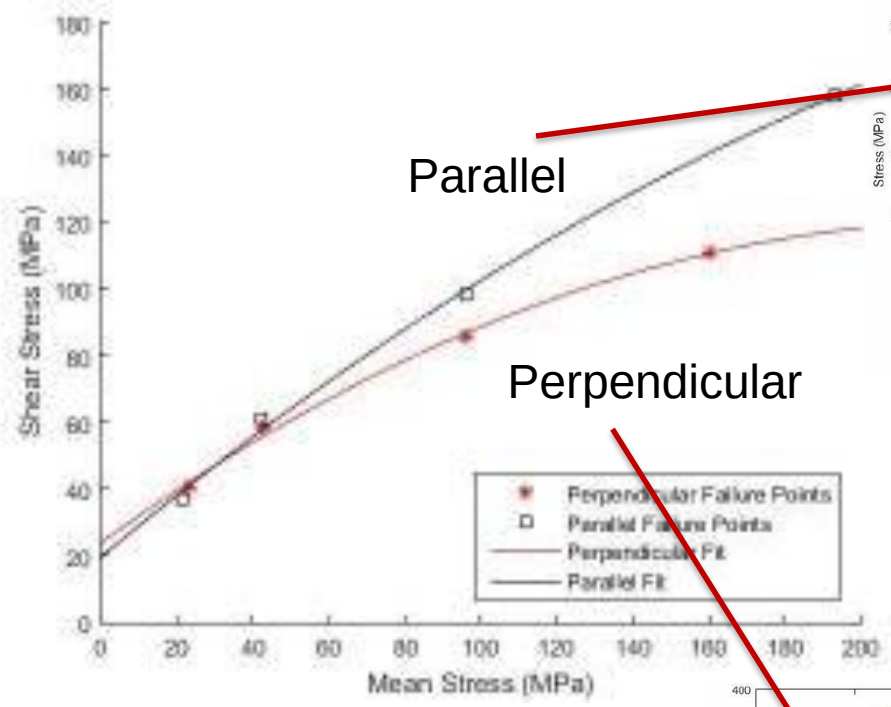


DEAL.II Open Source Finite Element Library



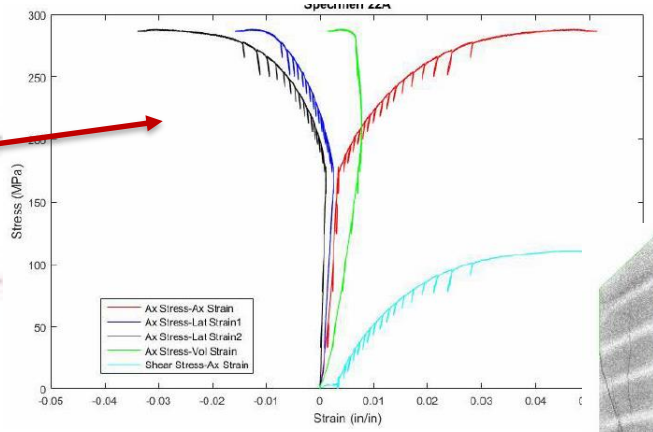
Na et al. (JGR 2017)

Axisymmetric Testing Results

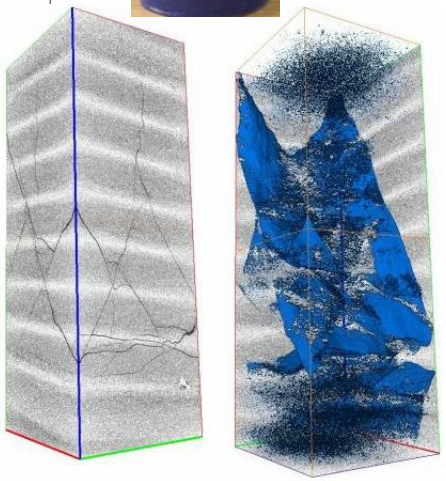


Parallel

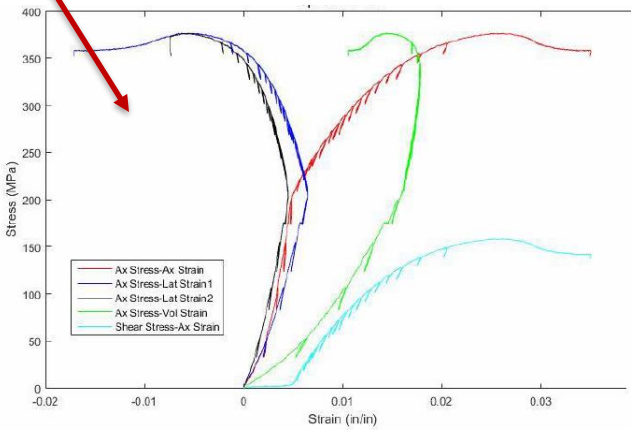
Perpendicular

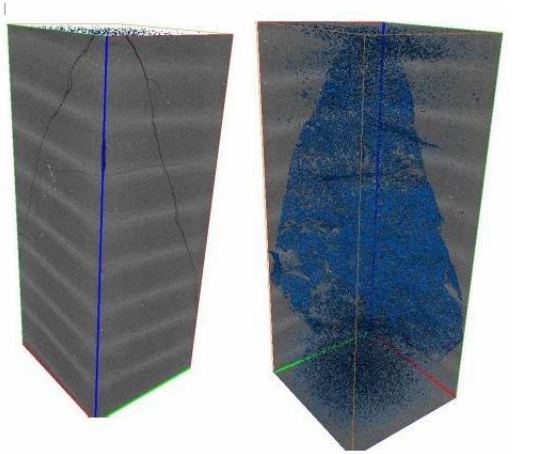


Loaded parallel to bedding



Loaded perpendicular to bedding

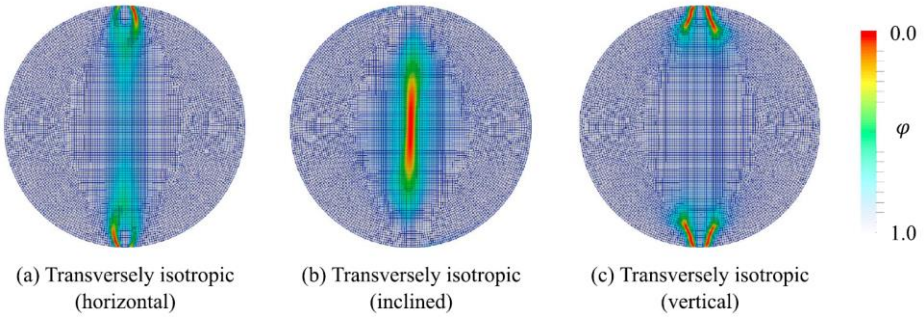


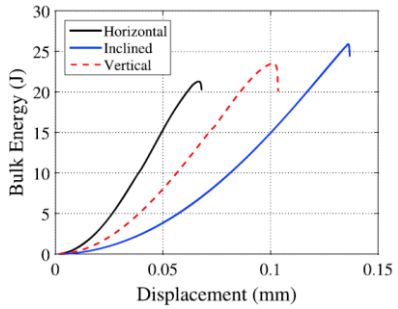


Ingraham at Sandia

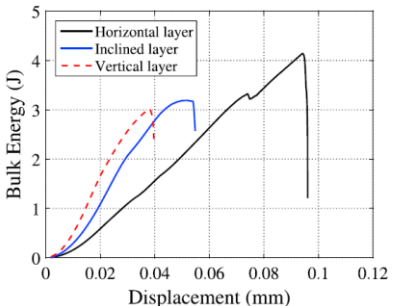
Effective Properties of Heterogeneous Materials

- 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)





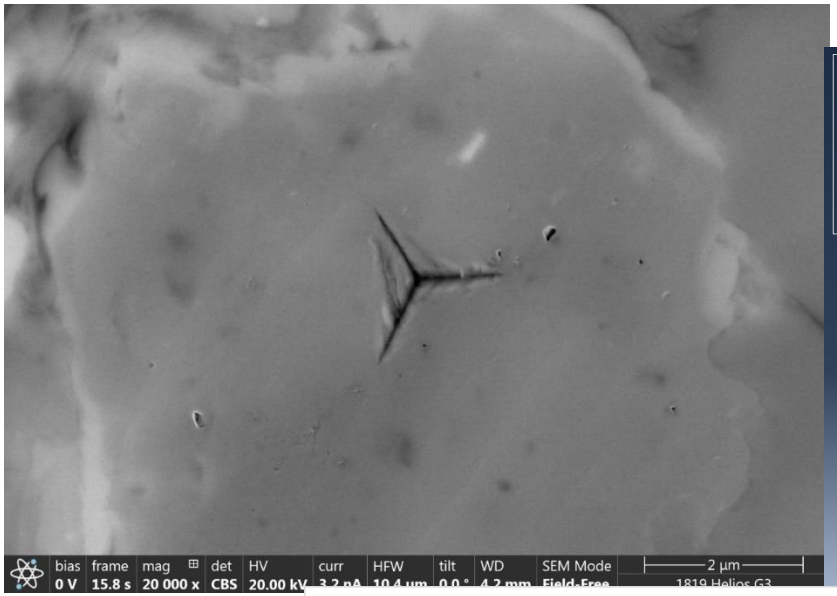
Transversely Isotropic

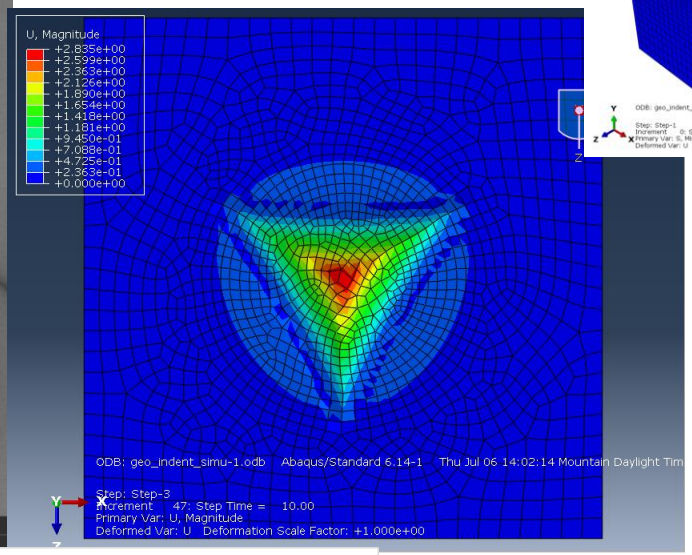


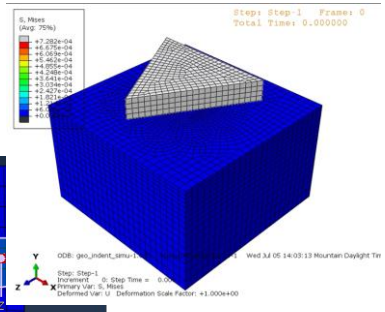
Layered Isotropic

Na et al. (JGR 2017)

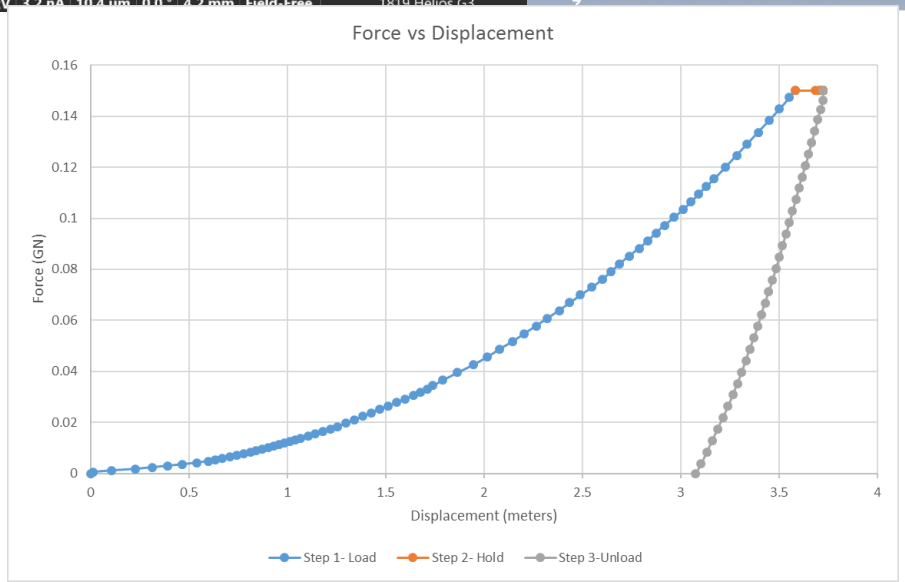
3D Mechanistic Modeling





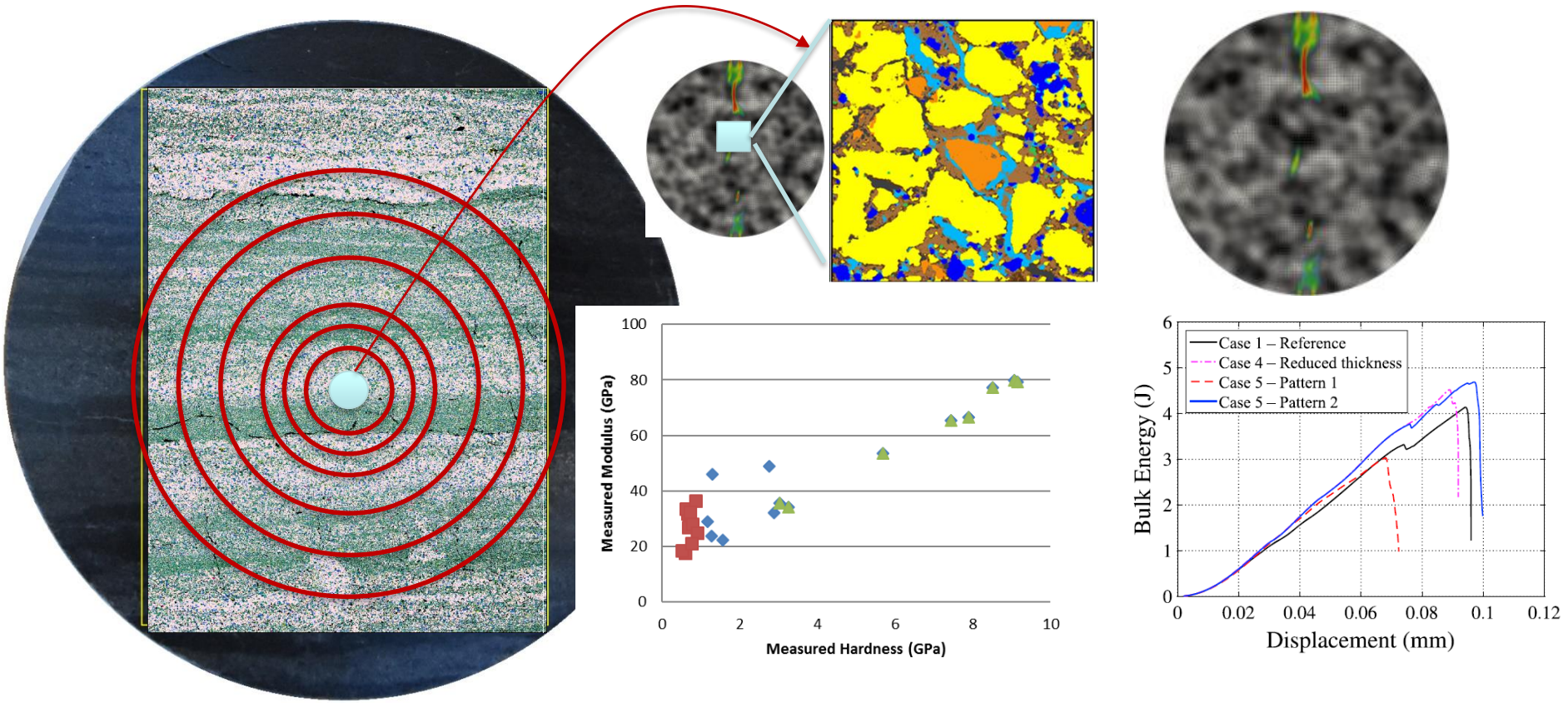


ABAQUS



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)

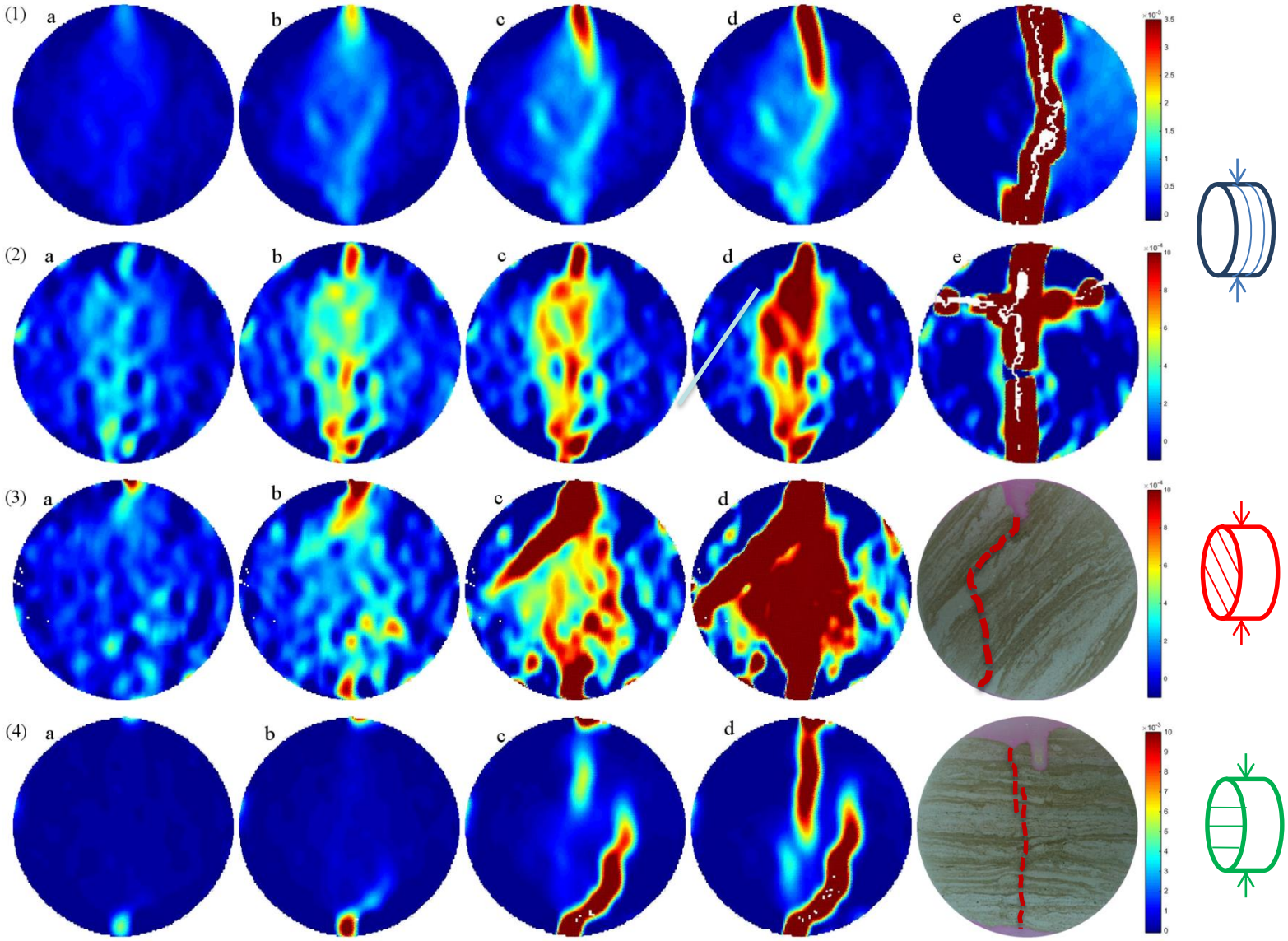


Summary

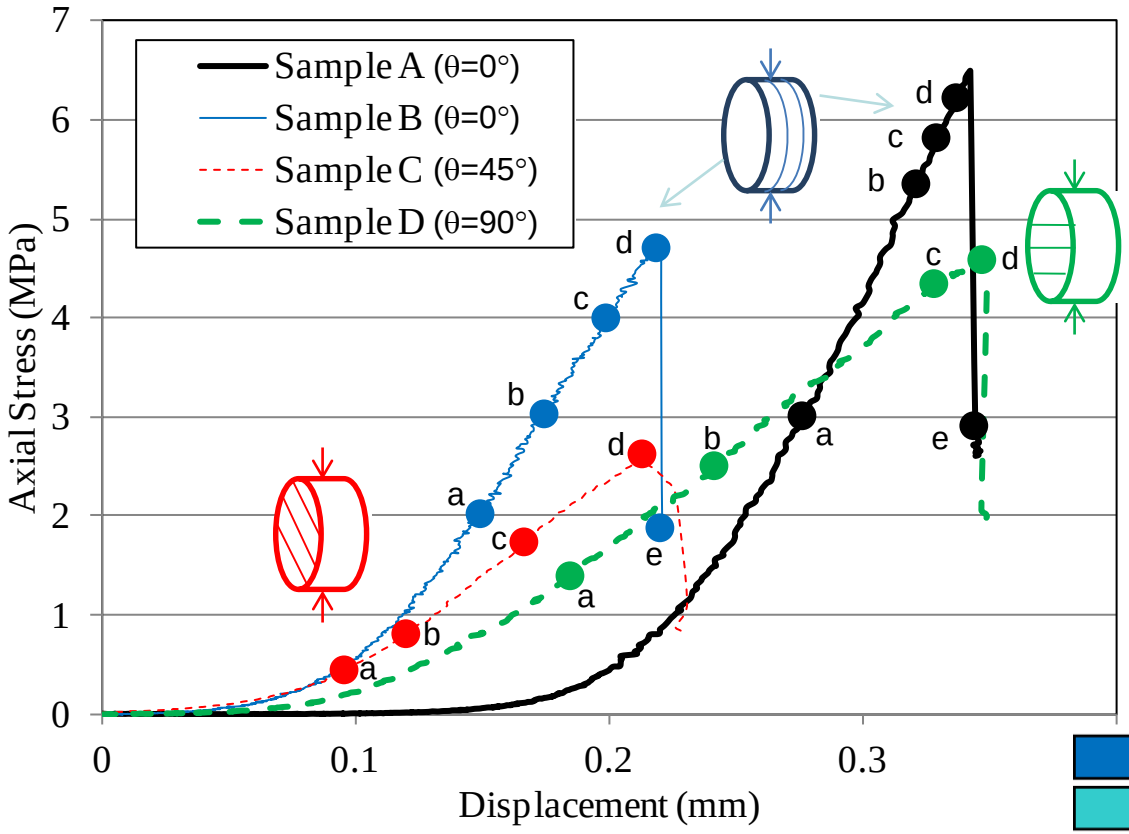
- Integrated multiscale imaging and mechanical testing with numerical simulation provides a robust approach to advancing our understanding of shale poro-mechanics
- Texture/mineralogical characterizations
  - Recent advances in mineralogical mapping with high resolution imaging over large area
  - Multiscale mineralogical and structural heterogeneity leads 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

Back Up

Tensile Strain Distribution (Digital Image Correlation)



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