IN21C-0055

Multiscale Pore Structure of Fine-grained Rocks: Evaluation of REV via High-throughput Scanning Electron Microscopy

Jason Heath[1], Thomas Dewers[1], Eric Shields[1], Hongkyu Yoon[1], Kitty Milliken[2]

[1]Sandia National Laboratories, Albuquerque, New Mexico; [2]Bureau of Economic Geology, University of Texas, Austin, Texas
email: jeheath@sandia.gov

2017 AGU Fall Meeting

Introduction

A foundational concept of continuum poromechanics is the representative elementary volume or *REV*: an amount of material large enough that pore- or grain-scale fluctuations in relevant properties (e.g., porosity or permeability) are dissipated to a definable mean, but smaller than length scales of heterogeneity. We determine equivalent 2D representative elementary areas (REAs) of pore areal fraction in mudrocks of three major types (i.e., calcareous, siliceous, and terrigenous) by applying multibeam scanning electron microscopy (mSEM) and novel multiscale image analysis methods. REAs based on coefficient of variance of pore areal fraction of the laminated calcareous and terrigenous samples are remarkably similar, with values on the order of ~400 micrometers and sub-laminae. The siliceous sample shows a much smaller REA at ~100 micrometers, reflecting differences in depositional environment. Pore eccentricity has remarkable consistency for all sample types, in which most pores bin in a broad distribution tending towards elongate shapes.

Research Questions and Methods

Research questions

- What sizes are REVs for mudrocks?
- Given the extreme heterogeneity of mudrocks (Lazar et al., 2015), is there a clear separation in scales between pore-scale variability and larger-scale heterogeneity for common mudrock types?
- Do mudrock REVs vary with composition, depositional environment, and burial diagenesis?

Approach

- Apply high-throughput mSEM imaging to examine nano-scale pores over areas up to square millimeters.
- Test mSEM on three major compositional types of mudrocks to evaluate usefulness of mSEM for multiscale mudrock studies.
- Extract pore characteristics from mSEM images and analyze for representative elementary area, and pore shape as a function of size using Matlab.

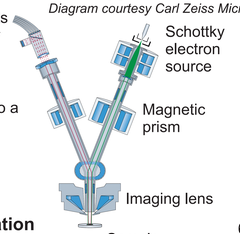
Multi-beam scanning electron microscopy

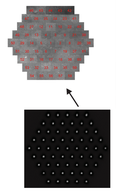
Projector column collects 61 separate ***secondary electron*** signals

8 image acquisition computers collect images and send them to a large image storage sever

Max throughput:
1.2 billion pixels/sec

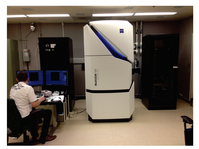
Diagram courtesy Carl Zeiss Microscopy

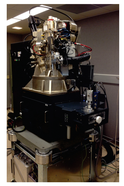




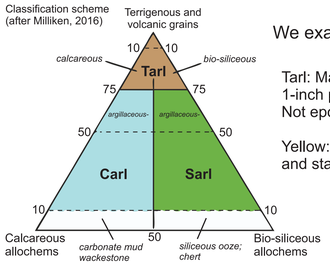
61 primary beams in hexagonal pattern

Zeiss MultiSEM





Shale samples and preparation



We examine Carl, Sarl, and Tarl mudrocks

Tarl: Mancos Shale
1-inch plug
Not epoxied

Yellow: region of EDS mapping and standard backscatter SEM

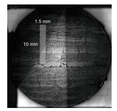
Broad ion-beam (BIB) polishing was preformed on all samples to obtain nm-flat samples

Carl and Sarl: non-epoxied Mesozoic siliceous shale sample was provided by Kitty Milliken, Bureau of Economic Geology, UT-Austin

mSEM Imaging and Pore Identification

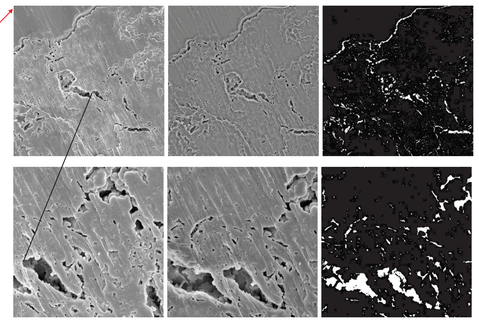
Tarl

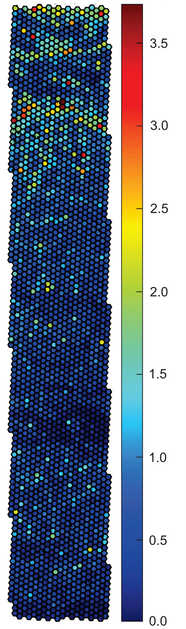
3.0 keV landing energy
Collection time: 104 min.
2,296 hexagons
140,056 images, 1.08 TB
4 nm resolution



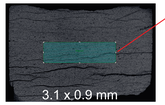
Raw mSEM mosaic

Deep Zoom Tri-viewer
Data to be posted at:
www.digitalrocksportal.org



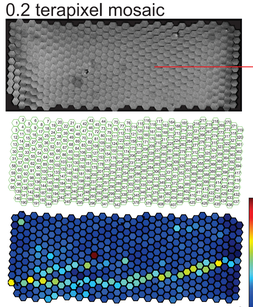


Sarl



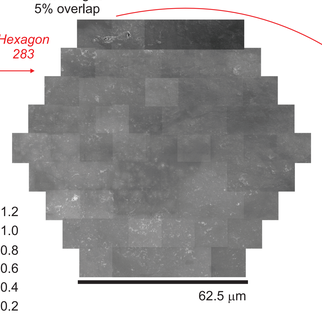
3 keV landing energy
Collection time: 16 min.
437 hexagons, 227 GB
4 nm resolution
26,657 images:
Single image:
12.5 μm x10.9 μm

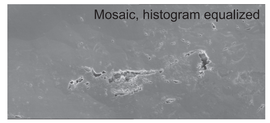
0.2 terapixel mosaic

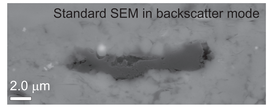


Hexagon 283

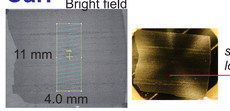
Raw images
5% overlap



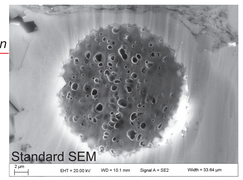




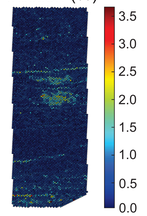
Carl



same location



Raw mSEM mosaic

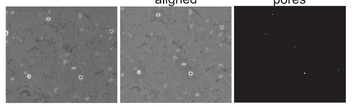


pore areal fraction (%)

3 keV landing energy
Collection time: 292 min.
6479 hexagons, 3.1 TB
4 nm resolution
395,219 images:
Single image: 12.5 μm x10.9 μm

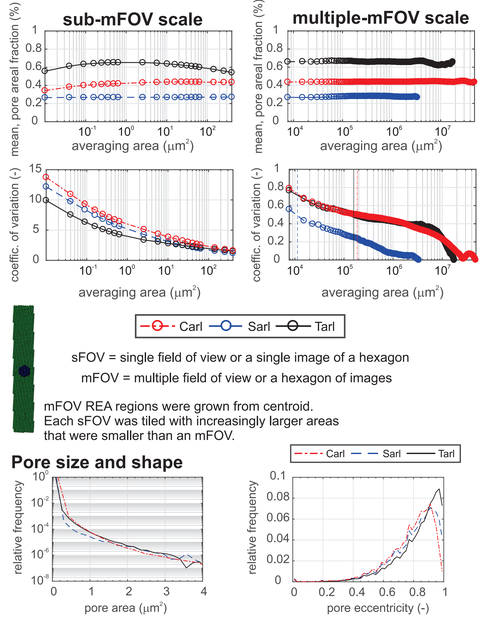
3.3 terapixel mosaic

Carl Triviewer example



Multiscale Porosity

Representative elementary area



sFOV = single field of view or a single image of a hexagon

mFOV = multiple field of view or a hexagon of images

mFOV REA regions were grown from centroid.
Each sFOV was tiled with increasingly larger areas that were smaller than an mFOV.

Pore size and shape

Conclusions & Future Work

- The REA for the carl and tarl mudrocks is on the order of ~ 400 μm, which is just below sizes of depositional lamina for both samples. The sarl has an REA of ~ 100 μm.
- The REA sizes indicate that standard FIB-SEM pore scale reconstructions of shale that are much smaller by 100 μm on a side are "in the noise."
- Pore size and shape distributions are remarkably similar, with elongated pores being more common.
- Future work will include analyzing representative elementary areas for organics, staining for improving secondary electron emission, and synthesizing with the mechanical data.

Acknowledgments
This work is funded by the Basic Energy Science program of the U.S. Department of Energy Office of Science under Award Number DE-SC0006883. We thank John Laughlin of Carl Zeiss Microscopy and Joseph Michael, Craig Nakakura, and Bonnie McKenzie of Sandia National Laboratories for facilitating collection of multibeam and standard SEM data. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.
SAND2017-XXXX C.