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geoConvention 2013

From Core to Pore: Multi-scale, Multi-dimensional characterization of fine-grained reservoir rocks

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Summary

Mineralogy and microstructure are key variables defining the physical properties of a rock. Rocks that have a heterogeneous mineralogy/microstructure will exhibit equally heterogeneous physical properties. Shale typically displays significant mineralogical and microstructural variation. Quantifying shale mineralogy and microstructure permits more accurate determination of a variety of physical properties important to modeling production potential: e.g. organic versus inorganic porosity, permeability, brittleness etc…

The microstructural elements that define properties of interest for a typical shale exist over 9 orders of magnitude in length-scale (nanometer to meter). Therefore, a main challenge exists in upscaling mineralogical and microstructural observations in a way that provides characterization of nanometer-scale observations that are representative of the rock at centimeter to meter-scales.

Three recent technological advances have paved the way for routine, accurate microstructural characterization from the nanometer to meter scales: Ultrahigh resolution scanning electron imaging, automated mineralogy and 3D imaging (X-ray computed tomography (CT) and FIB/SEM analysis). When combined, data from these sources can be used to quantify shale at a resolution that is only now possible.

We present examples of integrating multi-scale 2D and 3D electron imaging with proven automated mineralogy algorithms to more accurately evaluate the mineralogy/microstructure of shale and other fine-grained reservoir rocks. This multi-scale multi-dimensional workflow also provides a pathway for upscaling observations in a repeatable and quantifiable way. Integration of mineralogical and microstructural data provides a unique opportunity to evaluate shale and other fine-grained reservoir systems with unparalleled fidelity.

Introduction

As the physical and microstructural properties of fine-grained reservoir rocks continue to be refined, it is clear that these rocks comprise a surprising heterogeneity; especially at small length scales (Rickman et al., 2008; Curtis et al., 2010). Accurate modeling and prediction of production potential/stimulation of hydrocarbons from shale and other fine-grained reservoir rocks depends on a fundamental understanding of scaled variations in microstructures – from the length scale of individual pores to that of natural/induced fractures.

Gas shales typically contain dispersed organic matter. Gas transport in organic phases is limited by the dimension of pore throats (often <100 nm in diameter). The only method available for analysis of

feature of this scale is utilization of a FIB/SEM to produce high-resolution 3D reconstructions. The width of these analyses are generally <25 µm. Therefore, choosing locations representative of porosity structure are key in providing an accurate characterization of reservoir potential.

A workflow is presented that links centimeter to nanometer-scale observations with mineralogy and 3D reconstructions of representative microvolumes. This workflow provides a unique way to provide representative semi-automated analysis of mutliscale-multidimensional data. These tools allow for unprecedented access to exploring and quantifying microstructural/mineralogy variability in fine-grained reservoir rocks; thereby creating more realistic and accurate models of the physical and chemical attributes important to petrophysicists and reservoir engineers.

Methods

We used an Field emission (FEG) SEM for basic imaging and collection of automated mineralogy data and FEG FIB/SEM for ultrahigh resolution imaging and 3D analysis of shale microvolumes. All of our images were collected using backscattered electrons (BSE).

Automated Mineralogy

Automated mineralogical analyses were conducted using an algorithm-based mineral identification software package based on SEM/EDX technology. Mineral identification is done utilizing X-rays generated by the interaction of the electron beam with the sample in an SEM. X-rays are collected using an Energy Dispersive Spectroscopy (EDS) detector. The algorithm converts the X-ray signal to bin characteristic X-rays as elements and compares the data to an internal library of mineral spectra for identification (Lemmens et al., 2010).

Ultrahigh resolution scanning electron imaging

Ultrahigh resolution imaging is achieved by working at low beam currents and low beam accelerating voltages. Images produced under these conditions are the truest representation of a surface as influences of beam interaction volume are reduced. In addition, nanometer-scale resolution is achieved using the above conditions in part, because there is much less variation in beam energies.

We used a software package, which provides an automated routine that collects and stiches a mosaic of backscattered images. For the examples provided here, mosaics with a pixel resolution of 40 nm were collected over a 6x4 mm area. The stitched mosaic of images allows for the systematic exploration of fine-grained microstructure from the mm to the nm –scales.

3D microvolume reconstructions

A FIB/SEM was used to create 3D reconstructions of microvolumes. A focused ion beam (FIB) is used to mill successive slices of a sample. The FIB uses a Ga source which is focused on a sample. The Ga removes sample material through momentum transfer. The rate of sample removal (milling) is dependent on the mass and structure of the material of interest. 3D reconstructions are made by alternating between the SEM for imaging and the FIB for milling. The ion beam is well controlled and slices down to 5 nm thick can be acquired routinely. The quality and stability of FIB optics determine the ability to produce imaged surfaces of consistent quality and contrast.

Consecutive slices are aligned and stacked into a 3D model. SEM images are collected using BSE and therefore greyscale contrast in an image correlates with mineralogy (assuming the average atomic number for all minerals is not identical). Segmentation of 3D mineralogy is achieved by thresholding greyscale ranges for each mineral, organic material or porosity present. Visualization software can be used to investigate the 3D microstructure as well as output parameters for 3D modeling of physical properties such as permeability.

Examples

We present data from a core from the Eagle Ford formation as an example of the SEM-based workflow.

Automated mineralogical data for a horizon of the Eagle Ford formation is presented in spatial (Map) format (Figure 1A). Colors in Figure 1 are correlated to the each mineral identified (see legend). For the

mineral ID map in Figure1, organic and surface porosity (apparent) can also be easily measured for core samples. Mineral modal abundance is also presented. The data in Figure 1 were collected using 1 µm step sizes.

Investigation of the relationship between apparent porosity and organic matter can be seen in Figure 1B. Comparing Figure 1A and 1B reveals 2D porosity is concentrated in kaolinite foraminifera fills. However, porosity is also identified dispersed throughout the matrix. The dispersed porosity is associated with the phase 'lime mud', which is the second most abundant mineral phase. This phase represents a mixture of fine-grain clays and carbonates. Creating mixed phases is often a necessary solution for extremely fine-grained samples (<10 µm). All EDS based techniques will be limited by the volume of interaction between the beam and the sample. This size of the volume is determined by phase density and crystalline structure but can be as large as 5 µm. Combining mineralogical analysis with high-resolution imaging can shed light on the nature of rock microstructure for fine-grained samples (Figure 2).

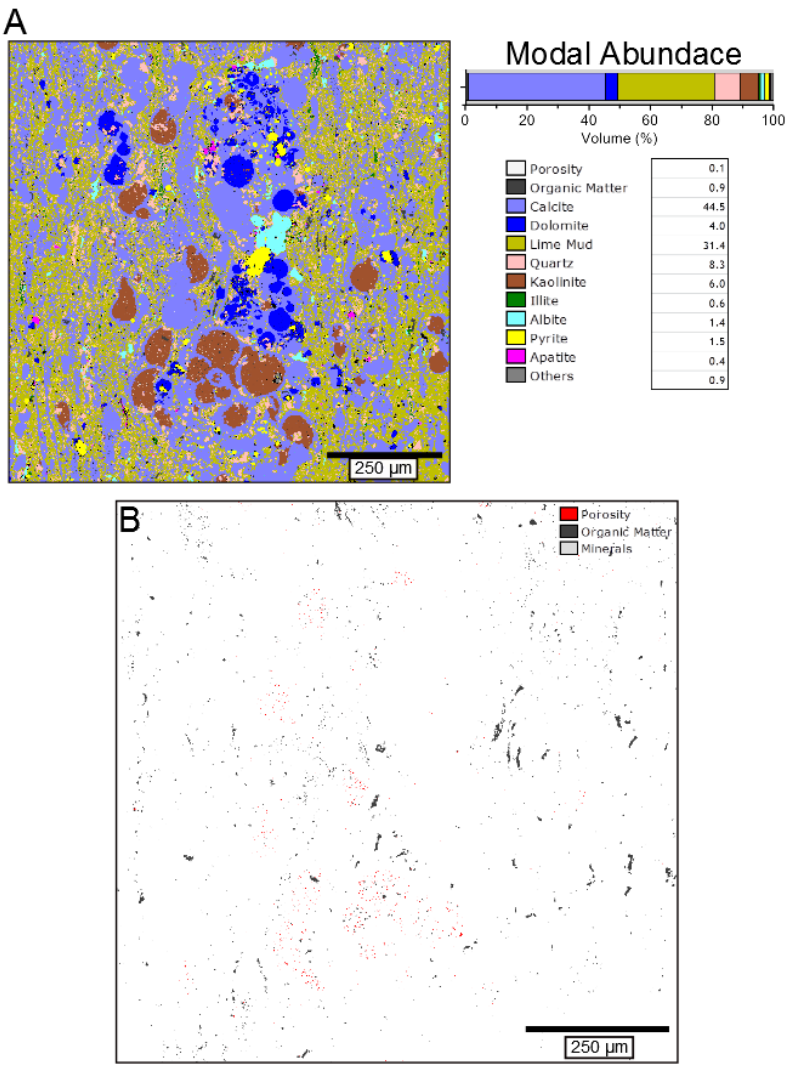

Figure 1

Figure 2

Figure 2A is a 6x4 mm BSE image comprised of a matrix of 1200 individual high-resolution images taken form the same core as the automated mineralogy data example above. The pixel resolution of this image is 40nm. One can examine this image interactively and zoom into areas of interest for inspection. For example, Figure 2B displays a zoomed in view of the larger image. Kaolinite within a foraminifera as well as an organic phase are seen in Figure 2B. Porosity picked up by the mineralogical analysis is clearly visible within the foraminifera. The organic phase on the right lacks any evidence of porosity. However, organics proximal to the foraminifera are extremely porous. Figure 2C illustrates the dominate matrix microstructure (Lime-mud in Figure 1A). The mineralogy of Figure 2C is clay, quartz and calcite. Organic matter is dispersed throughout the field of view, with an area of higher concentration of organic matter on the left hand side of the image that has high relative porosity. Examining the range of microstructures in this way highlights areas to target for more detailed 3D investigation.

Figure 3A illustrates example slices from a 3D microvolume reconstruction from the Eagle Ford core. The field of view of each image is 30 µm. This 3D volume characterizes the dispersed fine-grained organics associated with quartz, clay, calcite matrix. The volume illustrated in Figure 3A-B is a 30x20x5 µm volume. Figure 3B was created by segmenting out the mineral/organic phases of interest using the greyscale contrast present in the series of 2D slices shown in Figure 3A. Red colors illustrate the organic phases that appear dispersed in 2D are, in fact, highly interconnected.

Further work segmentation can then be used to quantify the interconnectivity of organic porosity. These data can be used as input values to model permeability at the nanoscale. Integrating observations and models from representative locations at the nanoscale with MicroCT and other laboratory data will provide a more complete and accurate estimation of organic and mineral microstructural variation and its impact on production potential.

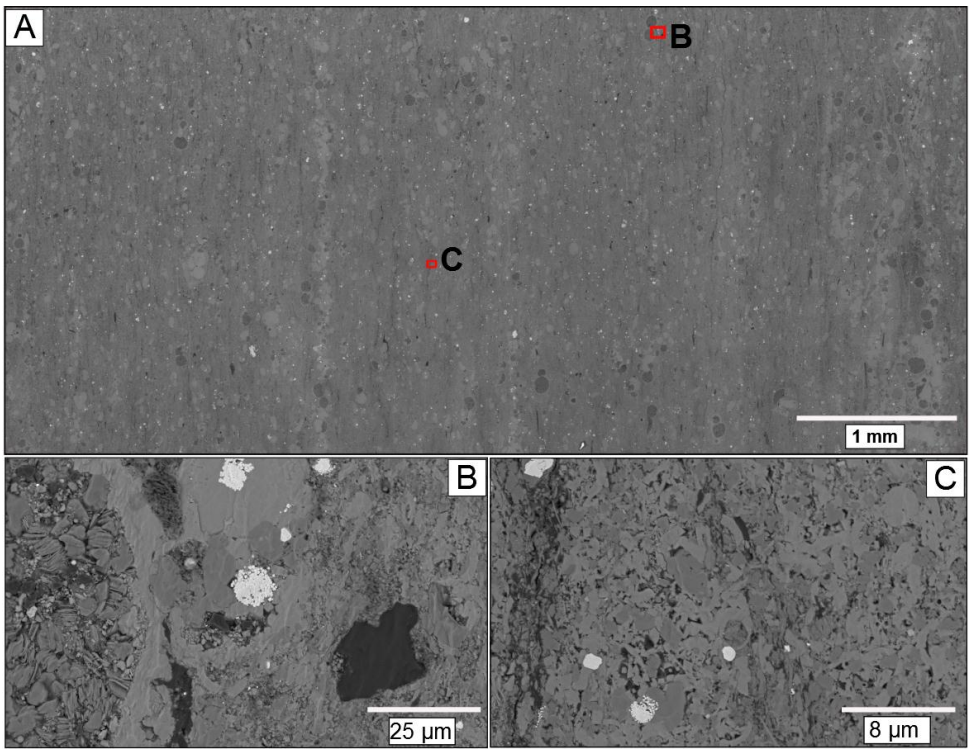

Figure 2

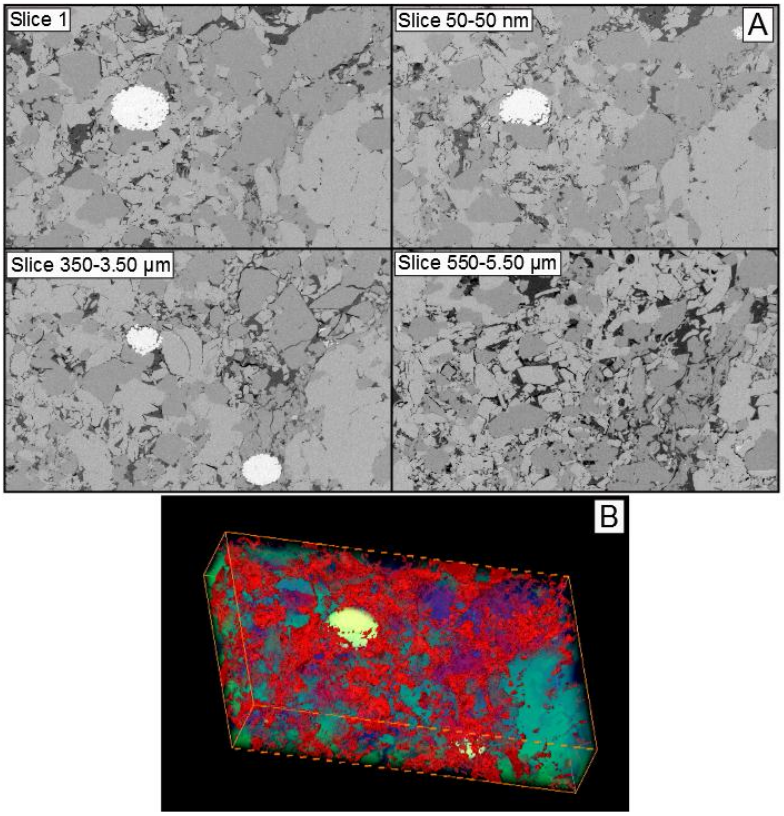

Figure 3

Conclusions

Integrating observations from automated mineralogy, high-resolution imaging and 3D reconstruction of microvolumes represents a new and promising method in evaluating the microstructural characteristics of fine-grained reservoir rocks. Preliminary work presented here, as well as examples from the literature illustrate that fine-grained rocks such as shales are extremely heterogeneous. Existing models for flow and fracture of these materials cannot be used. Applying mutiscale-multidimensional worflows, such as the one presented here, provides the opportunity to evaluate fine-grained materials with a fidelity that is only now possible. Data generated from studies such as these are necessary to develop better models of production and stimulation potential of fine-grained reservoir systems.

References

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