Microstructural Observations in Gas Shales

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Gas shales hold the promise to be a sustainable energy source for the foreseeable future. Success of the Barnett, Fayetteville, Haynesville, Marcellus and Woodford shale plays is driven by advances in technology. These technologies include massive hydraulic slickwater fractures, horizontal drilling and multiple-stage fracturing. The design and placement of these laterals and stages is largely empirical, and success is sometimes hit and miss. Stimulation response varies considerably; well life varies from play to play. To better exploit these shale gas plays, we need to know more about what controls gas in place (GIP) and delivery. The insight to these controls is found in the compositional and textural framework of the shale at multiple scales. Definition of the building blocks and controls on storage and deliverability starts at the nanoscale in shales. We have used macroscopic averaging measures such as nuclear magnetic resonance (NMR) relaxation and high pressure mercury injection along with nano-scale scanning electron microscopy (SEM) imaging to address these issues. Our findings suggest a dimensional consistency among the measurement techniques, *i.e.* the size of pore bodies inferred through NMR relaxations and the pore throat dimensions recovered from mercury injection span the range observed with the SEM. This dimensional consistency indicates that the objects controlling both GIP and delivery are small and these objects have been imaged in SEM studies.

Technologies and techniques required to study the microstructure of shales are different than those used on conventional reservoirs. Owing to the compositional variability, particularly the organic components, simple grinding and polishing are inadequate; they selectively remove the organics from the surface. The surface preparation method of choice is ion milling. This is a technique in which an Ar^+ ion beam irradiates a specimen surface and removes atoms from the surface by momentum transfer. When the composition of the specimen is uniform, milling times can be correlated with surface removal rates. However, shales present a challenge in that they vary in composition and thus milling times and surface removal rates are only correlated through experience. The surface of the shale is milled at an angle to create a slope and viewed perpendicular to the slope. A common milling artifact of standalone Ar^+ milling systems are “curtains” which are ridges produced in the milled surface. Refined procedures can reduce but not eliminate these ridges. An example of an ion-milled surface is presented in Figure 1.

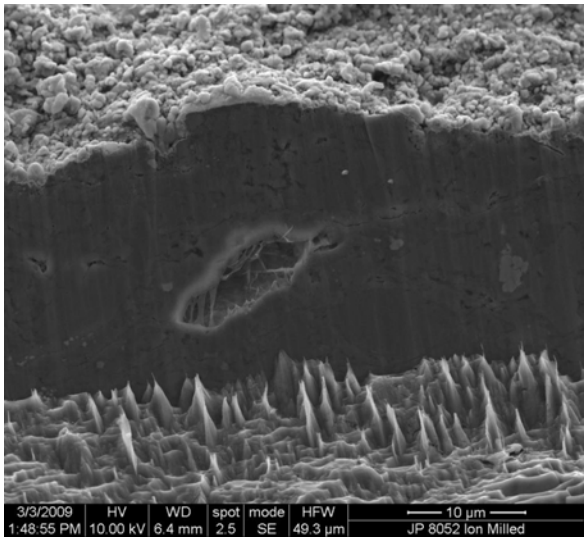

Figure 1: Shows an ion-milled slope in Barnett shale after oxygen plasma ashing to remove organics. The foreground and upper surfaces suffer from artifacts generated during the ion-milling process and vertical subparallel lines on the sloped surface are curtain artifacts of the milling process. Nevertheless, microstructural features generic to shale are visible. (Figure from Moncreiff, 2009)

A further improvement on this specimen preparation technique for SEM imaging is the use of a focused ion beam (FIB) integrated within a SEM. The FIB uses a Ga^+ ion source which can be accelerated at high voltage with variable ion beam current to image or remove material. In our studies, the FIB is used to remove material from the shale creating a cross-section and imaging of this surface is performed *in situ* using the SEM. To convince ourselves that we are not damaging organics, we conducted a series of experiments in which the ion beam current was varied and the milled surfaces imaged. The low and high current milled surfaces are shown in Figure 2a and b.

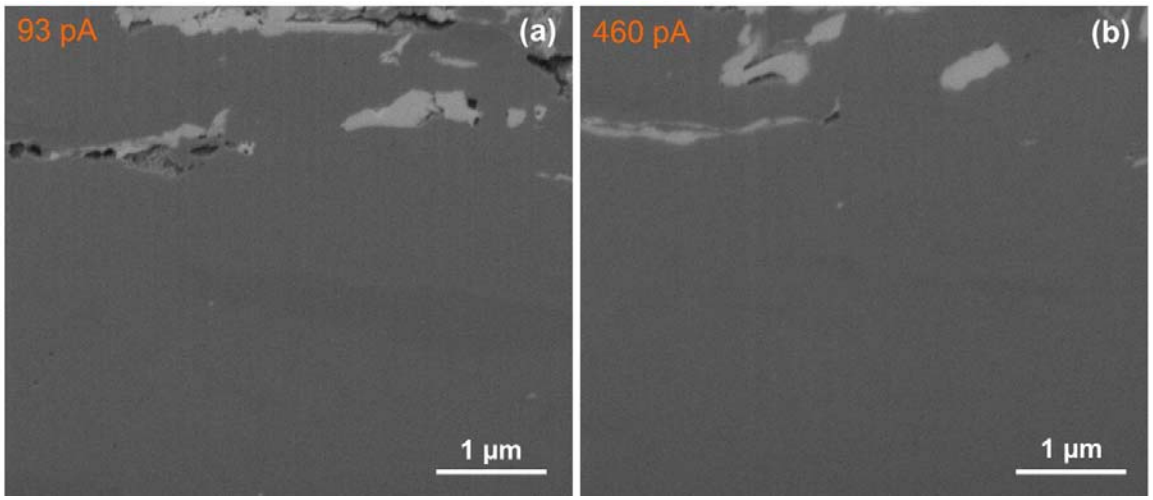

Figure 2: SEM images of Kimmeridge (40% TOC) shale prepared in cross-section by FIB at 30 kV accelerating voltage and 93 pA ion beam current (Fig. 2a). The darker material seen making up a majority of the image is kerogen. Figure 2b shows an area of the same shale sample that was prepared with a 460 pA ion beam current. No change in the structure of the kerogen can be seen nor is there visible porosity

We have used this technology to prepare surfaces to image the microstructure of a number of shales as shown in Figure 3. These images are "random" in the sense that we did not drive selection by any independent control such as mineralogy, porosity, TOC, etc. Each image was taken perpendicular to the bedding plane. The images were taken using backscattered electrons (BSE) to differentiate between the constituents of the shale. In the images, grains of inorganic material (lighter materials), typically clays, silica, carbonate, and pyrite, can be seen together with grains of organic kerogen (dark grey material). Pores can also be seen in the images with the size, shape, and location of the pores differing among the shale images. Pores can be seen within the kerogen itself.

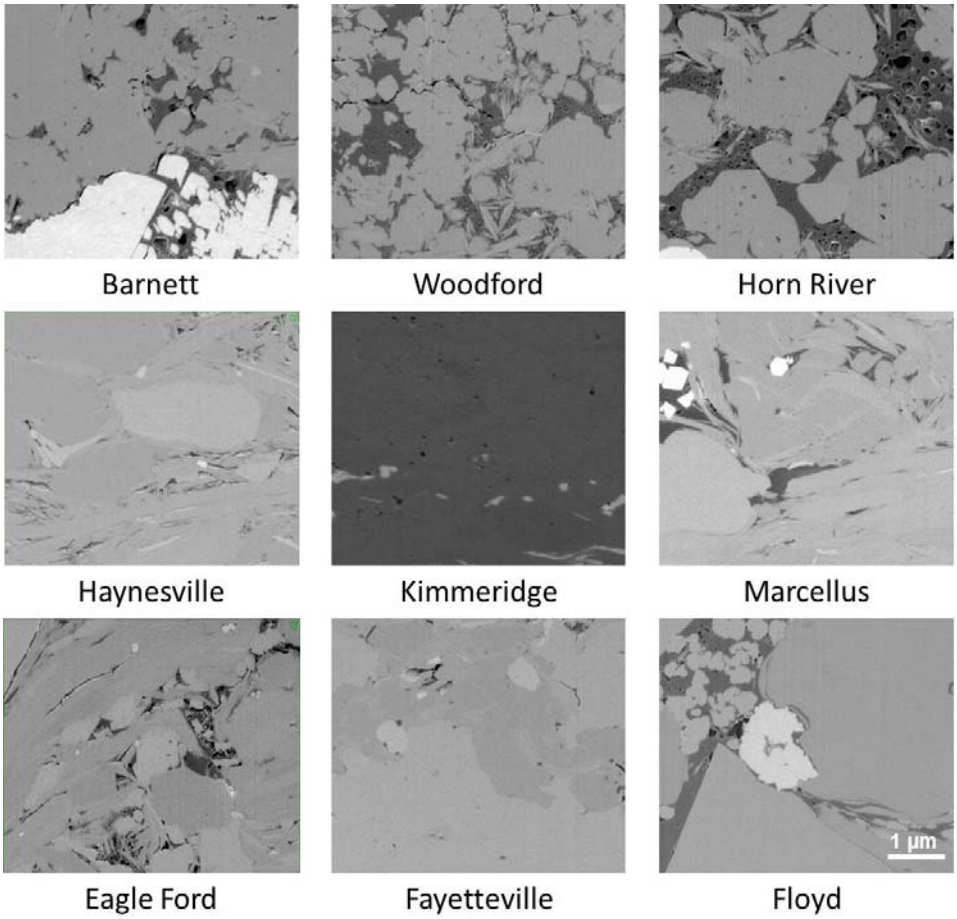

Figure 3: Mosaic of SEM images taken on FIB prepared samples from 9 different shale formations.

Figure 4 shows porous kerogen with an estimated porosity of 48-55%. A large number of these circular pores are below 20 nm in diameter with many pores possibly in a size regime below the resolution of the SEM. The location, shape, and size of the pores both within the kerogen and inorganic matrix will have a profound effect on many important aspects of shale gas production such as GIP and the physics of fluid flow through shale.

Common to most images in Figure 3 are components such as organics, pyrite, clays and quartz grains. Porosity appears to share similarities and differences. There are three classes of porosities: 1)crack-like, 2)phyllosilicate and 3)organophyllic. Not all shales display the crack-like pores. When organics are present in the more mature shales, they are typically associated with internal and bounding pore spaces. The magnitude of the internal organic porosity may be a function of maturity but seems to

vary among the images shown above. Clay platelets appear to conform to pyrite, grain and organic elements within each shale. The lack of conformity in detail contributes to phyllosilicate porosity. Pyrite is euhedral or framboidal. Internal to the framboids, we have observed additional organics and pores. Grain sizes even at this scale are variable between shales.

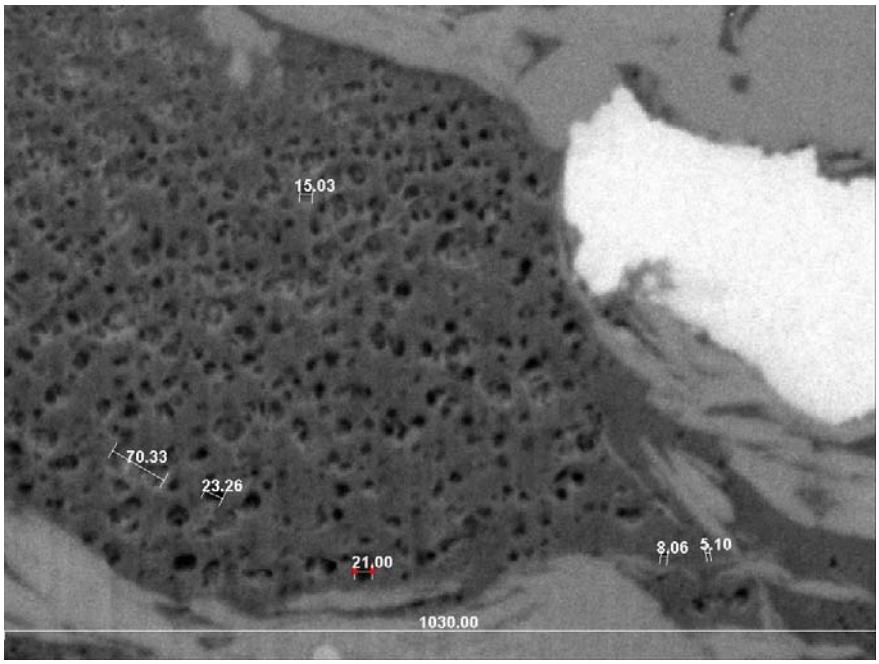

Figure 4: Organics showing 48-55% porosity in a Barnett shale sample. The bright object is pyrite. Holes within the organics are consistent with those resolved through MICP and NMR relaxation studies. (see Sondergeld, et al. 2010)

Conclusions:

New SEM imaging technologies are revealing important microstructures in shales which will allow classification and quantitative flow modeling. These images are combined with macroscale petrophysical measurements to correlate measurements with microstructure controls. Based on these images, we anticipate the seismic behavior of certain shales will be radically different than others forming the basis for detecting compartmentalization and prediction of over pressure. Moreover, these insights into shale microstructure allow investigators to ask questions previously unthinkable.

References:

Moncrieff, J. 2009. Microstructure of shale. Poster presentation at SPWLA Annual Meeting, Houston, Texas

Sondergeld, C.H., Ambrose, R. J., Rai, C.S., Moncrieff, J. 2010, Micro-Structural Studies of Gas Shales, SPE Unconventional Gas Conference, Pittsburgh, PA, SPE131771