P06

A Roadmap to Representative Data at the Nanoscale in Shale

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SUMMARY

The fine grained nature of shale makes scanning electron microscopy (SEM) an essential part of the core analysis suite of techniques. Both the imaging and the compositional analysis require a specific approach when working on shale samples.

In many shale reservoirs, the pores in the organic matter can be as small as 2nm. Imaging these small features comes at the cost of a very limited Field of View. The higher the magnification, the smaller the area that will be imaged. This raises serious questions about the representative nature of the SEM images. This paper describes a methodology where mosaics with increasing resolution and decreasing Field of View combined with EDS maps are analyzed digitally to uncover the representative features or building blocks that make up the rock fabric.

Introduction

The fine grained nature of shale makes scanning electron microscopy (SEM) an essential part of the core analysis suite of techniques. Typically, the SEM is used for both imaging and for elemental analysis by Energy Dispersive X-Ray spectroscopy (EDS). Both the imaging and the EDS analysis require a specific approach when working on shale samples.

In many shale reservoirs, the pores in the organic matter can be as small as 2nm, what would be the lower limit that can be visualized in the SEM. Imaging these small features comes at the cost of a very limited Field of View. The higher the magnification, the smaller the area that will be imaged. This raises serious questions about the representative nature of the SEM images. In the ideal case, one makes a mosaic by stitching as many SEM images as needed to have both the fine details and the fabric. However, it would take 900 million (2kx2k) pixel images to cover the entire surface with a resolution that sees the pores. While technically possible, it would not be practical.

Method and/or Theory

Mosaics of backscatter electron images with increasing resolution and decreasing Field of View combined with EDS maps are analyzed digitally to uncover the representative features or building blocks that make up the rock fabric. These building blocks are the prime candidates for subsequent 3D analysis at the nanoscale with the Focused Ion Beam Scanning Electron Microscope (FIBSEM). If mechanical properties can be related to these building blocks; and the spatial relationships known between the building blocks, then the mechanical properties of the core plug sample can be predicted. The EDS analysis itself, however, has some serious issues too. The interaction volume of the electron beam is a pear shaped figure with a diameter of between 1 and 2 micrometer. For a shale sample where grain sizes of clays, micas and organic particles are submicron, this means that the EDS spectrum can contain multiple phases. The EDS analysis is hence complicated not only by the clay minerals themselves but also because the spectra may contain different clays altogether.

This paper presents results in which the spectrum was analyzed in each pixel, and the mineral content and its relative proportions are known in each pixel. That gives insight in the association of the different minerals not attainable by any other technique. This provides statistically meaningful data on what phases are interacting spatially, and further refines the analysis of mechanical properties from images only.

Figure 1 Backscatter electron image with segmented dataset showing the porosity in black and the organic matter in red. This image is a mosaic of hundreds of individual SEM images; such that a large Field of View is combined with pore scale resolution.

Conclusions

Measurements at the submicron level are needed to characterize the pore space in gas shales. A combination of large area high-resolution imaging with composition analysis gives more representative data than randomly picked locations for high resolution imaging with limited Field of View.