Introduction

As shale gas has attracted substantial interests in recent years, characterization of unconventional mudstone reservoirs is becoming more and more important, especially for production planning. Sample based information such as the compositional and textural framework is one of the objects of typical analysis for shale-gas reservoir rocks. However, obtaining such shale information is challenging because of its multiple scales (micron- and nano-). Investigation of pore networks in mudrocks plays a vital role in shale characterization. In a common classification, major pore types include inorganic interparticle pores, inorganic intraparticle pores and organic-matter pores [1]. Especially, intraparticle pores within organic matter (kerogen) are related to thermal maturation of organic matter. Pores form when kerogen converts to hydrocarbon species such as liquids or gases. Due to this reason, a full study of organic matter pores on dimension, shape, location, porosity, connectivity and permeability is demanded.

Among a variety of characterization techniques for shale specimens, scanning electron microscope (SEM) offers a powerful imaging capability to investigate both mineral structures (typically in micron scale) and pores (down to a few nanometers) [2]. The modern FIB (focused ion beam)-SEM combines high resolution imaging capability with in situ nanoscale ion milling. It is able to create 3D reconstruction for connectivity and permeability studies [3, 4]. Such an expensive FIB-SEM workstation must require a stringent condition which is not possible for field application. In this note, we present a high throughput shale analysis procedure which includes high resolution imaging of ion milled shale specimens by using a compact field emission SEM and a quick software analysis for 2D porosity measurement.
Experiment

The shale specimen was prepared by embedding shale rock pieces in epoxy followed by mechanical polishing and ion milling in Fischione 1060 SEM Mill. Imaging was performed on Keysight 8500B SEM/EDS without metallic coating. The Keysight 8500B (Figure 1) is a compact field emission SEM/EDS, optimized for low voltage imaging (< 2 kV). It employs a novel all-electrostatic lens design eliminating any hysteresis effects. Keysight 8500B is able to achieve sub 10 nm resolution at 1 kV which provides a straightforward technique for high resolution imaging of uncoated rock specimens with enhanced surface details [5]. The compact dimension and high resolution imaging capability of Keysight 8500B hold the promise for potential SEM deployment in field. Image processing and analysis were performed using Pico Image (version 7.3) offered by Keysight.

Results

Figure 2 shows two typical backscattered electron (BSE) images of the shale specimen. Working at low voltages (e.g. 1 kV), backscattered electrons also carry high resolution information because of the reduced beam-specimen interaction volume. Without sample coating, charging-free images can be obtained with high resolution and excellent contrast. The bright and grey areas in Figure 2a correspond to pyrite and mineral, respectively. The dark grey areas are organic matter (kerosene). Pores including inorganic phyllosilicate intraparticle pores, crack-like pores and organic matter pores, indicated in arrows in Figure 2b, display in the darkest color. It can be estimated that the nanopores in organic matter are in the range of 10-100 nm.

The obtained raw SEM image was first adjusted with contrast and brightness on the MountainsMap Software. Then the enhanced image was applied with
thresholding to create a monochrome image, see Figure 3a. The blue areas indicating all pores display clearly on the dark background. The cracks in the specimen are normally featured with a larger area with a larger aspect ratio than pores. They could be induced by mechanical deformation during drilling or sample preparation. In order to remove those crack-like pores, the "sort grains" function based on grain area was applied. Figure 3b shows the pores with areas larger than a high threshold value (criteria). In SEM imaging, the smallest feature that can be seen is mainly determined by the spatial resolution of the microscope. Therefore, an assumption in this porosity measurement applies: no nanoscale pores with a dimension smaller than the resolution of the microscope (< 10 nm at 1 kV for Keysight 8500B) will be included. By another area-based pore sorting process, pores smaller than a low threshold value can be separated, as shown in Figure 3c. Figure 3d is the final image after pore sorting which exhibits most of nanopores that can be resolved by the SEM being used.

From Figure 3d, statistics over sorted pores can be obtained. As shown in Figure 4a, the total area occupied by the pores is 0.224 µm², and the porosity over the whole scanning area is 0.983%. The diameter distribution of the sorted pores is plotted in Figure 4b. The abrupt cut off at lower end is due to the resolution limit of the SEM.

By proper setting of the threshold value, the area of the organic matter can be obtained. Figure 5 shows a multi-color grain image. By selecting each grain, the area of each piece of the organic matter will be calculated. The total area of sorted pores divided by the total area of organic matters (5 pieces in Figure 5) will give a 2D porosity value over the organic matter, which is around 2.42%. It is believed that the magnitude of the organic porosity may be a function of maturity. The quick SEM imaging and porosity measurement of shale specimens could potentially provide useful microstructural information at the well sites.
Acknowledgement

We acknowledge Fischione Instruments and Digital Surf for collaboration in this work.

References


