Microstructural characterization of Gas Shales by electron microscopy

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Summary

The microstructure of gas shales was charaterized by state-of-the-art electron microscopy. Components were identified by Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) analysis of cross sectioned surfaces. Nano-pores in both organic and inorganic matters were imaged by Focused Ion Beam (FIB) and Transmission Electron Microscopy (TEM).

Introduction

Among the unconventional gas reservoirs that have been paid significant attention with increasing demand for stable resources for natural gas, gas shales is one of the most promising resources. Investigation of microstructure and chemical components of gas shales is very critical to help understand the microstructural controls on porosity and permeability.

Method

Electron microscopy has emerged as a very powerful tool for characterizing morphology and chemical composition of materials at the nanoscale. Here we applied various techniques (including SEM, FIB and TEM) to investigate typical shale samples. Proper sample preparation is critical to obtain high-quality images with minimal artifacts. Ar ion milling was used to produce cross sectional samples, and thin sections were prepared by FIB for high resolution TEM imaging.

Examples

1. Sample preparation:

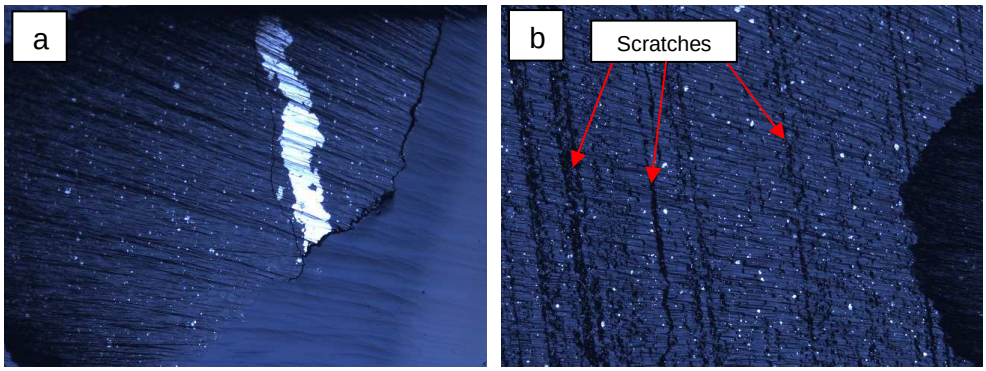

Figure 1. Ar ion milling (a) produces surface with less artifacts for SEM imaging, compared to (b) mechanical polishing.

2. SEM images and elemental mapping by EDX

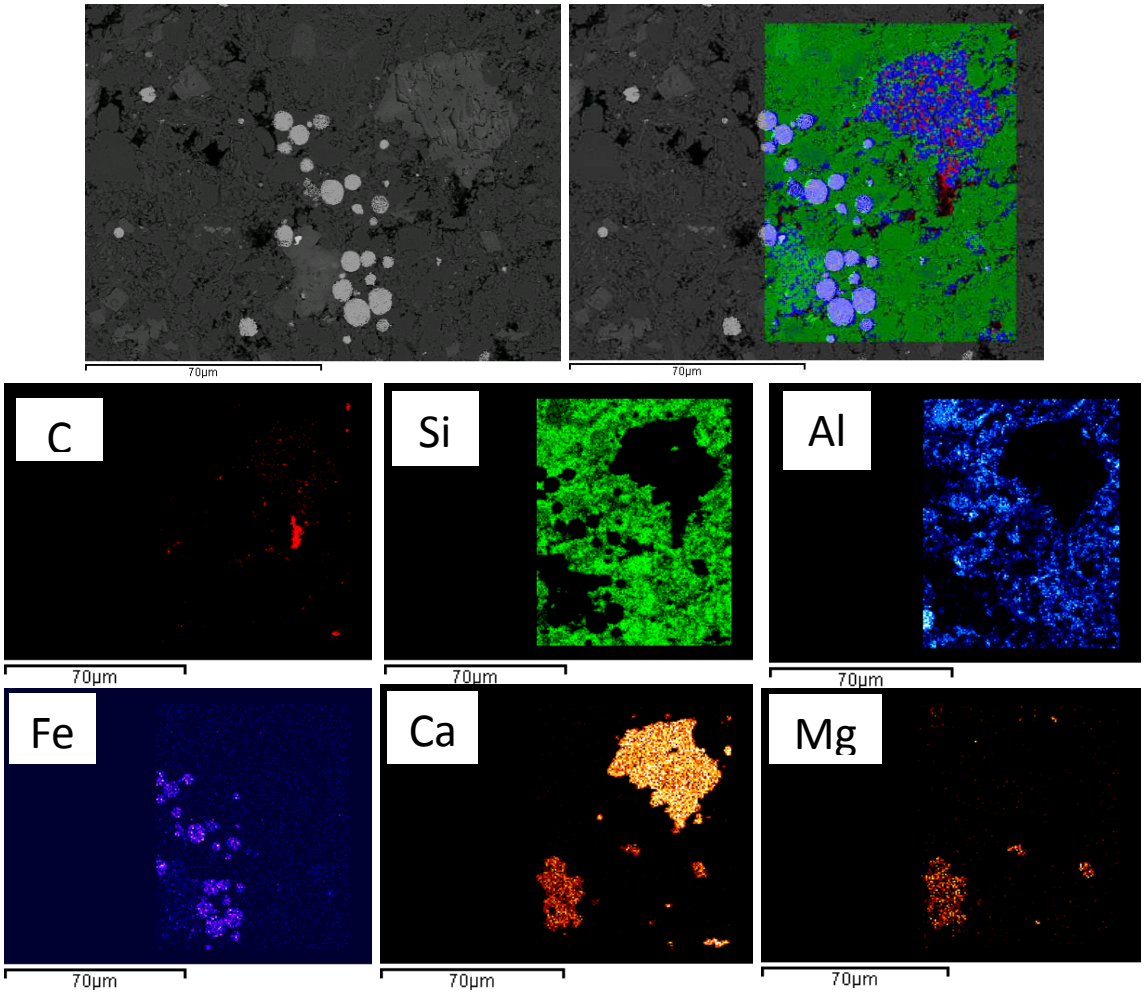

Figure 2. Backscattered electron (BSE) image and EDX mapping of an ion milled shale surface, showing typical organic and inorganic components.

3. Total organic-carbon (TOC) content analysis from BSE images

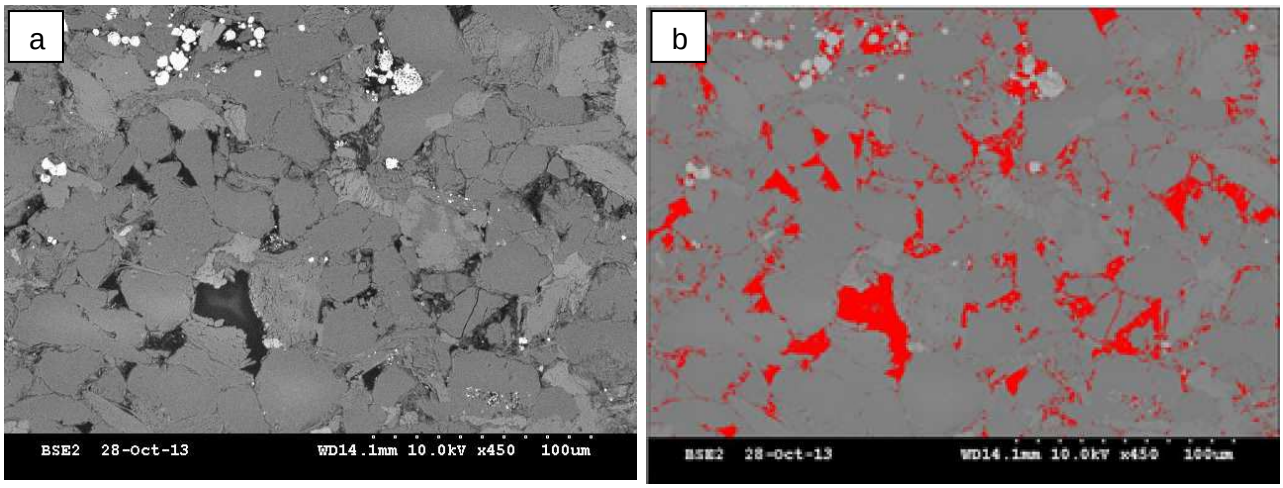

Figure 3. (a) a typical BSE image which provides contrasts proportional to the mean atomic number of the sample; (b) TOC analyzed directly from the image,
which is 9.3% in this image.

4. Porosity analysis by FIB and HRTEM

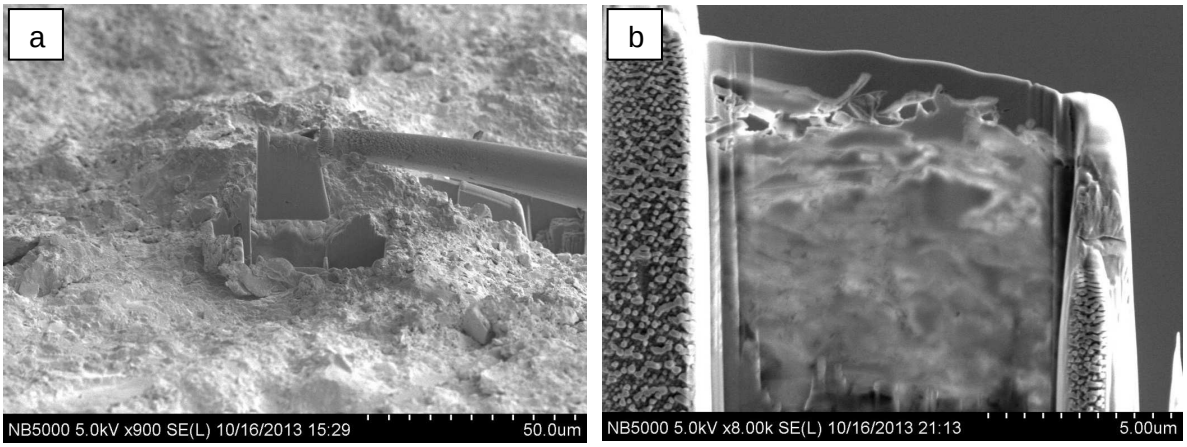

Figure 4. (a) a section was plucked out in a FIB and (b) thinned for TEM imaging.

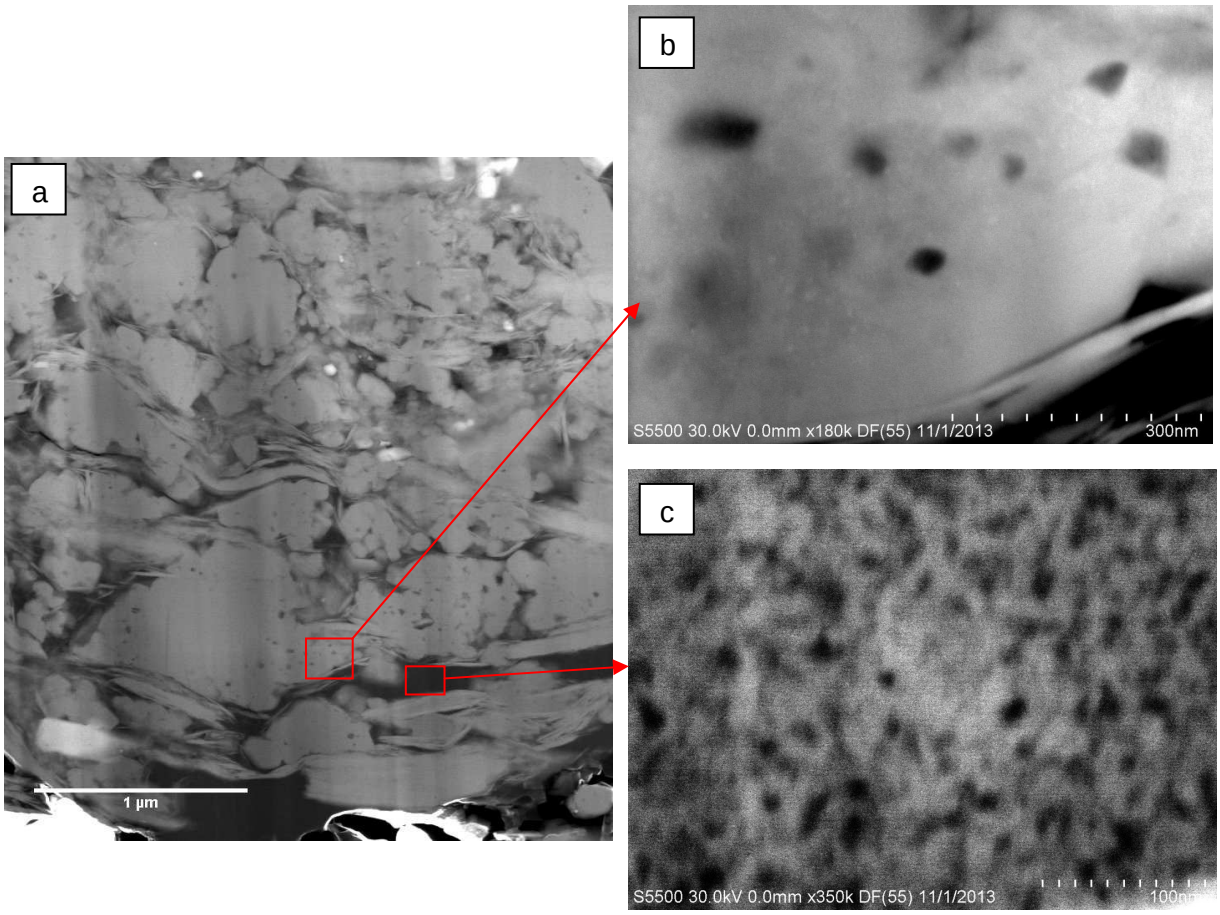

Figure 5. (a) Dark-Field STEM image of the thin section prepared by FIB as showed in figure 4, (b) nano-pores inside the quartz and (c) nano-pores inside the organic matter.

Conclusions

Electron microscopy techniques have been applied to image the microstructure of shale samples: SEM and EDX for chemical composition from cross sectioned surface; FIB and TEM for nano-pores in both organic and inorganic matters.