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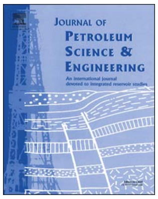

Microstructural and geomechanical analysis of Bakken shale at nanoscale

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ABSTRACT

With the development in production from shale oil and shale gas in North America during the last decade, more studies are being conducted in order to improve our knowledge of the shale characteristics. In this paper, samples from Upper and Middle Bakken Formation, which is an oil-bearing shale formation, were collected and analyzed. Permeability, porosity and saturation of the samples were studied in the lab. 2D XRD and EDX were used to study the mineral compositions, and FESEM was used to characterize the pore structure at micro and nanoscale. Implementing the image analysis method, the pore structure and pore size distributions (PSD) of the samples at nanoscale were quantified. In addition, nanoindentation method, which is a novel technique to investigate the geomechanical behavior of rocks, was applied to quantify the mechanical properties of the shale samples including Young's modulus, hardness, and fracture toughness at nanoscale.

1. Introduction

After the commercial boom of shale gas and oil in North America during the past decade, exploration of unconventional shale reservoirs has studied in different countries with potential economic hydrocarbon reserves. With the advancements of hydraulic fracturing technology applied to enhance production from unconventional reservoirs, a great increase in oil and gas production has been experienced (Li et al., 2015). This technique often, in combination with horizontal drilling, has allowed the United States to produce economically from unconventional reservoirs. Shale properties, such as pore microstructures and mechanical properties can influence the performance of hydraulic fracturing (Johnson and Rodgerson, 1998; Kissinger et al., 2013; Lamont and Jessen, 1963). The deep understanding of the pore structures and the mechanical properties can result in successful hydraulic fracturing operations and increased profits.

In order to obtain shale properties, in particular, mechanical properties, core plugs are used to carry out dynamic or static type experiments at the lab scale or the sonic logs are used to obtain the properties at larger scale representing the field (Shukla et al., 2013). Due to chemical and mechanical instability of shales, retrieving cores or a suitable volume of samples to perform standard experiments can be challenging. In addition, due to the presence of various minerals in shales, such as the presence of quartz, feldspar, calcite and clay minerals, field driven elastic properties (Young's modulus, Poisson's ratio) can be erroneous. Nanoindentation, which measures the applied load based on the depth of penetration of an indenter, shows promising applications to estimate the mechanical properties in different medium types. In petroleum and civil engineering, many researchers have applied the nanoindentation method to study shale properties (Kumar et al., 2012; Mason et al., 2014; Pal-Bathija et al., 2008). However, a majority of these studies have been focused on gas-bearing shale formations and organic matter properties.

The Bakken Formation is an Early Mississippian late Devonian organic-rich shale, which is located in the Williston Basin in Montana, North Dakota and southern Saskatchewan. It has become one of the largest shale oil plays in the world and currently produces around1 million bbls of oil daily. The Bakken Formation is composed of three typical members due to the lithology difference. The lithology of Upper and Lower Bakken members is similar with noncalcareous organic rich shales while Middle Bakken being the main production zone is highly variable in lithology and consists of several distinct lithofacies (Pitman et al., 2001). Pore structures and the mechanical properties of Bakken Formation still has not been fully understood.

In order to take a further understanding of the properties of Bakken Shale Formation at the nanoscale, in this paper, samples were taken and analyzed. Mineral compositions and microstructures were studied by using 2D X-Ray Diffraction (XRD) and a Field Emission Scanning Electron Microscope (FESEM) respectively. Then Young's modulus and hardness were derived using the nanoindentation method. Finally, based on the energy analysis method, fracture toughness of shale was calculated which noticeably extended the applications of nanoindentation in shales.

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Nomenclature

$h_{\max}$	Maximum displacement, nm	E_r	Reduced modulus, GPa
$P_{\max}$	Maximum indentation load, mN	U_t	Total energy, J
S	Contact stiffness	U_{pp}	Energy cost by plastic deformation, mN nm
h_S	Vertical surface deflection, nm	U_e	Energy cost by elastic deformation, mN nm
h_C	Contact depth, nm	U_{crack}	Energy cost by cracks, mN nm
h_f	Residual displacement, nm	G_C	Energy release rate, Nm/m^2
		K_{IC}	Fracture stress intensity factor, MPa$\sqrt{m}$

2. Samples preparation

For the analysis of the properties of Bakken Shale, we selected four samples. Sample 1, 2 are from the Upper Bakken Formation, and the other two are from Middle Bakken Formation. Small chips with the size around 2 cm length and 1 cm width parallel to the bedding were sampled. Because the roughness of the sample surface will affect the nanoindentation tests (ASTM, 2007), a certain sample preparation procedure needs to be followed before performing the tests. In this study, all samples were resin coated. When the resin became solid, sand paper with different sizes of grit from 600 to 1200 was used to polish the sample surface. Finally, we used diamond polishers with various grain sizes (5, 3 and 0.5 µm) to polish the sample to the desired smoothness. Fig. 1 shows the four shale samples after preparation procedure ready for the nanoindentation tests. Fig. 2 shows the 3D tomography of Sample 1 by AFM (atomic force microscope) with the surface roughness of 262 nm, which fits within the flat surface requirement of nanoindentation.

3. Experiments

3.1. Mineral composition analysis

X-Ray Diffraction (XRD) test was performed on the flat surface of the samples using Bruker D8 Discover 2D instrument. Comparing with the conventional 1D XRD, as the widely employed equipment in the petroleum industry which is confined to plane analysis of the sample surface, the 2D XRD can detect the whole and large portion of the diffraction rings and angles spectrum. In addition, the percent crystallinity measured with the conventional diffractometer is not consistent if the preferred orientation is not considered, while the sample orientation has no effect on the full circle integrated diffraction profile from a 2D frame. Also, 2D XRD system can measure percent crystallinity more accurately with consistent results (He et al., 2000; He 2003) for layered materials with preferred orientations such as shales.

In this experiment, we used 2D XRD to analyze the compositions of the samples. We set three frames with the scan width of 30° while each frame took 300 s to be covered. Then the signals from the three frames were combined and generated the mineral compositions by analyzing the signals. The procedure is explained in details in the following sections.

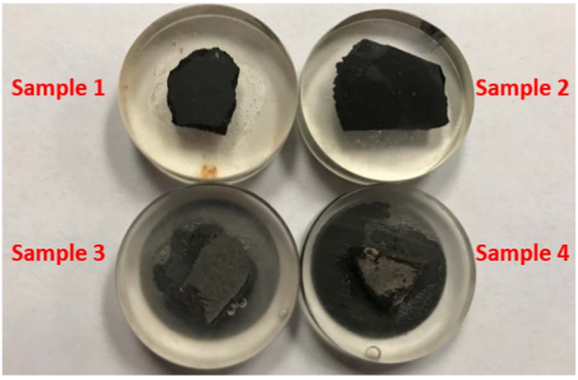

Fig. 1. Prepared samples for nanoindentation test.

3.2. SEM imaging

Field Emission Gun Scanning Electron Microscope (FESEM) JEOL

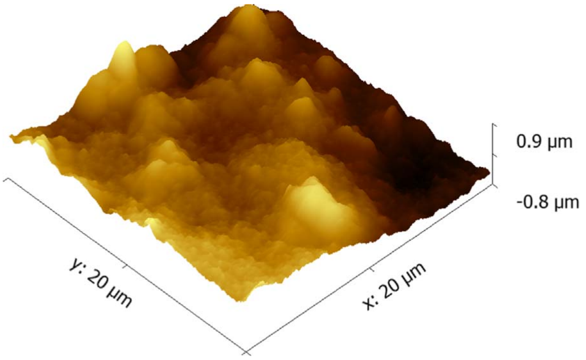

Fig. 2. 3D tomography of Sample 1 produced using the AFM.

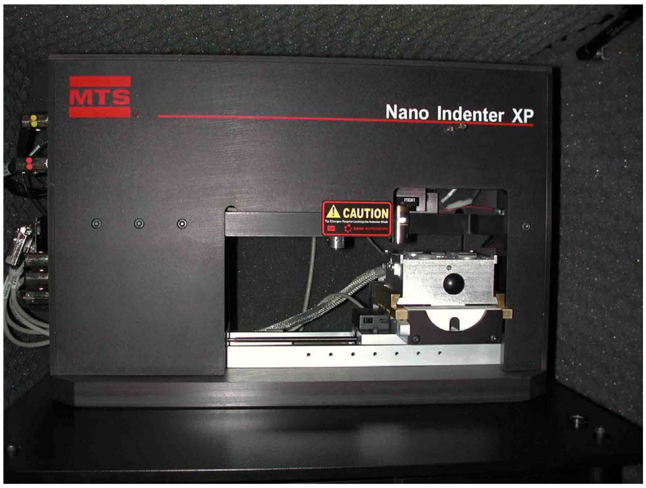

Fig. 3. MTS Nanoindenter XP.

Table 1
Core analysis results.

	Permeability	Porosity Helium	Bulk density	Saturation	
	md	%	gm/cc	Oil %	Water %
Sample 1	0.02	4.8	2.6	44.5	44.5
Sample 2	0.01	4.2	2.57	46.9	26.3
Sample 3	0.072	7.6	2.73	36.1	37.2
Sample 4	0.027	6.7	2.71	43.4	32