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Characterization of typical 3D pore networks of Jiulaodong formation shale using nano-transmission X-ray microscopy

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HIGHLIGHTS

- Nano-transmission X-ray microscopy is used to resolve different pore structures in shale.
- Pore morphology and interconnectivity were analyzed using 3D pore–throat model.
- Nanometer pores dominate in numbers while micrometer pores dominate in volume.
- Elliptical pore and micro cracks are the major pore structures.

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ABSTRACT

The microscopic pore structure is one of the most important factors to understand shale gas reservoirs. Recognized as a non-destructive method, nano-transmission X-ray microscopy (Nano-TXM) is sufficiently powerful to resolve nanometer pore structures and to quantify the effective network in shale. In this work, three dimensional (3D) pore networks of typical pore structures, such as organic matter pores (OM pores), interparticle pores (InterP pores), intraplatelet pores within clay aggregates (IntraP pores) and intercrystalline pores within pyrite (InterC pores), developing in Jiulaodong (JLD) formation shale in the Weiyuan 201 well (W201) in Sichuan Basin were reconstructed by Nano-TXM. Meanwhile, the pore morphology, pore size, porosity and interconnectivity were analyzed using Pore Network Modeling (PNM). The results indicated that the pore shape, pore size distribution, porosity and interconnectivity varied between the four pore types. Nanometer pores ranging from 150 nm to 1000 nm dominate the OM pores in the samples. However, pores with a sheet-like structure that are larger than 1000 nm are mainly found in InterP and IntraP pores. OM pores and InterP pores have larger porosities (35% and 23.7%, respectively) than the other two pore types. OM pores, InterP pores and InterC pores exhibit good and homogenous 3D connectivity, whereas IntraP pores have good extensity parallel to the clay mineral orientation but have no connectivity perpendicular to it. The 3D morphology and pore parameters suggest that the nano-pores in OM and InterC pores store absorbed gas and might be connected by micropores that existed in InterP and IntraP pores. The characterization of the pore structure in shale samples provides useful information for shale gas development.

1. Introduction

Shale gas has become one of the focuses of world attention after its rapid and successful commercial development in North America [6]. Shale gas explorations have been carried out and recently attained significant progress in Canada, England, China, Germany and other countries, especially in China [4,12,7,18]. The lower Paleozoic reservoirs of the Sichuan Basin in southwestern China

Abbreviations: Nano-TXM, nano-transmission X-ray microscopy; 3D, three dimensional; OM pore, organic matter pore; InterP pore, interparticle pore; IntraP pore, intraplatelet pores within clay aggregates; InterC pore, intercrystalline pores within pyrite; W201, Weiyuan 201 well; 2D, two dimensional; FE-SEM, field emission scanning electron microscope; FIB-SEM, focused ion beam scanning electron microscope; X-CT, X ray computed tomography; EDS, energy dispersive spectroscopy; NSRRC, National Synchrotron Radiation Research Center; PNM, Pore Network Model; PSD, pore size distribution.

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are typical gas-rich horizons with several sets of shale, indicating a tremendous potential for shale gas development [31,32]. In 2011, an industrial gas flow was first obtained by China National Petroleum Corporation at the Weiyuan 201 well (W201). As a typical tight gas reservoir, information regarding shale pore structure, which has a strong influence on the petrophysical properties of reservoir rocks, is necessary to understand storage and transport mechanisms [11,16]. The 2D-pore morphology, size and distribution are acquired by SEM and other methods [20,10,15]. Meanwhile, the shale pore size can reach the nanometer scale, which is much smaller than that quantified in sandstone and carbonate reservoirs [21,20,2]. Macropores, with a length lager than 50 nm, make great contributions to the total pore volume and porosity, as determined by nitrogen adsorption, high pressure mercury injection tests and FIB-SEM observations [9,29]. The microscopic structure is considered to be important for shale gas reservoirs; therefore, the two-dimensional (2D) pore structure characteristics of the JLD formation shale from W201 have been thoroughly researched [28]. From previous studies [20,9], there are four major types of pore structures in the JLD shale from W201 including organic matter pores (OM pore), interparticle pores (InterP pore), intraplatelet pores within clay aggregates (IntraP pore) and intercrystalline pores within pyrite (InterC pore). The InterP and IntraP pores are nanometer to sub-micrometer scale, are the most widespread forms in JLD shale, and are spaces for the storage of free gas. The OM pores, which consist of kerogen and hydrocarbons, are approximately 50 nm to ~1 μm and provide the main storage space for absorbed shale gas. The 2D pore structure characteristics of shale samples have been researched intensively but provide information on only the pore shape and size. However, its three-dimensional (3D) pore morphology and connectivity are still poorly understood and need further study.

Currently, many methods are used to directly characterize the nano-scale pore structure in shale gas reservoirs, such as field-emission scanning electron microscopy (FE-SEM) coupled with argon-ion-milled technique, focused ion beam scanning electron microscopy (FIB-SEM) and X-ray computed tomography (X-CT). FIB-SEM, with a maximum resolution of several nanometers, is used to visualize the nano-scale pore–throat structure in 3D, but it does unrecoverable destruction to the shale sample, making it impossible for further study with other methods [9,1]. Therefore, nano-transmission X-ray microscopy (Nano-TXM, also called Nano-CT) has received much more attention since the discovery of nano-pore in unconventional oil reservoirs. Recognized as a non-destructive method, X-ray computed tomography has been used to study pore structure, mineral contents and their spatial distributions as a function of different X-ray absorption coefficients. Meanwhile, permeability and porosity can also be calculated using relative computing software [23,14]. Additionally, Nano-TXM has a higher efficiency than FIB-SEM, while for example, a cube with a 10 μm side takes the latter at least 10 min to mill. With a resolution ranging from several micrometers to tens of nanometers, TXM is sufficient to resolve pore–throat structures down to the nanometer scale and to quantify the effective network in shale. When combined with an advanced dual-beam FIB-SEM milling-imaging technique, a typical area can be obtained by gallium ion milling, allowing SEM imaging of a newly milled shale surface in situ. Thus, the different types of pores that develop in shale can be clearly identified and chosen for Nano-TXM experiments to independently obtain their 3D structural information.

In this paper, the 3D pore networks of typical pore structures including OM pore, InterP pore, IntraP pore and InterC pore from a JLD formation shale sample from W201, Sichuan Basin were determined by Nano-TXM. Three dimensional models were built to intuitively show the pore spatial distributions. Furthermore, the quantitative information of pore size distribution (PSD), porosity and interconnectivity that are significant for shale gas accumulation and exploration were also calculated.

2. Samples and methods

2.1. Samples

The W201 shale samples from JLD formation were provided by Institute of Geology and Geophysics, Chinese Academy of Sciences. They are from the well that first provided the industrial gas flow by the China National Petroleum Corporation in 2011. The W201 shale samples were from approximately 2756 m in depth and had formed in a marine sedimentary environment, the lithology of

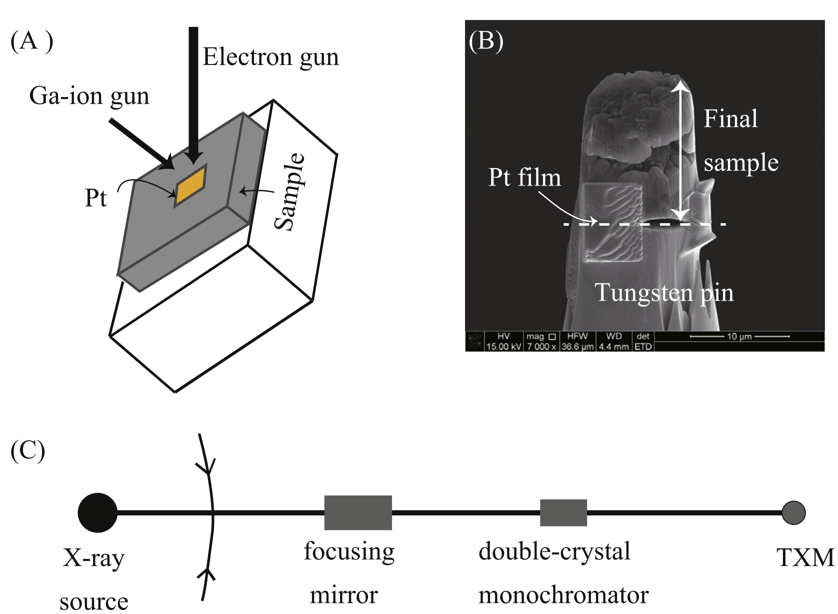

Fig. 1. (A) Schematic drawing of the milling procedure in a FIB dual-beam system. (B) Side-view of the final sample for Nano-TXM investigation. (C) Optical layout of beamline BL01B at the NSRRC.