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RESEARCH PAPER

Three dimensional characterization and quantitative connectivity analysis of micro/nano pore space

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Abstract: Evaluation is performed on pore connectivity of unconventional reservoirs using 3-D FIB-SEM imaging characterization and digital rock techniques. 3-D images are first obtained by FIB-SEM device and then transformed into digital pore structure model through shape correction, brightness correction, depth-of-field correction and phase distinguishing. Based on this model, a new connectivity evaluation method for micro/nano pores in unconventional reservoirs is proposed. This method differentiates dead and live pore space, grades live connected domains (1-grade is the worst and 3-grade is the best) and calculates the connectivity rates of all grades. Selected connected domains can be quantitatively characterized through statistical analysis of connected domain distribution, volume and shape. The applications on nano material, shale and carbonate get distinctive connectivity rates (96%, 22% and 82%) and characteristic differences on connected domain distribution, volume and shape. Through these statistical parameters (connectivity rate, connected domain distribution, volume and shape), the three demonstrated materials and reservoirs are quantitatively characterized and differentiated. Thus, validity of the proposed connectivity method in this paper is proved.

Key words: micro/nano pore space; pore structure; pore connectivity; unconventional hydrocarbon; Sichuan Basin; Junggar Basin

Introduction

Compared with conventional oil and gas, unconventional oil and gas are stored in smaller scale space, such as micro-pores and nano-pores, so the study of micro/nano pore space is of great significance for evaluating and developing unconventional oil and gas resources. Studies in China and abroad[1–5] show that the structure characteristics of micro/nano pores, the major reservoir space for unconventional oil and gas, is an important factor influencing physical property of the reservoir, so accurate and complete characterization of micro/nano pore structure has become an important aspect of unconventional reservoir study.

Currently, microscopic characterization methods for reservoirs can be roughly divided into two categories: (1) direct observation; and (2) indirect characterization that relies on pore structure characteristics. Commonly using laboratory devices such as high-resolution scanning electron microscope (SEM), atomic force microscope, nano-CT and FIB-SEM, the methods in the first category are visual, accurate and less affected by other factors, but small in characterization scale and limited by the resolution of the device used. Methods in the second category include constant-speed or high-pressure mercury intrusion test, gas adsorption test, nuclear magnetic resonance test and ultra-low permeability test. They have the advantages of large-scale sample and easy quantification of results, but these tests are not visual, difficult to verify, and strongly affected by anisotropy. Only through in-depth study of micro-pore characterization, can fluid distribution pattern and flow state in microscopic pores be more accurately described and analyzed.

With the decrease of pore scale, the permeability of tight reservoirs gets poorer. For micro/nano scale pores, permeability cannot accurately characterize permeable property of rock. Zou Caineng et al[4] proposed to describe the permeable property of nano-scale pore space with a new parameter, pore connectivity. Connectivity evaluation is an important part of the morphological study of porous-medium pore space. Connectivity evaluation of conventional pore space started quite early, for example, describing pore connectivity of soil and other porous media with the Euler characteristic[6]. This kind of quantitative connectivity evaluation method can provide quantitative parameter for pore network model and be used in permeability analysis. Researchers in China have carried out similar studies on pore connectivity[7–8]. An important

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means of connectivity evaluation is digital core technology (a general name [9–10] of rock microstructure description, digital model building, quantitative analysis of pore structure characteristics and petro-physical simulation techniques), which includes digital model building based on pore characterization, and analysis of resistivity and permeability etc.

In this study, based on previous research achievements, and using most advanced micro-pore structure characterization technology, the connectivity rate of unconventional reservoirs and statistical parameters of connected domain are studied using 3-D imaging characterization and digital core technology; and feasibility of this method has been verified by application.

1. 3-D characterization with ion-electron scanning microscope

1.1. 3-D Imaging of FIB-SEM

Focused Ion Beam (FIB) technology is often integrated with scanning electron microscope (SEM) into an instrument with 3-D imaging function. Under low-beam, FIB has microscopic imaging function similar to SEM, while under high-beam, FIB technology focalizes the ion beam spot to submicron, even nano-scale, and realizes micromachining through the deflection system.

Taking FEI Helios NanoLab 650 FIB-SEM as an example, its resolution can reach 0.9 nm at the accelerating voltage of 1kV. Besides ultra-high resolution, its most distinctive feature is that it can realize nano-scale erosion of tight reservoir rock and scanning layer by layer, reproduce 3-D images at nanometer resolution and accurately depict 3-D pore throat system of tight reservoir (Fig. 1). Imaging principle of FIB-SEM is: tilting the sample stage to make the sample surface perpendicular to the ion beam; carving the sample surface with the ion beam to expose the observation surface and perform the first imaging of the observation surface with the electron beam; setting energy parameters of the ion beam and carving the observation surface according to the corrosion thickness requirement of the observation surface; performing imaging of the new observation surface with the electron beam after

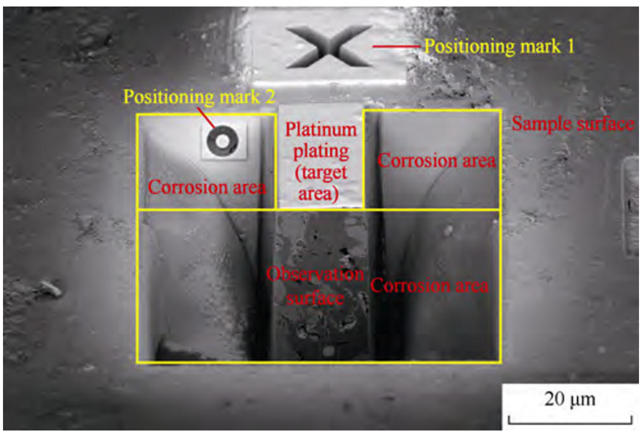

Fig. 1. Schematics of preparing 3-D imaging sample for FIB-SEM.

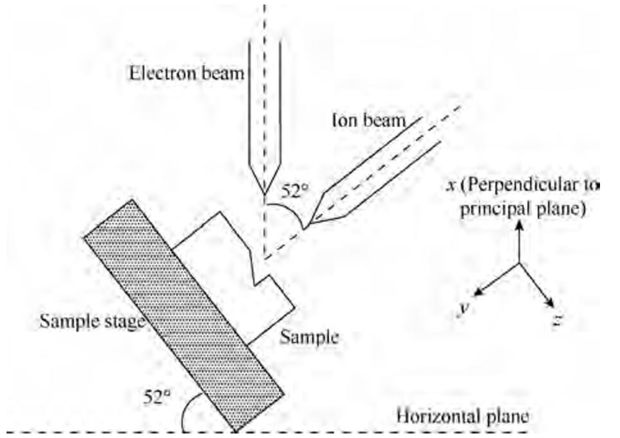

Fig. 2. Schematics of angle relationship between the electron beam, ion beam and the sample.

the corrosion; repeating this step once the imaging is completed (Figs. 1 and 2).

Basic steps for 3-D imaging of the rock sample using FIB-SEM are: (1) take small pieces of the rock sample, polish the surface, place them at the sample stage with conducting resin, and plate carbon on the surface; (2) revolve the work table in the working plane of the ion gun, mark the surface with the ion gun (positioning mark 1), and then plate platinum to protect the target area; (3) carve three sides of the target area to form an observation pit with a depth of 10-20μm and a width of about two times the width of the target area, and perform finishing of the observation surface; (4) revolve the working table in the working plane of the electron gun, mark the imaging plane of the electron gun with the ion gun (positioning mark 2) so that the imaging sequence has the same reference point; (5) revolve the working table in the working plane of the ion gun, select the imaging range of the electron gun and set imaging resolution, imaging time, sequential imaging footage and number of steps, start automatic denudation imaging after repeated corrections. In general, it takes about 6-20 h for 100 images.

1.2. 3-D digital rock technology based on FIB-SEM and related key issues

The original 3-D images captured by FIB-SEM must be digitally processed before quantitative analysis of pore structure. This process can be realized through digital rock technology, which mainly consists of three steps: digital image capture, digital model building and model analysis. Devices used for digital image capture are CT and FIB-SEM etc. Currently, FIB-SEM has higher resolution, but is only suitable for small samples. There are two kinds of methods to build digital model: one is directly building model according to the digital image, the other is building model based on the information reflected by the digital image in combination with special mathematical method. In model analysis, physical model is mainly used to solve the digital model to get the desired information.