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Short communication

Nanoscale pore structure and mechanical property analysis of coal: An insight combining AFM and SEM images

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ABSTRACT

Scanning Electron Microscopy (SEM) and Atomic Force Microscope (AFM), two easily acquired and widely applied image acquisition and analysis methods, have rarely been combined to study the pore structure for unconventional natural gas reservoir rocks. In this work, we present an investigation of nanoscale detection of the pore distribution and mechanical properties of coals using SEM and AFM observations, and conduct quantitative analyses on pore structure distribution, surface roughness and mechanical properties. The morphological characteristics of the coal surface can be revealed by both SEM and AFM methods, and the mechanical parameters of the selected position were obtained under the peakforce quantitative nano-mechanics (PF-QNM) AFM mode, including the Young's modulus, peak force error, deformation, and adhesion forces. By fusing 800 high resolution SEM images into one single image (named as MAPS), the pores morphology and distribution of different scales were acquired. And the studied coal shows different types of cellular pores and gas pores with multiresolution. The mechanical property difference between the matrix and minerals of coal are clearly observed, with the Young's modulus of organic component around 2 GPa, and that of the minerals generally higher than 10 GPa. The maximum adhesion force values range between 20 and 50 nN. The high values occurred where pores are developed. This work demonstrated that the combination of two dimensional (2D) SEM and three dimensional (3D) AFM results is effective in detection of surface properties, and is of significance in revealing the pore structure and mechanical properties at nanoscale.

1. Introduction

Detection of pore structure of unconventional natural gas reservoir rocks is continuously attracting extensive attention, as their nanoscale structure are of strong heterogeneity with no single equipment can fully illustrate the complex composition and structure variations [1–4]. Pore-scale modelling, e.g. "digital rock" technology, and modern imaging methods are emerging and this requires acquisition of pore structure in high resolution. Experimental methods are widely used including three dimensional (3D) imaging methods of micro- and nano- X-ray computed tomography (CT), Focused ion beam scanning electron microscopy (FIB-SEM) and Helium ion microscope (HIM) [5–8]. Moreover, the two-dimensional (2D) imaging methods, e.g. SEM and Atomic Force Microscope (AFM) are also widely adopted, because of their capability in rapid identification of maceral and mineral composition, pore size distribution and the surface properties [4,9–12]. The numerical simulation model for gas flow can be reconstructed also by 2D images using stochastic methods, such as simulated annealing method and multiple-point statistics method [13–14].

The SEM observation show that the adjacent micron-sized organic matter and maceral compositions sometimes differ greatly in porosity, which is caused by the innate differences in organic matter deposited as well as by different thermal evolution processes [15,16]. However, SEM cannot fully reflect the fluctuation of the sample surfaces, as the signals received generally shown in gray images, with no information on the surface roughness variation, or mechanical property [3]. AFM is effective in nanometer spatial resolution and force sensing sensitivity detection, which can not only measure the surface topography, but also reflect the mechanical and electrical properties of the tested samples, such as Young's modulus and surface potential [17–20]. As for coal and shale, two important organic rocks containing significant unconventional natural gas resources, their gas adsorption and flow capacity are

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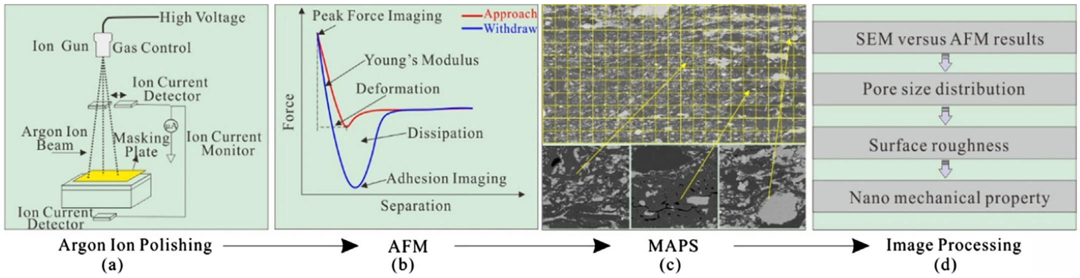

Fig. 1. Flow chart of the experiment. (a), argon ion polishing; (b), principle of the AFM test; (c), brief introduction to MAPS; (d), image processing procedures.

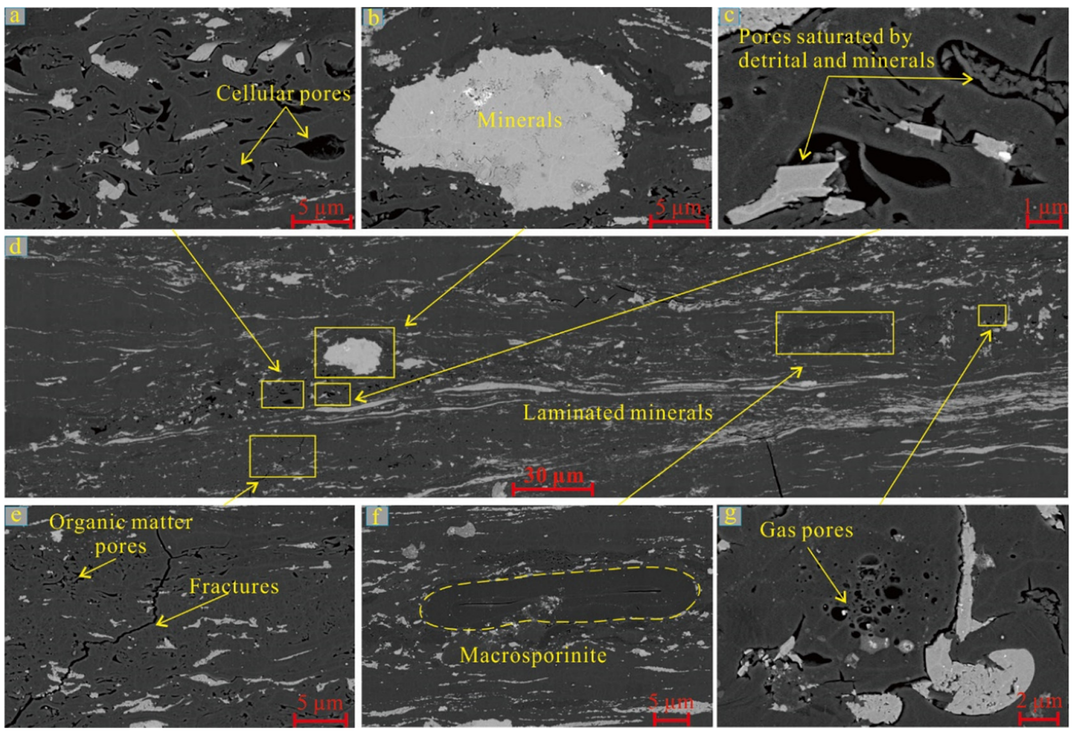

Fig. 2. Pores occurrence by SEM observations, (a), deformed cellular pores; (b), clay minerals; (c), pores filled by minerals and detrital; (d), MAPS image; (e), fractures in organic matter. (f), macrosporinite; (g), intensively gas pores in collinite.

closely related to its organic component as its abundance of micro- and nano- pores developed [1,21–24]. Mechanical property is tightly associated coalbed methane and shale gas production. The in-situ stress, bulk modulus, Poisson' ratio, axial and lateral strain, etc., are all related to the mechanical characteristics, affecting both fracturing efficiency and flow of gas in the reservoir [25]. Organic matter is a major component of coal and shale, hence fragile in nature. Thus, it is difficult to acquire samples of desire size to be experimentally tested for specific parameters [26]. Therefore, investigating the pores distribution and mechanical parameters is important for building robust geological models to access unconventional natural gas resources and enhance gas production efficiency [8].

As a complex issue with multiscale and multi-physics influencing factors, quantitatively and accurately revealing coal surface variation, including pore structure distribution, surface roughness, and its fractal characteristics, et al., has not yet been well understood [16,27]. In this study, we demonstrate the combination of SEM and AFM observations on nanoscale view firstly, providing direct and in situ mechanical characterization of coal matrix and minerals. Then discussion on the pores distribution and surface properties are performed. This approach allows evaluation of compositional and mechanical variations among different macerals (organic matter and minerals) and unravel the surface adsorption and flow capacity variations. The main innovation of this paper is 3-fold: (1) combining SEM 2D result and AFM 3D characterizations to study the nano pores morphology and surface characteristics comprehensively; (2) obtaining a large area of ultra-high-resolution SEM image by tilting thousands of SEM pictures (MAPS); (3) determining the mechanical property and pores distribution of the same location in a nano scale. The methods can be expanded to study the surface mechanical and pore structure variations under different maturity process and can be widely used in the shale reservoirs evaluation.

2. Methodology

2.1. Sample preparation and experimental procedure

The studied coal samples are collected from the Baode Coal Mines, northeastern Ordos basin, China, where the CBM have been successfully developed [28–30]. The electron beam bombardment on the surface of the sample in SEM may affect the sample to a certain extent. Thus to obtain the image of AFM and SEM in the same horizon, the AFM observation should be conducted first before the SEM experiment. In order to reduce the influence of human influencing factors during the experimental results, the following procedures were considered: (1) reduce the waiting time of the intermediate process; (2) scan more areas for sufficient data; (3) the optical microscope with the instrument was