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Pore structure characterization of coal by synchrotron radiation nano-CT

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ABSTRACT

For the significant impact of pore structure on gas storage and transport in coal seams, research on coal pore structure characterization has been a hotspot. Benefited from the high spatial resolution synchrotron-based nano-CT instrument, pore structure characterization of coal is investigated in nano scale. Image alignment and 3D reconstruction were completed at the platform designed by National Synchrotron Radiation Laboratory and Chinese Academy of Sciences. The segmentation of the unimodal grey-scale value histograms is solved by Between-class Variance Maximisation (BCVM) algorithm and the nano-CT images are segmented into three components, pore, organic components and mineral components. Based on the voxel number, components fraction is computed. Pore size distribution (PSD) presents bimodality. Pores with equivalent radius less than 60 nm account for 84% of the total pore number. Throats with equivalent radius less than 60 nm account for 89% of the total throat number. Throats with length less than 100 nm account for 58% of the total throat number and throats with length less than 400 nm account for 84% of the total throat number. Pore number decreases with the increase of coordination number. There are over 50% of pores without coordination pore and pore connectivity was analysed. Nanopore structure-based computational fluid dynamics (CFD) simulation was explored. The permeability in three coordinate axes directions presents anisotropy.

1. Introduction

Coal is a porous medium with complex pore structures [1]. Pore structures in the coal matrix affect the gas storage and transport in coal seams [2–4]. Pore size distribution significantly impacts the adsorption capacity of gas on coal, and pore interconnectivity influences the gas effective diffusivity of coal matrix [5–8]. Despite several studies have been presented in the literature on estimating coal microstructure properties and coal microstructure visualization [9–11], most of them assume the pore geometry in the interpretation of results (low pressure adsorption (LPA) using N_2 and CO_2, synchrotron small-angle X-ray scattering (SAXS), small angle neutron scattering (SANS)), and is either invasive (high pressure mercury intrusion (MICP)) or is limited to two-dimensional (2D) information (TEM and SEM) [12]. Most classical methods used for the characterization of coal porosity only provide information on the overall pore proportion and threshold pore size but little information on their real size or spatial distribution [13]. All the aforementioned techniques provide basic information (e.g. porosity and pore sizes), but they are constrained to precisely assess pore connectivity and spatial heterogeneity of the porosity [14]. X-ray computed tomography (CT) provides 3D images that can be processed and analyzed to get morphological information of the pore network such as pore shapes, sizes, connectivity, and tortuosity [15]. The limitation of the spatial resolution of the X-ray micro-CT represents a key issue in characterizing tight coal pore network. Recently, a new generation of laboratory-based nano-CT has been developed that provides nanoscale resolution [16]. However, these lab-based setups do not benefit from a monochromatic beam and high X-ray coherency, potentially causing some artifacts such as beam hardening [14]. As synchrotron-based nano-CT can achieve higher resolutions than laboratory-based nano-CT, authors of this paper have applied synchrotron-based nano-CT at the Beijing Synchrotron Radiation Facility (BSRF) to study the pore structure characterization of coal. Synchrotron-based nano-CT has been utilized for various porous media including cement [17,18], bone [19], and soil [20]. To our best knowledge, limited studies have been conducted on coal pore structure characterization by synchrotron-based nano-CT.

Pore structure characterization is critical in understanding the flow

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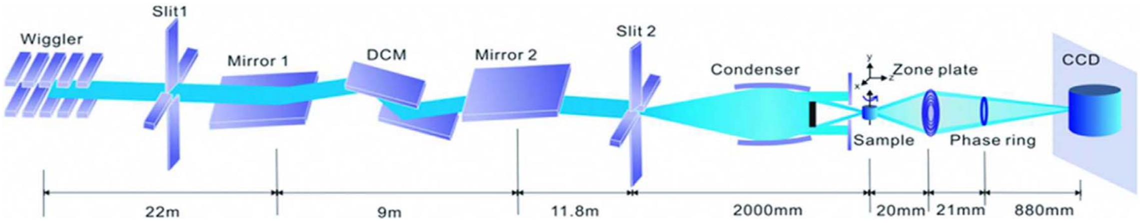

Fig. 1. Optical layout of the beamline and Nano-CT at BSRF. From Yuan et al. [29].

properties of tight porous media. Characterizing the flow properties at pore scale especially nanoscale by experimental approach is extremely challenging and difficult. A more straightforward approach to estimate the flow properties at pore scale is to use numerical models constructed using the CT images. This numerical approach can be divided into two groups [21]:

One is based on the pore geometry model, including lattice-Boltzmann method (LBM) [22], traditional mesh based computational fluid dynamics (CFD) method [23], and direct hydrodynamic simulator (DHD) which is based on the density-functional (DF) method applied for multiphase compositional hydrodynamics [24]. For the mesh based CFD method, the Navier-Stokes equation and full information of pore geometry and topology is implemented into the flow simulation. In order to accurately depict the complex pore structure obtained by high resolution CT instrument, the mesh size needs to be reduced significantly, which makes the CFD simulation computationally intensive and time consuming. In LBM simulation, the macroscopic behavior is described by modeling the particles propagate and interact at a mesoscopic level and then the macroscopic behavior is quantified by the weighted sums of the corresponding particle distribution functions [25]. As a result, LBM is also computationally intensive. It should be noted that the collision is calculated locally to handle the challenge of the geometric complexity, which makes LBM a good option for flow simulation in porous media with complex geometries.

The other is based on the pore network, which incorporates the complex pore space by a network of pore and throat (sphere-cylinder model) extracted from CT images [26]. The pore network is a lattice that consists of pore and throat size, coordination relationship between pores [27]. Based on the lattice, numerical simulation can be conducted. Pore network models illustrate complex pore structure using pore and throat with idealized shape (sphere and cylinder), which simplifies the simulation, but also losses some geometry details.

In this paper, considering the reliability and maturity of CFD method traditional mesh based CFD method, nanopore structure-based computational fluid dynamics (CFD) simulation was explored. Details in the CT images processing will be introduced, especially for the images alignment and 3D reconstruction and segmentation of raw CT images with unimodal grey-scale value histograms. Benefited from the high spatial resolution synchrotron-based nano-CT images, the nanopore structure characterization is investigated.

2. Experimental

2.1. Sample preparation and characterization

The coal sample used in this work was collected from No.11 seam at Xinzhouyao mine located at Shanxi, China. Analysis shows that carbon content of the sample is 56.67%, moisture is 1.56%, ash is 7.89% and volatile matter is 34.54%. The coal rank is bituminous. Vitrinite reflectance measurement shows $R_{o,\max}$ is 0.81%. Maceral analysis shows vitrinite group content is 75.4%, inertinite group is 20%, liptinite group is 1.8% and mineral matter is 2.8%. XRD analysis indicates that silica content is 0.6%, calcite is 1.9%, dolomite is 3.2%, siderite is 1.2%, amorphous substance is 90.8% and clay mineral is 2.3%.

Prior to synchrotron radiation nano-CT measurements, the coal sample was firstly pulverized down to the particle size less than 10 μm and then the particle was fixed on the tip of a pin. The pin was clipped in the sample turntable. A spherical gold particle with the diameter of 0.5 μm was mounted on the sample, which was used as the reference point for the image alignment (see Fig. 2(a)).

2.2. Experimental set-up

The synchrotron-based nano-CT employed in this study was designed and assembled at the Beijing Synchrotron Radiation Facility (BSRF), which is a first-generation synchrotron radiation facility that operates at 2.5 GeV. Nano-CT operates continuously from 5 to 12 keV with an achieved spatial resolution better than 30 nm [28]. In the case of the BSRF nano-CT, the beamline source size is 2.46 mm horizontally (H) × 0.84 mm vertically (V) (FWHM), and the beam divergence is 0.38 mrad (H) and 0.26 mrad (V) FWHM. The optical layout of beamline and nano-CT is shown in Fig. 1 [29].

As shown in Fig. 1, the synchrotron X-ray beam passing through slit 1 is collimated vertically by mirror 1 and then focused by the cylindrical bending mirror 2. A double-crystal monochromator with a pair of Si (1 1 1) crystals is set between the two mirrors, which tunes the X-ray energy in the range 5–12 keV. As a secondary source, slit 2 is set at the focus point of mirror 2. The Fresnel zone plate, installed after the sample position, serves as the objective imaging lens to produce a magnified image of the sample on the detector [29].

During imaging by nano-CT, the sample stage rotates discontinuously for well-defined angular intervals to obtain projection images at different angles. It takes approximately 30 s for the CCD camera to capture a single projection image and requires 1 s to transmit the data to the computer.

After the acquisition of raw dataset, alignment is vitally necessary because jittering of the rotation axis of the sample stage can provoke misalignments among the projection image series. These misalignments may cause rate blurring and streaking artifacts, which cause information loss and possibly faked structures in the reconstructed slices of the sample. The image alignment and 3D reconstruction were completed at the platform designed by National Synchrotron Radiation Laboratory and Chinese Academy of Sciences [28]. The first step of image alignment and 3D reconstruction is to determine the reference point (i.e. gold particle in Fig. 2(a)) location in different projection images. Two automatic methods can be used to locate the reference point in each raw projection image. One is the grey value barycenter (GVB) and the other is the circle fitting method (CFM) [28]. In some circumstances, neither GVB nor CFM generates a satisfactory alignment, manual correction module (MCM) is needed to align the raw images. In this study, manual correction module (MCM) was used. The image alignment is then carried out based on the coordinates of the reference point. Alignments are conducted in horizontal and vertical direction respectively. After finishing the alignment of the raw nano-CT dataset, pixels from the image shift are clipped. The images aligned can be used for 3D reconstruction. The inverse radon transform function iradon of the MATLAB software is applied to finish 3D reconstruction. Fig. 2(a) shows the sample reconstruction results.