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Permeability evolution and mesoscopic cracking behaviors of liquid nitrogen cryogenic freeze fracturing in low permeable and heterogeneous coal

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ABSTRACT

Fracking to approach permeability enhancement is indispensable to enhance coalbed methane recovery, which can significantly reduce greenhouse gas emissions and produce substantial clean energy. To access efficient fracking, samples of Permian low permeable coal reservoir were cracked into different sizes or scales of blocks and particles with liquid nitrogen cryogenic freeze fracturing (FF) by maximizing the underlying heterogeneity of coal. To investigate the crack efficacy, this study systematically examined the permeability evolution and mesoscopic cracking behaviors of coal with different water contents and cleat–fracture systems in the context of cryogenic FF. Results showed that the permeability enhancement and microcracking tended to occur with increasing water content of coal sample; and the efficacy of cryogenic FF on the tighter coal sample was more remarkable. However, the permeability does not strictly increase with the cycles of cryogenic FF and has a close relationship with water content, structural plane direction, efficacy of cryogenic FF, and porosity compaction. The mesoscopic cracking behaviors indicate that numerous smaller pores are iteratively, not strictly sequentially, cracked and become connective in this process. The permeability evolution of coal sample is identified as significantly associated with the mesoscopic cracking behaviors. Notably, the first cycle of cryogenic FF acted on the detected several scales of pores and micro fissures, and partially caused these structures to be opened and interconnected to be permeable. Two main changes were observed in the microcracking of the coal samples in this study: 1) nonuniform shrinkage deformation and micro fissure expansion; and 2) pores opening mostly in the macropore and mesopore scale. It is reasoned that thermal cracking and intermittent opening of seepage pores due to the phase transition of free water in pores or micro fissures ultimately contribute to the permeability enhancement in low permeable and heterogeneous coal.

1. Introduction

Since the 1990s, cryogenic fracturing has been attracting worldwide attention as a waterless or water-reduced fracturing technique for revolutionized production of unconventional energy resources such as shale gas, coalbed methane, and geothermal reservoirs buried in the deep Earth's crust [1–6]. Cryogenic fracturing exploits thermal gradient and resulting local tensile stress to initiate fractures/cracks on a surface exposed to cryogenic fluids such as liquid CO_2/noble liquid nitrogen (LN_2) [7–8]; it minimizes or even eliminates the use of freshwater and chemical additives, especially reduces formation damage [9–11], and lowers breakdown pressure [12–13]. LN_2 can be implemented to cost-effectively stimulate unconventional reservoirs [14]. Other fracturing techniques that use liquefied petroleum gas and cold compressed natural gas [15–16] or high-voltage electrical pulses [17] require additional rigorous safety measures.

Pore size and connectivity are vital factors in gas desorption and seepage. Therefore, reservoir stimulation involves increasing microcracks and fragmentation to connect the primary fissures and ensure gas desorption and seepage channels opening at a certain fracture aperture. If the phase transition of free water is considered, cryogenic freeze fracturing (FF) using LN_2 may be viable for permeability enhancement of low permeable and heterogeneous coal reservoirs. Coal mass fatigue and damage usually initiates at weak planes such as primary fissures and microcracks. However, just a single main hydraulically formed crack or fracture initiates approximately perpendicular to the least principal stress [18],

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which cannot fully stimulate the reservoirs by maximizing the heterogeneity of coal seam. Alternatively, the effect of low temperature and phase transition of free water caused by LN_2 cryogenic FF can compensate for this underlying defect.

LN_2 cryogenic FF can be performed by injecting pressurized pure LN_2 after pumping water into the reservoirs for wetting (if water is indispensable). Thereafter, water infiltrates the discontinuities and pores and then freezes as the temperature sharply decreases to below approximately −77 K. After the commencement of freezing, the volume of infiltrated water significantly increases by 9.1% and generates a frost heave stress up to ~207 MPa [19], which is considerably higher than the fracture initiation stress of coal seam. With the passage of time, the pores will generate microcracking and will start becoming connective. Gasification fracturing is another favorable factor. At 294.15 K and atmospheric pressure, the volume of LN_2 increases 696 times with a latent heat of vaporization of 5.56 kJ/mol [19–20]. LN_2 undergoes gasification and produces tremendous gas pressure that the reservoirs cannot overcome. The LN_2 gasification fracturing process involves gas fracturing, thermal shock fracturing, rock embrittlement fracturing, and nitrogen expansion fracturing [21]. In addition, the dynamic viscosity of gaseous methane decreases at low temperature. As a result, the reservoir methane flows more easily [22] with nitrogen extrusion and flooding as the higher adsorbing gaseous or supercritical CO_2 displaces it [23–24]. It has been reported that the permeability of coal is enhanced by 48.89–93.55% after super-cooling of LN_2 [25]. Several similar studies have examined the permeability evolution of rock subjected to LN_2 cooling based on fast Lagrangian analysis of continua [26] and under true triaxial confining stresses [27]. Furthermore, permeable stratification and its orientation significantly influence the permeability of reservoirs [28–30].

However, these studies rarely considered anisotropy and/or water content; and the influence mechanism of the mesoscopic cracking behaviors on permeability evolution remain unclear in the context of LN_2 cryogenic FF. The mechanism of pore cracking has also not been adequately exploited. Several problems such as the influence of water content, anisotropy, freezing time, and cycles of cryogenic FF must be considered and resolved. Thus, this study aims to further understand the characteristics of permeability evolution and microscopic cracking of low permeable and heterogeneous coal to bridge the gap between fundamental research and engineering application of the technique of cryogenic FF. Based on this, the coal industry can evaluate the feasibility of cryogenic FF in similar coal and coalbed methane reservoirs for significantly reducing greenhouse gas emissions and contributing to much more natural gas production.

2. Materials and methods

2.1. Sample preparation and characterization

Coal samples were extracted from the Ji_{15}–24080 working face at a depth of 750–960 m in the No. 10 mine of Pingdingshan Tian'an Coal Industry Co., Ltd. located in the Pingdingshan coalfield of Henan Province. The sampled coal seam belongs to the Permian formation and has low permeability. First, the coal cores were drilled from the coal blocks, then cut and polished at dimensions of Φ 50 mm × 100 mm (for the tests of core flooding and uniaxial compressive strength) and Φ 50 mm × 25 mm (for the uniaxial tensile strength tests according to the Brazilian disk splitting method) with an end-surface nonparallelism of ≤0.02 mm. A batch of coal particles with dimensions of approximately 5–8 mm was also prepared for the test of pore size distribution based on high-pressure mercury intrusion and the test of heterogeneity based on Raman scattering. Then, these coal samples were dried/saturated (or without treatment) until the mass of the coal samples remained constant; i.e., the mass change was ≤0.002 g between twice adjacent weighing at a time interval of 24 h. Subsequently, the coal samples were wrapped with plastic clingfilm to maintain constant water content and prepared for cryogenic FF. The AG – 250 Shimadzu electronic precision material testing apparatus was used to load the samples at 0.002 mm/s for uniaxial/tensile compressive strength measurements. The average uniaxial and tensile compressive strength of the coal samples were at 8.02 MPa and 0.73 MPa, respectively.

The characteristic Raman spectra can be used to effectively characterize the size and degree of structure order of carbonaceous material to some extent. The characteristic Raman spectra of carbonaceous materials are usually of two orders; the first order mainly includes two bands, which are D1 peaks with a shift of ~1350 cm^{-1} indicating lattice defects of carbon atoms, and G peaks with a shift of ~1580 cm^{-1} indicating in-plane stretching vibration of carbon atoms sp^2 hybridization, i.e., order degree of carbon atoms lattice. The two bands represent the characteristic Raman spectra of carbon atomic crystals i.e. ideal graphitic lattice. Therefore, Raman spectra are related to organic matter maturity of coal samples, and was employed as a non-graphical technique to assess the heterogeneity characteristics of coal and coal rank (positively correlated with the degree of metamorphism) [31–33]. The characteristic Raman spectra of the fracture surface of the coal sample were measured with a laser confocal Raman spectrometer at room temperature with a reduced laser power of 5 mW to avoid sample heating and photoinduced damages. As shown in Fig. 1 (a), the D1 peaks of the coal sample was fairly dwarf with a larger full width at half maximum (FWHM) than that of the G peaks. Only the D1 peaks and the G peaks of the 5 peaks in the first order were distinct, while the other 3 subcomponents of the secondary order were difficult to distinguish in low rank coal [44], which indicates that the order degree of the carbon atom lattice structure is poor. In addition, the Raman photographs were captured by a high-speed, high-resolution, non-shaking scan of linear Raman scatter beam focused by a slit. The imperfections are very clear, as indicated by the dark gray texture shown in Fig. 1 (b). As a consequence, the degree of structural order of the coal samples tested in this study showed a strong heterogeneity and has a low degree of metamorphism. The maximum vitrinite reflectance (R^0_{max}) was measured in the range of 1.28–1.53%. The coal samples belong to fat coal with an in-situ water moisture of ~1.37%, ash content of ~18.86%, and volatile matter of ~23.27%.

2.2. Experimental apparatus and procedure for core flooding test

The apparent permeability of the coal samples was measured using a self-developed triaxial seepage experimental apparatus [34] as shown in Fig. 2 (a). According to Section 2.1, the prepared coal samples were evenly coated with TY-704 RTV–1 silicone rubber on the lateral surface and then tightly wrapped with a heat-shrinkable tube to prevent gas overflow and hydraulic oil infiltration. Gas for core flooding comprised 99.99% gaseous methane which was supplied by the high-pressure gas cylinder and controlled by the relief valve. After passing through the gas inlet of the upper steel pillar and the coal sample, gaseous methane aggregated and flowed out of the lower steel pillar with circular aperture and gas outlet. During the core flooding test, the gas pressure was kept constant at 2.23 MPa according to gas geological conditions of the No. 10 mine, and the minimum and maximum principal stress were loaded or unloaded at a rate of 0.05 MPa/s. The load was realized by the triaxial seepage equipment apparatus (Fig. 2 (a)) controlled by an invisible electro-hydraulic servo control system. The axial and radial deformation was collected by the axial displacement sensor of the upper steel pillar and radial extensometer submerged in hydraulic oil of the triaxial pressure chamber, respectively. All measurements including the gas pressure and flux, deformation, ambient temperature of the sample chamber, as well as stress change over time were automatically monitored and controlled in real time with a special software for data acquisition as shown in Fig. 2 (b).