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X-ray micro-tomography for investigation of meso-structural changes and crack evolution in Longmaxi formation shale during compressive deformation

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ABSTRACT

Shale is a typical heterogeneous geomaterial and investigation of the meso-structural changes and crack evolution is beneficial to shale gas development. Understanding the microscopic failure mechanism is undoubtedly crucial to hydraulic fracturing treatment. In this paper, real-time high-resolution X-ray Computed (micro-) Tomography (X-ray CT) with was used for the first time to deform shale sample experimentally under unconfined axial compression. Organic matter, pyrite, and micro-fractures were volumetrically rendered with a resolution of 11.27 μm, and 2D/3D image investigations enabled us to explore the structure and fracture evolution of the sample due to in-situ compression. This work reveals that the stress-induced deformation of shale is found to be dependent on organic matter compaction, pyrite spatial evolution, and micro-fracture initiation, propagation, and coalescence. The volume of organic matter decreases with the increase of axial stress; and the spatial distribution of pyrite changes at different loading stages, but the effective volume is almost the same. The formation of macroscopic fractures ∼20° oblique to the loading direction was observed. The cracks initiate in the tensile cracks between the bedding plane at the bottom of the sample and shear fractures are composed of tensile cracks connected by an "X" shape. The layered deposited structure and weak cementation between layers are the main factors controlling the failure mechanism.

1. Introduction

The rock mechanical properties of shale are important basic parameters in rock engineering, such as for slopes or mining, ground excavations, building foundations or for petroleum engineering (e.g., hydraulic fracturing, well infectivity improvement, borehole stability assessment, and the evaluation of stable mud weight windows for drilling) (Rybacki et al., 2015, 2016; Yuan et al., 2017, 2018; Yuan, 2018). Especially, in the oil-gas field, a good understanding of the shale mechanical properties is helpful to form fracture networks and enhance production rates (Rybacki et al., 2015; Zheng et al., 2016; Ren et al., 2016; Yuan et al., 2017a). Currently, many scholars have conducted a series of macroscopic laboratory compressive tests (Heng et al., 2014; Masri et al., 2014; Rybacki et al., 2015, 2016), tensile tests (Mokhtari et al., 2013, 2014), three-point bending tests (Heng et al., 2015a), direct shear tests (Heng et al., 2015b), and numerical tests (Mokhtari et al., 2013; Suarez-Rivera et al., 2013) on the shale outcrop and reservoir specimens. They have not only studied the macroscopic failure mechanism of shale under a static strain rate (Heng et al., 2014; Masri et al., 2014), but also under medium strain rate (Rybacki et al., 2015), high strain rate (Liu et al., 2015), cyclic loading and unloading (Wei et al., 2015), and high temperature (Islam and Skalle, 2013; Masri et al., 2014) conditions. The widely performed experiments show that the failure mode, strength, static elastic module, and ultrasonic velocities of these materials varies with the orientation of the specimen with respect to the principal stresses (e.g., Naumann et al., 2007; Kuila et al., 2011; Zhubayev et al., 2015; Wang et al., 2017), temperature (Islam and Skalle, 2013), and strain rate (Rybacki et al., 2015). Many works have also been done to investigate the time-dependent mechanical behaviors of shale through creep tests. By using multi-stage triaxial creep tests on several reservoir shales, Li and Ghassemi (2012) found that the pseudo-steady state creep rate increased linearly with deviator stress, the strain can be described by a power-law

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function of time. Sone and Zoback (2013) have performed a series of triaxial creep experiments, and they found that a strong correlation exists between the shale mineralogical compositions, the volume of clay plus kerogen, and the intact rock strength, frictional strength, and visco-plastic parameters. Rybacki et al. (2017) conducted triaxial creep experiments on Posidonia Shale at high pressure and temperature conditions, and found that the creep behavior was influenced by the bedding plane orientation, composition, and water content, the long-term creep properties of shale was correlated with composition-based brittleness. Liu et al. (2018) have conducted nano-dynamic mechanical analysis of creep behavior of shale, the results showed that the minerals with various mechanical properties strongly influenced the creep behavior.

Many efforts have been done to investigate the strength, deformation, and failure mode of Longmaxi shale under various macroscopic experiments. From the results of the direct shear test by Heng et al. (2015), the strength, cohesion and internal friction angle change for samples with various orientations. Liu et al. (2015) proposed a dynamic damage constitutive model for anisotropic shale material based on the statistical damage theory and the SHPB impact test at high strain rate. Wei et al. (2015) conducted experimental study on shale deformation under cyclic loading, and the results show that the loading deformation modulus and the unloading deformation modulus firstly increase and then monotonously decrease with the increase of the cyclic times. Wang and Li (2017) studied the P- and S- wave velocity anisotropy of a Longmaxi shale by real-time ultrasonic and mechanical experiment. They found that the P- and S-wave velocity decrease with increasing orientation and the pronounced bedding planes of shale govern the intrinsic P- and S-wave velocity anisotropy. By conducting tensile testing, the failure modes and tensile strength of shale are strongly affected by the bedding plane orientation and loading rate, the tensile strength is always a maximum when the loading direction is perpendicular to the bedding plane (Wang et al., 2017). There are few reports about the internal fracture evolution of shale under compressive deformation. Reports about visualizing and digitizing the fracture evolution using X-ray computed tomography (CT) are rarely published, but the meso-structural changes and crack evolution behaviors of shale is critical to shale gas development. Hence, the fracture behavior prediction and establishment of effective mechanical modeling for gas shale under in-situ deformation are problematic.

To explore the evolution characteristics of the internal damage and failure pattern of rocks during deformation, macroscopic mechanical testing are usually effective (Zhou et al., 2008). By using normalized strain gauge measurements (axial strain, lateral strain, or volumetric strain), it provides us with a relatively simple mean to obtain the macroscopic deformation characteristics, while the micro-cracks initiate and propagate for rock samples. During sample deformation, we can obtain a lot of fracture information such as plastic strain, crack growth and nucleation, and crack coalescence of brittle rock material. The failure morphology of shale can be observed after the experiments. However, it is difficult to throw deep insight about the micro-meso failure mechanism and micro-fracture development from the direct strain measurement using macroscopic mechanical experiments. The micro-fractures in rock samples during deformation are in the form of a three-dimensional distribution, yet most microscopic approaches on rock deformation have been restricted to the investigation of thin sections using a scanning electron microscope (SEM) (Janssen et al., 2001; Moore and Lockner, 1995) with subsequent explanation of the 2D results to get a 3D image. An alternative technique to study the deformation and failure behaviors of shale is the acoustic emissions (AE) monitoring experiments, which can provide a direct measurement of fracture events in brittle rock, including pore closure, grain boundary movements, crack initiation, propagation and coalescence (Holcomb et al., 1990; Lei et al., 2000). Moreover, the AE actives data were analyzed to classify the crack modes (i.e., shear crack, tension crack). This technique is able to reflect the initiation, propagation, and coalescence nucleation of cracks in the form of 3D images as well as the crack geometry morphology (e.g., width, length, area) inside the sample (Lei et al., 2000; Reches and Lockner, 1994; Zabler et al., 2008), but it cannot visualize and quantitatively obtain the fracture characterization of rock specimens.

X-ray CT is a non-destructive technique with the ability of imaging detailed material information down to the micrometer and even to the nanometer. More than that, we can clearly observe the structural and composition evolution of a rock sample by a series of 2D/3D images during in-situ loading to determine where and how cracks initiate, propagate, and coalesce. For shale material, due to the lack of effective and non-destructive approaches that can resolve down to the micrometer or nanometer, it is difficult for us to describe the pores, micro-flaws, and crack evolution behaviors. Because of the strong anisotropy and heterogeneous characteristics of shale, analytical and experimental characterization of the deformation and failure characteristics are very irreproducible. Therefore, the mechanical behavior of shale under loading is strongly affected by the nonuniform distribution of natural fractures, bedding plane, grain boundaries, fossils, inclusions, mineral cleavage planes and micro-cracks. To better understand the fracture process from millimeters to kilometers, including crack initiation, propagation and coalescence, the fracture behaviors of shale on micrometer scale should be studied.

As X-ray CT techniques became a feasible and effective method in laboratory investigations, plenty of studies on the 2D/3D pore characterization (McCoy et al., 2006; Polacci et al., 2010), 2D/3D grain analysis (Masad et al., 2005; Dewanckele et al., 2009; Cnudde et al., 2011), fracture analysis (Keller, 1998; Ketcham et al., 2010), multi-scale imaging (Wildenschild and Sheppard, 2013), ore analysis (Kyle et al., 2008), monitoring structural dynamic processes (Raynaud et al., 2010) and fluid flow analysis (Ketcham and Iturrino, 2005) have been performed. The studied material includes not only natural rock (Ohtani et al., 2001; Hirono et al., 2003), but also "rock-like" materials (Otani and Obara, 2003) over the last ten years. Tth the increase of the number of investigations and increase in the resolution of the X-ray CT (Desrues et al., 2006) has shown powerful functions in the extraction of hierarchical size of the voids and minerals among many orders of magnitude, e.g., from 0.5 mm to 0.5 m. Based on the basic principle of cold and/or thermal neutron sources, the neutron tomography and radiography methods are widely used to analyze the 3D imaging of micro-cracks as well as mineral phases. However, it is not capable of imaging the very small voids due to the restraint of spatial resolution of neutron tomography (100 μm) (Fredrich et al, 1995). As a result, even though the typical wavelength of the X-ray from 0.5 nm to 0.02 nm, it is difficult to image the crack and mineral distribution in the rock with details on the micrometer level due to the limitation of hard X-ray beamlines. Studies among those imaging experiments, with a resolution ranging from 10 to 20 μm are still rarely reported (Nakashima et al., 2004; de Argandoña et al., 2009). Zabler et al. (2008) have used high-resolution X-ray CT with a spatial resolution of ~10 μm for greywacke and limestone samples; however, the studied rock samples have relatively large mineral grains compared to shale materials.

In this work, a novel setup with a maximum spatial resolution of 700 nm and the capability of imaging micro-structures, voids, and cracks in the 5–10 mm sized samples was used for characterizing the in-situ deformation of the shale sample. A type of well characterized Longmaxi black shale, which is a typical heterogeneous sedimentary rock, is selected to study the fracture evolution under deformation. The results of this study, by comparing the 2D/3D CT images, organic matter, pyrite, and micro-fracture distribution during the deformation process, determined the microscopic fracture characteristics of shale material.

2. Tested materials

Samples were prepared from Longmaxi Formation in the Lower Silurian located in Shizhu County Chongqing, China. It is a natural extension of the Longmaxi formation in shale gas block of Pengshui play. The trend of the formation is 327°, and dip is about 36°. Two sets of natural fractures can be observed from the outcrop. The geographical