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Three-point bending test investigation of the fracture behavior of siltstone after thermal treatment

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ABSTRACT

The influence of temperature on the fracture behavior of siltstone is investigated in detail by mode I fracture toughness tests under three-point bending in situ SEM observations. A total of 27 specimens subjected to thermal pre-treatment have been tested. Experimental results indicate that effects of temperature on siltstone fracture behavior are obvious, not only on failure mechanism, but also on mechanical parameters like peak failure loads, fracture toughness and modulus of elasticity. The failure mechanism changes from intergranular fracture to mixed intergranular and transgranular fractures, and finally to intergranular fracture and thermal cracking with temperature from 25 to 60 °C. Fracture toughness K_{IC} decreases slightly from room temperature 25 to 100 °C, and then increases significantly from 100 to 125 °C, and then gradually declines from 125 to 600 °C. A new numerical elastic modulus estimation method is proposed, considering a series of fluctuated experimental data. The variation of the elastic modulus with the temperature is similar with that of fracture toughness.

1. Introduction

It is necessary to evaluate quantitatively the safety and stability of rock engineering based on the physical and mechanical parameter of rocks. Rock fracture toughness expresses the rock's resistance to crack propagation or fracture energy consumption rate required to create new surfaces. Thermally assisted liberation, which induces the failure of tunnels from deep mining and nuclear waste disposition, and the collapse of the cores from oil engineering and geothermal engineering, has been suggested as a possible mechanism to reduce fracture energy. Axial compression tests have been widely applied to determine the effect of thermal pretreatment on intrinsic strength parameters of rock materials. Some studies have clearly indicated that the strength of major rocks at macro-scale decreases with the increasing temperature. In addition, the decreasing trend is related to rock types, humidity, specimen thickness and size, and experimental confining pressure [1–3]. Numerous investigations have mainly discussed the effects of temperature on rock deformation and failure at macro-scale. Although thermally assisted liberation has been suggested to reduce fracture energy, the mechanism of how to reduce is not fully investigated. For example, more attention should be paid to temperature effects on the failure mechanism and fracture toughness of cracks of rock at micro/meso-scale.

Investigation of fracture toughness of rocklike materials is a complicated problem due to their heterogeneous nature. Many experimental methods have been developed to test fracture toughness of geomaterials, such as a Straight Edge Cracked Round Bar Bend (SECRBB) method [4], Chevron Bend (CB) test [5], Semi-Circular Bending (SCB) test [6], and a Chevron Notched Semi-Circular Bending method [7]. Due to the easiness of specimens' preparation and simplicity of testing configurations three-point bending (TPB) and related testing techniques are attractive for K_{IC} determination. Mode I fracture toughness of semicircular specimen of water-saturated synthetic mudstone through TPB has been investigated in [6]. And they conclude that TPB technique provided reliable results that are comparable with more established Mode I fracture testing methods. Experimental results of Kimachi siltstone indicate that fracture toughness is influenced by different temperatures through a single edge-notched round bar in bending (SENRBB) [8]. The fatigue behavior of marble under three/four-point bending has been investigated in [9]. Static tests on smooth and notched specimens are carried out to determine the tensile strength and fracture toughness of Carrara marble. Recently, a notched disk bending method has been developed to determinate model I fracture toughness of disk specimens of andesite and marble subjected to TPB loads [10]. Experimental results are compared to those well-known testing methods on K_{IC} which prove their methods effectively [4–7,11]. Some corresponding

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numerical simulation methods have also been developed to study brittle materials failure behavior [12–15]. SEM is a type of electron microscope that produces digital images of a sample by scanning it with a focused beam of electrons, which has been widely applied in the evaluation of failure mechanism and fractography of materials. However, most of SEM systems can only observe failure behavior of materials after the destruction. The whole failure process or in situ failure with loading is difficult to be obtained. in situ observation of material failure process with real-time loading is of great significance for us to understand the deformation and failure of the materials. Although great progresses for evaluation of the fracture toughness of rocklike materials have been obtained, in situ SEM investigation on micro/meso failure mechanisms and fracture toughness subjected to thermal effects for rock materials have rarely been reported.

Recent studies indicate that, sandstone failure behavior at micro/meso-scale may be greatly different from those results at macro-scale, especially subjected to thermal effects [16–18]. Micro/Meso failure mechanisms have been reported to influence the failure behavior of thermal–mechanical coupled effects. For example, the effects of temperature on siltstone fracture toughness at meso-scale have been reported through uniaxial tensile test [17]. And we have noticed that the effects of temperature can be adequately exhibited at micro/meso-scale. In this paper, to understand the meso failure mechanism of rock after thermal effects, in situ SEM experiments have been employed to study the failure mechanism and fracture toughness of Pingdingshan siltstone with the single edge notched specimens through TPB tests.

2. Specimen size and experimental procedure

Test specimens used here are Pingdingshan siltstones. X-ray diffractometry (XRD) analysis revealed that Pingdingshan siltstone mainly consists of quartz, potassium, feldspar, calcite, dolomite, siderite and other clays. Their mineral compositions are as follows: quartz 54.7%, potassium feldspar 17.0%, calcite 2.0%, dolomite 3.6%, siderite 3.1%, and argillaceous clay minerals 19.6%. The physical and mechanical behaviors of siltstone are mainly determined by three compositions.

Tests were carried out mainly using three-point bending Single Edge-Notched (SEN) specimen. All specimens are obtained from the same large rock block, and prepared in the same direction, the purpose of which is to avoid or minimize the errors generated due to sampling as much as possible. The geometry of the specimen is 25 mm × 10 mm × 5 mm ($L \times W \times B$) according to the requirement of the loading system and fracture test's criteria of SEM, the middle effective span $l=20$ mm, as shown in Fig. 1.

Although V-notched specimens have been widely used in the tests of fracture toughness for metal materials, V-shape notch is very difficult to fabricate for rock materials. Most of mineral particle diameter of siltstone mainly ranges between 0.06–0.30 mm and even more. Therefore, it is very hard to obtain the strict V-shape notch specimen because big mineral particles make the crack tip to be a U-shape notch. Therefore, we processed the specimen with a U-shape notch in this work. The specimens were strictly made according to the requirement of ISRM's suggested methods [11]. According to the standards of TPB fracture mechanism test [19], the ratio of the length of crack to specimen height is designed as 0.5. Though high quality SEM observations were carefully carried out on siltstone, no significant indication of FPZ was observed. This might be due to the relatively smaller grain size of siltstone structure as compared to concrete or metallic materials. Therefore, ratio of 0.5 can be considered as acceptable.

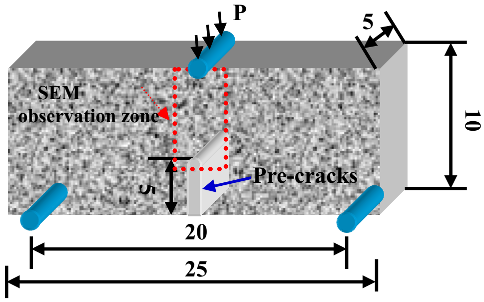

Fig. 1. Specimen size of three-point bend test (Unit: mm). (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

It is also convenient for in situ observation of the crack propagation and fracture behavior of siltstone. The length and width of pre-crack are about 5 mm and 0.4 mm, respectively, which are elaborately processed in the middle of the specimen. As a result of processing errors, the crack's length is slightly different. Pre-crack lengths of all specimens are listed in Table 1. The errors of specimen height, specimen width and pre-cracks length are less than 0.6%, 3.6% and 11.6%, respectively. The height and width of specimens basically have no error. The length of pre-cracks has slight error. Considering the effects of mineral particle sizes, we can accept the processing error of siltstones in our research works. The observing surface of specimens is polished in order to observe the evolution of micro-structure and the propagation of cracks.

The TPB tests are carried out by meso-scale test system with SEM in State Key Laboratory of Coal Resources and Safe Mining, China University of Mining and Technology (Beijing). The system can be used to observe the deformation and damage evolution of micro-structure in the surface of materials in situ under different load conditions. The siltstone specimens have been heat-treated in advance. The rate of heating is strictly controlled at 3 °C/min. After the designed temperature is achieved, it is kept for 1 h. Test temperatures are room temperature (25°C), 50, 100, 125, 150, 175, 200, 300, 400, 500 and 600°C, respectively. Cool the stove after the heat treatment using rate of 3°C/min, then put the specimen into the TPB clamps of SEM experimental system for testing. First, put the specimen into the specimen clamp of TPB, and then adjust the position of the specimen to make the pre-cracking in the middle of the specimen coincide with the centerline of the loading cylinder. In order to reduce the influence of load clamps to experiments, the loading head should be a columned head with large rigidity and high smoothness, which itself contacts the specimen linearly in order to guarantee the accuracy of the TPB test. After adjusting the location of the specimen, a pre-load of about 3–5 N is applied to fix the specimen at first, and then push the loading table to the scanning electron microscope room. The loading test can begin after the vacuum in the electron microscope room is pumped. Because the rock usually shows characteristics of brittleness, we use the displacement mode in the loading process. The loading rate is 10^{-4} mm/s which is the minimum of the test equipment. Because of the existing pre-crack, it causes the stress surrounding the crack to be amplified where the magnification is dependent upon the orientation and geometry of the crack and heterogeneous of siltstone. Therefore, the in situ observation zone can focus on the pre-crack tip, as shown in the red dashed box in Fig. 1. In this way, the in situ initiation and propagation of pre-crack tip can be clearly and completely observed through SEM.

3. in situ observation on fracture mechanism and whole failure process of siltstone

Although there are many literatures discussing the failure mechanism and crack propagation of rocks, the majority are