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An experimental study of the mechanical properties of granite after high temperature exposure based on mineral characteristics

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ABSTRACT

The macroscopic mechanical properties of granite collected from the Fujian Province, China were measured using uniaxial compression and three-point bending after the granite was exposed to high temperatures. The stress-strain relationship was measured and mechanical properties including the Young's modulus, uniaxial compressive strength (UCS) and fracture toughness were calculated. In general, the uniaxial compressive strength (UCS), Young's modulus and fracture toughness of the granite specimens decrease with an increase in heat treatment temperature up to 800 °C, above which there is no obvious change. The microstructure of the failed specimens was explored using scanning electron microscopy (SEM) and stereomicroscopy. After heat treatment, the minerals within the granite were analysed using X-ray diffraction. The failure mechanisms and the mechanical characteristics of the granite are explained in terms of the microstructure and the minerals present. The results of the experiments indicate that the crystal structure of the phlogopite in granite transforms to a more stable structure between 400 °C and 600 °C with an associated increase in volume. A dehydroxylation reaction also occurs in the phlogopite at 400 °C, after which the uniaxial compressive strength (UCS) decreases and the ductility increases. Using SEM and stereomicroscopy, it was observed that the connection between the minerals within the granite becomes weaker with increasing heat treatment temperature. Intercrystalline cracking is the main failure mode for samples exposed to temperatures below 800 °C, whereas transcrystalline cracking can be observed in samples exposed to 1000 °C.

1. Introduction

An understanding of the mechanical properties of rocks after exposure to fire is essential for mine engineering, oil exploration, nuclear waste disposal and restoration of underground construction. Deterioration of crystalline rocks due to temperature variation is one of the most important issues in this field, as thermal attack induces new microcracks and mineralogical changes in the material. In addition, microstructural changes, especially microcracks development in the rock, affect its mechanical properties (Takarli et al., 2008).

The physical and mechanical properties of rocks after heat treatment have been investigated extensively. Bauer and Handin (1983) proposed that thermal expansion has a significant effect on thermal conductivity, permeability and strength. Since then, a great number of experiments on selected rocks from all over the world have been performed to study the effect of high temperature on the various physical and mechanical properties including density, mass, P-wave velocity, porosity, elastic modulus, compressive strength, and peak strain (Liang et al., 2006; Rao et al., 2008; Keshavarz et al., 2010; Sygała et al., 2013; Chen et al., 2014; Zhang et al., 2015; Roy and Singh, 2016; Zhang et al., 2016).

Apart from the general mechanical properties mentioned above, Zuo et al. (2012) analysed the meso-scale tensile properties of sandstone from Pingding Mountain and found that the tensile strength of the sandstone increased between 20 °C and 150 °C. Chen et al. (2012) conducted fatigue tests with stepping amplitude on granite specimens by using ZWICK-100HFP5100 test apparatus and derived the fatigue life. In addition, the effect of temperature variation on the fracture characteristics and fracture toughness of rocks have been studied by laboratory tests (Chen and Zhang, 2004; Nasseri et al., 2007; Zuo et al., 2014; Mahanta et al., 2016; Aliha et al., 2016). Some studies have also involved the in-situ measurement of fracture toughness at elevated temperatures (Meredith and Atkinson, 1985; Balme et al., 2004; Funatsu et al., 2004).

Mineral composition, microstructure, and phase changes have been found mainly responsible for the decrease in mechanical properties of rocks under thermal attack (Heard, 1980; Rao et al., 2007; Sun et al., 2013; Cao et al. 2015; Liu et al., 2016; Zhao, 2016). Limestone, marble and granite were found to have a poor resistance to thermal cycling,

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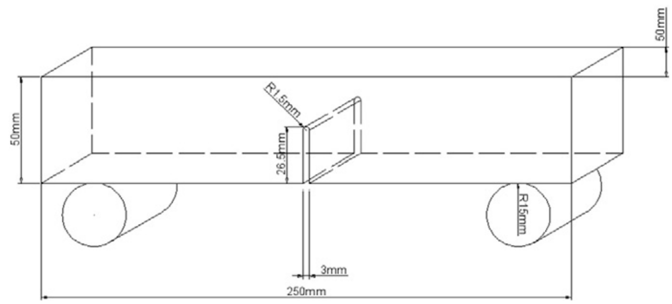

Fig. 1. Geometry of three-point bending test specimens.

while rhyolite and quarzitic sandstone showed excellent stability after thermal cycling (Tiskatine et al., 2016). Zhu et al. (2006), Sun et al. (2013), and Xi (1994) analysed mineral changes in different rocks after high temperature exposure and their results showed that quartz was the main mineral which influenced the mechanical properties.

So far, most studies focused on one particular thermal effect or some effects on rock performances with only limited studies concerning the connections between macro-mechanical properties as uniaxial compressive strength (UCS), stress-strain behaviour, Young's modulus, fracture toughness and fundamental physico-chemical mechanisms at a micro-scale. In this research, granite specimens were subjected to heat treatment at temperatures between 20 °C and 1000 °C followed by uniaxial compression and three-point bending test. The change in the mineral content of the granite after high temperature exposure was measured using X-ray diffraction and the effect on the mechanical properties was discussed. The microstructure of the granite after uniaxial compression was observed using scanning electron microscopy (SEM) and stereomicroscopy. The effect of the microstructure on the mechanical properties was discussed accordingly.

2. Experimental tests

2.1. Specimen preparation

The granite used in this research was collected from an outcrop at the depth of 2 m in Nan'an City, Fujian Province, China. The diameter and the height of the specimens used for uniaxial compression were 50 mm and 100 mm respectively, in line with the recommendations of the International Society for Rock Mechanics (ISRM). The geometry of the specimens used for three-point bending test is shown in Fig. 1. Single Edge-Notched (SEN) specimens were used with a length, height and thickness of 250 mm, 50 mm and 50 mm respectively. The precast crack was located midway between the clamps and had a depth of 26.5 mm.

Fig. 3. Surface appearances of specimens after heating.

2.2. Test equipment

An SMF1900-50 box-type furnace was used for heat treating the samples. The uniaxial compression was carried out using the SANS electro-hydraulic servo compression test machine manufactured by Xi-an LETRY Testing Machine Co. with a maximum load capacity of 2000 kN, shown in Fig. 2(a). The P-wave velocity and the Young's modulus of the granite specimens were measured using the V-METER III ultrasonic pulse velocity tester, which has an accuracy of 0.1 μs, shown in Fig. 2(b). The mineral content of the samples was determined using the X-ray diffractometer (Bruker/D8 Advance) shown in Fig. 2(c). The microstructure of the granite was examined using a VEGA3-SBH scanning electron microscope and the SteReo Discovery V8 stereomicroscope from Carl Zeiss shown in Fig. 2(d).

2.3. Test procedure

Uniaxial compression tests were conducted on 30 granite specimens which were divided into 6 groups based on the temperature of heat treatment. The Young's modulus and longitudinal wave velocity for each of the 30 granite specimens were measured before heating. The specimens were then heat treated at room temperature, 200, 400, 600, 800, and 1000 °C and atmospheric pressure. Samples were heated at a rate of 5 °C/min until the nominal temperature was reached. The temperature was then maintained for 6 h before the specimen was cooled naturally in the furnace. After cooling, the mass, longitudinal wave velocity and Young's modulus of each of the specimens were measured. Uniaxial compression tests were performed under load control at a rate of 0.5 kN/s until the point of failure. After uniaxial compression tests, the failure modes were determined and the mineral composition and microstructure of the samples were obtained using XRD analysis and SEM in order to understand the failure mechanisms under uniaxial compression.

Three-point bending tests were also carried out on 24 specimens in 6 groups prepared in the same way as the samples for uniaxial compression tests. Three-point bending test was carried out under load control at a rate of 50 N/s until the point of failure. The cracks and micro-cracks were observed by stereomicroscope and the fracture toughness K_{IC} was

(a) (b) (c) (d)

Fig. 2. Testing equipment: (a) SANS compression test machine, (b) V-METER III ultrasonic pulse velocity tester, (c) X-ray diffractometer, (d) SteReo Discovery V8 stereomicroscope.