

Thermal damage pattern and thresholds of granite

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Abstract High temperature may lead to the development of new microcracks or growth of pre-existing microcracks within granite, varying its physical and mechanical properties. Experiments were conducted to study the evolution of the physical and mechanical properties of granite specimens from room temperature to 800 °C. The specimens were heated in heating furnace and uniaxial compression tests were done using MTS servo-controlled testing machine. The results indicate five phases in the variation of physical and mechanical properties with temperature: from room temperature to 100, 100–300, 300–400, 400–600, and 600–800 °C. The first phase corresponds to the vaporization-escaping interval of adhered water, bound water, and structural water. Larger changes of physical and mechanical parameters in the temperature range of 300–600 °C, mostly 400–600 °C, are probably caused by the transition from the brittle state to plasticity (or ductility) of granite, and 400 °C may be a critical threshold of its thermal damage. These results confirm the important link among physical and mechanical properties in response to thermal treatment.

Keywords Thermal damage · Physical and mechanical properties · Micro-mechanism · Phase transformation · Critical threshold

Introduction

Thermally induced microcracks can significantly change the physical and mechanical properties of rock. Knowledge of the variation of these mechanical and physical properties with temperature is important to understand and model many processes in engineering projects, geological disasters, and geological structure formation, such as rock drilling (Nasseri et al. 2007, 2009), rock fragmentation, ore crushing, underground oil or gasification (Chen and Wang 1980; Chen et al. 1999; Liu et al. 2005), extraction of geothermal energy, deep petroleum boring, underground repositories of nuclear wastes (Zhang et al. 2001, 2008), protection of rock building or rocky cultural relics (Hajpal 2002), earthquake (Foulger 1995; Yang et al. 1997; Ramdani 1998), folding (Shimamoto and Hara 1976; Parish et al. 1976; Anderson and Bridwell 1980), geothermal activity (Mereer 1973), magmatic intrusions (Koide and Bhattacharji 1975; Knapp and Norton 1981), and plate tectonics (Pracht 1971; Heuze 1983; Björnsson 2008; Albaric et al. 2009; Craig et al. 2012).

Information about the evolution of physical and mechanical properties with temperature may be also used to analyze the thermal damage and identify the critical thresholds of rocks. Numerous studies have shown that some properties of granite are correlated to thermal damage, such as mechanical strength (e.g., compressive and tensile strength), Poisson's ratio, elastic modulus, porosity, acoustic velocities, permeability, wave velocity, fracture toughness, and fracture roughness (Bauer and

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Johnson 1979; Chen and Wang 1980; Géraud et al. 1992; Jones et al. 1997; Yang et al. 1997; Xu and Liu 2000; Liu et al. 2005; Dwivedi et al. 2008; Nasser et al. 2009; Xu et al. 2010; Xi et al. 2011; Lokajíček et al. 2012; Zhi et al. 2012; Chen et al. 2012; Yin 2012). Furthermore, exposure of fault planes to increased temperature has been found to reduce the friction coefficient (Stesky 1978; Lockner et al. 1986; Blanpied et al. 1998). Hence, the growth of cracks with temperature may lead to different level of thermal damage.

The important preoccupation of our study is to know the pattern of variation of physical or mechanical parameters with temperature in thermally cracked granites, and understand the underlying mechanism. In this paper, uniaxial compression tests were conducted on granites mined from Jining, Shandong, China. Before the tests, the granite specimens were heated to typical temperatures and then cooled, and then their stress–strain curves and peak stress under uniaxial compression, porosity, and P-wave velocity were measured and analyzed.

Experimental tests and results

Granite samples with average density of 2.76 g/cm^3 at room temperature were cut into $\varnothing 50 \times 100 \text{ mm}$ cylinders, which were then heated up to designated temperatures (25, 50, 100, 200, 300, 400, 500, 600, 700, and 800 °C) in a high temperature furnace (type MTS652.02). The heating rate was 30 °C/min , and each designated temperature was kept constant for about 2 h. The power was cut off and the specimen was allowed to cool naturally with the temperature of the furnace.

The mass, volume, porosity, and P-wave velocity of these specimens were tested before and after heating. The porosity was measured by a Microporous structure analyzer apparatus (type 9310) produced by Micromeritics equipment Co., Ltd., and P-wave velocity was collected by TICO test machine, at the same time with this action.

Uniaxial compression tests of these specimens were carried out on an electro-hydraulic servo-controlled testing machine (MTS815). These tests were strain controlled at the rate of 0.0015 mm/s .

X-ray diffraction (XRD) (type D/Max-3B) analysis showed that feldspar, illite, and pyroxene are the main components (Fig. 1), accompanied by a small amount of other minerals. However, the experimental results of Zhang et al. (2010) showed that the Luhui granite (mined from Linyi, Shandong, China) contains feldspar, quartz, illite, calcite, and siderite.

The porosity and stress–strain curves of granite specimens heated up to different temperatures are shown in

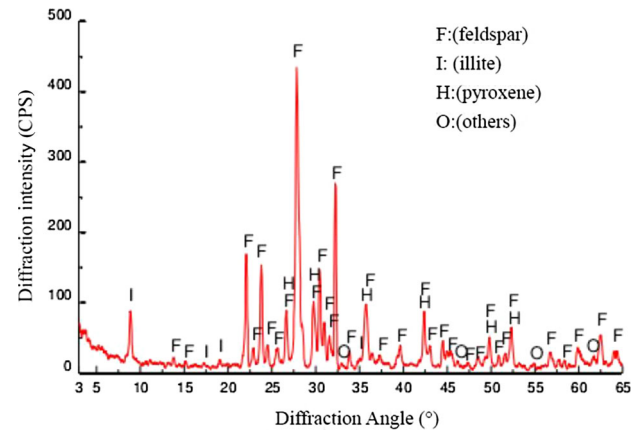


Fig. 1 XRD spectrum of granite sample (under 25°)

Table 1 Porosity under different temperature

Temperature °C	Porosity/%			
	Sample 1	Sample 2	Sample 3	Average value
25	0.97	0.96	0.72	0.88
50	0.73	0.81	0.70	0.75
100	0.87	0.68	0.66	0.74
200	0.91	1.06	1.05	1.01
300	1.04	1.11	1.37	1.17
500	1.49	1.37	1.82	1.56
800	2.62	2.42	2.68	2.57

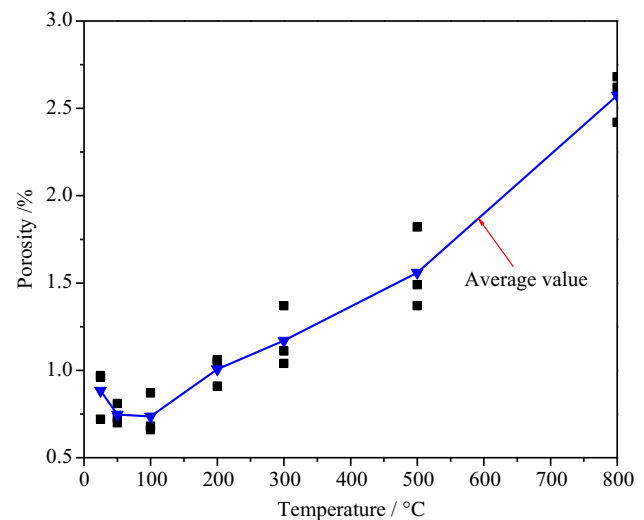


Fig. 2 Porosity of stress–strain of granite samples after different temperature

Table 1, Figs. 2 and 3. With the increase of temperature, especially above 400 °C, the mass loss continues to increase, as shown in Fig. 4.

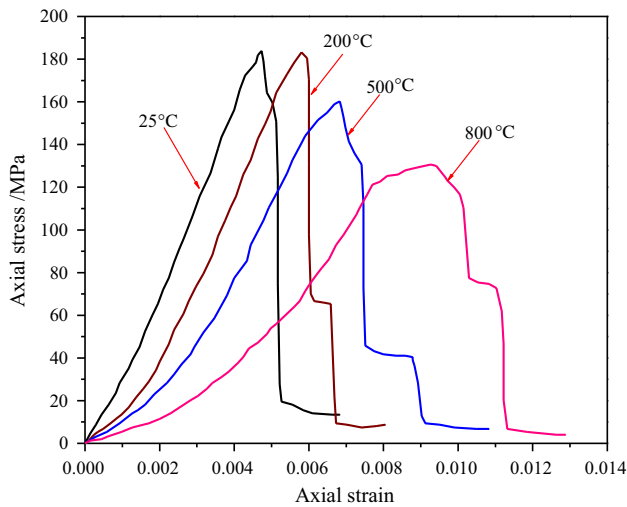


Fig. 3 Curves of stress–strain of granite samples after different temperature

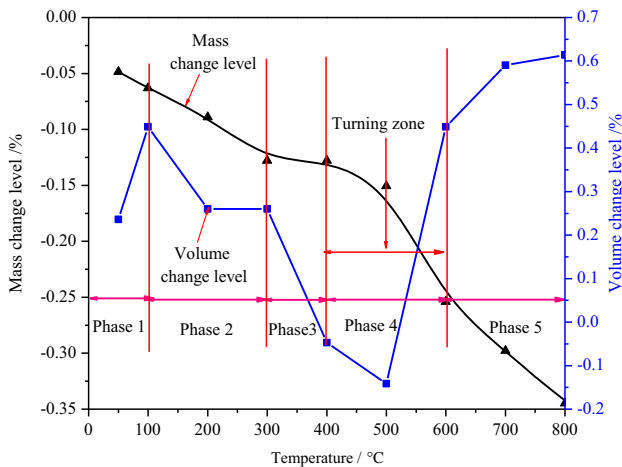


Fig. 4 Relationship between mass, volume, and temperature of granite

The results of uniaxial compression tests are shown in Figs. 3 and 5. Generally, the peak stress decreases and the peak strain increases as the temperature increases. Figure 3 demonstrates the variation of stress–strain curves with heating temperature. In each stress–strain curve of heated granite, four stages can be identified: (1) the compaction stage, when microcracks are folded by external loads; (2) the elastic stable cracking stage with continuous compaction at the beginning and micro-fractures developed later; (3) the yielding stage, when the stress reaches the maximum value at the end; (4) the softening stage, when the stress–strain curve declines steeply and the rock specimen fractures rapidly. Figure 5 shows that when the heating temperature is below 400 °C, the effect of temperature on the peak stress and elastic modulus of the specimen is relatively small; when the heating temperature

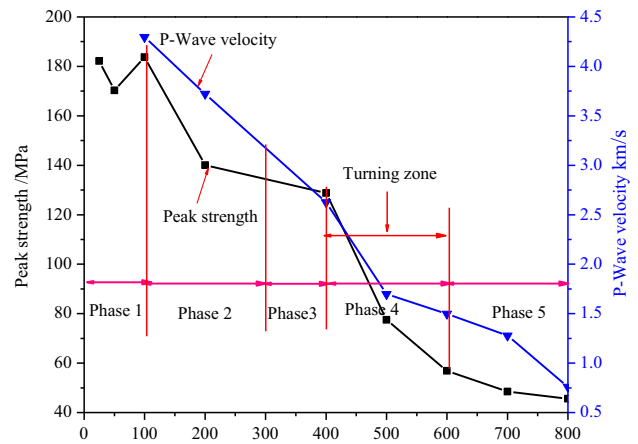


Fig. 5 Relationship between strength, P-wave velocity, and temperature

exceeds 400 °C, the peak stress and elastic modulus of the specimen decreases significantly with the increase of heating temperature.

Analysis and discussion

This decrease in the strength of heated granite is caused by the variation of internal structure induced by heat. Since granite is composed of mineral particles with different thermal expansion coefficients and thermo–elastic characteristics, high temperature may lead to inhomogeneous thermal expansion of mineral particles or phase transition of some mineralogical components, generating internal stress and microcracks in granite.

With the increase of temperature, internal defects would grow and change the physical and mechanical properties of granite. Moreover, in the process of heating, the water inside granite changes its existing form, i.e., the absorbed water, bounded water, and mineral water (e.g., crystal water, structural water, or zeolite water) would escape from granite under different temperature. It is known that the absorbed water would escape around 100 °C; the bounded water escape between 100 and 300 °C; crystal water would escape below 400 °C; and structural water of mineral would escape above 300 °C (Sun et al. 2013). The loss of crystal water and structural water leads to the damage of mineral crystal lattice skeleton, increasing the defects of granite.

Accordingly, variation of the measured compressive strength, P-wave velocity, and mass quality with temperature may be divided into five phases (as shown in Figs. 4, 5):

1. Room temperature –100 °C. In this phase, the absorbed water would be lost and the mineral grains of

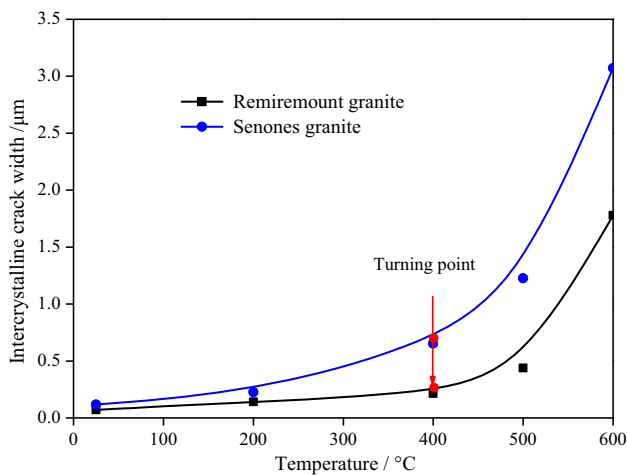


Fig. 6 Intercrystalline crack widths variations of Senones and Remiremont granite as a function of the temperature (Etienne and Poupert 1989)

granite be expanded, so porosity and P-wave velocity decrease slightly.

- 100–300 °C. In this phase, bounded water and crystal water escape, porosity increases slightly, and P-wave velocity decreases slightly.
- 300–400 °C. In this phase, the crystal water and structural water of mineral escape, so porosity increases significantly and P-wave velocity decreases. A review of previous work on thermally treated granites found that the permeability has increased significantly (as shown in Fig. 6, Etienne and Poupert 1989), which was considered to be caused by increased defects and connectivity due to the loss of crystal and structural water. Moreover, in this phase, the water reaches its critical temperature (i.e., 374 °C), at which the water may turn into supercritical fluid, causing internal stress in the granite and increased thermal damage. The existing form of water around the critical temperature has significant influence on the solubility, physical, and chemical properties of granite minerals. The test of Zhang et al. (2008) showed fluctuation in the dissolution rates when passing the critical state of water, i.e., over the temperature range of 300–400 °C. Experiments show that the maximum release rate of Si is released at 300 °C. Variation of the water properties and kinetic behavior of water–rock interactions also affect other features of granite mineral, such as the release of Silica, and breaking of silicate framework of minerals.
- 400–600 °C. In this phase, the physical and chemical features of granite minerals would change (shown in Figs. 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18). Between 400 and 600 °C, especially 500 and 600 °C, the minerals (such as ankerite, siderite, magnetite,

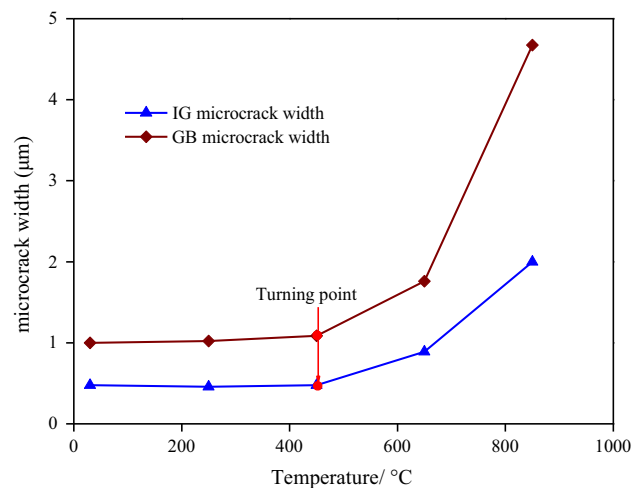


Fig. 7 Evolution of intra-granular (IG) and grain boundary microcrack (GB) widths as a function of the temperature for Westerly granite (Nasseri et al. 2007)

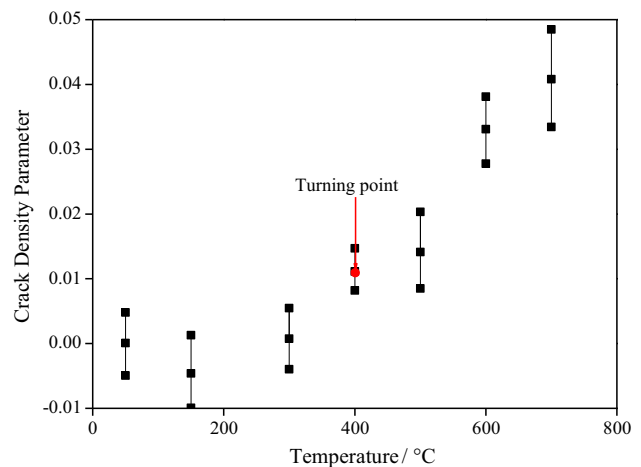


Fig. 8 Change in crack density parameter calculated from the compressional and shear wave velocities (Jones et al. 1997)

pyrrhotite, pyrite, illite, and kaolinite) of granite have chemical changes (Jana and Agnes 2012). At roughly 573 °C and under atmospheric conditions, quartz has a phase transformation from α phase to β phase, which can be used to explain the large variation of mechanical and physical properties.

- Above 600 °C. Under the influenced of solid mineral inflation and fracture of metallic bonding (such as Al–O, K–O, Na–O, and Ca–O), the strength and wave velocity continue to reduce, and the permeability and porosity continue to increase. In this phase, part of the minerals would melt, leading to enlarged defects.

In the first three phases, the variations of compressive and tensile strength are different from what was found in previous studies. Uniaxial compressive strength and elastic

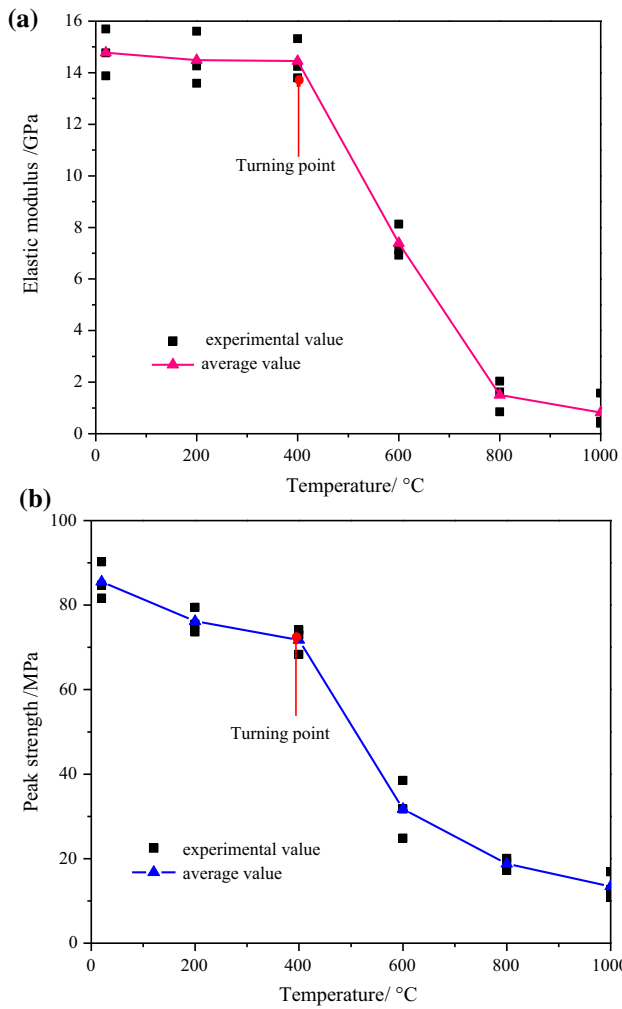


Fig. 9 Relationship between strength, elastic modulus, and temperature (Chen et al. 2012). **a** Elastic modulus against the temperatures. **b** Peak strength against the temperatures

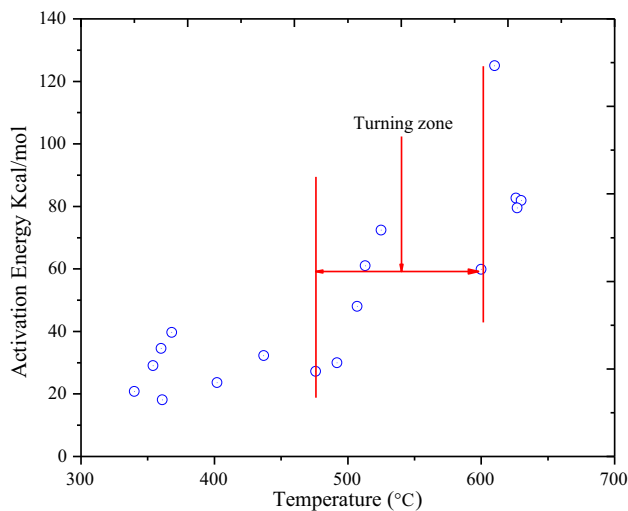


Fig. 10 Experimental activation energy at different temperature for sliding at 2.5×10^5 kPa pressure (Stesky 1978)

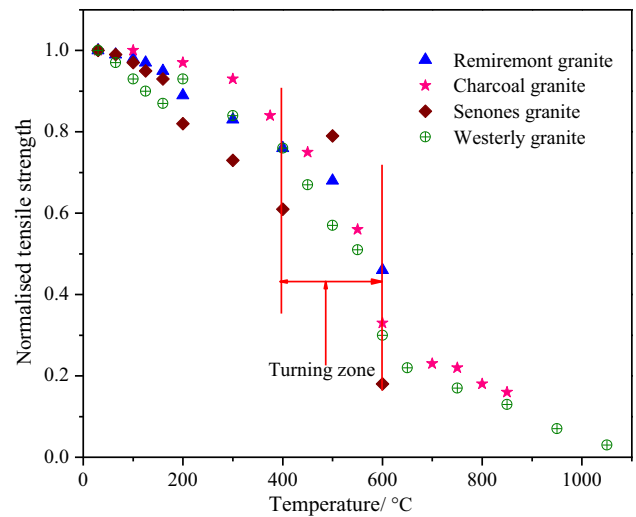


Fig. 11 Normalized tensile strength vs. temperature at atmospheric pressure (Dwivedi et al. 2008)

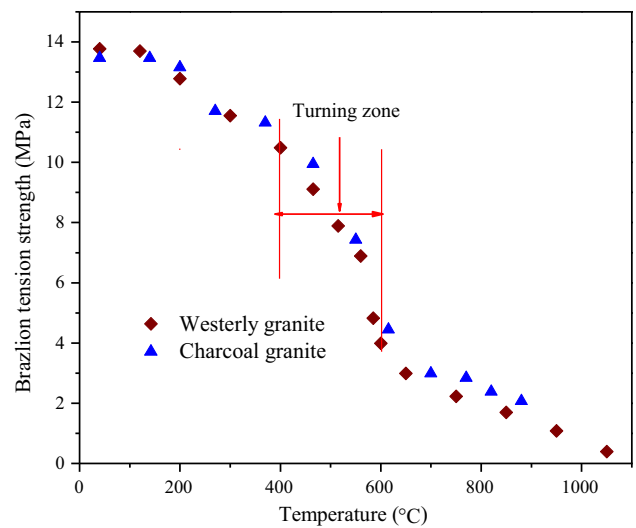


Fig. 12 Tensile strength of granites vs. temperature (Heuze 1983)

modulus decrease with temperature until 400 °C, while porosity, permeability, and AE significantly increase (Bauer and Johnson 1979; Etienne and Poupert 1989; Géraud 1994; Du et al. 2004; Chaki et al. 2008).

However, interestingly, numerous studies have shown that when the temperature is over 400 °C, some physical and mechanical properties of granite, such as density of microcracks, porosity, permeability, wave velocity, fracture roughness, fracture toughness, and peak strength (as shown in Figs. 5, 16), would change significantly, consistent with our experimental result. Bauer and Johnson (1979) pointed out that although microcracking starts in granite at about 80–120 °C, most of the mineral grains are microcracked at about 400 °C. Chaki et al. (2008) observed a small increase in porosity between 105 and 500 °C, and a

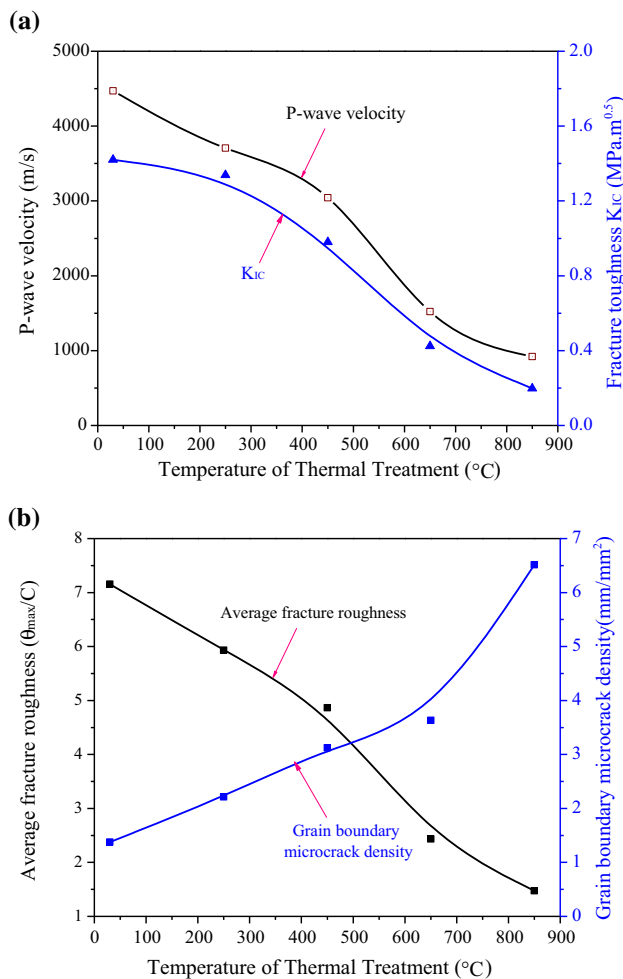


Fig. 13 Variation of the average fracture roughness, fracture toughness, P-wave velocity, and grain boundary microcrack density as a function of the temperature of thermal treatment (Nasseri et al. 2009). **a** Variation of the average fracture toughness and P-wave velocity. **b** Variation of the average fracture roughness and grain boundary microcrack density

significant increase between 500 and 600 °C (anisotropic expansion linked to the α/β quartz transition which occurs at 573 °C). Géraud et al. (1992) showed that the porosity of granite increases above 300 °C. The largest increase of surface roughness occurred between 450 and 600 °C, corresponding to the largest decreases in both V_p and K_{IC} .

Bauer and Johnson (1979) observed that the strength of granite heated up to 400 °C was slightly lower than that of the granite at room temperature. Etienne and Poupert (1989) pointed out that the Remiremont granite showed slightly higher strength at 200 °C, but the Sennones granite showed a larger increase in strength at 400 °C. The increase of strength at 400 °C was also observed by Alm et al. (1985), although Rao and Murthy (2001) showed that the strength of granite at 400 °C and room temperature is comparable.

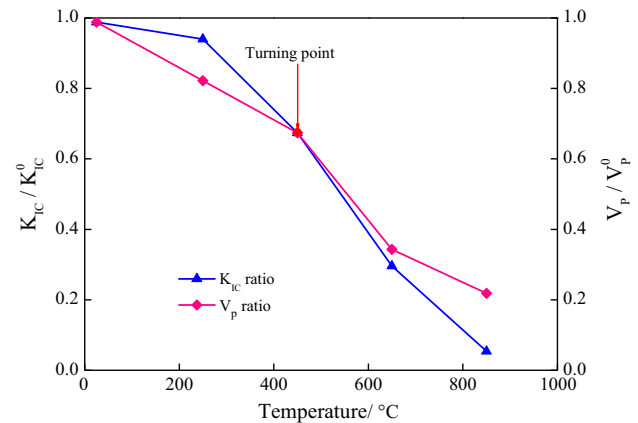


Fig. 14 A comparison of the fracture toughness (K_{IC}) and the mean P-wave velocity as a function of temperature treatment. Both K_{IC} and the V_p were normalized to that of the untreated specimens (K_{IC}^0 and V_p^0) (Nasseri et al. 2007)

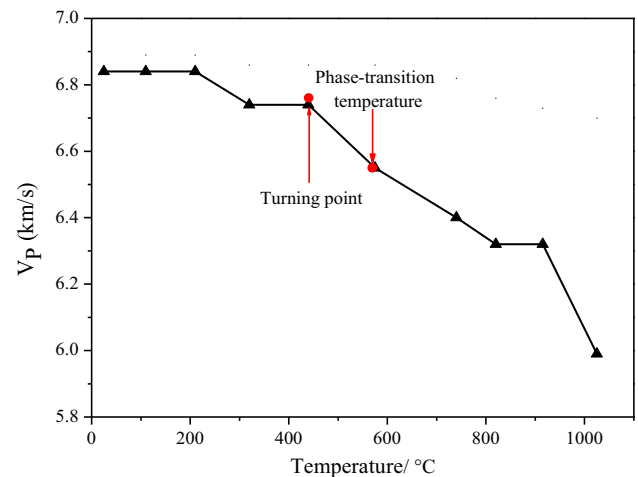


Fig. 15 Evolution of P-wave velocity vs. heat treatment temperature of Huanan granite (type I) under 500 MPa (Yang et al. 1997)

The mechanical behavior of rocks essentially depends upon their mineralogy, structure, temperature, and stress history (Etienne and Poupert 1989). Granite is polycrystalline containing minerals with different thermal expansion coefficients, so inter-granular compressive and tensile forces are generated under heating. When these forces exceed the local strength, microcracks are generated. Depending on the temperature, thermal cracking can occur either between adjacent crystalline grains (inter-granular cracks) (Jason et al. 1993) or within grains (intra-granular cracks) such as the case of α/β phase transition in quartz (Glover et al. 1995). Analysis of mineral–mineral contact types along the test fracture path in thermally treated granite specimens showed that pre-existing thermal damage mainly around grain boundaries leads to a larger volume of rock affected by fracture propagation (Fredrich and

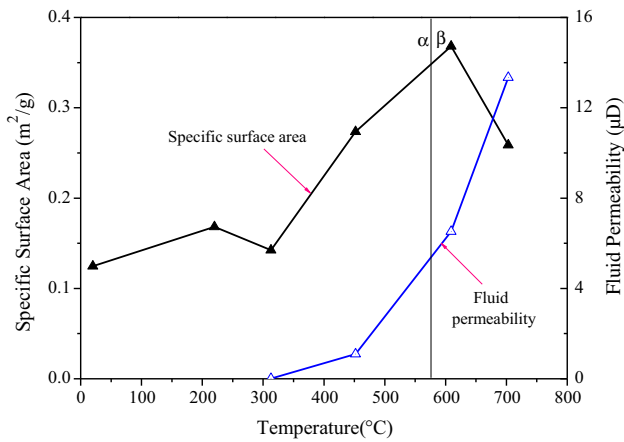


Fig. 16 Measurements made upon samples of La Bresse mylonite (DLB1 and DLB2). The variation of BET total internal surface area and fluid permeability with heat treatment temperature (Glover et al. 1995)

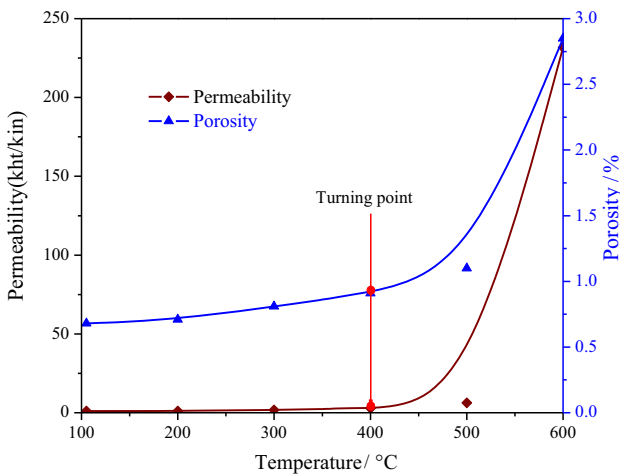


Fig. 17 Evolution of porosity and gas permeability vs. heat treatment temperature (Chaki et al. 2008)

Wong 1986; Nasseri et al. 2007). With the growth of microcracks due to thermal damage, the mechanical strength, elastic modulus and wave velocity decrease, plastic deformation, acoustic emission (AE), and permeability increase.

Chen et al. (2012) pointed out that 400 °C may be a critical value for the strength of granite. Nasseri et al. (2007, 2009) reported that above 450 °C, grain boundary opening and cracking, and intra-granular cracking and mineral grain dissection linked to the quartz α/β phase transition, induced a significant increase in the total density of cracks.

Our experimental results also suggest that 400 °C could be a critical threshold of the thermal damage of granite, corresponding to the transition from the brittle state to plasticity (or ductility). Therefore, significant changes in

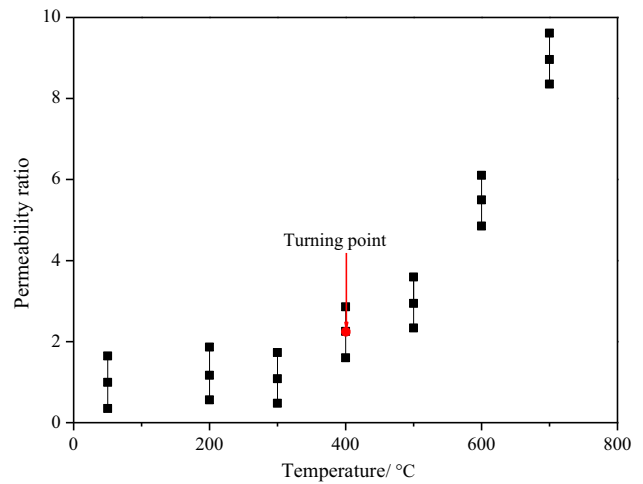


Fig. 18 Permeability change after heat treatment (Jones et al. 1997)

the physical and mechanical properties of granite specimens were observed in the temperature range of 300–600 °C, especially 400–600 °C.

Conclusion

In order to find out how the mechanical and physical properties of granite vary with temperature, uniaxial compression tests of granite specimens were conducted to measure the mechanical properties of granite rocks heated up to 800 °C, and their physical properties such as porosity, permeability, and ultrasonic wave propagation are also measured.

Based on the results and the data reported in previous studies, the process and critical threshold of the thermal damage of granite are discussed, and the following conclusions can be drawn:

1. temperature has a significant impact on the physical and mechanical properties of granite.
2. Generally, the temperature range of 300–600 °C, especially 400–600 °C, corresponds to the transition from the brittle state to plasticity (or ductility) of granite, and 400 °C maybe a critical threshold of the thermal damage of granite.
3. The results indicate five phases in the variation of physical and mechanical properties with temperature: from room temperature to 100, 100–300, 300–400, 400–600, and 600–800 °C. The first phase corresponds to the vaporization-escaping interval of adhered water, bound water, and structural water. Larger changes of physical and mechanical parameters in the temperature range of 300–600 °C, mostly 400–600 °C, are probably caused by the transition of granite from the brittle state to plasticity (or ductility). Between 400 and

600 °C, especially from 500 to 600 °C, the minerals (such as ankerite, siderite, magnetite, pyrrhotite, pyrite, illite, and kaolinite) in granite have chemical changes, which are demonstrated as volume increase, reduction of bearing capacity, increased connectivity, and abrupt change of wave velocity.

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