Experimental analysis on physical and mechanical properties of thermal shock damage of granite

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ABSTRACT. The purpose of this study was to explore the changes of mechanical and physical properties of granite under different thermal loading effects. Uniaxial compression experiments studying the rules of the influence of temperature load on mechanical properties of granite were carried out. After high-temperature heating at above 600 °C, granite tended to have stronger ductility and plasticity as well as declined peak stress and compressive strength. Thermogravimetry - differential scanning calorimetry (TG-DSC) analysis results showed that, thermal load at different temperatures induced reactions such as water loss, oxidation and crystallization in the microstructure of granite, which led to physical changes of granite. Hence it is concluded that, heating can significantly weaken the mechanical performance of granite, which provides an important support for the optimization of heating assisted processing of granite. It also reveals that, heating assisted cutting technique can effectively lower energy consumption and improve processing efficiency.

KEYWORDS. Granite; Damage; Differential scanning colorimetry analysis; Uniaxial compression thermogravimetry analysis.

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INTRODUCTION

Granite, a kind of highly hard and brittle material, is featured by high strength and stable physical and chemical performance. Granite products, which look elegant and beautiful, have been widely applied in fields such as architectural ornament, precision machine tool manufacture, etc [1-3]. Currently, the processing of granite becomes more precise and artistic and stone machining industry develops rapidly. However, stone material is a nonrenewable resource. Under the tendency that the society appeals to save industrial resources and protect environment, how to exploit and utilize stone materials efficiently has become a bottleneck in stone material industry [4-6].

Ogilvie SR [7] analyzed the microstructure of granite under the condition of uniaxial compression using image analysis method and found stronger aeolotropy of internal structure. By using different test specimens, it was found that, larger anisotropy inside specimens resulted in more significant directional property in the shape of micro-grains and the grain size had a remarkable influence on macroscopic strength of specimens.

Under different thermal loads, real-time changes happen to temperature field and stress filed inside granite, which can result in the changes of structure and mechanical performance of granite. This study explored the rules of temperature load influencing the physical performance of granite by carrying out a thermal load difference experiment and a uniaxial compression experiment.

PHYSICAL PROPERTIES UNDER THERMAL LOADING

In the actual grinding processing of granite, the intensive friction between cutter and workpiece generates a large amount of heat, due to the high hardness of granite. Diamond crystals may lose cutting ability at the temperature above 800 °C due to carbonization. Hence cutter needs to be cooled using water [8, 9].

In this experiment, the cutter was cooled using water after high-temperature heating up to 800 °C.

Experimental equipment and methods

The workpiece used was granite G630 with a size of 100 mm × 50 mm × 20 mm. After the determination of the specification, two types of treatment named plan A and B were performed in the following. Eight specimen groups were set up for each plan. In each group, five granite rectangle blocks were used. The temperature for the groups was set as 100 °C, 200 °C, 300 °C, 400 °C, 500 °C, 600 °C, 700 °C and 800 °C. Heating stopped after 30 min at constant temperature. Granite specimens of plan A were cooled with water for 5 min and then dried with air for 24 h, while granite specimens of plan B were cooled under natural conditions. The experimental set up is shown in Fig. 1.

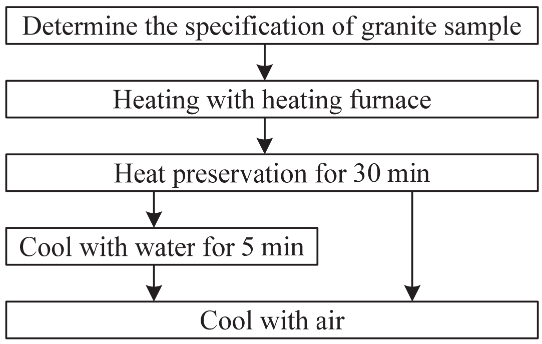

Figure 1: Experimental set up.

Analysis of experimental results

Quality changes of granite after water and air cooling are shown in Fig. 2. When the temperature was not higher than 400 °C, heating dried up the water stored on the surface or in the gap of the granite. When the temperature was higher than 400 °C, weight loss became more obvious. It was because that, micro-damage generated and micro-cracks increased in rocks under stress, which resulted in the evaporation of the water on the surface of rock cracks. Through comparing the data of the groups treated by air cooling and thermal shock, we found that, when the temperature was lower than 500 °C, the weight loss of the granite processed by air cooling was less remarkable than that of the granite processed by water cooling. It was because that, water entered into the cracks of the workpiece during water cooling and then was stored inside after cracks closed with the decreasing of temperature. When the temperature was higher than 600 °C, the weight lost in air cooling was less than the weight lost in water cooling. It was because of the excessive loss of crystal water caused by the sharp increase of micro-cracks of the granite.

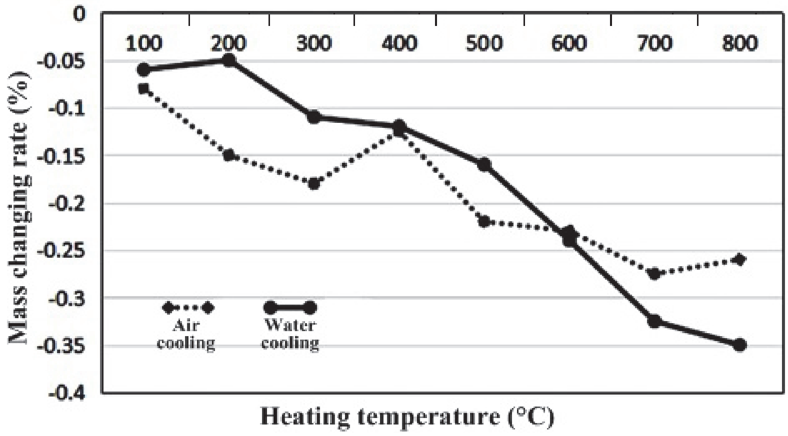

Figure 2: Mass changing rate after heating.

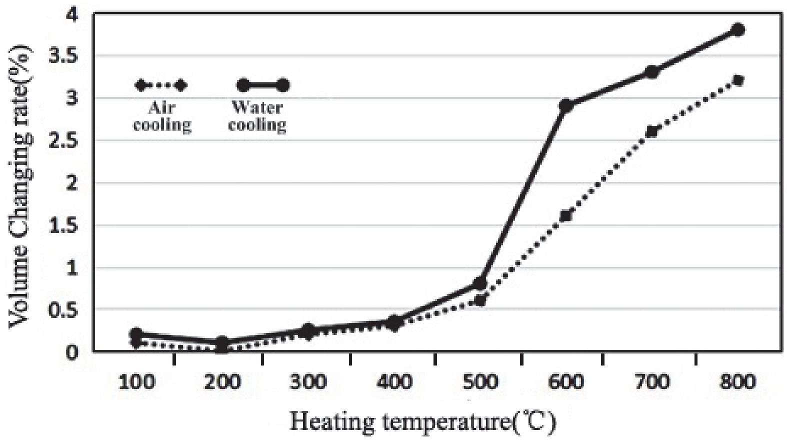

Figure 3: Volume changing rate after heating.

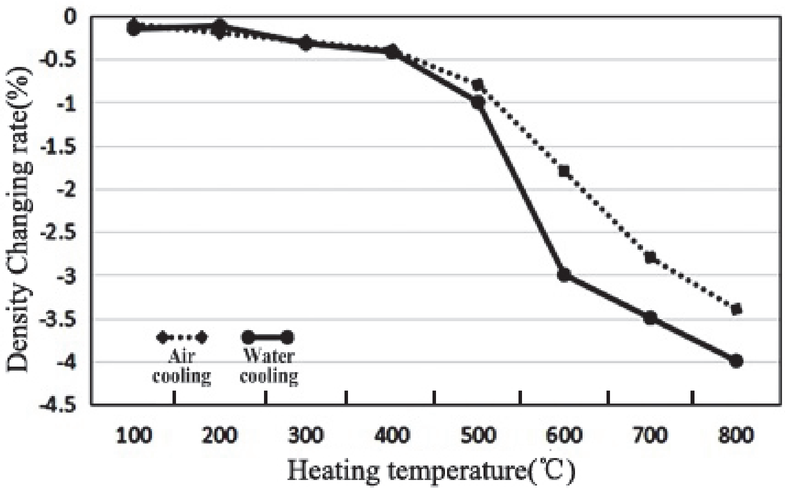

Figure 4: Density changing rate after heating.

Grains with different compositions, structures and coefficients of linear expansion expand when the granite is heated at certain temperature. As a result, the stress inside the rock exceeds yield strength limit of grains, thus causing damage on crystal. Therefore, cracks grow in the place where stress concentrates or preexisting defects generate and expand. When the temperature exceeds 400 °C, the internal stress increases sharply and the number and length of cracks increase faster, which indirectly results in the expansion of the granite.

It can be noted in Fig. 3 that, when the temperature exceeded 400 °C, the volume expansion rate of the granite processed by water cooling was higher than that of the granite processed by air cooling. It was because that, the sharp decreasing of temperature outside rocks led to the significant temperature gradient inside and outside rocks, and cracks expanded when the stress inside grains exceeded the yield limit. The above findings suggest that, increasing temperature gradient can promote the expansion of cracks and the generation of new cracks, which is beneficial to refine grains.
It can be noted in Fig. 4 that, when the thermal temperature was not higher than 400 °C, the rock density declined slightly. It was because of the evaporation of both free water in granite cracks and the expansion of micro-cracks of the granite. When the temperature exceeded 400 °C, micro-cracks further expanded, a large number of preexisting defects generated and expanded, and some crystal water was separated out, leading to the further decrease of density.

UNIAXIAL COMPRESSION TEST

Experimental equipment and methods

This experiment was carried on an electro-hydraulic servo universal testing machine [10, 11]. The selected hydraulic cylinder had a maximum axial output of 2000 KN and a piston movement speed of 0 ~ 85 mm/min. The axial loading of the cylinder sample which was cooled down after high temperature heating was controlled by the displacement generated by the hydraulic cylinder. Axial loading was performed on cylinder samples by means of hydraulic cylinder displacement control. An axial compression load was exerted under a constant displacement speed up to 0.001 mm/s till samples collapsed and lost the weight-carrying ability. Then a force-displacement curve was output. After numerical conversion, a stress-strain curve and parameters such as elastic modulus were obtained.

Experimental procedure

The heating processing and heating speed of the uniaxial compression test were the same as those of the thermal load difference experiment. The heating temperature was set as 200 °C, 400 °C, 600 °C and 800 °C. After temperature preservation for 30 min, the granite was taken out and then cooled with normal temperature water for 5 min. Then the granite specimens were dried up with air at normal temperature for 24 h. There were totally five groups. In each group, there were three cylinder granite specimens. A uniaxial compression test was performed on the fully cooled samples. The experimental process is shown in Fig. 5.

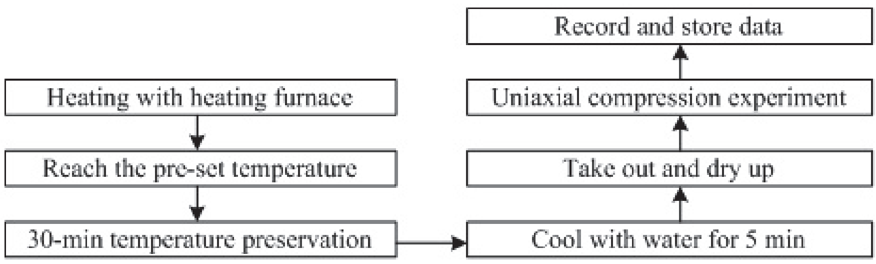

Figure 5: Experimental process.

Analysis of experimental results

Fig. 6 shows the axial stress-strain curve of the cylinder granite processed by different heating temperatures. The stress-strain curve of the granite could be divided into five stages.
(A) Compression stage (OA)
In this stage, most of the preexisting defects except cracks, whose initial arrangement direction was parallel to axial stress, were compressed to close and the slope of the axial strain curve increased gradually. Compression deformation in this stage was nonlinear: the structure and properties of the rock had no reversible changes and were at an equilibrium state when loading and unloading experiments were carried out in this area. Whether this stage was distinct or not depends on the density and initial distribution direction of preexisting defects.
(B) Elastic stage (AB)
As most of the preexisting defects were compressed in the last stage, the stress in this stage, though, could promote relative sliding between crack surfaces, but was not large enough to promote the expansion of cracks. Therefore, rock samples could be regarded as linear and isotropous elastic deformation bodies. The slope of axial strain and horizontal

strain remained unchanged. In this stage, there was no macro irreversible process and the deformation state was uniform. Hence the elastic stage was at an equilibrium state.

(C) Initial growth stage (BC)

With the increase of stress, new cracks developed in the site where the tip and local stress of the preexisting defects concentrated. The stress in this stage was 20% ~ 40% of the peak stress. But as the number of cracks was limited, the deformation of the specimens was still approximate to elastic deformation.

(D) Stable growth stage (CD)

With the increase of load, some cracks which had closed began to open and even expand when the stress was 36% ~ 90% of peak stress. Some weak grain boundaries generated new cracks. Both the original cracks and the new cracks expanded towards the direction parallel (or approximately) to the maximum major stress. In this stage, horizontal strain curve deviated from the original direct line and the growth speed of horizontal strain was faster than that of axial stress. Therefore, volume growth speed slowed down, which meant that dilatation occurred. Cyclic loading and uploading experiments suggested that, the generated permanent deformation could be ignored when axial stress was loaded to a level higher than 90% of the peak stress and then unloaded. Such a phenomenon could be observed under different confining pressures. Hence, it was considered that, the granite was still in a state of elastic deformation in the stable growth stage of cracks, but elastic modulus and Poisson ratio had been deteriorated due to the stable expansion of micro-cracks.

(E) Post-peaking stage (DE)

The micro-fracture surface inside rocks developed into a connective structural surface. When the exerted load was larger than the bearing capacity of rock samples, deformation became severer and friable rocks might even burst. As shown in Fig. 6, when the heating temperature was set between normal temperature and 400 °C, the stress-strain curve of samples suggested that, the deformation was similar to elastic deformation. When the load reached the peak stress, stress sharply declined, leading to the crushing of the rock body. Nearly no plastic deformation happened. When the temperature was between 600 °C and 800 °C, the rock body showed obvious plastic deformation; the samples at that moment still had certain bearing ability, but became soften when the stress slowly declined and the strain rapidly increased. Finally, the samples were damaged after severe deformation.

With the increase of thermal loading temperature, the compressive strength of the granite gradually declined. Combining with the heating processing results, we found the increase of micro-cracks of rocks resulted in the reduction of overall bonding area of grains, and intergranular cracks were more likely to generate during loading, considering the decline of strength.

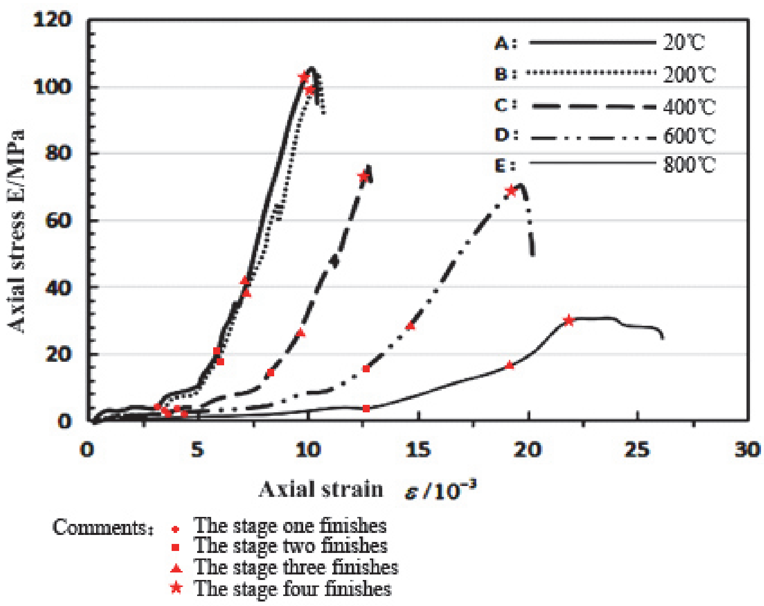

Figure 6: Stress-strain curves of granite specimens heated at different temperatures in uniaxial compression test.

Through comparing the stress-strain curves of granite specimens heated at different temperatures, it can be noted that, with the increase of heating temperature, the strain capacity of the granite increased, peak stress decreased, overall elastic-brittle performance weakened and ductile-plastic performance strengthened. When the heating temperature was lower than 400 °C, the stress-strain curve of samples was similar to the curve of deformation of brittle materials. Brittle damage occurred to rock samples when stress reached the peak value, and the stage of elastic deformation did not last long. When the temperature was higher than 600 °C, the rock body showed significant plastic deformation and strengthened ductility. Strain of rock samples increased rapidly after the stress reached the peak value, and then the stress decreased slowly. Eventually, the deformation similar to plastic deformation happened.

Effects of temperature on peak stress-strain and elastic modulus

Changes of microstructure of granite heated at different temperatures were reflected as the decrease of both macro-peak stress and elastic modulus and the increase of strain.

(1) Changes in peak stress of granite

Fig. 7 shows the changes of uniaxial compressive strength of granite σbc along with the increase of heating temperature. Results with relatively large deviation caused by personal error, instrumental error and calculation error were removed; the average values of the remaining peak stress points were calculated and connected with a line.

It can be noted in Fig. 7 that, when the temperature was not higher than 200 °C, compressive strength was above 90 % of compressive strength of granite at normal temperature, showing no obvious reduction. It suggested that, internal thermal stress and thermal damage were small and compressive strength showed no significant decreasing when the temperature was lower than 200 °C. When the temperature reached 400 °C, compressive strength decreased from 98 MPa to 78 MPa. When the temperature reached 600 °C or 800 °C, compressive strength sharply decreased to 62 MPa and 30 MPa respectively, and consequently the compressive strength was 64% and 30% that of granites at normal temperature, respectively.

When the temperature was higher than 400 °C, mineral grains inside samples expanded and cracks rapidly grew under the effect of internal stress, which resulted in the remarkable decrease of compressive strength.

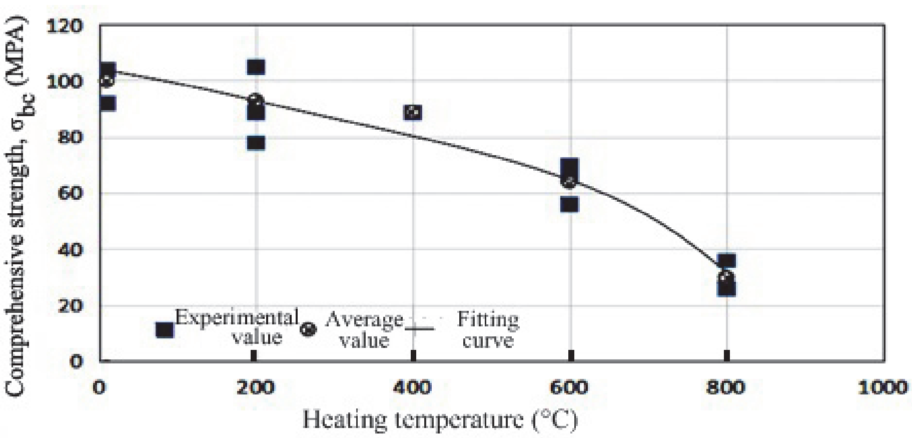

Figure 7: Relationship between compressive strength and heating temperature.

(2) Changes of peak strain of granite

Fig. 8 shows the curve for the relationship between compressive peak strain of granite and heating temperature after water cooling. The average peak strain value of temperature gradient in different groups was calculated and then a curve was drawn. With the increase of heating temperature, peak strain of granite tended to be higher. When the temperature was not higher than 200 °C, mineral grains inside rocks expanded and preexisting defects gradually closed. When the temperature was higher than 200 °C and continued to increase, thermal expansion became more and more intensive. Due to the different compositions and forms of grains, coefficient of linear expansion was also different.

Therefore, interaction generated between grains promoted the intensive expansion of preexisting defects at the point where stress concentrated. When the temperature was lower than 400 °C, peak strain increment was relatively small; but when temperature exceeded 400 °C, peak strain increment suddenly increased. Peak strain increment at temperature 600 °C and 800 °C was 66.7% and 94.5%, respectively. On one hand, higher temperature resulted in stronger thermal motion of molecules inside rocks and the active molecular motion weakened the combination of grains, which led to high risks of sliding deformation of grains and generation of more intergranular micro-cracks. On the other hand, with the increase of temperature, the action force between grains strengthened, leading to the increase of stress concentration points which exceeded grain ultimate strength and sharp increase of transgranular cracks. As a result, the number and area of micro-crack inside rocks increased rapidly and peak strain increment during uniaxial compression became larger, generating an increase of compressive deformation of rock samples at macro level.

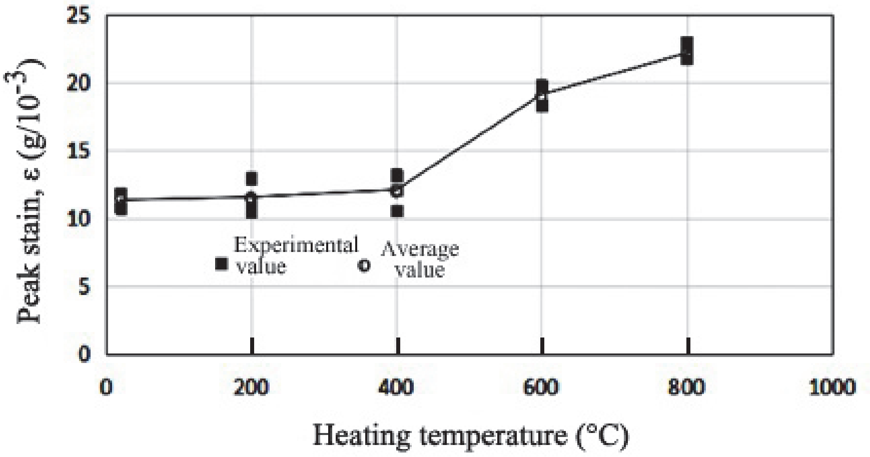

Figure 8: The relationship between peak strain and heating temperature.

(3) Changes of elastic modulus of granite
Elastic modulus, E, could be obtained by fitting approximate straight line segment on the stress-strain curve before peak stress. The elastic modulus of granite against the increase of temperature is shown in Fig. 9. Such a figure suggests that, elastic modules of granite gradually declined with the increase of temperature. When the temperature was not higher than 200 °C, such a decreasing was not remarkable, but when the temperature was higher than 400 °C, elastic modulus declined rapidly. At 400 °C and 600 °C, elastic modulus reduced by 50% and 75% respectively. Elastic modulus of granite heated at above 800 °C was only 10% that of elastic modulus of granite at normal temperature. The above curve indicates that, high temperature had a significant influence on the elastic modulus of granite, reflected as decreased rigidity and strengthened plasticity.

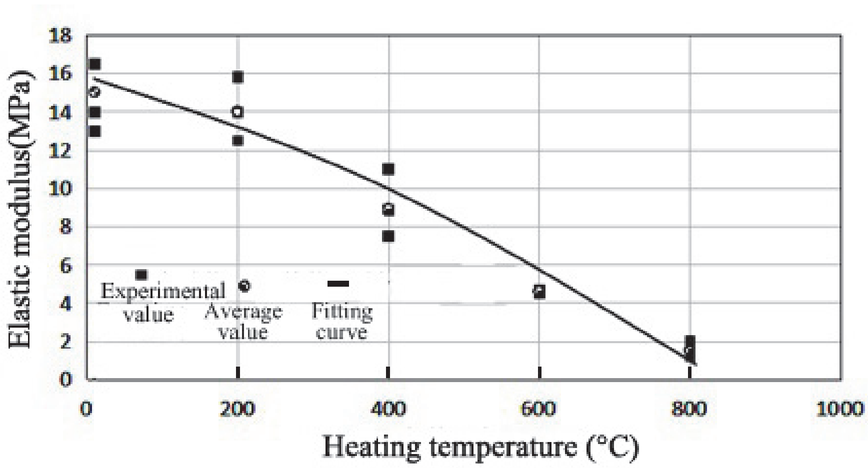

Figure 9: The relationship between elastic modulus and heating temperature.

Granite contains multiple mineral components. Different thermal expansion coefficients of various mineral particles and different thermoelastic properties of different crystalline orientations of anisotropic grains induce uncoordinated thermal expansion across grain boundary, which results in tensile stress or pressure stress between grains or inside grains. The generated stress promotes the generation of micro-cracks.
During high temperature heating, both constraining force promoting and inhibiting deformation between mineral grains lead to the increase of micro-cracks in granite. Structure thermal stress induced by temperature can cause damages to rock structure and thus weaken rock strength.
In these tests, G603 cylinder granite with a diameter of 60 mm and a height/diameter (H/D) of 2 was used. Diameter of 50 mm and H/D of 2 are usually regarded as the calculation basis of tests in most studies in China and abroad. Therefore,

we applied size amend coefficient K_D in the calculation of standard elastic modulus value of the sample. Standard size amend coefficient of diameter of 60 mm was 1.033. Standard elastic modulus could be obtained by amending elastic modulus measured, as shown in Tab. 1.

Temperature T/°C	Elastic modulus E[MPa]
20	17.18
200	14.37
400	8.95
600	4.33
800	1.64

Table 1: Standard elastic modulus obtained after amendment.

THERMAL ANALYSIS OF GRANITE

Thermal analysis is defined as a technology for analyzing the relationship between physical properties of substances and temperature by International Confederation for Thermal Analysis (IC-TA). Thermal analysis includes therogravimetry (TG), derivative thermogravimetry (DTG), differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermomechanic analysis (TMA), evolved gas analysis (EGA) and thermal expansion analysis. Among them, TG, DTA and DSC are the ones commonly used. The theory of thermal analysis can be simplified as:

$$P = f(T) \tag{1}$$

where T refers to temperature which is a function of time, and P stands for a physical property (function of temperature) of tested samples.

DSC [12-14] and TG were used in this experimental campaign. TG [12-14] is mainly used for analyzing the relationship between substance quality and temperature.

TG can be used for understanding thermal reaction of substance, for example, loss of crystal water and quality changes induced by changes of structure or crystalline state in thermal decomposition reaction.

DSC [15, 16] is a technology used for measuring the relationship between the power difference of substance and reference compound and temperature. The deviation of curve from baseline represents the speed of heat absorption or heat release (unit mJ/s). The area surrounded by peak or valley in a curve represents changes of heat.

In this test, a differential thermal analyzer was used to make thermal analysis on powder particles of granite, with which, TG curve and DSC curve can be obtained. Performance index of the analyzer was: sensitivity < 0.2 μg. Room temperature during heating was about 1500 °C.

In the test, grey white granite grains were used. The granite was grinded into even fine powder, with an initial weight of 13.1 mg and the weight turned to be 12.6 mg when the powder was heated from 20 °C to 1000 °C with an increasing speed of 10 K/min.

It can be seen from Fig. 10 that, the DSC curve and TG curve had three obvious heat absorption valleys and three obvious weight loss steps. The first heat absorption valley of the DSC curve appeared at 55 °C; when the temperature was 55 °C, water adhered on the surface of micro-crack of granite evaporated under the effect of heat, and weight loss rate was 0.21%. The second heat absorption valley appeared at 200 °C; water adhered on the surface of micro-cracks of granite evaporated under the effect of heat and the weight loss rate was 0.69 %. Moreover, interlayer water of granite was dehydrated in this stage. Heat release peak at temperature from 250.4 °C and 466.7 °C was smooth and heat release peak at temperature 264.7 °C was the highest; the weight loss rate was 1.49% in this stage. Crystal water of mineral crystal lattice lost when the temperature was between 590.7 °C and 670.4 °C; a weight loss rate of 3.87% appeared though there was no significant heat exchange. When the temperature exceeded 800 °C, mineral crystal lattice was damaged and showed phase change. A heat absorption valley appeared at 890 °C on the DSC curve.

We can know from physical and chemical changes of granite during heating that, absorbed water and interlayer water on the surface or in the cracks of granite evaporated when the heating temperature was between 100 °C and 200 °C. Micro-crack inside granite lost support and closed due to the loss of interlayer water, which led to increased density and stronger mechanical performance of the granite. When the temperature was between 200 °C and 466.7 °C, oxidation and crystallization reaction occurred to granite, i.e., granite released heat and lost much weight. When the temperature was between 466.7 °C and 800 °C, granite lattice lost crystal water and thus much weight lost; temperature had a large span in this stage, but there was no obvious thermal exchange. Moreover, in this stage, macro-mechanical performance of granite significantly decreased, cracks of grains grew rapidly and severe thermal damage generated. When the temperature exceeded 800 °C, silica grains inside granite showed phase changes, mica melted, the structure of granite was severely damaged, and mechanical performance intensively declined.

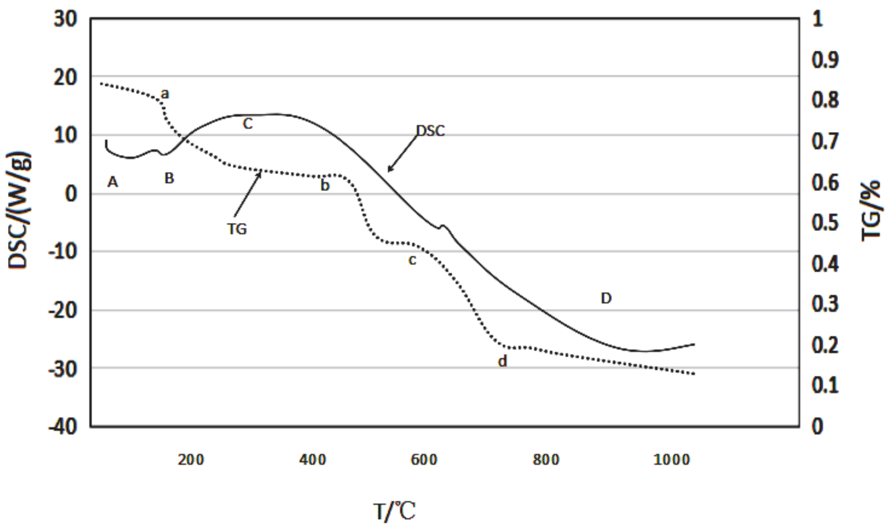

Figure 10: DSC-TG curve of granite.

CONCLUSION

In this study, on the premise of not considering homogeneous polycrystal, mechanical performance under different temperatures was obtained through carrying out heat processing experiment and uniaxial compression experiment. The increase of temperature resulted in lower density, volume expansion, decrease of compressive strength and elastic modulus and increase of peak strain.

With the increase of temperature, more weight of granite lost, cracks grew and crack volume increased due to the concentration of internal stress, leading to the permanent expansion of macro-volume and a density decrease. When the heating temperature exceeded 600 °C, the ductility and plasticity of granite strengthened, peak stress declined and compressive strength of granite declined. TG-DSC analysis showed that, thermal load induced reactions such as water lost, oxidation and crystallization in the microstructure of granite specimens, resulting in the changes in physical properties of rocks.

The research results of this study reveal that, heating can significantly lower mechanical performance of granite, which provides an important data support for the optimization of heating assisted processing of granites. Moreover, heating assisted cutting technology is proved to be effective in reducing energy consumption and improving processing efficiency.

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