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Analysis of triaxial compression deformation and strength characteristics of limestone after high temperature

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Abstract

Understanding the mechanical behavior of rock under conditions of high temperature and pressure is critical when it comes to implementing underground coal gasification, deep disposal of highly radioactive nuclear waste, development and utilization of deep underground space, development and utilization of geothermal resources, etc. To understand the effect of confining pressure on limestone at high temperature, uniaxial and triaxial compression testing was conducted on limestone under high temperature (20–800 °C) using the MTS 815 rock mechanics testing system. Using this system, the change in the strength and deformation parameters of the limestone at varying temperature and confining pressure conditions were studied. The results show that the triaxial compression stress-strain curve of limestone at high temperatures is divided into five stages: compaction stage, elastic stage, plastic deformation stage, post-peak failure stage, and residual stage. In addition, the rock brittleness and ductility decrease as the temperature increases. The peak stress and residual strength of the limestone at high temperatures increase as the confining pressure increases, and the strength of the rock specimens decreases as the temperature increases. The internal friction angle (φ) first increases and then decreases as the temperature increases, but the relationship of the cohesive force (c) to temperature is opposite that of φ , indicating that the shear strength of limestone at high temperatures is determined by both c and φ . The elastic modulus of limestone at high temperatures increases with the increase in confining pressure and decreases as the temperature increases, and the peak strain of limestone at high temperatures increases as the confining pressure and temperature increase. The effect of temperature on the failure of limestone is not obvious, and the failure of rock specimens during uniaxial compression testing was mostly through axial splitting, while the failure of rock specimens under triaxial compression testing was mostly through shear failure. As the confining pressure increases, the fracture type of the rock specimens gradually changes from brittle tensile fracture to shear fracture, and the instability type of the rock specimen changed from the sudden instability type to the progressive failure type. Our research results are especially useful in areas where engineering is performed on rocks under high temperature.

Keywords High-temperature rock · Strength and deformation characteristics · Triaxial compression testing · Brittleness and ductility . Failure form

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Introduction

Rock is formed through lengthy and complicated geological processes, including tectonic movements under the influence of various factors. It is also the main constituent of the crust and mantle. Rock is a natural aggregation of one or several rock-forming minerals and has a specific structure, chemical composition, and mineral composition (Song et al. [2019\)](#page-13-0). In recent years, there has been much attention paid to the mechanical properties of rock under high temperature since rocks are present at these temperatures in projects such as underground gasification of coal, exploitation of geothermal resources, disposal of high-temperature nuclear waste, and

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repair and reconstruction of underground chambers and tunnel projects (Heuze [1983](#page-13-0); Glover et al. [1995;](#page-13-0) Heap et al. [2009;](#page-13-0) Su et al. [2015;](#page-13-0) Sun et al. [2016;](#page-13-0) Zhang and Sun [2018;](#page-13-0) Liu et al. [2016;](#page-13-0) Liu et al. [2018](#page-13-0)). In high-temperature rock engineering projects, such as underground mining and underground energy storage projects, rock is present in a particular temperature and stress environment. The mechanical parameters of rock occurring at high temperature are the basis for stability analysis and support design of rock after events such as fire. Excessive temperature can cause a rock to deteriorate dramatically, and therefore, it is especially necessary to study the strength characteristics of rock under various temperature and three-dimensional stress states (confining pressure). These studies have important theoretical significance and engineering application value when it comes to understanding the influence of high temperature on the strength and deformation characteristics of rock.

Temperature (T) is an important factor affecting the mechanical properties of rock. A large number of theoretical and experimental studies have been performed on the influence of temperature on the mechanical properties of rock (Brotons et al. 2013; Bejarbaneh et al. [2013](#page-13-0); Mahanta et al. [2016;](#page-13-0) Tiskatine et al. [2016;](#page-13-0) Meng, et al. [2019\)](#page-13-0). Alm et al. [\(1985\)](#page-13-0) reviewed the mechanical properties of granite subjected to different heat conditions and discussed the rock fracture process under different temperatures. Tullis and Yund [\(1977\)](#page-13-0) performed uniaxial compression testing of granite at a temperature of 25–1000 °C and showed that the uniaxial compressive strength and elastic modulus of granite decreases gradually as the temperature increases. Zhang et al. [\(2009\)](#page-13-0) and Mao et al. [\(2009\)](#page-13-0) studied the mechanical properties of marble, limestone, and sandstone under different high temperatures through uniaxial compression testing. They reviewed the stress-strain curve characteristics of three kinds of rock and presented the manner in which the peak stress (uniaxial compressive strength), peak strain, and elastic modulus change with temperature. Their research results indicate that the peak stress and elastic modulus of marble show an undulating change within the range of 20–400 °C, and when the temperature is above 400 °C, the peak stress and elastic modulus show a gradual decline. Within the range of 20–200 °C, the peak stress and elastic modulus of limestone decreases as the temperature increases; they do not change much within the range of 200–600 °C; and the peak stress and elastic modulus show a sharp decline when the temperature is above 600 °C. Within the range of $20-200$ °C, the peak stress of sandstone tends to decline; within the range of 200–600 °C, the peak stress of sandstone increases; within the range of 20–600 °C, the elastic modulus of sandstone does not change much; when the temperature is above 600 °C, the peak stress and elastic modulus of sandstone drop sharply. The peak strain of limestone does not change much within the range of 20–600 °C, and when the temperature is above 600 °C, the peak strain rises sharply. Within the range of 20–200 °C, the peak strain of marble and sandstone decreases as the temperature increases; and when the temperature is above 200 °C, the peak strain increases rapidly. Chen et al. ([2012](#page-13-0)) studied the mechanical properties of granite at high temperatures through uniaxial compression testing and found that the peak stress and elastic modulus of granite gradually decrease, and the peak strain increases as the temperature increases. Ranjith et al. ([2012](#page-13-0)) carried out uniaxial compressive strength testing (UCS) after a 25–950 °C high-temperature treatment on Hawkesbury sandstone, and presented the variation in the uniaxial compressive strength of sandstone at different temperatures. Brotóns et al. [\(2013\)](#page-13-0) studied the influence of temperature on the mechanical properties of carbonate rock and showed that when the temperature is increased from 105 to 600 °C, the uniaxial compressive strength, elastic modulus, and Poisson ratio decrease as the temperature increases. Wu et al. ([2013](#page-13-0)) performed tests on sandstone at a temperature of 20–1200 °C and obtained the stress-strain curve of sandstone at high temperatures; a notable point from this study is that the peak stress and elastic modulus of rock specimens after a specific high temperature gradually decrease as the temperature increases. Shao et al. [\(2015\)](#page-13-0) conducted uniaxial compression testing on Strathbogie granite; in this study, at approximately 800 °C, the granite stress-strain curve showed obvious plasticity and post-peak strain-softening behavior, i.e., the brittle-plastic transition was observed to occur between 600 and 800 °C under uniaxial compression. Yang et al. [\(2017\)](#page-13-0) carried out uniaxial compression testing on granite treated at a high temperature of 200–800 °C with the TAW-1000 servo-controlled rock mechanics experimental system; the results of this study show that the uniaxial compressive strength and elastic modulus of granite first increase and then decrease as the temperature increases to a limit of 300 °C; this study also analyzed the strength and deformation failure behavior of granite at high temperatures. Masri et al. [\(2014](#page-13-0)) conducted triaxial compression testing under confining pressure ($\sigma_3 = 0$ –20 MPa) on the Tournemire shale after a 20– 250 °C temperature exposure and analyzed the effect of temperature and confining pressure on the mechanical properties of the Tournemire shale. Xu and Zhang ([2016](#page-13-0)) carried out triaxial compression testing under different temperatures ($T = 25-1000$ °C) and confining pressures ($\sigma_3 = 0-$ 40 MPa) through the MTS 815 rock mechanics testing system and analyzed the influence of the temperature and confining pressure on the strength and deformation characteristics of rock specimens; the study showed that the peak stress, shear strength, residual strength, and plastic deformation of rock specimens at high temperature increase with the increase in the confining pressure; in addition, the cohesive force of the limestone specimen decreases linearly as the temperature increases, and the internal friction angle first increases and then decreases as the temperature increases. Su et al. [\(2017](#page-13-0))

treated marble samples at 0–900 °C with the MXQ1700 heating furnace and conducted triaxial compression testing under confining pressures of 0–15 MPa with the MTS 815 rock mechanics testing system; this study revealed the effect of temperature, confining pressure, and joint direction on the strength and deformation characteristics of the marble specimens.

There are a large number of theoretical and experimental studies covering the influence of temperature on the mechanical properties of rock, but most have focused on the physical properties and characteristics of rocks under uniaxial compression testing at high temperatures. Relatively few studies have been performed using triaxial compression testing of rocks at high temperatures. In addition, due to the differences in mineral compositions and compositional structures of different types of rocks, the deformation and strength characteristics of different rocks vary under different temperatures and confining pressures; even the same rock under different geological conditions behaves differently since the pressure and temperature conditions are different. Differences in the mineral composition, crystal transition, microstructure, etc. of different rocks existing at high temperatures require that further studies should be undertaken to understand the behavior of these different rocks under different conditions. Adequately understanding rock mechanics under different temperature and confining pressure conditions is required when it comes to underground gasification of coal and oil shale, development of geothermal resources, treatment of high-temperature nuclear waste, etc. Therefore, it is necessary to study the effect of both pressure and temperature on the mechanical properties of rock. Keeping this in mind, in this study, triaxial compression tests were performed on limestone samples after hightemperature treatments at 20–800 °C to understand the effect of high temperature and confining pressure on the samples. The research results can be used as a reference for stability analysis and for restoration and reinforcement design related to high-temperature rock engineering.

Material and methods

Material and sample preparation

Limestone from Xuzhou, Jiangsu, China, was selected as the test material. This limestone is gray in its natural state and mainly composed of calcite, dolomite, illite, etc. The limestone has a uniform overall texture and has an average density of 2.71–2.73 $g/cm³$. The rock specimens were processed into cylinders with a diameter of 50 mm and height of 100 mm using rock processing equipment such as coring machine, rock sawing machine, rock grinding machine, and others. The rock specimens were heat-treated at high temperatures with a GWD-02A high-temperature furnace

(Fig. [1a](#page-3-0)). With this equipment, the temperature can be raised to (T) 20 °C (normal temperature), 200 °C, 400 °C, 500 °C, and 800 °C at a rate of 10 °C/min. A constant temperature was maintained for 4 h after the specified temperature was reached to ensure uniform heat levels inside the rock specimens. After being cooled to normal temperature, the specimens were taken out from the oven. The curve of the rate of rise in temperature is as shown in Fig. [1](#page-3-0) b, and Table [1](#page-3-0) presents details of the high-temperature limestone specimen testing program.

For the uniaxial and triaxial compression testing of the limestone at high temperatures (Fig. [1c\)](#page-3-0), the MTS 815 rock mechanics testing system, which is composed of loading, testing, and control systems, was used. This system can be used for axial force or stress control, axial displacement or strain control, etc. and meets the requirements for rock testing. The stiffness of the testing machine is 2.6×10^9 N/m, the axial load is 1459 kN, and the maximum travel of the axial actuator is 100 mm; the sensitivity of the servo valve in this system is 290 Hz, the data acquisition frequency is 5 kHz–1 MHz, the loading strain rate is 10^{-7} – $10^{-2}/s$, and the minimum sampling time is 50 μs. Before the testing began, Vaseline (lubricant) was evenly applied on the upper and lower surfaces of the rock specimens to reduce the influence of friction between the bearing plate of the testing machine and the rock specimens on the testing results.

Experimental methods

The strength and deformation properties of rock form the basis of theoretical calculations and engineering design when it comes to researching the mechanics of rock. Uniaxial and triaxial compression tests of rock are the basic testing methods for studying the mechanical properties of rock (Tembe et al. [2008](#page-13-0); Yang et al. [2011;](#page-13-0) Nicksiar and Martin [2012\)](#page-13-0), and these testing methods and processes are briefly described below:

(1) Uniaxial compression testing method and process: The diameter and height of the rock samples are measured at the upper, middle, and lower ends of the rock specimens, or the left, middle, and right ends of the end surface, etc. with a Vernier caliper, and the average value is taken as the diameter or height of the rock specimen; the loading indenter is placed on the upper and lower ends of the rock specimens, and then the rock specimens are placed at the central position of the bearing plate of the testing machine; the axial and circumferential displacement sensors, etc. are then installed; the axial displacement loading control mode is adopted, the upper limit of the compression displacement is set, the axial load is applied at a loading rate of 0.003 mm/s, and the testing is stopped when the failure of the rock specimens occurs.

Fig. 1 a–c Testing temperature curve and testing equipment

Table 1 High-temperature rock specimen testing program for limestone

Temperature $(^{\circ}C)$	σ_3 (MPa)					
	Ω	5	15	20	25	30
20	No. 1, no. 2,	No. 19, no. 20,	No. 22, no. 23,	No. 25, no. 26,	No. 28, no. 29,	No. 31, no. 32,
	no. 3	no. 21	no. 24	no. 27	no. 30	no. 33
200	No. 4, no. 5,	No. 34, no. 35,	No. 37, no. 38,	No. 40, no. 41,	No. 43, no. 44,	No. 46, no. 47,
	no.6	no. 36	no. 39	no. 42	no. 45	no. 48
400	No. 7, no. 8,	No. 49, no. 50,	No. 52, no. 53,	No. 55, no. 56,	No. 58, no. 59,	No. 61, no. 62,
	no. 9	no. 51	no. 54	no. 57	no. 60	no. 63
500	No. 10, no. 11,	No. 64, no. 65,	No. 67, no. 68,	No. 70, no. 71,	No. 73, no. 74,	No. 76, no. 77,
	no. 12	no. 66	no. 69	no. 72	no. 75	no. 78
600	No. 13, no. 14,	No. 79, no. 80,	No. 82, no. 83,	No. 85, no. 86,	No. 88, no. 89,	No. 91, no. 92,
	no. 15	no. 81	no. 84	no. 87	no. 90	no. 93
800	No. 16, no. 17,	No. 94, no. 95.	No. 97, no. 98,	No. 100, no. 101,	No. 103, no. 104,	No. 106, no. 107,
	no. 18	no. 96	no. 99	no. 102	no. 105	no. 108

(2) Triaxial compression testing methods and processes: After the diameter and height of the prepared rock specimens are measured with a Vernier caliper, the periphery of the rock specimens is covered with a thin heat shrinkable tube and uniformly wrapped with waterproof adhesive tape to prevent the hydraulic oil from immersing into the rock specimen and affecting the testing results; the rock specimen is placed in the triaxial pressure chamber, and air in the pressure chamber is then discharged. A confining pressure is applied at a loading rate of 0.1 MPa/s and the confining pressure value is kept constant at confining pressures of 5, 15, 20, 25, and 30 MPa. The axial displacement loading control mode is adopted, the upper limit of the compression displacement is set, and the axial load is applied at a loading rate of 0.005 mm/s until the rock specimen fails.

Analysis of triaxial compression testing results of limestone at high temperatures

The triaxial compression testing stress-strain curves of limestone rock specimens at high temperatures are shown in Figs. [2](#page-5-0) and [3](#page-6-0), and Table [2](#page-7-0) gives the results of triaxial compression testing of limestone rock specimens at high temperatures. Figures [2](#page-5-0) and [3](#page-6-0) show that the triaxial compression testing stress-strain curve of limestone rock specimens at high temperatures can be divided into the following stages: compaction, elastic, plastic deformation, post-peak failure, and residual (Martin and Chandler [1994;](#page-13-0) Martin [1997\)](#page-13-0). (1) Compaction stage: under the effect of a load, the microcracks or pores in the rock shrink gradually until they close (Guo et al. [2019\)](#page-13-0); the increase in the amplitude of the stress is relatively slow, while the increase in the amplitude of the strain is relatively fast; the stress-strain curve is concave, and this stage mainly reflects the closing of the microcracks in the rock specimens. (2) Elastic stage: the curve in this stage basically increases linearly, and the stress and strain are in a direct proportional relationship, i.e., the deformation of rock after unloading in the elastic stage are recoverable elastic deformation. (3) Plastic deformation stage, which is also known as the yield stage: with the further increase in the load, the closed microcracks in the rock specimens re-open and expand continuously, while, at the same time, new cracks occur; the microcracks in the rock specimen continue to expand, connect, and penetrate under the effect of load; in this stage, the stress-strain curve shows a non-linear relationship, and the process mainly reflects the yield process in which the internal material of the rock specimens gradually reach the limit of their bearing capacity. Due to the discontinuity and heterogeneity of rock, the stress-strain morphology near the bearing limit (peak point) of the rock sample is different, which reflects the differences in the distribution of the internal material

strength of the rock specimens. In general, during the axial compression process, when the strength distribution of internal materials of the rock specimen is homogeneous, the internal materials of the rock specimens reach their bearing capacity limit almost simultaneously, so that the bearing capacity limit of the rock specimen may become a stress cusp (peak point); when the difference in the strength distribution of the internal material of the rock specimen is large, the yield process is long, and a gentle yield platform is often formed in the stress-strain curve. (4) Post-peak failure stage: when the load is increased to the limit of the bearing capacity of the rock specimens, the interaction between cracks is enhanced and the internal cracks of the rock specimen penetrate to form a macrofracture, which causes the rock specimen to lose its capacity to bear the load, and thus, the rock fails. This stage represents the continuous weakening of the bearing capacity of the rock specimens. (5) Residual stage: After the macrofracture surface of the rock is generated, the bearing capacity of the rock does not completely disappear but is reduced to a certain extent (residual strength), and it still shows a certain bearing capacity at the macroscopic level. At this stage, after the rock has failed, a certain amount of bearing capacity is maintained due to the friction between the broken rock blocks. Of course, the bearing capacity of the rock specimen after failure during uniaxial compression testing is very low.

There are some differences in the characteristics of the stress-strain curves of the rock specimens under the combined action of temperature and confining pressure. During uniaxial compression testing, the stress-strain curve of the rock specimen quickly decreases after reaching the peak stress point (σ_f) . The rock specimen loses its bearing capacity in a short time, and brittle failure of the rock occurs. As the confining pressure increases, after the stress-strain curve reaches the peak stress point, the stress decreases with the increase of strain. Meanwhile, the plastic deformation of the rock specimen gradually increases, and the brittle-ductile transition occurs. As the temperature rises, the rock specimen shows an obvious yield platform in the stress-strain curve, and the failure state of the rock specimens gradually changes from brittle failure to ductile failure (Heard [1960;](#page-13-0) Wawersik and Fairhurst [1970;](#page-13-0) Fredrich et al. [1990](#page-13-0)). As can be seen from Fig. [2](#page-5-0), at the same temperature, both the peak stress and strain of the limestone increase with the rise of confining pressure, and the post-peak stage of the stress-strain curve of the limestone after high temperature is transformed from a strain-softening characteristic to ideal plasticity. That is to say, when the confining pressure increases to a certain value, the stress-strain curve of the limestone shows a yield platform (under the action of axial load, the strain of the rock specimen increases continuously, while the stress remains basically unchanged), exhibiting a plastic flow state, i.e., an ideal plasticity of the rock specimen is achieved at the post-peak. At this time, the corresponding confining pressure is called the transformation

Fig. 2 Stress-strain curve for triaxial compression testing of limestone specimens under different confining pressures. a $T = 20 \degree$ C. b $T = 200 \degree$ C. c $T = 200 \degree$ C. 400 °C. d $T = 500$ °C. e $T = 600$ °C. f $T = 800$ °C

confining pressure. When the confining pressure is lower than the transformation confining pressure, there is an obvious peak stress point (stress cusp) on the stress-strain curve of the rock; when the confining pressure continues to increase

and is higher than the transformation confining pressure, there is no longer an obvious peak point on the stress-strain curve (the stress continues to be maintained at a certain numerical level). As can be seen from Fig. 2 a, for the rock specimens at

Fig. 3 Stress-strain curve in triaxial compression testing of limestone rock specimens at different temperatures. a $\sigma_3 = 0$ MPa. b $\sigma_3 = 5$ MPa. c $\sigma_3 = 5$ 15 MPa. d $\sigma_3 = 20$ MPa. e $\sigma_3 = 25$ MPa. f $\sigma_3 = 30$ MPa

normal temperature ($T = 20$ °C), when the confining pressure is higher than 25 MPa, there is an obvious yield platform near the peak strength, and the peak stress point is not obvious; after the yield platform lasts for a period of time, the stress begins to decrease slowly with obvious elasticity characteristics. As can be seen from Fig. [2](#page-5-0) b–f, for the rock specimens subjected to temperatures of 200–800 °C, when the confining pressure is higher than 15 MPa, there is an obvious yield platform near the peak strength, and the amount of stress decreases after the yield platform gradually slows down as the

Table 2 Triaxial compression testing results of limestone specimens after high temperature

The data in the table is the average value of three rock specimens

temperature increases, which means that the brittleness of the rock decreases and the plasticity increases as the temperature increases. As can be seen from Fig. [3](#page-6-0), under the same confining pressure, the strength of the limestone (peak stress) gradually decreases and the strain gradually increases as the temperature increases; when $T = 500$ °C and $\sigma_3 = 0$ MPa, the strength of the rock specimens drops sharply with a decrease amplitude of − 18.66%; when $T = 500$ °C and $\sigma_3 = 15$ MPa, the strength decrease amplitude of the rock specimen is − 15.58%; when $T = 500$ °C and $\sigma_3 = 30$ MPa, the strength decrease amplitude of the rock specimens is − 14.60%. In summary, when $T = 20$ °C, the stress-strain curve of the rock specimens shows obvious brittle failure characteristics; when $T = 200$ °C, the stress-strain curve of the rock specimens shows brittle failure characteristics under the low confining pressure conditions, and certain ductile failure characteristics under the high confining pressure conditions, i.e., as the strain increases, the speed of the stress reduction becomes slower, the plastic deformation of the rock specimen increases gradually, and the rock specimen transitions from brittle failure to plastic deformation; when $T > 400$ °C, the rock specimen shows obvious ductile failure characteristics, and an obvious yield platform appears on the stress-strain curve.

Discussion

Analysis of strength characteristics of limestone at high temperatures

The strength of a rock is what prevents failure of the rock under external force, and it is one of the most important characteristics of the mechanical properties of a rock. The strength of a rock is usually expressed by peak stress and residual strength; the peak stress refers to the maximum load on the rock specimen during uniaxial or triaxial compression testing (i.e., failure resistance capacity of the rock), and it is closely related to environmental factors such as confining pressure and temperature. Residual strength refers to the bearing capacity provided by the friction force on the fracture surface after the failure of rock sample. Data about the peak stress and residual strength of the limestone specimens used in this study after being subjected to high-temperature conditions during uniaxial and triaxial compression testing were statistically analyzed, and the relationship between the peak stress, residual strength (residual stress), confining pressure, and temperature was obtained, as shown in Figs. [4](#page-8-0) and [5](#page-8-0). As can be seen from Figs. [4](#page-8-0) and [5](#page-8-0), the peak stress and residual strength of the limestone specimen, after being subjected to high temperatures, increase as the confining pressure increases; as the temperature increases, the strength of rock specimens decreases.

Under sufficient compressive stress, shear failure (compression shear failure) of rock-like materials occur, and the shear strength is related to the strength parameters of the material (cohesive force and internal friction angle), and thus the strength characteristics can be indicated by the internal cohesion and internal friction angle of the rock. The general expression (Yang et al. [2012](#page-13-0); Bejarbaneh et al. [2015](#page-13-0)) of the Mohr-Coulomb (M-C) in Eq. 1 is:

$$
\tau = c + \sigma \tan \varphi \tag{1}
$$

where τ is the ultimate shear stress (shear strength) of rock (MPa); σ is the normal stress of rock (MPa); c is the internal cohesion of rock (MPa); and φ is the internal friction angle of rock (°).

When the principal stress is adopted, the formula of the Mohr-Coulomb strength criterion is as follows:

Fig. 4 Relationship between peak stress, residual stress, and confining pressure of limestone specimens. **a** $\sigma_f \sigma_3$. **b** $\sigma_r \sigma_3$

$$
\sigma_1 = \sigma_c + \xi \sigma_3 \tag{2}
$$

where σ_1 is the maximum principal stress (MPa); σ_3 is the minimum principal stress (MPa); σ_c is the theoretical uniaxial compressive strength of rock (MPa), $\sigma_c = 2c \cos\varphi/(1-\sin\varphi)$; ζ is the slope of the strength line, $\xi = (1 + \sin \varphi)/(1 - \sin \varphi)$.

The strength characteristic parameter c and φ of the rock can be obtained from Eq. [1](#page-7-0):

$$
\begin{cases}\nc = \sigma_c \frac{1 - \sin \varphi}{2c \cdot \sin \varphi} \\
\varphi = \sin^{-1} \frac{\xi - 1}{\xi + 2}\n\end{cases}
$$
\n(3)

From Eq. 3, the relationship between the cohesive force, internal friction angle, and temperature of limestone specimens after being subjected to high temperatures can be obtained, as shown in Fig. [6](#page-9-0).

Fig. 5 Relationship between peak stress, residual stress, and temperature of limestone specimens. **a** σ_f T. **b** σ_r -T

It can be seen in Fig. [6](#page-9-0) that when $T \leq 400$ °C, φ of 19.728° at 20 °C increases to 20.99° at 400 °C with an increase in amplitude of 6.43%. When $T > 400$ °C, φ gradually decreases; and when the temperature reaches 800 °C, the internal friction angle decreases to 18.76°, which is an amount of -4.87% as compared to that at 20 °C. However, the relationship of c with temperature is opposite to that of φ . The true reason for this is that before the temperature reaches 400 °C, different thermal expansion coefficients of minerals cause irregular deformation in the rock, enhance the mutual friction and mutual interlocking between rock particles, and cause the internal friction angle to increase as the temperature increases. When the heating temperature reaches 400 °C, due to the rise in the temperature, water inside the rock specimens evaporates and the minerals dehydrate, so that the internal porosity increases; in addition, the thermal stress between mineral particles inside the rock specimens is further increased, and microcracks continuously merge and penetrate, resulting in larger microcracks

Fig. 6 Relationship between cohesion, internal friction angle, and temperature of limestone specimens at high temperatures

and a decrease in friction between the rock specimens, and, thus, the internal friction angle is reduced. In general, the internal friction angle and cohesion force are indicators used to indicate the shear strength of a rock. The cohesive force reflects the cohesive degree of the interior of the rock, and is related to the particle size, cementing compositions, and cementation degree of the mineral particles present in the rock. The changes in cohesive force and internal friction angle are roughly opposite to the changes in temperature, which indicates that the shear strength of the rock specimens is determined by the cohesive force and internal friction angle.

Limestone usually contains many microcracks. The axial deformation caused by triaxial compression includes elastic deformation of the mineral particle framework, slippage of cracks, and closure of the pores. The plastic deformation of rock sample during triaxial compression includes friction slip between particles and pore closure. The confining pressure can make the cracks in the rock sample close partly and slip between the cracks. As the confining pressure increases, the cracks inside the rock specimens become more closed, there are fewer cracks that slip, and the axial deformation of the rock specimen is smaller, so that the rock specimen has a higher elastic modulus, which shows that the deformation resistance of rock specimens gradually increases as the confining pressure increases. In addition, under a confining pressure, the pores or microcracks in the rock are compacted and closed again (Guo et al. [2019](#page-13-0)), the normal stress on the crack surface increases, and the residual strength of the rock specimen increases. Because limestone is an aggregation composed of different minerals, there is a certain difference between the thermal expansion coefficients of various minerals, and the deformation characteristics after heating during triaxial compression loading includes frictional slippage, pore closure, etc. between the particles. The confining pressure can partially seal the internal cracks of rock specimens and cause different

slippage between different cracks. When the structural stress reaches or exceeds the strength limit of the rock, microcracks appear between the mineral particles at the interface. The rapid expansion of organic components in the rock specimens leads to microcrack expansion and rock damage. Simultaneously, some minerals in the rock specimen may recrystallize and form new minerals, the factors of decrease of stiffness of cement between particles may affect the deformation of the rock specimens, and finally, the deformation resistance of limestone specimens decreases. Furthermore, as the temperature increases, the stress between or within particles further increases, causing more microcracks on the rock specimen or expansion, opening, and connection of the primary cracks leading finally to the deterioration in the mechanical properties of the rock specimens. The deterioration of the mechanical properties of the limestone after being subjected to high temperatures is the result of the combination of structural thermal stress and expansion formed due to factors like mineral crystal transition, crystal expansion, coefficient difference, and organic matter expansion.

Analysis of deformation characteristics of rock at high temperatures

The elastic modulus of rock material, which is an important performance parameter in geotechnical engineering design, determines the stiffness characteristics of the rock. At the macroscopic level, the elastic modulus of a rock is the measurement of the deformation resistance of the rock material. At the microscopic level, the elastic modulus of the rock reflects the bonding strength between the microstructures, crystal structures, etc. of rock material, and affects the factors related to the elastic modulus of rock material, such as crystal structure, chemical composition, microstructure, stress, and temperature. In this study, the elastic modulus (Young's modulus, E) of the rock specimens was calculated using the slope of the approximate straight segment on the stress-strain curve of the limestone at different temperatures, as shown in Fig. [7](#page-10-0) a–b. As the confining pressure increases, the plastic deformation near the peak stress of the rock increases, and, therefore, there must be some relationship between the confining pressure and peak strain. The relationships between peak strain of the limestone specimens at high temperature and confining pressure conditions and the temperature are as shown in Fig. [7](#page-10-0) c–d. The peak strain described in this paper refers to the corresponding axial strain value (ε_t) when the rock specimen reaches the peak stress. As can be seen from Fig. [7](#page-10-0) a–d, the elastic modulus of limestone at high temperatures increases with the increase in the confining pressure and decreases as the temperature increases, and the peak strain of the limestone specimens at high temperatures increases as the confining pressure increases and temperature increases.

Fig. 7 Relationships between the elastic modulus, peak strain, confining pressure, and temperature of limestone specimens. a E - σ_3 . b E - T . c $\varepsilon_r \sigma_3$. d ε_r - T

Limestone is an aggregation of mineral particles with obvious heterogeneity. During axial compression, frictional slippage may occur along the internal cracks of the rock specimen. Obviously, there are a few cracks along which slips may occur when the confining pressure is increased. As a result, the axial deformation of the rock specimens is small, and consequently, limestone has a high elastic modulus (Yang et al. [2012](#page-13-0)). At high temperatures, there are failures of different degrees inside the limestone. As the temperature increases, fusion and phase change, and fracture of metal bonds further increase the stress within particles and thermal stress between or within particles occurs on a lot of minerals inside the rock, which causes more microcracks to form in the limestone or the expansion, opening, and connection of the primary cracks. Consequently, the strainsoftening characteristics of the limestone increase, and, as a result, the peak strain of the limestone increases as the temperature increases. At the macroscopic level, limestone shows deterioration in its mechanical properties and the elastic modulus of limestone decreases as the temperature increases (Ranjith et al. [2012](#page-13-0); Sun et al. [2016\)](#page-13-0).

Analysis of modes of failure of limestone at high temperatures

Uniaxial and triaxial testing of limestone specimens at high temperatures show that the modes of failure of rock specimens are closely related to their physical and mechanical properties, confining pressure, temperature, etc. (Baud et al. [2000](#page-13-0); Chang and Lee [2004;](#page-13-0) Yang et al. [2012](#page-13-0)). The failure forms of limestone in this study during uniaxial and triaxial compression testing at high temperatures are shown in Fig. [8](#page-11-0).

In summary, (1) the failure forms of limestone specimens in uniaxial compression testing at a temperature of 20–800 °C are basically through axial fracture parallel to or almost parallel to the axial direction, i.e., the failure form after failure of rock specimens is mainly through

Fig. 8 Failure forms of limestone specimens at high temperatures

failure based on axial splitting (columnar splitting). After the failure of the rock specimens, there are many cleavage planes along the axial direction, and these cleavage surfaces are parallel to or almost parallel with the axial direction of the rock specimens, and after the failure of the specimens, multiple long strip rock blocks are formed. (2) At a temperature of $20-800$ °C, most of the failure forms of the rock specimens during triaxial compression testing are through shear failure, and in general, obvious shear failure surfaces exist in the rock specimens. Most failure forms of the rock specimens after failure are through single shear surface failure, and the rock specimens are divided into both upper and lower triangular blocks due to a shear failure surface passing through most axial sections of the rock specimens. This shear failure surface exists in the destroyed rock specimens. On the interface (shear failure surface) of the two rock blocks, there are obvious friction marks, which are caused due to secondary shear failure formed by the stress concentration at the partial rough place of the shear surface during shear slippage of the upper and lower rock blocks. The instability types of the rock specimens include abrupt instability type and progressive failure type. The abrupt instability is characterized by a sudden drop of the stress and the simultaneous release of a relatively large sound; the stress-strain post-peak curve shows a straight line that falls sharply in the post-peak failure stage. In the progressive failure type, the stress slowly decreases or remains at the same level as that at the post-peak failure stage; the sound is smaller than that occurring during the abrupt instability type, and the stress-strain curve is a gently descending curve in the post-peak failure stage. As the confining pressure increases, the fracture mode of the rock specimens gradually changes from brittle tensile fracture to shear fracture, and the instability type of the rock specimens change from the sudden instability type to the progressive failure type. Within the designed ranges of temperature and confining pressure in this study, the primary factor affecting the mechanical properties of limestone is temperature followed by confining pressure. However, the influence of temperature on the instability type of limestone is not obvious, and the instability type of limestone mainly depends on the confining pressure.

Conclusions

Triaxial compression testing under a confining pressure of 0– 30 MPa was conducted on limestone specimens at temperatures of 20–800 °C with the MTS 815 rock mechanics testing system. The stress-strain curve of limestone under different temperature and confining pressure conditions was analyzed,

and the change in the stress-strain curve shape, strength, and deformation parameters of limestone at different temperature and confining pressure conditions were analyzed. The main conclusions are as follows:

- (1) The triaxial compression stress-strain curves of limestone at high temperatures can be divided into 5 stages as compaction stage, elastic stage, plastic deformation stage, post-peak failure stage, and residual stage. When $T = 20$ °C, the stress-strain curve of the rock specimen shows obvious brittle failure characteristics; when $T = 200$ °C, the stress-strain curve of the rock specimen presents brittle failure characteristics under low confining pressure conditions, and shows certain ductile failure characteristics under high confining pressure conditions, and the failure (deformation) mode of rock specimen gradually changes from brittle failure to plastic deformation; when T>400 °C, the rock specimen indicates obvious ductile failure characteristics, and an obvious yield platform occurs. As the temperature increases further, the confining pressure of brittle-ductile transition decreases.
- (2) The peak stress and residual strength of limestone at high temperatures increase as the confining pressure increases; as the temperature increases, the strength of the rock specimens decreases. When $T \leq 400 \degree C$, φ increases as the temperature increases; and when $T > 400$ °C, φ decreases as the temperature increases. However, the relationship of c with temperature is opposite that of φ , and this study shows that the shear strength of limestone at high temperatures is determined by the cohesive force and internal friction angle. The elastic modulus of limestone at high temperatures increases with the increase in confining pressure and decreases as the temperature increases, and the peak strain of limestone at high temperatures increases as the increase of confining pressure and temperature.
- (3) Most of the failure forms of limestone specimens during uniaxial compression testing at a temperature of 20– 800 °C are through axial splitting parallel or approximately parallel with the axial directions, and most of the failure forms of rock specimens during triaxial compression testing are through shear failure. As the confining pressure increases, the fracture mode of the rock specimens gradually changes from brittle tensile fracture to shear fracture, and the instability type of the rock specimen changes from sudden instability type to progressive failure type. The influence of temperature on the instability type of limestone is not obvious. The instability type of limestone mainly depends on the confining pressure.

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References

- Alm O, Jaktlund LL, Shaoquan K (1985) The influence of microcrack density on the elastic and fracture mechanical properties of Stripa granite. Phys Earth Planet Inter 40:161–179
- Baud P, Zhu W, Wong TF (2000) Failure mode and weakening effect of water on sandstone. J Geophys Res-Sol Ea 105:16371–16389
- Bejarbaneh BY, Armaghani DJ, Amin MFM (2015) Strength characterisation of shale using Mohr-Coulomb and Hoek-Brown criteria. Measurement 63:269–281
- Bejarbaneh V, Tomás R, Ivorra S, Alarcón JC (2013) Temperature influence on the physical and mechanical properties of a porous rock: San Julian's calcarenite. Eng Geol 167:117–127
- Brotóns V, Tomás R,Ivorra S, Alarcón JC (2013) Temperature influence on the physical andmechanical properties of a porous rock: San Julian's calcarenite. Eng Geol 167:117–127
- Chang SH, Lee CI (2004) Estimation of cracking and damage mechanisms in rock under triaxial compression by moment tensor analysis of acoustic emission. Int J Rock Mech Min 41:1069–1086
- Chen YL, Ni J, Shao W, Azzam R (2012) Experimental study on the influence of temperature on the mechanical properties of granite under uniaxial compression and fatigue loading. Int J Rock Mech Min 56:62–66
- Fredrich JT, Evans B, Wong TF (1990) Effect of grain size on brittle and semibrittle strength: implications for micromechanical modelling of failure in compression. J Geophys Res-Sol Ea 95:907–920
- Glover PWJ, Baud P, Darot M, Meredith PG, Boon SA, LeRavalec M, Zoussi S, Reuschlé T (1995) α/β phase transition in quartz monitored using acoustic emissions. Geophys J Int 120:775–782
- Guo JN, LIU JF, Li Q, Chen X, Chen ZQ, Huang BX, Chen SL (2019) Variation law of coal permeability under cyclic loading and unloading. Therm Sci 23(3b):1–8
- Heap MJ, Baud P, Meredith PG (2009) Influence of temperature on brittle creep in sandstones. Geophys Res Lett 36:L19305
- Heard HC (1960) Transition from brittle fracture to ductile flow in solenhofen limestone as a function of temperature, confining pressure, and interstitial fluid pressure. Geol Soc Am Bull 79:193–226
- Heuze FE (1983) High-temperature mechanical, physical and thermal properties of granitic rocks- a review. Int J Rock Mech Min Sci Geomech Abstr 20:3–10
- Liu JF, Song Y, Skoczylas F, Liu J (2016) Gas migration through watersaturated bentonite-sand mixtures, COx argillite and their interfaces. Can Geotech J 53:60–71
- Liu JF, Ni HY, Chen YG, Wu Y, Song SB, Cao XL (2018) Research on water retention and microstructure characteristics of compacted GMZ bentonite under free-swelling conditions. Environ Earth Sci 2018(77):583–594
- Mahanta B, Singh TN, Ranjith PG (2016) Influence of thermal treatment on mode I fracture toughness of certain Indian rocks. Eng Geol 210: 103–114
- Mao XB, Zhang LY, Li TZ (2009) Properties of failure mode and thermal damage for limestone at high temperature. J China Univ Min Technol 19:290–294
- Martin CD, Chandler NA (1994) The progressive fracture of Lac du Bonnet granite. Int J Rock Mech Min Sci Geomech Abstr 31:643– 659
- Martin CD (1997) Seventeenth Canadian Geotechnical Colloquium: the effect of cohesion loss and stress path on brittle rock strength. Can Geotech J 34:698–725
- Masri M, Sibai M, Shao JF, Mainguy M (2014) Experimental investigation of the effect of temperature on the mechanical behavior of Tournemire shale. Int J Rock Mech Min 70:185–191
- Meng QB, Wang KC, Liu JF, Zhang MW, Lu MM, Wu Y (2019) Physical and micro-structural characteristics of limestone after high temperature exposure. B Eng Geol Environ:1–16. [https://doi.org/10.1007/](https://doi.org/10.1007/s10064-019-01620-0) [s10064-019-01620-0](https://doi.org/10.1007/s10064-019-01620-0)
- Nicksiar M, Martin CD (2012) Evaluation of methods for determining crack initiation in compression tests on low-porosity rocks. Rock Mech Rock Eng 45:607–617
- Ranjith PG, Viete DR, Chen BJ, Perera MSA (2012) Transformation plasticity the effect of temperature on the mechanical behavior of Hawkesbury sandstone at atmospheric pressure. Eng Geol 151:120–127
- Shao SS, Ranjith PG, Wasantha PLP, Chen BK (2015) Experimental and numerical studies on the mechanical behaviour of Australian Strathbogie granite at high temperatures: an application to geothermal energy. Geothermics 54:96–108
- Song SB, Liu JF, Yang DS, Ni HY, Huang BX, Zhang K, Mao XB (2019) Pore structure characterization and permeability prediction of coal samples based on SEM images. J Nat Gas Sci Engin 67:160–171
- Su HJ, Jing HW, Mao XB, Zhao HH, Yin Q, Wang C (2015) Size effect of sandstone after high temperature under uniaxial compression. J Cent South Univ 22:1901–1908
- Su HJ, Jing HW, Yin Q, Yu LY, Wang YC, Wu XJ (2017) Strength and deformation behaviors of veined marble specimens after vacuum heat treatment under conventional triaxial compression. Acta Mech Sinica-Prc 33:886–898
- Sun Q, Zhang WQ, Su T, Zhu S (2016) Variation of wave velocity and porosity of sandstone after high temperature heating. Acta Geophys 64:633–648
- Tembe S, Baud P, Wong TF (2008) Stress conditions for the propagation of discrete compaction bands in porous sandstone. J Geophys Res 113:B09409
- Tiskatine R, Eddemani A, Gourdo L, Abnay B, Ihlal A, Aharoune A, Bouirden L (2016) Experimental evaluation of thermo-mechanical performances of candidate rocks for use in high temperature thermal storage. Appl Energy 171:243–255
- Tullis J, Yund RA (1977) Experimental deformation of dry westerly granite. J Geophys Res 82:5705–5718
- Wawersik WR, Fairhurst C (1970) A study of brittle rock fracture in laboratory compression experiments. Int J Rock Mech Min Sci Geomech Abstr 7:561–575
- Wu G, Wang Y, Swift G, Chen J (2013) Laboratory investigation of the effects of temperature on the mechanical properties of sandstone. Geotech Geol Eng 31:809–816
- Xu XL, Zhang ZZ (2016) Fractal characteristics of rock fracture surface under triaxial compression after high temperature. Adv Mater Sci Eng 3:1–10
- Yang SQ, Jing HW, Li YS, Han LJ (2011) Experimental investigation on mechanical behavior of coarse marble under six different loading paths. Exp Mech 51:315–334
- Yang SQ, Jing HW, Wang SY (2012) Experimental investigation on the strength, deformability, failure behavior and acoustic emission locations of red sandstone under triaxial compression. Rock Mech Rock Eng 45:583–606
- Yang SQ, Ranjith PG, Jing HW, Tian WL, Ju Y (2017) An experimental investigation on thermal damage and failure mechanical behavior of granite after exposure to different high temperature treatments. Geothermics 65:180–197
- Zhang LY, Mao XB, Lu AH (2009) Experimental study on the mechanical properties of rocks at high temperature. Sci China Technol Sc 52:641–646
- Zhang WQ, Sun Q (2018) Identification of primary mineral elements and macroscopic parameters in thermal damage process of limestone with canonical correlation analysis. Rock Mech Rock Eng 51: 1287–1292