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Experimental study on mechanical properties of granite after reaction with supercritical carbon dioxide at high temperature and high pressure

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Experimental study on mechanical properties of granite after reaction with supercritical carbon dioxide at high temperature and high pressure

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Abstract: In order to study the effect of supercritical carbon dioxide (ScCO₂) on the mechanical properties of granite located in and near the core of the CO₂-based enhanced geothermal system (EGS) region, fluid–rock interaction experiments were conducted at 210, 240 and 270 °C. Three test conditions were used: (1) ScCO₂ and dry granite; (2) ScCO₂, water vapor and granite; and (3) ScCO₂ and granite soaked in water for 24 h. The P-wave velocity, uniaxial compressive strength (UCS), and Young's modulus of all ScCO₂-treated granite samples and one untreated granite sample were obtained by carrying out the wave velocity tests and uniaxial compression tests. The wave velocity tests showed that the P-wave velocities of all ScCO₂-treated granite samples were reduced compared to that of the untreated sample. The uniaxial compression test showed that the UCS and Young's modulus were almost not affected. From the failure mode, it can be seen that the untreated granite more likely presented the brittle tensile failure, while the treated sample showed more likely shear failure. As the temperature increased, the failure mode became more and more close to shear failure. Experimental results showed that the ScCO₂ induced slight damage to the granite under dry or a little water condition, causing a slight decrease in the brittleness, and a small increase in the plasticity. The P-wave velocity decreased slightly and the impact on the granite strength can be negligible. Therefore, the interaction of CO₂–rock will not cause obvious effect on the mechanical properties of granite located in and near the core of the CO₂–EGS region.

Keywords: CO₂; enhanced geothermal system (EGS); granite; high temperature and high pressure; mechanical property

1 Introduction

In the past decade, the world energy crisis and environmental problems are becoming more and more serious. The development of new and environmentally friendly alternative energy sources with large reserves is an important way to mitigate environmental problems and ensure energy supply^[1]. As one kind of clean energy, the geothermal energy characterized by stable operation and wide distribution has been gained more and more attention worldwide^[2]. In the past four decades, the scientific community and industry have been working on the development of enhanced geothermal system (EGS). The traditional EGS projects generally use water as the heat transfer fluid^[3–6]. Brown (2000) first proposed that the supercritical carbon dioxide (ScCO₂) can be used not only as the fracturing fluid in the hydraulic stimulation, but also as the heat transfer fluid in the EGS for the geothermal energy production. Compared with the traditional water-based EGS, the ScCO₂-based EGS has significant advantages^[7]. Meanwhile, the CO₂–EGS project can store the CO₂ in the subsurface geological formations, which is of great significance to mitigate the global warming effect^[7]. However, the variation of physico-mechanical properties of rock after reacting with ScCO₂ fluid in the ScCO₂–EGS has not been understood yet, and the related experimental research

is still limited^[8].

In the supercritical state, the volumetric mass of liquid CO₂ is the same as that of the saturated gaseous CO₂, thus the interface between liquid and gas disappears. The state of ScCO₂ is between gas and liquid at this moment, and it has the dual characteristics of gas and liquid. The density of ScCO₂ is close to that of liquid CO₂, and the viscosity is close to that of gaseous CO₂. The diffusion coefficient of ScCO₂ is nearly 100 times that of liquid CO₂^[8]. When ScCO₂ is used as the heat transfer fluid of EGS, the long-term ScCO₂–water–rock interaction may occur^[9], which will further change the physical, chemical and mechanical properties of the rock. As shown in Fig.1, the CO₂–EGS reservoir is mainly composed of three regions from the inside to the outside^[10]: (1) The central area (zone 1) is the main seepage and heat transfer area of CO₂, where all free water has been displaced by continuously circulating ScCO₂, and only the single phase of ScCO₂ exists in the reservoir. (2) The middle area (zone 2) is not the main heat exchange area of ScCO₂, where the ScCO₂ does not completely replace all the initial water, thus the reservoir fluid is composed of a small amount of water and ScCO₂. (3) In the peripheral area (zone 3), a small amount of ScCO₂ diffuses into this area, but the circulation of ScCO₂ does not form, thus the reservoir fluid consists of the water with a small amount of CO₂ dissolved^[10]. Numerous

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scholars around the world have conducted extensive research on the interaction between CO₂–water–rock in different regions of CO₂–EGS reservoirs through laboratory and field tests and establishment of reactive solute transport numerical models. In zone 1, the CO₂ in the anhydrous state is not an ionic solvent, hence there is a small possibility for mineral dissolution and precipitation^[7]. In zones 2 and 3, different degrees of reaction among ScCO₂–water–rock will lead to the dissolution of minerals such as chlorite and feldspar, and the precipitation of kaolinite and secondary carbonate minerals. When the temperature range is 200–300 °C, the dissolution rate of the original minerals is lower than the formation rate of new minerals. The dissolved CO₂ in zones 2 and 3 can be fixed in the rock by forming secondary minerals^[11–13].

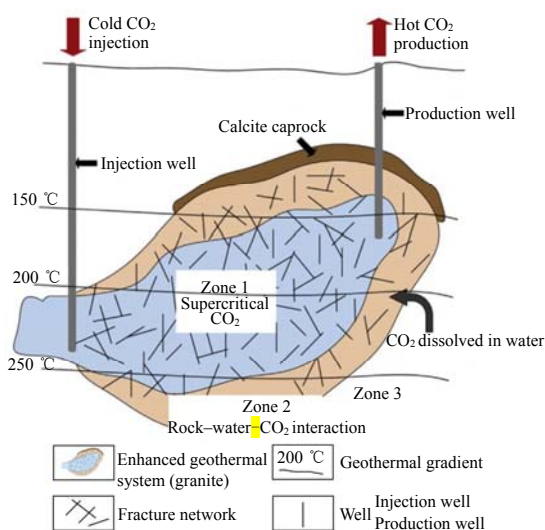


Fig. 1 Schematic diagram of zones in the CO₂-EGS reservoir^[10]

Under high temperature and high pressure conditions, ScCO₂–water–rock reactions occur in the presence of sufficient water, and the mechanical properties of the rock will change after the reaction. The ScCO₂–water–rock reaction can cause the dissolution of cements, fillings and granular minerals in the rock, resulting in the enlargement of rock pores, the destruction of cements, the loosening of structures, and even the generation of micro-cracks. The rock strength is greatly reduced accordingly^[14–15]. For example, when the shale sample is soaked into the solution with 20% NaCl and ScCO₂ at 40 °C for 30 d, the uniaxial compressive strength (UCS) and elastic modulus of the shale decreased by 70.47% and 62.45%, respectively^[16]. When the granite samples are reacted with water at temperatures of 100, 200 and 300 °C with the confining pressure of 30 MPa for 6 months, the triaxial failure strengths of granite samples were reduced by 3.83%, 2.59% and 1.44%, respectively^[17]. The granite samples prepared for the triaxial compression tests are treated in three conditions (i.e. no fluid injection, water injection, and CO₂ injection). It can be seen that both water injection and CO₂ injection reduce the strength and elastic modulus of granite^[18].

It implies that the ScCO₂–water–rock reaction can reduce the rock strength to a certain extent in the peripheral region of EGS in the presence of sufficient water.

The research into the effect of the interaction of heat transfer fluid and rock in CO₂–EGS reservoirs on rock mechanical properties mainly considers the condition with sufficient water (zone 3). But there are few studies on the changes of rock mechanical properties in the absence of water (zone 1) or with only a small amount of water (zone 2). The previous research only inferred that the possibility of mineral dissolution and precipitation is low due to the fact that CO₂ is not an ionic solvent in an anhydrous state, and the ion content is very low in the case of a small amount of water. However, the direct interactions between ScCO₂ and rock under the above two conditions were not investigated experimentally to obtain accurate results. In view of the shortcomings of previous studies, the fluid–rock interaction in the zones 1 and 2 of the CO₂–EGS reservoir is of great significance to the heat recovery efficiency and working life of the reservoir. Therefore, in this paper, the hot dry rock in zones 1 and 2 is studied, and the high temperature and high pressure reaction vessel is used to carry out experiments of the ScCO₂–water–granite reactions at temperatures of 210, 240 and 270 °C. The mechanism and variation law of mechanical properties of granite samples after reactions are discussed.

2 Experimental methods

The granite samples taken from Yueyang, Hunan Province, China were used in the experiments. They have good macroscopic homogeneity and no obvious defects. The granite samples are mainly composed of quartz (SiO₂), albite (NaAlSi₃O₈), potassium feldspar (KAlSi₃O₈), biotite (K(Fe, Mg)₃AlSi₃O₁₀(OH)₂) and a small amount of anorthite (CaAl₂Si₂O₈). The rock samples were processed into the size of $\phi 50 \text{ mm} \times 100 \text{ mm}$. The test is divided into two parts: (1) the interaction of granite and fluid under different temperature and fluid conditions is carried out; and (2) the mechanical properties of the granite samples after different types of chemical interactions are tested.

During the fluid–rock interaction process, no mass is input or output. The test system designed in this paper is mainly composed of three parts: (1) CO₂ injection system, which injects CO₂ into the reactor until the pressure in the reactor reaches the designed pressure; (2) reactor is the high temperature and high pressure reaction vessel, which has the heating function; and (3) monitoring system, which can record and regulate the temperature and pressure values in the reactor in real time. The schematic diagram of the whole system is shown in Fig.2. The working pressure of the reactor used in the test is 0–40 MPa, the working temperature ranges from the room temperature to 350 °C, and the inner size of the reactor is $\phi 75 \text{ mm} \times 160 \text{ mm}$. The reactor is heated by a heating steel jacket. In order to prevent the overpressure during the heating process in the reactor, the reactor is equipped with a pressure relief valve and the pressure is set at 40 MPa, i.e. the pressure

is automatically released when the inner pressure of the reactor exceeds 40 MPa. The stirring speed of the magnetic stirrer is adjustable from 0 to 300 rad/min.

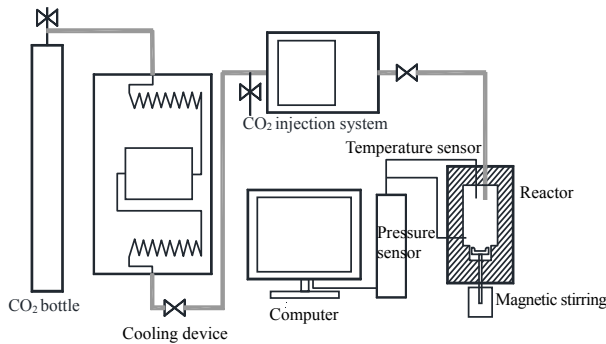


Fig. 2 Experimental system for fluid–rock interaction under high temperature and high pressure

The test process is described as follows: at first, the granite samples are placed in the material basket of the reactor and the reactor is covered tightly. Then the reactor is heated until the designed temperature is reached. Keep this temperature for more than 1 h to ensure that the rock sample is heated to the test temperature. Turn on the CO₂ injection system to inject CO₂ into the reactor to ensure that it is filled with CO₂. Increase the injection pressure gradually so that the pressure in the reactor reaches the set value. When the temperature and pressure in the reactor are higher than 31.06 °C and 7.39 MPa, respectively, the CO₂ in the

reactor is at the supercritical state and the ScCO₂–rock reaction starts. During the experiment, CO₂ is replenished in time to offset the consumption caused by the reaction or the effect of pressure reduction.

Three experimental conditions are designed in this work, as shown in Fig.3. The details of the three test conditions are described as follows:

(1) The interaction of dry granite samples with ScCO₂ is regarded as the first reaction condition, which is used to simulate the water-free zone 1 in the CO₂–EGS system, as shown in Fig.3(a).

(2) When ScCO₂ interacts with dry granite samples, a small amount of water is placed at the bottom of the reactor simultaneously, which is acted as the second condition, as shown in Fig.3(b). Under this condition, although the water does not directly contact with the granite sample, the water vapor will form after stirring with the electromagnetic stirrer and then it will be mixed with ScCO₂ to interact with the rock, thus it can be used to simulate the zone 2 reservoir that contains a small amount of water in the CO₂–EGS system.

(3) The interaction between the granite sample soaked in distilled water and ScCO₂ is regarded as the third condition. There is free water inside the soaked granite sample. But the amount of water participating in the chemical reaction is limited, because the granite is relatively dense and has low porosity. Therefore, it can also be used to simulate the zone 2 containing a small amount of water in the CO₂–EGS system, as shown in Fig.3(c).

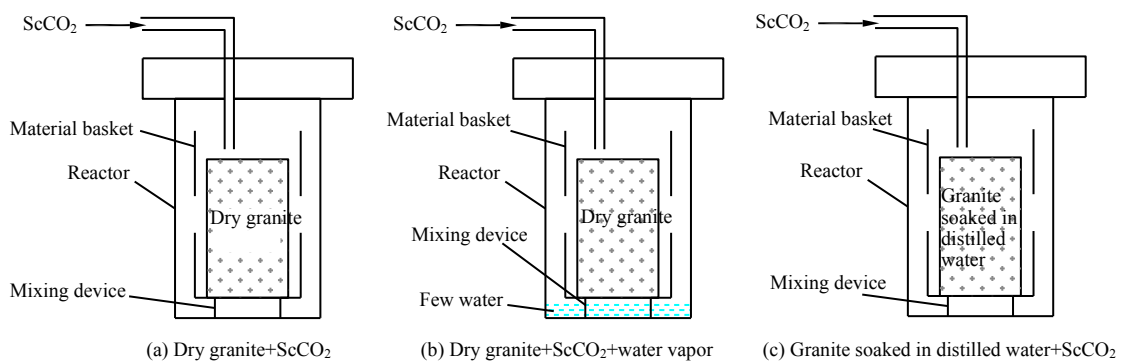


Fig. 3 Three different test conditions for ScCO₂–rock interaction at high temperature and high pressure

To sum up, we design a test condition for zone 1 (the first condition), and two test conditions for zone 2: the water vapor is mixed with ScCO₂ (the second condition), and the water is attached to the granite (the third condition).

In the second and third conditions, the CO₂ injected into the reactor is first dissolved in water. With the continuous injection of CO₂, the water in the reactor is saturated with CO₂ during the test. The density of the solution saturated with CO₂ is higher than that of ScCO₂. Hence in the second reaction condition, the aqueous solution saturated with CO₂ is distributed in the lower part of the reactor, it does not directly contact with the granite, and the ScCO₂ fluid is located at the upper part of the reactor.

On the basis of the above three test conditions, three test temperatures are set for each condition, i.e. 210, 240 and 270 °C. The pressure in the reactor is set to 37 MPa, and the reaction period is 120 h. Meanwhile, the reference sample, i.e. a granite sample without any heating and reaction treatment, is set up. The different experimental conditions designed in this paper are listed in Table 1.

After interaction with ScCO₂, the granite sample is cooled down to room temperature. Then the rock sample is taken out after the pressure is released, and the sonic wave velocity test is carried out. Afterwards, the conventional uniaxial compression test is performed on the above 10 rock samples using INSTRON 1342 mechanical testing system. During the loading process, the parameters

such as axial force and axial displacement are recorded.

Table 1 Experimental conditions of ScCO₂ and granite interaction

Sample No.	Temperature /°C	Initial pressure /MPa	Reaction period /h	Experimental conditions
#1	210	37	120	Dry granite+CO ₂ (See Fig. 3(a))
#2	240			
#3	270			
#4	210	37	120	Water vapor+CO ₂ +dry granite (See Fig.3(b))
#5	240			
#6	270			
#7	210	37	120	Granite soaked into distilled water for 48 h+CO ₂ (see Fig.3(c))
#8	240			
#9	270			
#10	—	—	—	Reference granite sample, without reaction with ScCO ₂

3 Results and discussion

3.1 Strength of granite samples

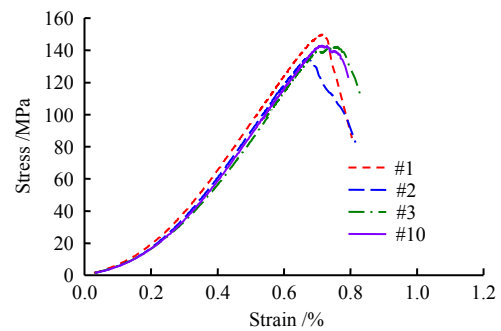
Figure 4 shows the uniaxial compressive stress–strain curves of granite samples under different test conditions. It can be seen that each granite sample has experienced four typical stages before reaching the peak strength: the compression and closure of micro-cracks, the elastic deformation, the stable expansion of cracks and the rapid expansion of cracks.

At the initial stage, the micro-cracks in the granite samples are compressed and closed under the action of external force, and the axial deformation of the sample is relatively large. When the cracks are completely closed, the axial and radial strains of the granite samples approximately change linearly with the increase in stress. Afterwards, the internal cracks gradually initiate and develop steadily. When the stress nearly reaches the peak strength, the expansion rate of the internal cracks increases, and the axial stress–strain curve does not show obvious yielding, but the strain rate in the radial direction gradually increases. Finally, the granite sample reaches the peak failure and enters the post-peak stage^[19–20].

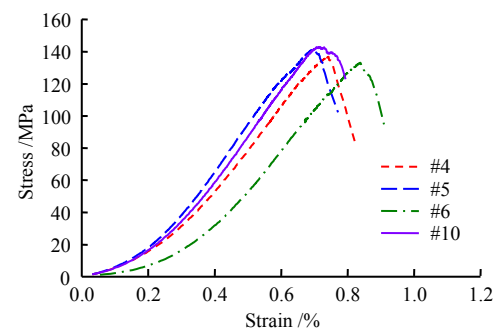
Figure 4(a) shows the stress–strain curves of the granite samples #1, #2 and #3 under the first test condition. It can be seen that the slopes of the stress–strain curves of the three granite samples are similar to those of the sample #10 at the elastic stage. The curve shapes of these granite samples are similar, and the variation range of the peak stress is within 5%. This shows that the elastoplasticity of granite does not change after the interaction with ScCO₂ in the absence of water, and the stress–strain characteristics of the granite basically do not change with the temperature in the range of 210–270 °C.

Figure 4(b) shows the stress–strain curves of the granite samples #4, #5 and #6 under the second test condition. The slope of the elastic stage of these three rock samples gradually decreases with the increase in temperature. The peak stress of the rock sample gradually decreases by 7% at most. The slope of the straight line segment of the rock stress–strain curve at the three

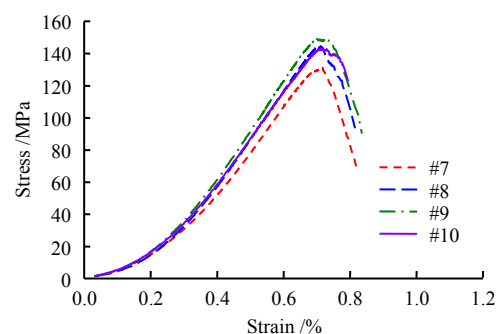
temperatures is basically unchanged, i.e. the elasticity of the granite does not change significantly. The strain corresponding to the peak stress increases with the increase in temperature, indicating that the plasticity of the granite increases gradually. The stress–strain curves of the rock samples #4, #5 and #10 are similar, and there is a significant lag at the compression stage of micro-cracks for the rock sample #6. It is speculated that the effect of water vapor and ScCO₂ on granite is much stronger at 270 °C than that at 210 and 240 °C.



(a) Dry granite+ScCO₂



(b) Water vapor+ScCO₂+dry granite



(c) Granite soaked in water after 48 h+ScCO₂

Fig. 4 Stress and strain curves of granite after interaction with ScCO₂ under different test conditions

Figure 4(c) shows the stress–strain curves of the rock samples #7, #8 and #9 under the third test condition. These three rock samples have similar slopes as the rock sample #10 at the elastic stage, the peak stress variation range is within 8%, and the elastoplasticity of the rock samples has no obvious variation. It is inferred that the amount of water involved in the chemical reaction is small under this condition, and the reaction mainly occurs on the surface of the rock sample, thus the fluid–rock reaction has little influence

on the deformation characteristics of the granite.

Table 2 shows the uniaxial compression and wave velocity test results of each granite sample (see also in Fig.5). According to the mechanical parameters of granite before and after the fluid–rock interaction test, it shows that after the reaction of dry granite and ScCO₂, the maximum variation ranges of the UCS and elastic modulus of granite are about 5% and 4%, respectively. Under the condition of dry rock sample, water vapor

and ScCO₂, the maximum variation ranges of the UCS and elastic modulus of granite are about 7% and 9%, respectively. The maximum variation ranges of the UCS and elastic modulus of granite that is soaked in the distilled water are about 8% and 4%, respectively. These changes are basically within the experimental error and the distribution of general experimental values, i.e. the UCS and elastic modulus of the granite samples are basically unchanged.

Table 2 Mechanical properties of granite before and after chemical reaction with fluid

No.	Temperature /°C	Pressure /MPa	Experimental conditions	P-wave velocity /($m \cdot s^{-1}$)	UCS /MPa	Elastic modulus /GPa
#1	210		Dry granite+CO ₂	3 067.077	149.583	29.27
#2	240	37		2 909.565	135.243	29.13
#3	270			2 834.085	142.214	27.86
#4	210		Water vapor+ CO ₂ +dry granite	2 926.377	136.891	26.96
#5	240	37		2 949.412	141.088	30.07
#6	270			2 682.667	133.146	26.23
#7	210		Granite soaked in distilled water after 48 h+CO ₂	3 130.000	131.254	27.85
#8	240	37		3 127.500	144.523	29.91
#9	270			3 033.330	149.234	29.80
#10	—	—	Reference granite sample without any treatment	3 232.580	142.846	28.93

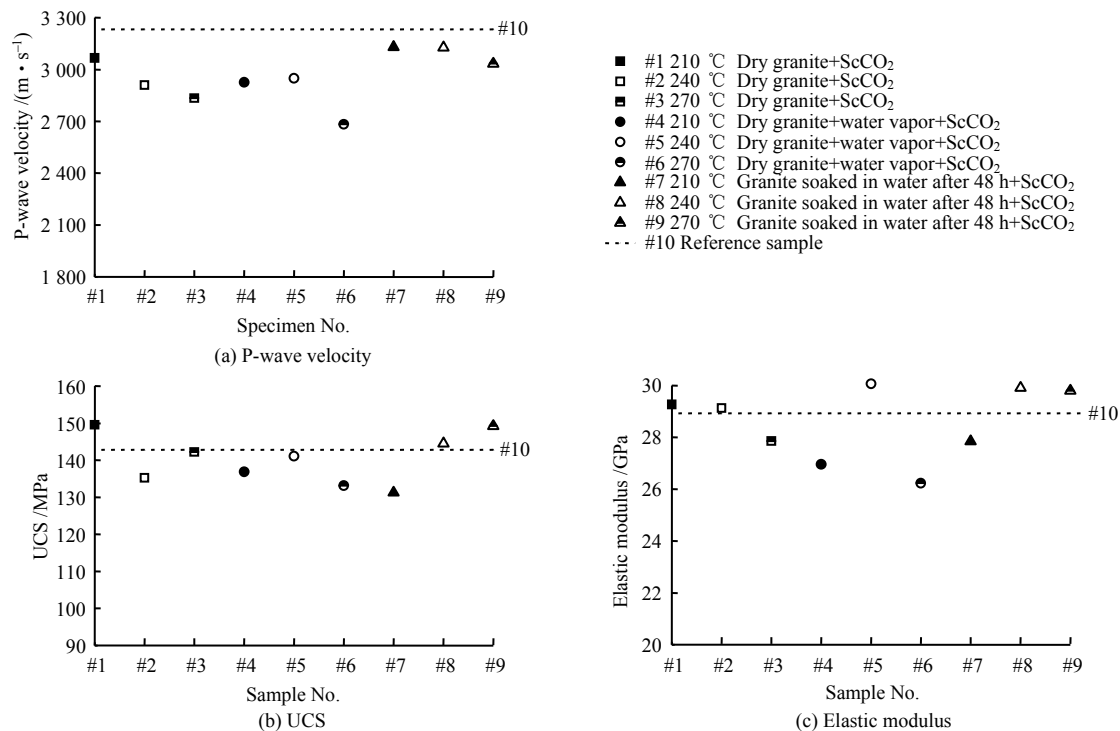


Fig. 5 Mechanical properties of granite under different test conditions

Under the above three test conditions, the P-wave velocity of the granite samples all decreases. However, it is difficult to find the regularity considering the measurement error and the small decrease value of P-wave velocity. The experimental results show that the granite is slightly damaged after interaction with the solutions presented in this paper, the integrity and rigidity of the granite are deteriorated, resulting in a small decrease in the P-wave velocity, but their impacts on the mechanical strength of granite can be ignored.

3.2 Macroscopic failure morphology of granite

There are two possible failure modes for rocks under uniaxial compression: shear failure and tensile

failure. In the shear failure mode, the failure surface is oblique to the axial direction and cuts thorough the cylindrical rock sample. The tensile failure is characterized as the tensile shedding failure of the outer parts of the cylindrical rock sample, while its central part is not completely destroyed.

Figure 6 shows the failure mode of the sample in the experiments and its schematic diagram. It can be seen that the failure mode of granite under different test conditions varies. The rock sample #10 without any treatment mainly presents tensile failure and brittleness characteristics. The failure mode of the dry granite after the action of ScCO₂ is analyzed. When the

reaction temperature is 210 °C, the failure mode is dominated by tensile failure. When the temperature increases to 240 and 270 °C, the failure mode gradually changes into shear failure, and a large amount of debris is generated during the failure process. The dry rock samples also have similar damage characteristics after reacting with water vapor and ScCO₂. Under different experimental temperatures (210, 240 and 270 °C), the failure mode of the granite samples that are soaked in distilled water for 48 h and ScCO₂ is mainly shear

failure. The failure mode of the granite samples in the range of 210–270 °C does not change significantly with temperature. Compared with the reference sample, it shows that the temperature and mixed fluid have certain influences on the failure mode of granite. The reference granite is characterized as the tensile failure and strong brittleness, while the granite samples that are reacted with the fluid is mainly characterized as the shear failure with decreased brittleness and increased toughness.

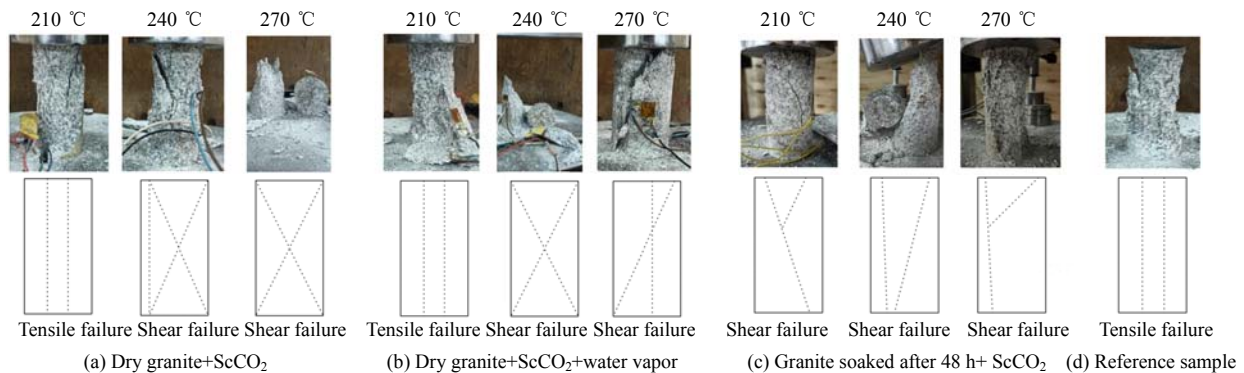


Fig. 6 Failure mode of granite and simplified diagram

3.3 Discussion

The interaction process and mechanism of ScCO₂–water–rock are relatively complex and have not been clearly understood. In the presence of sufficient water, the ScCO₂–water–rock reaction usually includes three processes: carbonation of water, mineral dissolution and mineral precipitation. This study focuses on the interaction between dry granite and pure ScCO₂, and the interaction between granite and ScCO₂ in the presence of a small amount of water. The test results show that the mechanical properties of the granite have no significant change under these two conditions. There is no mineral dissolution and precipitation phenomenon on the rock surface by observation, i.e. the reaction between ScCO₂ and the granite is very weak. The specific reason can be speculated based on previous studies: in zone 1, since CO₂ in anhydrous state is not an ionic solvent, there is small possibility of causing mineral dissolution and precipitation in granite^[7]. In zone 2, ScCO₂–water–rock occurs to a certain extent, resulting in dissolution of minerals such as quartz and feldspar and the precipitation of kaolinite and secondary carbonate minerals. However, since the amount of water is very small, the reaction effect is very weak, and the effect on the rock properties is negligible.

Compared with ScCO₂, when water is used as the heat transfer fluid, the reaction between water and rock may change the rock mechanical properties. The natural granite with the UCS of 142.3 MPa is soaked in pure water and slowly reacts with the pure water, respectively. After 4855 h of the chemical reaction, the mechanical damage values are 10.68% and 12.09%, respectively^[21]. In the presence of sufficient water, quartz dissolves and reacts with the water rather than with the CO₂^[22–23]:



The dissolution and precipitation process of minerals is one of the important problems of water-based EGS, which reduces the permeability and porosity of the reservoir, making the fracture network eventually closed and the exhaustion of thermal recovery^[24]. There is no water or only a small amount of water in the heat exchange region of ScCO₂–EGS and its adjacent areas. Therefore, the dissolution and precipitation of minerals are very weak, which is helpful for the long-term heat transfer effect of the EGS reservoir.

4 Conclusions

In this paper, ScCO₂–water–granite interaction experiments at 210, 240 and 270 °C are carried out to simulate the geological conditions in the core zone 1 without water and the peripheral zone 2 with only a small amount of water in the EGS rock mass. The reacted granite samples are then subjected to sonic tests and uniaxial compression tests. Based on the experimental results, main conclusions are drawn as follows:

(1) After reacting with the ScCO₂ fluid, the variation range of the strength parameters of the dry granite, such as UCS, elastic modulus and P-wave velocity, is within 9% on average. There is no obvious deterioration phenomenon and change law.

(2) When the dry granite reacts with the ScCO₂ in the presence of water vapor, the UCS and elastic modulus of the granite also change insignificantly, and the P-wave velocity decreases slightly. It means that the mixed fluid of limited amount of water vapor and ScCO₂ has a slight effect on granite under high temperature and high pressure conditions.

(3) When the wet granite samples react with the ScCO_2 , the changes of UCS, elastic modulus and P-wave velocity are still small, meaning that the mechanical properties of granite samples are not significantly affected in the zone 2 of the EGS reservoir.

(4) Under the above three test conditions, the uniaxial compression failure of granite samples all shows the decreased brittleness and increased toughness.

(5) In the core heat exchange region of CO_2 -EGS and adjacent areas, the effect of CO_2 on the rock is very weak because there is no water or very limited amount of water. The dissolution and precipitation of minerals are very weak, and the variation of mechanical properties of granite samples can be ignored. The strength and opening of rock fractures cannot be affected, which is helpful for keeping the connectivity of the fracture network of geothermal reservoirs in the long term, improving the heat recovery efficiency and the working life of EGS.

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