Research Article

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Effect of hydrothermal coupling on energy evolution, damage, and microscopic characteristics of sandstone

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Abstract: Heat–cool (H–C) cycle is a serious natural weathering mechanism for rock engineering in temperate desert climate; meanwhile, engineering rocks usually involve responses to impact loads arising from blasting operation, mechanized construction, and seismic oscillation. Considering the universality and destructiveness of rock failure caused by H–C cycle weathering coupled with dynamic loading, split Hopkinson pressure bar tests were conducted for sandstone with various H–C cycles. Additionally, hydrothermal coupled damage (D) was defined based on variation of total import strain energy. Energy evolution, damage, and microscopic characteristics of sandstone after different H–C cycles were studied. Finally, the microcosmic structure changes of sandstone after various H–C cycles are compared by means of scanning electron microscopy (SEM) and energy dispersive spectrometer (EDS) technology. Results show that decreasing rate of total import strain energy in high temperature group is significantly larger compared with that in low temperature group and moderate temperature groups. Repeated H–C cycles produce the thermal stress at

the mineral boundary constantly and fracture along the boundary of the mineral particle according to the SEM and EDS results.

Keywords: split Hopkinson pressure bar, hydrothermal coupling, energy evolution, heat–cool cycles, damage

1 Introduction

Diurnal temperature difference and annual temperature difference is enormous in temperate desert climate. Meteorological records indicate that rock materials will experience a typical repeated heat–cool process, which is attributed to the high low temperature cycling in summer and winter. Rock materials degradation is a typical process of rock fracture and mineral metamorphism under the combined effects of radiation, biology, and geology [1–4]. Rock materials are commonly applied building materials in construction engineering, which always suffers from repeated heat–cool (H–C) cycles weathering. Additionally, engineering rock typically involves responses to dynamic impact loads arising from blasting excavation, mechanized construction, and seismic oscillation [5]. The study of mechanical properties of rock materials under harsh weathering conditions has engineering significance to the design, construction, and application of rock materials in temperate desert climate regions. Previous investigation demonstrated that water content and freezing temperature significantly affect the damage degree of rock weathering in H–C cycles. Rock is a heterogeneous composite porous material, which contains joints, cracks, and natural flaws [6–8]. Rock is repeatedly eroded by water and the clay mineral in rock is hydrophilic, which weakens the cohesion and slip each other, leading to the weakening of the cementation between mineral particles inside rock materials [9,10]. Hence, water inside rock undergoes water phase transition of solid (ice)–liquid (liquid water)–gas (steam) with

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temperature changing coupled with water [11]. When temperature is below freezing point, 9% volume expansion for pore water occurs inside rock with solid–liquid phase change, resulting in the damage of pore structure inside rock; in contrast, when temperature is above freezing point, the freezing water thaws and migrates between pores inducing reduced cohesion between rock particles. The frequent freeze–thaw (F–T) of water expands the original fissures and voids inside the rock and induces new microcracks, which causes great damage to rock engineering [12–14]. Temperature increment during H–C cycles leads to secondary thermal stress in rock due to various coefficients for different mineral particles. The main mechanism of H–C cycles on rock is the loss of water and inner structure damage caused by thermal reactions [15–17]. By summarizing previous theoretical and experimental investigations it can be observed that recent developments in the field of strength and deformation characteristics of rock after F–T and thermal shock (TS) cycles have led to an increased interest. The main representative study results are as follows: Yavuz [18] performed 10 F–T cycles and 50 TS cycles on andesites, and test results found that with the increase of cycle numbers, the velocity of P-wave, compressive strength, and hardness of andesites specimen decrease, while both the porosity and water absorption increase. Demirdag [19] compared the physical and mechanical properties of filled and unfilled travertine after different F–T and TS cycles (e.g., 10, 20, 30, 40, and 50 cycles). Investigation results showed that both F–T and TS cycles affect the rock weathering degree on the mechanical resistance properties. Ghobadi and Babazadeh [20] studied the differences in the accelerated weathering processes of sandstone among F–T, salt crystallization (SC), H–C, and heating, cooling, and wetting (H–C–W) cycles. Wang and Xu [6] tested the quasi-static and dynamic splitting tensile of red sandstone after 10, 20, 30, and 40 TS cycles. Test results demonstrated that the antigrain fracture increased with the increase of strain rate based on the scanning electron microscopy (SEM) results, and TS cycle reduced the adhesion of cement and the bond between particles, reducing the splitting tensile strength of red sandstone after TS cycle. The mechanical response of marble under coupled high temperature and dynamic loads was investigated using SHPB device, and the temperature threshold was observed based on the changes of stress–strain curves, peak stress, peak strain, and failure mode of marble [21], in addition, the effects of impact load rate and high temperature on strain rate were also analyzed [22,23], and test results indicated that under

the same load rate, the strain rate of rock showed an obvious increase trend with the increase of high temperature.

By summarizing the theoretical and experimental studies achievements of predecessors, it can be found that researches, in terms of the physical and mechanical changes of rock materials after F–T and TS cycles, mainly concentrate on changes in physical and mechanical properties. However, research has consistently shown that its energy dissipation and damage mechanism have not attained an adequate understanding. Moreover, study on the influence of cycle temperature on the dynamic mechanical property of rock materials after H–C cycles is still insufficient. In this research, the influence of treatment temperature and cycle number on physical property (i.e., P-wave velocity, density, and porosity), dynamic energy evolution, damage, and microscopic properties of rock during the weathering process. SEM and energy dispersive spectrometer (EDS) technique were used to detect microscopic damage of sandstone, and the damage is defined according to the variation of total impact strain energy. Finally, the relationships between energy characteristic index and cycling number are also discussed.

2 Test materials and methods

2.1 Sample preparation

The fine-grained sandstone was collected in the Zhujidong coalmine located in Huainan city, Anhui Province, China. Samples with similar P-wave velocity were selected and cylindrical specimens with diameters of 50 mm and heights of 25 mm were obtained by boring, cutting, and polishing the slate chunks according to the suggested methods and standard requirements of ISRM [24].

According to the treatment temperature in H–C cycles, the test sandstone samples were divided into four groups: high temperature (H-T) group from -20°C to 400°C , moderate temperature (M-T) group from -20°C to 100°C , low temperature (L-T) group from -20°C to 50°C , and the control group. For L-T group or M-T group, four experimented sets are prepared according to 10, 20, 30, and 40 H–C cycles. Because of the fast deterioration rate of the high temperature group, three experimented H–C cycles (4, 8, and 12) were set. Figure 1 is the scheme of the testing procedure. There are five specimens in each experimented set to fulfill the repeatability conditions.

Figure 1: Scheme of the testing procedure.

2.2 H–C cycling and testing equipment

In this test, SX-5-12 box-type resistance furnace, which could heat rock specimen to 1,200°C, was adopted as the heating device, which was composed of control box and electric furnace. Sandstone specimens were heated at a rate of 6°C/min to desired temperature. The low temperature adopted the freezing temperature test box. The temperature of test chambers drop from 20°C to –20°C need 40 min.

The experiment samples were immersed in water for 12 h, and then put into freezing temperature test box at –20°C for 6 h, after that, put samples in a high temperature box at a predetermined temperature heating for 6 h. The above test process was treated as one H–C cycle and lasts for 24 h. The basic physical properties (mass, volume, and ultrasonic P-wave velocity) of sandstone specimens before and after cycling were tested and shown in Table 1.

Dynamic impact loading tests of sandstone were carried out involving the SHPB system with a diameter of 50 mm under 0.4 MPa impact air pressure. In SHPB test, sandstone specimens after different H–C cycle conditions are placed between incident and transmitted bars, and two strain gauges are mounted on incident and transmitted bar to collect the stress waves, respectively. Strain acquisition instrument is adopted to collect the

origin strain signals. Moreover, the calculation method of dynamic stress, strain, and strain rate has been reported in the previous investigation [5]. The stress equilibrium of sandstone specimen after H–C cycle is checked and shown in Figure 2. It can be clearly noticed that sandstone specimen is basically satisfied stress equilibrium during impact process.

3 Test results and analysis

Thermodynamics laws indicate that the transmission and transformation of energy is combined with the entire failure process of material. Notably, energy dissipation reflects the evolution of the microcracks inside rock, which leads to strength weakening and failure of rock materials. Hence, investigation on the rock damage from energy perspective can denote the whole failure process under external load [25,26]. As an open system, rock specimen continuously transfers and transforms energy to the environment during the loading failure process. Environment works on the rock system, and the import energy is absorbed by rock system. As shown in Figure 3, the process of management transfer and transformation

Table 1: Test results on the basic physical properties of sandstone after H–C cycles

Test number	Number of sample group	Cycling number	Average P-wave velocity (m/s)	Average density (g/cm ³)	Average porosity (%)
1	Room temperature	0	2974.78	2.53	2.23
2	Low temperature (L-T) group	10	2835.83	2.53	2.53
3		20	2739.13	2.50	3.12
4		30	2726.04	2.50	3.33
5		40	2686.87	2.49	3.44
6	Moderate temperature (M-T) group	10	2841.87	2.51	2.93
7		20	2631.97	2.48	4.31
8		30	2554.25	2.48	4.56
9		40	2515.61	2.48	5.12
10	High temperature (H-T) group	4	1752.51	2.47	5.29
11		8	1484.20	2.45	6.70
12		12	1412.15	2.45	7.34

Figure 2: Stress equilibrium of sandstone specimen after H–C cycle.

of loaded rock system can be divided into four main forms: energy import, energy accumulation, energy dissipation, and energy liberation.

3.1 Energy evolution of rock after H–C cycles

3.1.1 Computational formula

Previous theory reveals that there is no heat exchange between rock and environment during SHPB test. Considering the deformation of one rock element under external load, the total input energy, dissipated energy, and the releasable elastic strain energy of a rock unit can be calculated based on the first law of thermodynamics [27].

$$U = U^e + U^d \quad (1)$$

where U is the total import strain energy of one rock unit generated by the external load, U^d and U^e are the dissipated energy and the releasable elastic strain energy of a rock unit, respectively. The units of U , U^d , and U^e are all MJ/m³, equivalent to the stress unit, MPa. The calculation scope of them is from initial point to residual point, in addition, the value of them under different stages is calculated from initial point to the end of the stage point.

Taking axial stress and strain into consideration [27], energy evolution equation of a rock unit in principle stress space can be expressed as

$$U = \int_0^{\varepsilon} \sigma d\varepsilon$$

$$U^e = \frac{1}{2} \sigma \varepsilon^e \quad (2)$$

$$U^d = U - U^e$$

where σ , ε , and ε^e are the axial stress, strain, and elastic strain of the rock unit, respectively.

3.1.2 Variation in total import strain energy with cycling number

The energy evolution curves of sandstone specimens after H–C cycles are obtained by calculation. The representative stress–strain curve and energy evolution curve without cycle are adopted for description, as shown in Figure 4. It is obviously noticed that the whole process of energy evolution can be divided into four stages:

Figure 3: Four main forms of energy transfer and transformation during rock impact failure.

compression stage, linear deformation stage, yield stage, and failure stage. In addition, Figure 4 also illustrates that the energy evolution curve for H-C cycles damaged sandstone specimen under impact compression process shows obvious stage characteristics. In compaction stage, with the increase of strain, the total import strain energy, both the releasable elastic strain energy and dissipation energy increases, and the rise rate increase gradually. At linear elastic deformation stage, the rise rates of total import strain energy and releasable elastic strain energy are relatively fast, while the dissipative energy is relatively slow. In yield stage, the rise rate of the released

elastic strain energy begins to slow down gradually and reaches the peak value, while both dissipation energy and total import strain energy are continuously increasing. In the failure stage, both the total import strain and dissipative energy increase continuously, while released elastic strain energy decreases. The dissipation energy increases continuously and approaches the total import strain energy.

The results show that the total import strain energy decreases with increasing cycling number for L-T, M-T, and H-T groups. The total import strain energy (U) under different test conditions is shown in Table 2.

(a)

(b)

Figure 4: Dynamic energy curves of sandstone specimens after H-C cycles: (a) diagram of the staged stress-strain curves; (b) energy evolution curve of sandstone (room temperature).

Table 2: The total import strain energy (U) under different test conditions

Test number	Number of sample group	Cycling number	Total import strain energy (U /MJ/m ³)
1	Room temperature	0	2.043
2	Low temperature	10	1.985
3	(L-T) group	20	1.649
4		30	1.525
5		40	1.371
6	Moderate	10	1.664
7	temperature (M-T)	20	1.555
8	group	30	1.244
9		40	1.160
10	High temperature	4	0.924
11	(H-T) group	8	0.697
12		12	0.599

Table 2 reveals that the total import strain energy of L-T group decreases gradually with the increase of cycling number, and values of the total import strain energy are 2.04, 1.99, 1.65, 1.53, and 1.37 MJ/m³, respectively, which are corresponding to 0, 10, 20, 30, and 40 H-C cycles. The decreased values of total import strain energy are 2.84%, 19.29%, 25.36%, and 32.89% compared with that of 0 H-C cycles, respectively. The values of total import strain energy are 2.04, 1.66, 1.56, 1.24, and 1.16 MJ/m³, decreased by 18.55%, 23.89%, 39.11%, and 43.22%, respectively, for M-T group. From the data of three groups it is found that under the same H-C cycles, the decrease rate of rock for H-T group is larger than that of other two groups, specifically, the value of U for H-T group decreases to 0.60 MJ/m³ after 12 H-T cycles. Finally, from the comparative analysis it can be noticed that with the increase of cycling number, both the total import strain energy and total dissipation energy of sandstone in the whole impact compression and failure process gradually reduce.

3.2 Relationship between energy characteristic index and cycling number in different stages

The above analysis illustrates that energy evolution process of rock under dynamic loading can be divided into four stages: compression, linear deformation, yield, and failure. Moreover, energy characteristic indexes (i.e., U , U^e , and U^d) are different with various dynamic

compression deformation and failure stages of rock. Energy distribution characteristic and damage degree are varied corresponding to four stages [26,27]. According to equation (2) the energy characteristic indexes for L-T, M-T, and H-T groups are obtained and shown in Figure 5.

Figure 5 illustrates that: (1) three energy characteristic indexes (i.e., U , U^e , and U^d) are affected by cycling number, deformation and failure stage, and treatment temperature. Additionally, similar effects of treatment temperature on energy characteristic index for three cycle groups are observed. (2) With increasing cycle numbers, energy characteristic index (i.e., U , U^e , and U^d) gradually decreases. For example, the U^d at yield stage in M-T group decreases from 1.264 to 0.695 MJ/m³ with cycle number increasing from 0 to 40. (3) Furthermore, the decrease degree is quite different under different deformation and failure stages, the decrease degree for U and U^d at failure stage exhibited the maximum value compared with the other three stages. However, the decrease degree for U^e at failure stage shows the minimum value. (4) Additionally, the treatment temperature also has significant effect on the energy characteristic index (i.e., U , U^e , and U^d) at the same deformation and failure stage.

The energy characteristic index is constantly changing and transforming into each other at different deformation and failure stages. In compaction stage, test results indicate that $U > U^e > U^d$, and the total import strain energy in this stage is converted into elastic energy. The elastic energy and dissipation energy of each specimen exist simultaneously in linear elastic deformation stage, while the total import strain energy is converted into elastic energy and dissipated. Meanwhile, the total import strain energy of sandstone begins to dissipate in this process and a small percentage is converted into elastic energy in yield stage. At failure state, dissipation energy in this stage is larger than the total import strain energy, while the elastic energy is approximately zero, indicating the elastic energy stored in pre-peak stage is released.

3.3 Hydrothermal coupling damage caused by H-C cycles

With the increase of H-C cycles number, the damage degree of sandstone gradually increases, which shows

Figure 5: Relationship between energy characteristic index and cycling number in different stages: (a) L-T group. (b) M-T group. (c) H-T group.

more easily failure under the same conditions. From the above analysis it can also be noticed that the total import strain energy gradually reduces with the increasing cycling numbers. To further describe the hydrothermal

coupling damage of rock caused by H–C cycles, the hydrothermal coupled damage (D) is defined and calculated based on the variation of total import strain energy required for the compression deformation process after different H–C cycles [27] and the function can be expressed as:

$$D = 1 - \frac{U_n}{U_0} \quad (3)$$

where D is the hydrothermal coupled damage value of sandstone after H–C cycles; U_n and U_0 are the total import strain energy of sandstone samples subjected to n and 0 H–C cycles, respectively.

According to above calculation method, fitting curves between damage of rock and cycling number are shown in Figure 6. The variation of H–C cycle damage with cycling numbers of sandstone can be found in formulas (4)–(6):

L–T group:

$$D = -1.211 \times (1 - e^{0.006n}) \quad R^2 = 0.939 \quad (4)$$

M–T group:

$$D = 0.608 \times (1 - e^{0.032n}) \quad R^2 = 0.980 \quad (5)$$

H–T group:

$$D = 0.708 \times (1 - e^{-0.365n}) \quad R^2 = 0.999 \quad (6)$$

Figure 6 and formulas (4)–(6) reveal that damage of rock specimen caused by H–C cycles gradually increase with the increase of cycling number. Under the same cycle number, rock damage for H–T group became more serious compared with that for M–T and L–T groups. After

10 H–C cycles, the damage value for H–T group is 0.69, which is much higher than that of 0.16 and 0.07 for M–T and L–T groups.

3.4 Effect of H–C cycle on microstructure changes of rock

The microstructural changes have significant effect on the variations of physical and mechanical properties of rock materials after different H–C cycles, which is beneficial to better understand the H–C damage mechanism for rock materials. H–C cycle is a complex three-field coupling damage process of the interaction of water (ice and steam), heat (temperature), and force (temperature force) in rock media. To further investigate the mechanical damage of H–C cycles on dynamic strength and energy dissipation of rock, SEM and EDS tests are conducted in this section to observe the microstructural damage of rock.

3.4.1 Thermal-water coupling damage mechanism

3.4.1.1 Water–rock interaction mechanism

Physical mechanism. Sandstone is composed of different kinds of minerals (such as albite, quartz, and calcite) [28–30]. Figure 7 shows expansion process of clay mineral. In this test, sandstone belongs to sedimentary rock, the whole structure of which is mainly clastic structure. The clay minerals, such as kaolinite, which has small particle size and high cohesion force can bond with each other and on the surface of other minerals, such as quartz, albite, and calcite. Hence, clay minerals act as binder mediums to stabilize rock structure. Additionally, kaolinite accounts for the main part of clay minerals in sandstone specimen, which shows better hydrophilic ability, and it shows hydration and expansion characteristics because of the effect by capillary force, surface hydration, and osmotic hydration [31].

It can be seen from Figure 7 that clay mineral polymer absorbs water giving rise to the expansion of volume, which produces stress on mineral particles and affects the direction of clay mineral, resulting in original equilibrium state being broken. Water molecules and hydrated ions fill the clay mineral aggregates, which weakens the cohesion and slip each other, leading to the weakening of the cementation between sandstone mineral particles [32],

Figure 6: Relationship between damage and cycling number in H–C cycles.

Figure 7: Diagram of expansion process of clay mineral polymer.

resulting in the macroscopic damage of rock after water-rock reactions.

Chemistry mechanism. According to the mineral composition of sandstone, the chemical changes that

occur between sandstone and water are mainly albite hydrolysis, calcite weight carbonation, and colloidal dispersion of quartz insoluble minerals [9]. The chemical reaction formulas are calculated and shown in (7)–(10).

Figure 8: Diagram of thermal damage mechanism in H-C cycle process.

Figure 9: Microstructure of sandstone: (a) 20°C (×2,000), SEM results. (b) 20°C, EDS results. (c) After 12 cycles in H-T group (×2,000), SEM results. (d) After 12 cycles in H-T group, EDS results. (e) After 10 cycles in L-T group (×2,000), SEM results. (f) After 10 cycles in L-T group, EDS results. (g) After 40 cycles in L-T group (×2,000), SEM results. (h) After 40 cycles in L-T group, EDS results. (i) After 10 cycles in M-T group (×2,000), SEM results. (j) After 10 cycles in M-T group, EDS results. (k) After 40 cycles in M-T group (×2,000), SEM results. (l) After 40 cycles in M-T group, EDS results.

The above reaction formulas illustrate that the materials exchange between aqueous solution and rock minerals occur continuously. Specially, the ion concentration close to the mineral particles is much larger than that in free water. Hence, large thermodynamic potential energy is generated between them, which promotes the chemical reaction.

Formulas (7)–(10) also illustrate that the chemical reaction products are easier to dissolve or decompose in water compared with chemical reactants. Hence, with the

increase of H–C cycles, more and more chemical reaction product generates, which makes rock minerals become looser and more fragile.

3.4.1.2 Thermal damage mechanism

Rock is a typical multimineral complex material; the thermal damage mechanism in H–C cycle process is shown in Figure 8. From the above figure it is noticed that in saturated process, all the pores and cracks inside rock are filled with water, and there is no thermal damage in this process. In cooling process, part of water freezes and expands about 9% of the original volume, and this expansion induces tensile stress concentration and damages the micropores. In heat process, mineral particles generate uncoordinated deformation caused by the difference in thermal-expansion coefficient [33,34], leading to the thermal force, which will damage the rock structure. Recurrent H–C cycles gradually lead to the

accumulation of damage inside rock and further weaken its mechanical properties.

3.4.2 Microstructure characteristic of rock after H–C cycle

During SEM observation, the most crucial issue is how to distinguish the cracks and pores between mineral particles. Hence, EDS technology is adopted to further study the damage characteristics (i.e., cracks and pores). Typical microstructure characteristics of sandstone specimen after different H–C cycles are displayed in Figure 9.

Figure 9 illustrates that at 20°C (room temperature), only very few initial pores and microcracks can be observed in the rock (Figure 9(a) and (b)). In the L–T group, when sandstone specimen experiences 10 H–C cycles, some initial microcracks and pores propagation are induced by the freezing expansion and thermal expansion of mineral particles which improves the microstructure of sandstone, as shown in Figure 9(e) and (f); after 40 H–C cycles, due to the disharmony of thermal deformation between minerals particles, the thermal stress concentrates at the edge of the mineral particle (Figure 9(g) and (h)); after repeated H–C cycles, the thermal stress at the mineral boundary is constantly produced, leading to the fatigue rupture along the boundary of the mineral particle, which indicates that the microstructure inside rock is weakened by the thermal stress. The variation of microstructure of rock after H–C cycles in M–T group is similar to that in L–T group, the cracks propagate and pores expand with increasing H–C cycles. Additionally, new pores and cracks between the mineral particles begin to be concentrated and interlinked (Figure 9(i)–(l)). In contrast, by observation and comparison of EDS results under room temperature of L–T group, M–T group, and H–T group, a large number of cracks and pores appear inside rock after 12 H–C cycles in H–T group (Figure 9(c) and (d)); study results show that the presence of clay minerals, such as kaolinite, may lose water with hydroxyl groups and change the structure of rock after 400°C treatment [35–38].

With increasing cycling number, thermal expansion of sand particles inside the crystal increases the volume of sandstone specimen. The internal free water and organic matter in the sandstone gradually evaporate, which leads to decrease in the P-wave velocity, density (Table 1), and total import strain energy (Table 2) and increase in the porosity (Table 1) and damage (Figure 6).

4 Conclusions

- (1) The whole energy evolution process of sandstone after H–C cycle treatment can be divided into four stages. With the increase of cycle times, the total import strain energy decreased. The decreasing rate of total import strain energy in H–T group was significantly larger compared with that in L–T group and M–T groups.
- (2) In the dynamic compression deformation and failure process of sandstone, the total input of strain energy, dissipation energy, and the characteristics of releasing projectile are different at different stages, the decrease degree for U and U^d at failure stage exhibited the maximum value compared with other three stages. However, the decrease degree for U^e at failure stage shows the minimum value.
- (3) Under the same cycle number, rock damage for H–T group became more serious compared with that for M–T and L–T groups. After repeated H–C cycles, the thermal stress at the mineral boundary is constantly produced, leading to the fatigue rupture along the boundary of the mineral particle.

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