Experimental Study on Creep of Sandstone under Hydrochemical Corrosion

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Abstract. Corrosion creep of sandstone has great influence on the safety and stability of geotechnical engineering, and it is of great significance to study the creep characteristics of sandstone under the action of hydrochemical corrosion for the long-term stability of geotechnical engineering. Through creep test of corroded rock, the characteristics of rock deformation and strength are analyzed. Combined with electrochemical impedance spectroscopy (EIS), the microcracks of corroded sandstone during creep process are detected, and the variation law of equivalent circuit parameters and the development trend of microcracks during creep process of corroded sandstone are analyzed. The test results show that the creep characteristics of rock samples are related to the hydrochemical environment; The medium-and long-term strength of the solution with high pH value is relatively small, and the difference with uniaxial compressive strength is large; Moreover, there is a certain correlation between creep of rock samples and equivalent circuit parameters. In this paper, the influence of creep deformation, mechanical properties and hydrochemical environment on sandstone creep is analyzed, and the research results can provide reference for stability analysis of related geotechnical engineering.

1. Introduction
Geotechnical engineering is an engineering relying on soil or rock mass, which mainly tends to foundation, slope and underground engineering. From design, construction, completion and use, and even later maintenance, the mechanical properties of rock mass change throughout the life cycle of geotechnical engineering. The effect of occurrence environment on rock mass is a long-term and cumulative process, which reflects the timeliness of deformation-failure. In-situ stress, groundwater, temperature and chemical environment are the four main factors forming complex geological environment. Rocks in groundwater seepage field and chemical environment for a long time, on the one hand, bear the static and dynamic water pressure of water, on the other hand, water acts as a carrier to bring chemical substances into rock pores, and some elements in rock minerals react with chemical substances, breaking the balance state between water and minerals, which makes the dissolution and migration of rock minerals change the mineral composition and structural composition of the rock itself, and weakens the cementation between rock mineral particles, resulting in the change of rock mineral composition and dissolution holes in the rock\textsuperscript{[1-2]}. With the long-term action of the underground stress field, the rock is constantly deformed, the internal cracks are generated, the micro-cracks are penetrated and expanded, and the existing micro-cracks are gradually expanded under the
action of chemical solution. Serious rock mass damage will eventually have an irreversible impact on the stability of geotechnical engineering [3-5].

At present, direct detection method and indirect detection method are often used to detect rock microcracks. Direct methods mainly include microscopy, scanning electron microscopy, and laser holographic interferometry. Most samples need to be prepared before direct detection technology, and the process of sample preparation also aggravates the damage of rock mass. Although it can visually display and detect the damage state of rock mass materials, it is limited to plane measurement, and there are still limitations in the application process. Indirect detection methods mainly include mass density change detection method, acoustic emission technology, mercury intrusion detection, nuclear magnetic resonance, ultrasonic wave, rock damage CT, etc[6]. Indirect detection technology mostly compares the test results of samples before and after or tests in specific indoor conditions. Some methods are limited by the conditions of rock samples, and the detection accuracy is not high, so most methods cannot realize real-time detection.

Based on the study of the influence of hydrochemical environment on the instantaneous strength of rock, the creep test of sandstone under graded loading in hydrochemical environment is carried out in this paper, combined with the detection method of electrochemical impedance spectroscopy[7], in order to understand the creep deformation of sandstone, the mechanical properties and the influence of hydrochemical environment on sandstone creep, and also hope to provide reference for practical engineering.

2. Experimental Design

2.1. Test Materials and Equipment
The rock block used in the test is Shi Ying feldspar sandstone. Sandstone samples are mainly composed of detritus, matrix, accessory minerals and secondary minerals. After the rock block is taken from the construction site, the sample with diameter d of 50mm is drilled by the rock core drilling machine, and the height of the sample is intercepted by the rock cutting machine, and the sampling height h is 60mm; Grind both end surfaces of the sample on a rock grinding machine to ensure that the cross section of the sample is parallel, flat and vertical. After sampling, place the sandstone sample in a ventilated place for natural air drying. The design of hydrochemical solution is shown in table 1.

| Solution Number | Ingredient | Solution Concentration/mol/L | pH |
|-----------------|------------|-----------------------------|----|
| 1               | HCL        | 0.1                         | 2  |
| 2               | HCL        | 0.1                         | 5  |
| 3               | Distilled Water | /            | 7  |
| 4               | NaOH       | 0.1                         | 10 |
| 5               | NaOH       | 0.1                         | 13 |

Several samples were taken from each group every 30 days for rock creep test. Creep test of corroded rock is carried out on YR-2000 rock creep tester in Henan Mechanical and Engineering Structure Test Center. The creep tester is controlled by TestExpert software and can provide different loading modes and different loading rates. The main technical parameters are shown in table 2.

| Maximum axial load | Axial force measurement accuracy | Dismeasurement range | Dismeasurement accuracy | Deformation limit value Axial and Radial direction | Demeasurement resolution |
|--------------------|----------------------------------|----------------------|------------------------|-----------------------------------------------|-------------------------|
| 2000kN             | <=±1%                            | 1-100mm              | <=±1%                  | 5mm，2.5mm                                    | 0.001mm                 |
Using the displacement control method, the designed displacement load rate of 0.5mm/min, damage detection threshold is 50%, and it is believed that when the strain of the rock sample reaches the peak strength, the strain has weakened to 50% believes that the rock sample has damaged [16].

2.2. Experimental Process and Matters Needing Attention

(1) According to the above, select samples from rock samples with different treatment methods. Boil distilled water for 30min, and dilute chemical solution remains.

(2) After the rock sample is naturally dried, it is put into an oven for drying at 105°C for 24 hours, and the dried sample is placed at room temperature.

(3) Add a rigid cushion block with a height of 30mm and a diameter of 50 on the lower bearing platform of YR-2000 rock creep instrument, and put the rock sample on the upper cushion block.

(4) Graded loading is set for the test. In this creep test of corroded rock, constant speed and step-by-step loading are adopted. The grading parameters of this test are set as follows: at the beginning of the first stage, displacement control is adopted, the displacement speed is controlled at 0.5mm/min, and the target value is set at 1mm or 5kN. When the receiving displacement of the rock creep gauge sensor reaches 1mm or the receiving axial stress reaches 5kN, it is considered that the first stage begins and remains there for 30min. Second stage: force control is adopted in this stage, the loading rate is controlled at 1kN/min, the target axial stress is 10kN, and this stage is maintained for 4h; In the last five stages, force control is adopted, the acceleration rate is controlled at 1kN/min, the target axial stress is 20, 30, 40, 50 and 60kN respectively, and it is maintained for 10h; hours in no stage.

(5) In the later stage of the test, because of the individual differences of sandstone samples, the number of segments is different, and the target axial stress in the last stage is taken according to the uniaxial compressive strength; For force majeure such as power failure during creep test, stress shall be loaded step by step, and shall be maintained in the accident section for corresponding time before entering the next stage.

(6) Protective measures should be taken in advance before the test. First, prevent the sudden destruction of rocks and the flying of cuttings.

3. Experimental Results and Analysis

3.1. Effect of Hydrochemical Solution on Uniaxial Creep Curve of Sandstone

Several sandstone samples were selected from different hydrochemical solutions for uniaxial compression creep test. The creep curves of rock samples under different hydration solutions and different corrosion time are integrated and compared. Considering that rock samples in different pH solutions are taken from different rock blocks and different rock blocks, only the creep stress-strain curves of rock samples in the same pH solution are characterized. As shown in figure 1-5.

![Figure 1](image-url) Creep curve of rock samples corroded by different time in pH=7 hydrochemical solution.
Figure 2. Creep curve of rock samples corroded by different time in pH=10 hydrochemical solution.

Figure 3. Creep curve of rock samples corroded by different time in pH=2 hydrochemical solution.

Figure 4. Creep curve of rock samples corroded by different time in pH=13 hydrochemical solution.

Figure 5. Creep curve of rock samples corroded by different time in pH=5 hydrochemical solution.

Figure 1-5 shows the creep curves of rock samples under various working conditions obtained from uniaxial compression creep test of corroded sandstone. Because the rock samples in different working conditions adopt the same loading mode, the creep curve shows the same trend, which mainly includes instantaneous stress loading deformation section and constant stress deformation section [8]. The main characteristics of creep curve of rock samples are as follows:

(1) Time effect. The creep end time of rock samples in the same hydrochemical solution is advanced with the increase of corrosion time. The longer the corrosion time is, the load instantaneous strain and creep of rock samples will increase correspondingly.

(2) Corrosion effect. Under different working conditions, the creep failure time of rock samples in the same corrosion time is gradually shortened with the increase of pH value of hydrochemical solution. Taking corrosion for 30 days as an example, the corresponding creep failure time of water chemical solution from low to high pH value is 65.75h, 86h, 157h, 126.65h and 85.8h, respectively.

(3) Instantaneous strain appears in each sample after each stress level is recorded. It can be seen that with the increase of loading stress, the deformation of rock sample generally increases. Under the action of low stress, the rock sample is mainly subjected to instantaneous deformation, and the creep strain is small. During this period, the creep of the rock sample presents a stable trend. When the stress increases gradually, the creep of the rock sample increases gradually, and the strain increases sharply during the last loading [9]. It can be considered that the rock sample has internal damage and cracks due to stress concentration.
3.2. Long-Term Strength Characteristics of Corroded Sandstone

According to the data, the isochronous stress-strain curve of corroded sandstone is drawn. Take the rock samples corroded for 30 days at pH=2 as an example. Firstly, the graded loading creep curve of rock sample is transformed into the separately loaded creep curve [10], as shown in figure 6, and then the isochronous stress-strain curve of rock sample is made according to the method in figure 7.

![Figure 6. Creep curve under separate loading.](image)

![Figure 7. Method drawing of isochronous stress-strain curve.](image)

According to relevant research, the inflection point in the isochronous stress-strain curve can be expressed as the characteristic point of rock sample's transition from viscoelasticity stage to viscoplasticity, that is, the yield point of rock sample at each time point, which is reflected in the isochronous stress-strain curve as the characteristic point of curve trend from steep to slow [11]. It can be seen from fig. 8 that the inflection point appears approximately at 0.14% strain. According to the test data, the loading stress corresponding to 0.14% strain is 22.6MPa, and it can be considered that the long-term strength of rock sample is 22.6MPa.

According to the above method, the long-term strength of rock samples under various working conditions is calculated as shown in table 3.
### Table 3. Long-term strength of rock samples under various working conditions.

| Hydration environment | Time/d | Compressive strength/MPa | Long term strength/MPa | strength ratio% |
|-----------------------|--------|---------------------------|------------------------|-----------------|
| pH=7                  | 30     | 96.39                     | 76.95                  | 79.8            |
|                       | 60     | 93.57                     | 69.45                  | 74.2            |
|                       | 90     | 89.57                     | 62.07                  | 69.3            |
|                       | 30     | 64.232                    | 40.864                 | 63.6            |
| pH=5                  | 60     | 56.174                    | 30.934                 | 55              |
|                       | 90     | 47.095                    | 22.058                 | 46.9            |
|                       | 30     | 80.94                     | 58.276                 | 71.9            |
| pH=10                 | 60     | 76.434                    | 50.43                  | 65.9            |
|                       | 90     | 63                        | 37.75                  | 59.9            |
|                       | 30     | 51.53                     | 22.6                   | 44.3            |
| pH=2                  | 60     | 30.19                     |                        |                 |
|                       | 90     | 25.295                    |                        |                 |
|                       | 30     | 56.3                      | 33.78                  | 60              |
| pH=13                 | 60     | 29.063                    | 15.46                  | 53              |
|                       | 90     | 26.07                     |                        |                 |

It can be clearly seen from the test results in Table 3 that the long-term strength of rock samples in all working conditions is smaller than the uniaxial compressive strength of rock samples, with a drop of 40%–80%. The long-term strength of rock samples corroded by pH=2 for 30 days is the lowest, followed by rock samples corroded by pH=13 for 60 days. The long-term strength of rock samples in distilled water is relatively high, but the decrease is that the corrosion time increases by 30.7%. It can be seen that the long-term strength of rock samples changes with the properties of hydrochemical solution and corrosion time.

Where there is no data in table 3, the creep time of rock samples corroded by pH=2 water chemical solution at 60 and 90 and rock samples corroded by pH=13 water chemical solution for 90 days is shorter, and the isochronous stress-strain curve is linear without obvious inflection point.

#### 3.3. Change Law of Electrochemical Impedance Spectroscopy in Creep Process of Corroded Sandstone

According to the application of electrochemical impedance spectroscopy (EIS) in microscopic description of rock samples, this paper attempts to apply EIS testing method to describe the development of microstructure in creep process of rock samples. Combined with the reaction of sensitivity of rock samples to hydrochemical solution in various working conditions summarized above, the rock samples corroded in hydrochemical solution with pH=2 for 60 days were selected for electrochemical detection of creep process. It is designed that the stress maintenance time of each level of loading is 10 hours, and the loading is divided into four levels, and the loading stress of each level is 10kN. The detection stage is only the creep stage within one hour of each level of stress loading.
In table 4, $Z_1$ and $Z_2$ represent the coordinates of the start and end segments of high-frequency arc in Nyquist diagram. $\alpha$ represents the average overlooking angle of high frequency band; $\varphi_{Z_1}$ and $\varphi_{Z_2}$ represent the top-down angles of the start and end sections of the high-frequency arc. $R_1$ and $R_2$ represent the projection of $Z_1$ and $Z_2$ on the real axis in Nyquist diagram.

The electrochemical impedance spectroscopy of rock samples during creep is described and analyzed as follows:

It can be seen from fig. 9 that the radius of frequency band arc in Nyquist diagram is 2h, 12h, 22h and 32h respectively, which indicates that the internal crack of rock sample begins to develop under load;
(1) According to Bode diagram, the value of high-frequency phase angle measured by stress loading for 8 hours is the smallest; Bode diagram, it can be clearly seen that the difference between the first stage and the second stage of loading is larger than that between the third stage and the fourth stage of loading, and the impedance value decreases gradually with the increase of time. It can be considered that the microcracks of rock samples expand rapidly during initial loading, and then the stable development stage of cracks appears. When loading the last stress, the cracks develop again and the microcracks expand completely.

(2) Combined with the high-frequency technical parameters in Nyquist diagram, it can be concluded that the variation law of rock microstructure is as follows: ① The change of high-frequency arc phase angle index in Nyquist diagram is 0.1, 0.08, 0.06 and 0.04 respectively according to the increase of corrosion time, which shows that the micro-cracks in rock samples increase obviously under long-time load. ② In ②Nyquist diagram, the changes of real impedance at the beginning and end of high frequency arc period with the increase of corrosion time are 16.97, 12.5, 9.7 and 4.2, respectively, indicating that the internal structure of rock samples changes obviously during creep process, and some voids gradually penetrate.

Similarly, the equivalent circuit is used to fit the creep electrochemical impedance spectrum of rock samples, and the parameters are shown in table 5.

Table 5. Fitting equivalent circuit parameters.

| Parameter | Time/h | \( R_s \) | \( CPE - T \) | \( CPE - P \) | \( R_p \) |
|-----------|--------|----------|-------------|-------------|--------|
|           | 2h     | 219      | 2.0\times10^4 | 0.53        | 768    |
|           | 12h    | 192.5    | 2.2\times10^4 | 0.55        | 726.3  |
|           | 22h    | 179.1    | 3.1\times10^4 | 0.56        | 671.1  |
|           | 23h    | 151.6    | 5.2\times10^4 | 0.58        | 509    |

In table 5, \( R_s \) represents the resistance of solution in rock samples; \( CPE - T \) represents electric double layer capacitance; \( CPE - P \) is dispersion angle parameter, \( \varphi = \pi(1-CPE - P)/2 \) is dispersion angle; \( R_p \) represents the connection resistance.

![Figure 11. Relationship between rock sample strain and \( R_s \).](image1)

![Figure 12. Relationship between rock sample strain and \( CPE - T \).](image2)

According to the data in table 5, with the increase of stress loading grade, \( R_s \) decreases gradually as the creep time continues. When electrochemical test shows that the ion concentration of pore solution in the system remains unchanged, there is the following relationship between pore solution resistance and test substance particles [12]:

\[ y = 316.54x + 221.76 \]
\[ R^2 = 0.9471 \]

\[ y = 1.6395e^{4.992x} \]
\[ R^2 = 0.9933 \]
\[ R_s = K \left( \frac{\rho_s}{1 - \psi_s} \right) \]  

(1)

In which \( \rho_s \) is the resistivity of pore solution, and \( \psi_s = a \varphi_s \) is the relationship between solid fraction and volume fraction. The relationship between volume fraction of \( \varphi_s \) and porosity of \( P \) is \( \varphi_s = 1 - P \), so \( R_s \) can be expressed as:

\[ R_s = \frac{K \rho_s}{1 - (1 + a) P} \]  

(2)

It can be seen that is inversely proportional to. Therefore, it can be seen that the porosity of rock samples increases gradually during creep. The change trend of microstructure of rock samples can be directly reflected. When pores expand, electrolytic solution gradually fills the pores, and the corresponding capacitance increases. In addition, Burtman et al. analyzed the induced polarization mechanism of rocks and tested the IP response of sandstone, and found that the electric double layer is related to the size and shape of pores filled with electrolyte [13]. Compared with the discrete phenomenon in the third chapter, there is a certain regularity in creep electrochemical test, that is, with the development of creep deformation of rock samples, it shows a decreasing trend. It can be considered here that due to the load, the developed fissures make the electrolytic solution communicate and the current path open. Micro-cracks in rock samples are the main cause of deformation and fracture with time effect. Therefore, through the relationship between deformation and deformation of rock samples, it can be considered that creep deformation of rock samples has a certain correlation with them.

4. Conclusion

Through uniaxial creep test and electrochemical test of rock samples under different working conditions, the following conclusions are obtained:

(1) The creep characteristics of rock samples are related to the hydrochemical environment. The creep amount of rock sample increases gradually with the increase of pH value of hydrochemical solution, and its creep rate increases. At the same time, the creep of rock sample has corresponding time effect, and the longer the corrosion time, the greater the creep deformation.

(2) The isochronous stress-strain curve method is used to determine the long-term strength of rock samples under different working conditions. It is found that the long-term strength of the solution with high pH value is relatively small, and the difference with the uniaxial compressive strength is large.

(3) Applying electrochemical impedance spectroscopy to detect creep deformation of rock samples, it is found that there is a certain correlation between creep amount of rock samples and equivalent circuit parameters.

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