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Research Article

Study of Influence of Environmental Factors on Deep Shale Creep Properties

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In order to study the influence of environmental factors on creep properties of deep shale, a series of creep tests of deep black shales are performed under different environmental conditions, including stress deviation, temperature, and chemical pH value. The influence of these conditions on creep properties of deep shale is studied. The results show that the creep and creep rate of shale will grow with the increase of temperature, stress difference, and acidity-alkalinity. We get nonlinear creep model of deep shale when pH = 4 and $T = 30^{\circ}$C. The critical stress difference of deep shale is no larger than 31.04 MPa when the chemical pH value is 4 and the temperature is 30°C. By scanning shale after corrosion, we know that the effects of chemical pH value on the creep characteristics are mainly determined by the feldspar dissolution and corrosion caused by chemical action. Our work has important theoretical significance and practical value for evaluating rock engineering stability.

1. Introduction

Deformation and failure of the underground engineering in Shandong Tangkou Coal Mine showed that the engineering was mainly located in shale and mudstone area. The overburden of surrounding rocks is under the influence of gravity and coupled with the complex geological environment by the threat of high ground temperature, high pressure, and groundwater erosion, which produce rheological phenomena as time increases. If the rock is buried deeper, this condition will be more obvious [1–5]. Shale is one of the primary surrounding rock materials of deep roadway, which exhibits strong rheological properties and significant deformation characteristics when it interacts with the underground complex environments, i.e., high temperature, high stress, and chemical corrosion [6–8], which badly threatens the stability of subterranean rock engineering and may even lead to serious accidents.

In recent years, more and more researchers have investigated creep characteristics of rock. Most of the research focuses on the creep properties of rock under condition of the single stress field [9–11]. Zhang et al. performed triaxial creep tests of mudstone and indicated triaxial creep of weak rock is nonlinear: the creep deformation is three times more than the transient deformation. Xu et al. studied lateral strain and axial strain variation with time under different confining pressures and the rupture mechanism of rheological properties of rock. They established a new seven-component nonlinear viscoelastoplastic rheological model of rock. Li et al. performed triaxial creep study of shale and concluded that creep produces two phases: steady creep and transition creep and also obtained creep variables related to time at the steady-state creep stage. By studying two fields, such as the temperature field and stress field [12–14], Li et al. implemented uniaxial and triaxial creep experiments on sand stone and found that, as temperature increases, the total creep deformation, initiative creep deformation, instantaneous thermal strain, and creep velocity increase. In terms of temperature field and stress field coupling [15, 16], Wang et al. studied the effects of chemical corrosion on creep under uniaxial compressive. The results showed that the creep and creep strain rate increase with the degree of chemical corrosion. According to failure criteria of Mohr–Coulomb and Griffith, the primary factor of great deformation failure for a roadway is the main stress deviation [17, 18].

The above studies of creep characteristics of rock are based on single stress field or two fields, and most of them are developed for shale, greenschist, sandstone, etc. The creep behaviors of rock under the action of multiple fields are seldom studied. Therefore, the creep characteristics of deep shale under the combined action of temperature, stress, and chemical corrosion are rarely reported.

There is plenty of shale with clear bedding and high chemical activity in roadway of 1000 m deep in Tangkou Coal Mine. The creep characteristics of shale are inevitably affected by the chemical action of the ground water, which are accompanied by the influence of ground stress and temperature of the ambient environment. Therefore, study of the influence of stress difference, temperature, and chemical corrosion on creep characteristics of deep shale can provide theoretical guidance and technical approach for the support design, disaster prediction, safety assessment, and long-term maintenance of deep mines under complex environment in this area. It can also be used as a reference for other underground tunnels, undersea tunnels, and deep roadways in other areas.

By simulating different conditions of stress deviation, temperature, and chemical corrosion, we carry out laboratory experiments of creep characteristics of deep shale and find the creep behaviors under various environmental factors.

2. Creep Test of Shale

2.1. Shale Properties and Sample Preparation. The shale samples used in this study were taken from the tunnel at a depth of 1000 m in the Shandong Tangkou Coal Mine. Samples are black shales with clear bedding and low hardness, which are integral and dense. Because some rock cores were disconnected for bumpy road from the Tangkou Coal Mine, all samples were processed into cylinders of 55 × 80 mm. Figure 1 shows all test samples.

To study the chemical corrosion effects on the creep properties, each sample was immersed in the chemical solution of the same volume and the pH value of 4, 7, or 10 (Table 1), which was tightly sealed. After being soaked for 15 days, the sample reached natural saturation and was removed and sealed with plastic wrap. To protect the chemical field of the shale from the impact of hydraulic oil, heat-resistant tape was twined, and then shrink film was put by heat drying with a hair dryer in order to seal tightly.

2.2. Test Methods. Test equipment is the triaxial creep test machine, which was jointly developed by Qingdao University of Science and Technology and Lear Ltd. of Yantai in Shandong (Figure 2). The axial loading system is composed of a common tension and compression test machine used in the course of mechanics of materials, which is the axial force source equipment of the test machine. The triaxial environmental chamber is a core unit of the triaxial creep test machine. Oil in the test chamber provides temperature and confining pressure by the server control system for the sample.

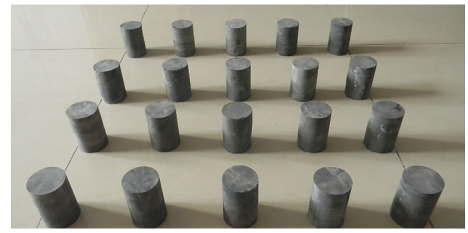


Figure 1: All shale samples used for creep test.

Table 1: Hydrochemical environment.

Serial number	Hydrochemical environment	pH value
1#	HCL solution	4
2#	Distilled water	7
3#	NAOH solution	10

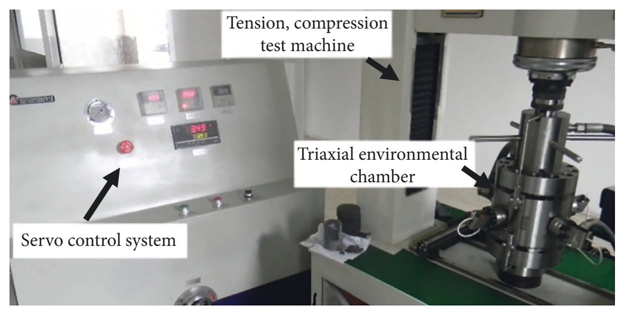


Figure 2: Triaxial creep test machine.

We set the test temperature as three levels: 30, 50, and 70°C. Each temperature was rated to a group, and each group had three different pH value samples. Previous experiments have concluded that the average uniaxial compressive strength of shale is respectively 63.69, 38.80, or 47.30 MPa when the chemical pH value is 4, 7, or 10. This experiment adopted the classification loading mode, and the total load series was 5. And the confining pressure was 3 MPa. The final-level stress deviation was 38.8 MPa. Therefore, the first-level stress deviation was 7.76 MPa. In addition, stress deviation was controlled at 7.76, 15.52, 23.28, 31.04, and 38.8 MPa (Table 2). A heating rate of 2°C/min was applied. To ensure uniform heating of the sample, we heated the oil to a predetermined temperature and kept the constant temperature for 3 h.

The test used the displacement control mode, which simultaneously applied confining pressure and axial pressure to a predetermined value at a loading rate of 0.005 mm/s, which remained constant for 24 h. Confining pressure remained 3 MPa during the test process, and axial pressure used the classification loading mode. We used a 5 mm displacement sensor to measure the axial displacement of the sample.

3. Effect Factors on Creep Properties of Shale

3.1. Effects of Stress Deviation. We conducted creep tests of different stress difference of 7.76, 15.52, 23.28, 31.04, and

Table 2: Schemes of creep test.

T (°C)	pH value	$\sigma_1 - \sigma_3$ (MPa)
30	4	7.76, 15.52, 23.28, 31.04, 38.80
30	7	7.76, 15.52, 23.28, 31.04, 38.80
30	10	7.76, 15.52, 23.28, 31.04, 38.80
50	4	7.76, 15.52, 23.28, 31.04, 38.80
70	4	7.76, 15.52, 23.28, 31.04, 38.80

38.80 MPa when the pH value is 4 and the temperature is 30°C for 24 h. The creep strain duration curves are shown in Figure 3(a), and the development curves of creep rate are demonstrated in Figure 3(b) by calculating the tangent of each point of the creep curves of Figure 3(a).

When the stress deviation is 7.76 MPa, the creep strain of shale is small, and the creep rate is slow and soon stable. The experimental data fit well with Burgers model, and the creep constitutive equation is as follows:

$$\varepsilon = \frac{\Delta\sigma}{E_1} + \frac{\Delta\sigma}{E_2}\left(1 - e^{-(E_2/\eta_1)t}\right) + \frac{\Delta\sigma}{\eta_2}t, \quad (1)$$

where ε is the total strain of Burgers; $\Delta\sigma$ is stress deviation; E_1 and E_2 are the elastic modulus; and η_1 and η_2 are viscosity coefficients.

The correlation coefficient of fitting is 0.98, and the model parameters are shown in Table 3.

When the stress deviation is 23.28 MPa, creep curve shows no sudden destruction trend, and the experimental data still fit well with Burgers model. However, the creep strain rates are larger, and the time that the shale takes to enter the stable creep stage is relatively longer.

When the stress deviation is 38.80 MPa, the creep characteristics are completely different from that of the low-stress deviation. Creep strains develop rapidly with time, and creep rates are very large. The strain-time curve becomes more and more concave to the strain axis, which shows creep with obvious nonlinear characteristics. Under this condition, the experimental data cannot fit well with Burgers model. Therefore the creep equation of Burgers model cannot fully satisfy the creep characteristics of shale.

Replacing nonlinear element with the conventional linear element in order to describe the nonlinear rheological properties, the creep equation is as follows:

$$\varepsilon = \frac{\Delta\sigma}{E_1} + \frac{\Delta\sigma}{E_2}\left(1 - e^{-(E_2/\eta_1)t}\right) + \frac{\Delta\sigma}{\eta_2}t + \frac{\Delta\sigma - \sigma_s}{\eta_3}t^n, \quad (2)$$

where σ_s is the yield strength of the rock and η_3 is the viscosity coefficient of the nonlinear viscoplastic model.

By using nonlinear least square method of MATLAB and programming, the correlation coefficient is 0.96, and the parameters of the model are shown in Table 3.

Isochronous stress-strain curves of creep test are shown in Figure 3(c). An inflection point occurs when the stress difference is greater than a threshold value, which indicates that the creep deformation of shale has a steady development trend when the stress difference is smaller than this threshold value, and creep strain increases sharply when the stress difference is greater than this threshold value. We define the D-value of stress as the critical stress deviation value of creep. This value can be identified from Figure 3(c). For underground roadway design, the critical stress deviation value of creep is no greater than 31.04 MPa under ambient condition of pH = 4 and T = 30°C.

3.2. Effects of Temperature. The strain-time curves are shown in Figures 4(a) and 4(b) at pH value of 4 under different temperatures and stress deviations.

We can more clearly see the effects of temperatures on the creep of shale by drawing the stress deviation-creep strain curves under different temperatures (Figures 4(c) and 4(d)). The ambient temperature affects the critical stress deviation value. The critical stress difference for temperature lower than 50°C is no greater than 31.04 MPa, and it is reduced to 23.28 MPa when temperature is 70°C.

Table 4 gives creep strains of shale after the loads stabilize for 1 h and 10 h under different temperatures. As seen from the above charts and table, the rising temperature only causes an increase of the creep deformation under low-stress deviation, but there is not much effect on the creep duration curves. Therefore, the creep equation is still simulated by the Burgers model. However, when the stress deviation increases to 31.04 MPa, the ambient temperature not only leads to a sharp increase of creep, but also significantly changes the other creep characteristics (Figure 4(b)). Creep curves at 70°C are significantly nonlinear curves. When the stress deviation continues to increase to 38.80 MPa, the effects of temperature on creep characteristics are more remarkable, and the creep and creep rate of shale are very great, and shale quickly reaches a state of destruction.

3.3. Effects of Chemical pH Value. The strain-time curves are given at T = 30°C under different pH values and stress differences in Figures 5(a) and 5(b). Strain-time curves of different pH values at a stress deviation of 38.80 MPa (Figure 5(c)) are plotted. We can identify the characteristicsc from the isochronous stress-strain curves (Figures 5(d) and 5(e)). We can see that the curves of pH = 7 are approximately a family of lines, and curves of pH = 10 and the higher stress deviation are approximately a family of nonlinear curves, which show the creep of shale has nonlinearity at the viscoplastic stage. As is apparent from a comparison with the previous correlation curves of pH = 4, the creep and creep rate of shale will grow with the increase of stress difference or acidity-alkalinity.

4. Creep Mechanism of Shale under Temperature and Chemical Corrosion

4.1. Creep Mechanism of Shale under Temperature. Temperature changes the creep properties by changing the physical properties of the rock. In researching the relations between temperatures and creep properties, we need to combine the temperature with physical properties of rock. Shale has the smaller particles and the lower porosity. A smaller temperature range was applied in this article. Shale produces microcrack by swelling around the pore of rock when the temperature rises. The crack will grow with the

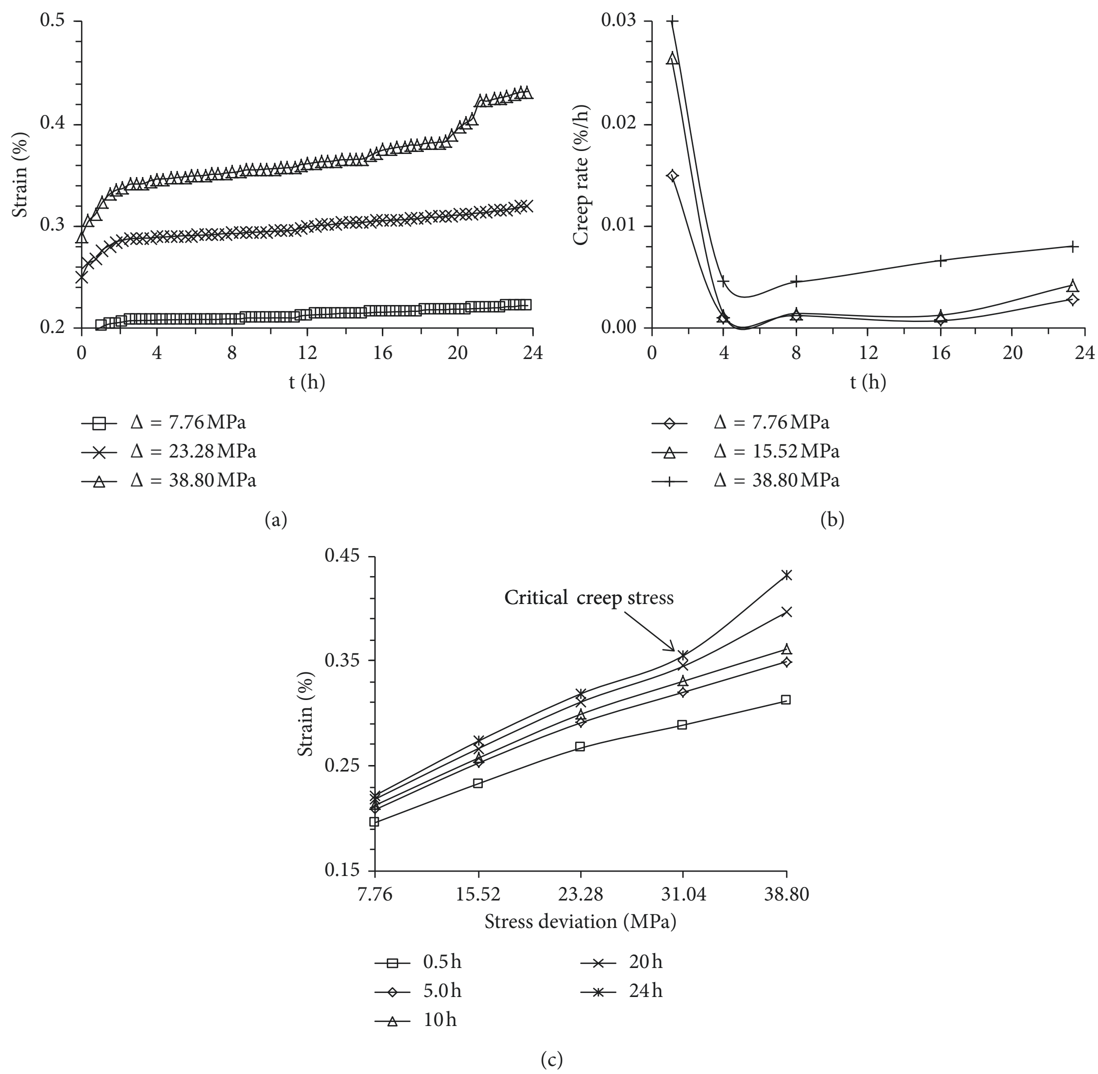


FIGURE 3: Influence of different stress deviations on creep properties of shale (pH = 4, $T = 30^{\circ}$C). (a) Strain-time curves. (b) Creep rate-time curves. (c) Isochronous stress deviation-strain curves.

TABLE 3: Creep parameters of deep shale at pH = 4 and $T = 30^{\circ}$C.

Stress deviation (MPa)	Model parameters	$T = 30^{\circ}$C
$\Delta\sigma = 7.76$	E_1/GPa	4.122
	E_2/10 GPa	4.887
	η_1/10 GPa·h	3.820
	$\eta_2/10^3$ GPa·h	1.087
$\Delta\sigma = 38.80$	E_1/GPa	13.477
	E_2/10 GPa	9.585
	η_1/10 GPa·h	7.441
	$\eta_2/10^3$ GPa·h	2.440
	σ_s/MPa	38.799
	$\eta_3/10^3$ GPa·h	36.013
	n	5.231

increase of temperature. The increase of temperature causes instantaneous elastic strain. The compressibility of shale is larger under constant stress, it takes a longer time to enter stable creep stage, and creep after 24 h is greater.

4.2. Creep Mechanism of Shale under Chemical Corrosion. To observe the damage of chemical solutions to rock, we soaked samples in the chemical solution of pH = 4 and 7 before creep test. In this experiment, equipment of scanning electron microscopy of JSM-6700F was adopted with a multiple of 10000 times (Figure 6). Figure 7 shows the identification results for the microstructure sheets of the shale under hydrochemical environment of pH = 4 and 7 for 15 days. As seen from Figure 7(a), shale particles are dense and evenly distributed, and there are no obvious hole under pH = 7. The apparent erosion phenomenon occurs in the hydrochemical environment of pH = 4. The shale from Shandong Tangkou Coal Mine is mainly composed of quartz and feldspar debris. The shale is eroded and produces voids due to the hydrochemical solution corrosive effects. The higher acidity-alkalinity, the greater the instantaneous elastic strains at each level of the load. Hydrochemical corrosion causes different initial damage to shale, and it has an aftereffect on the creep characteristics of shale. With the

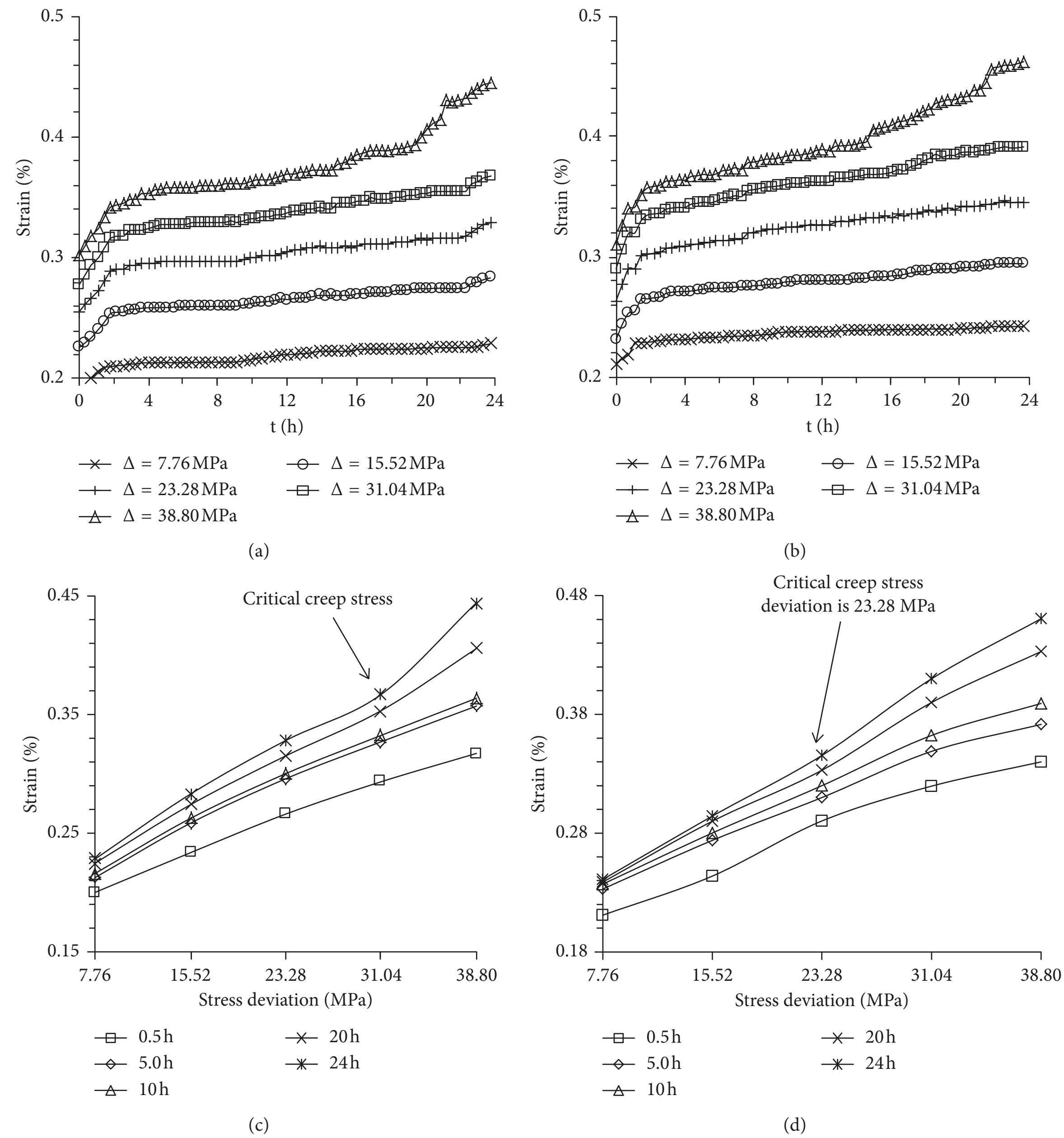


Figure 4: Influence of different temperatures on creep properties of shale. (a) Strain-time curves (T = 50°C). (b) Strain-time curves (T = 70°C). (c) Isochronous stress deviation-strain curves (T = 50°C). (d) Isochronous stress deviation-strain curves (T = 70°C).

Table 4: Creep strains of shale under different stress levels and temperatures (unit: %).

Time (h)	Temperature (°C)	Creep strain under different stress deviations (MPa)				
		7.76	15.52	23.28	31.08	38.80
	30	0.201	0.241	0.293	0.299	0.323
1	50	0.206	0.240	0.273	0.300	0.325
	70	0.216	0.253	0.291	0.320	0.341
	30	0.210	0.254	0.295	0.326	0.356
10	50	0.216	0.262	0.300	0.332	0.363
	70	0.237	0.278	0.325	0.360	0.384

greater the initial damage, the larger the void and the compressible space of shale, which leads to the longer time of entering the stable creep stage and the larger creep of 24 h.

5. Conclusions

From performing systematic indoor creep tests of deep shale under different temperatures, stress deviations, and chemical pH values, we get the following conclusions:

(1) Creep characteristics of shale closely associate with environmental factors, such as temperature, chemical corrosion, and stress difference. The creep

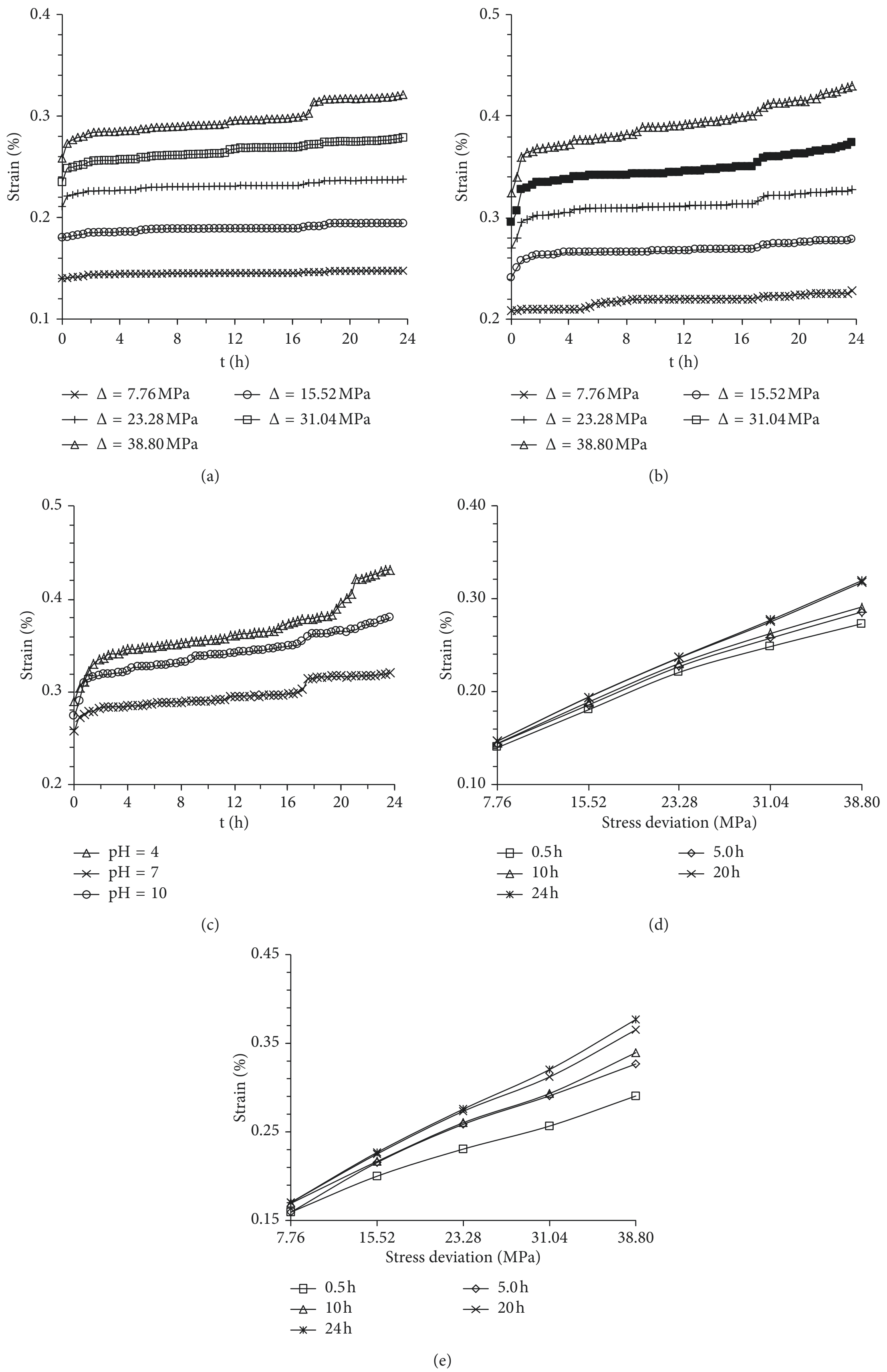
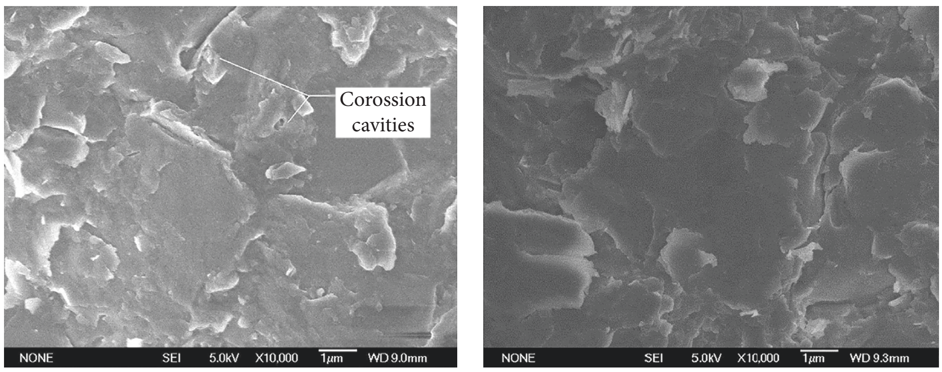


Figure 5: Influence of different pH values on creep properties of shale. (a) Strain-time curves (pH = 7). (b) Strain-time curves (pH = 10). (c) Creep strains under different pH values (stress deviation is 38.80 MPa). (d) Isochronous stress deviation-strain curves (pH = 7). (e) Isochronous stress deviation-strain curves (pH = 10).

Figure 6: Scanning electron microscopy of JSM-6700F.



(a) (b)

Figure 7: Micrographs of shale under the hydrochemical environment for 15 days. (a) pH = 4. (b) pH = 7.

and creep rate of shale will grow with the increase of temperature, stress difference, and acidity-alkalinity.

(2) The effects of temperatures and stress differences on shale creep are linked, i.e., the higher the temperature, the lower the critical stress deviation. The critical deviation stress values of $T = 20$ and 50°C are not larger than 31.08 MPa, but when the temperature is raised to 70°C, the critical stress difference value is larger than 23.28 MPa.

(3) The effects of chemical acidity-alkalinity and stress differences on the creep characteristics of shale are interrelated. The critical stress deviation will reduce with the increase of acidity-alkalinity. The critical difference of stress of pH = 4 is no larger than 31.08 MPa, but when the pH value is 7, the isochronous stress-strain curves are approximately linear. However, when the pH value is 10 and the stress deviation value is higher, it presents a nonlinear trend, though it is not obvious. Therefore, in practical engineering applications, it should be noted that the allowable design strength is reasonably determined by the creep critical stress deviation based on local actual underground environment.

(4) By using scanning electron microscopy structure, the effects of chemical pH on the creep characteristics in this paper are mainly determined by feldspar dissolution and corrosion caused by chemical action.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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