

Experimental Study on Influences of Chemical Solutions on Mechanical Properties of Brittle Limestone Under Triaxial Compression

YAO Huayan ^{1,2}, ZHANG Zhenhua ²

1. School of Civil Engineering, Hefei University of Technology, 230009, P.R.China

2. Key Laboratory of Geological Hazards on Three Gorges Reservoir Area, China Three Gorges University, Ministry of Education, 443002, P.R.China
yaohuayan@163.com

ABSTRACT: A series of triaxial compression tests on dry and saturated limestone samples (soaked in chemical solutions of Na₂SO₄ and CaCl₂ with 0.1mol/L ionic concentration and varying pH values, and distilled water for 240 days) have been carried out. The corresponding complete stress-strain curves are obtained. The influences of chemical solution on rock mechanical behaviors are studied. Results from these tests indicate that: (1) Dry limestone samples have obvious brittle feature. Even if the confining pressure reaching 20MPa, the sample is still ruptured suddenly. After soaked in chemical solutions for 240 days, the deformation and failure behaviors were changed from brittle to ductile manner. (2) Chemical corrosion is an important reason for the strength deterioration of specimens. With the same soaking time and confining pressure, the strength degradation level of samples depends on ionic components and pH value of chemical solutions. Acidic solutions have the most significant influence on samples' mechanical properties. The fracture modes of samples could be divided into 7 types, which depend on chemical environments and confining pressures. The study has a good reference to researches of deep rock mechanics and water-rock interaction.

KEYWORDS: Rock mechanics, Triaxial compression test, Mechanical properties, Limestone, Chemical corrosion, Stress-strain curve

1 INTRODUCTION

Generally, rockmass is under triaxial stress and suffered with long-time water erosion and weathering. In order to estimate the stability of rock engineering structures in the long term, it is important to investigate the influence of triaxial stress and groundwater on rock mechanical properties.

At present, many scholars have launched a series of research work on water and rock interaction from different aspects. The previous results have shown that water and water chemistry are important factors in terms of effects on the deformation and strength of rock ^[1]. For example, sandstone samples saturated with chemical solutions display great weakening in comparison with the dry samples ^[2-3]. And some researchers have found that in wet conditions the fracture toughness will be lower than that in the dry condition and the crack propagation velocity will be increased ^[4-6].

Feng have reported experimental researches on the damage evolution properties of a sandstone subjected to chemical corrosion for the first time by using the real-time CT testing technique ^[7], and the influences of water corrosion on rock crack growth were also studied ^[8]. Li have investigated the deformation and strength properties of sandstone varying with soaking time and corrosion intensity ^[9]. In a word, the fact has been well-known that chemical reactions can lead to rock failure acceleration and instability ^[1-14].

In fact, there are many factors that affect the triaxial

strength of rock, for example, mineral composition, texture, specimen size, loading rate, and so on. Different minerals itself have different strength, and rock with different minerals have different mechanical properties under water environment. For example, the effects of water on sulfate rock differ from those on carbonate rock ^[15]. Therefore, in practice, it is necessary to perform relevant system studies according to different kinds of rock ^[16].

In the present paper, based on a series of triaxial compression tests, the effects of the ionic species, pH of water solutions, and confining pressure on properties of limestone are investigated. The results are analyzed to provide a guide for considering the long term effects of rock engineering.

2 EXPERIMENTS

2.1 Sample

The rock sample for the experiments is dolomitic limestone from the deep tunnel areas in Enshi, Hubei Province, China. The main mineral components are 73% calcite, 25% dolomite, and less than 1% hydromica and biodetritus, and a little pyrite, limonite, quartz, and carbon.

2.2 Test method and process

The diameters of the specimens for all tests were 50mm, and the lengths were 100mm. Different chemical solutions, such as CaCl₂, and Na₂SO₄, were selected to investigate the influence of chemical corrosion on strength of limestone.

The solutions were of 0.1mol/l concentration with pH values of 2, 7, 9, and 12. The chemical solutions are shown in Table 1. The results are compared with the air and the distilled water cases.

All specimens were dried at the temperature of 105°C for 24 hours at first, and cooled to room temperature, then

soaked in distilled water and the solutions shown in Table 1 for 240 days until required for testing at room temperature. Five specimens were prepared for each type of solution, and the confining pressure for the triaxial test are arranged 2.5, 5, 10, 20 and 40MPa, respectively.

Table 1 Chemical solutions

Chemical solution	Ionic concentration /mol·L ⁻¹	pH value
CaCl ₂	0.1	2, 7, 9, 12
Na ₂ SO ₄	0.1	2, 7, 9, 12

3 RESULTS

3.1 Influence of chemical corrosion on deformation of limestone

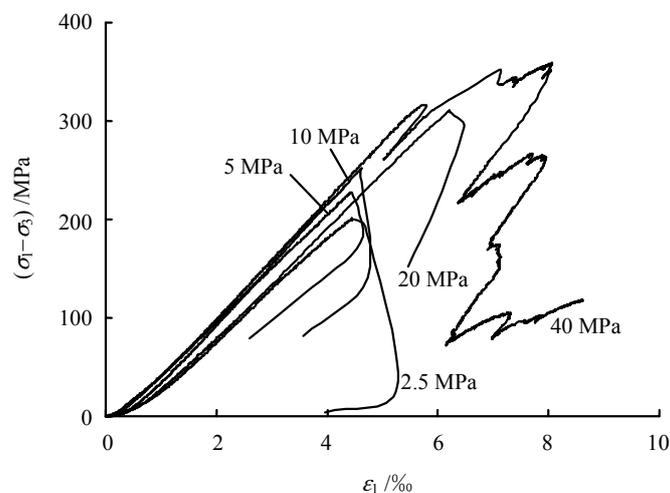
Figure 1(a) shows typical stress–strain curves for dry specimens. We can see that, when the confining pressure is less than 20MPa, the curve is almost straight before peak strength. There was not obvious yield stage before failure. The specimen broke down abruptly and lost bearing capacity when it reached the peak strength. When the confining pressure at 40MPa, the specimen had yield stage, but still ruptured quickly as soon as it reached the peak strength. As mentioned above, it seems that the limestone failure process is obvious brittle manner.

The deformation characteristics of specimens occur distinct changes after soaked in chemical solutions. Figure 1(b) shows typical stress–strain curves for specimens soaked in CaCl₂ solution with ionic concentration of 0.1mol/l, pH2 for 240 days. When the confining pressure was 2.5MPa, the limestone failure process was still brittle, but had residual strength after failed. The results indicate

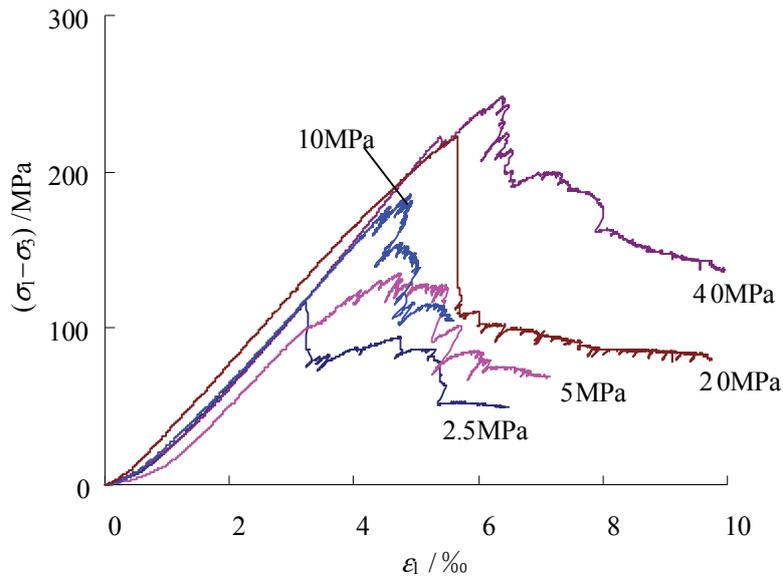
that, the specimens become ductile. When the confining pressure increasing, there had obvious plastic stage before peak strength. Although the axial stress dropped sharply after the peak strength, the specimens had residual strength.

From the experiments results, it may be concluded that the deformation and failure behaviors can be changed from brittle manner to ductile under chemical corrosion. The possible explanation is that with the chemical corrosion the sudden release of strain energy becomes weaker.

Figure 2 shows typical stress–strain curves for specimens soaked in Na₂SO₄ solution with ionic concentration of 0.1mol/L, pH value of 2, 7, 9 and 12, with confining pressure of 2.5MPa. When pH value at 2, chemical reactions were acting much more extensively, making the rock specimen more ductile. As a result, plastic deformation took significantly increase. There was no plastic deformation before peak strength when pH value at 12, but the specimen still had residual strength; instead, the specimens break down abruptly and lost bearing capacity when it reached peak strength when pH value at 7 and 9, the failure manners still were brittle.



(a) Dry specimens



(b) Specimens soaked in CaCl₂ solutions with 0.1mol/L, pH 2

Figure 1 Triaxial compressive stress-strain curves for limestone

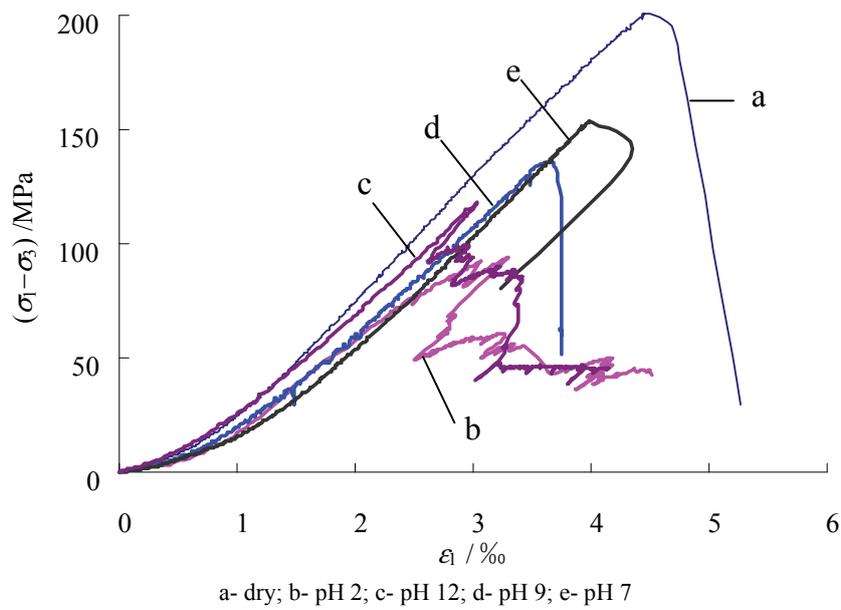
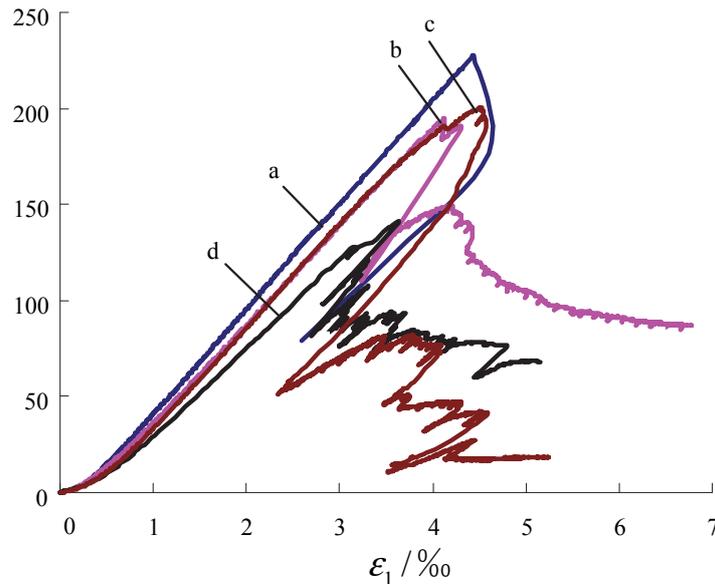


Figure 2 Triaxial compressive stress-strain curves for specimens soaked in Na₂SO₄ solutions of 0.1mol/l for 240 days with 2.5MPa confining pressure



a-dry; b- distilled water; c- CaCl₂ solution of 0.1mol/l ,pH 7; d- Na₂SO₄ solution of 0.1mol/L pH=7

Figure 3 Triaxial compressive stress-strain curves for specimens under different environments with 5MPa confining pressure

There are some differences in deformation characteristics of specimens soaked in different solution with the same pH value. Figure 3 shows typical stress-strain curves for specimens under different environments (dry, soaked in distilled water, and CaCl₂, Na₂SO₄ solution with ionic concentration of 0.1mol/l, pH value of 7 for 240 days). The characteristics of stress-strain curves after peak strength are different from each other. This is due to the fact that there are different chemical reactions between chemical solutions and limestone, and thus results in somewhat differences in the chemical corrosion effect on specimens.

It is clear on the basis of the data presented above, that the fluid environment has a substantial effect on the deformation. Generally, two factors seem to be important: the pH value and ionic components.

3.2 Influence of chemical corrosion on strength of limestone

The chemical corrosion is one of the most important factors to degrade the strength of rock. The compressive strength varied with the pH values of chemical additives.

Figure 4 shows strength results for specimens under

different environments (dry, soaked in distilled water, and CaCl₂, Na₂SO₄ solution with ionic concentration of 0.1mol/L, pH value of 2, 7, 9 and 12 for 240 days) with 10 MPa confining pressure. After soaked in Na₂SO₄ solution with ionic concentration of 0.1mol/l, the triaxial compression strength varied significantly, and become lowest with pH 2, 46.04% reduction compared to the strength of dry specimen. The reduction of the peak strength are 24.0%, 20.24%, 24.77% respectively, corresponding to the solutions with pH 7, 9, 12. The results show that pH value is a very important factor to decrease the strength of limestone with the same ionic concentration.

It is also found that the triaxial strength is dependent on the ionic components of chemical solutions. Due to the limit of space, the authors present here only the results of peak strength for specimens soaked in chemical solutions for 240 days with 10MPa confining pressure, as is shown in Figure 4. When pH keeps at a certain value, the corrosion effects of Na₂SO₄ solutions with ionic concentration 0.1mol/L are more significantly than CaCl₂ solutions with the same ionic concentration.

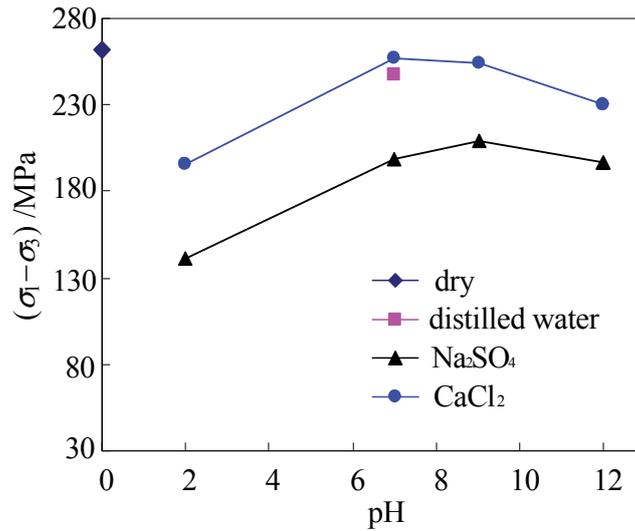


Figure 4 Peak strength for specimens soaked in chemical solutions for 240 days with 10MPa confining pressure 3.3 Fracture mode

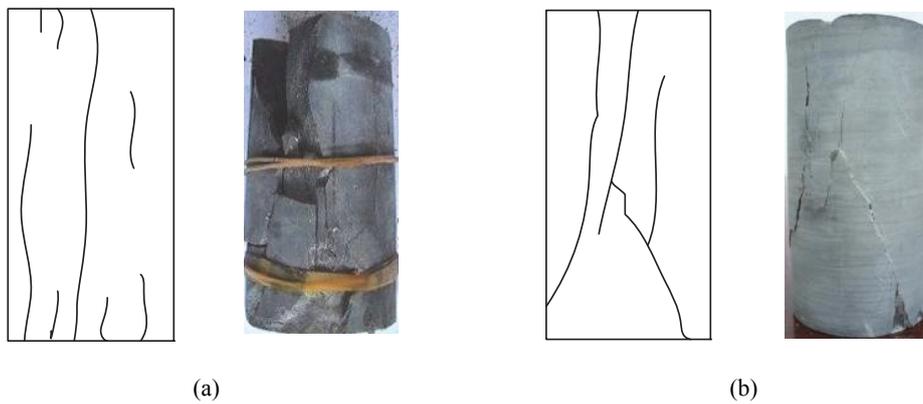
Being a typical inhomogeneous material, factors of chemical and environment let rocks inner accrue microcosmic and macroscopical defects. Cracks will be generated around these defects and extended under loading, which would lead to the final unstable failure of rocks.

The fracture modes of specimens depend on physical and mechanical properties of rock, confining pressure, chemical corrosion conditions. It could be divided into 7 types according to the location, main direction, density of cracks, as shown in Figure 5:

(a) There have many tension cracks aligned along the

axial direction. It's brittle tension failure. The specimen fell to pieces after experiment. This type occurred when the specimens were subject to low confining pressure and slight corrosion, for example, dry samples or samples soaked in alkaline solutions under 2.5MPa confining pressure.

(b) There have both tension and shear cracks. During the process of coalescence of these cracks, the specimen was broken. This maybe occurred in case of the specimens subject to slighting corrosion with confining pressure varying between 2.5 and 20MPa.



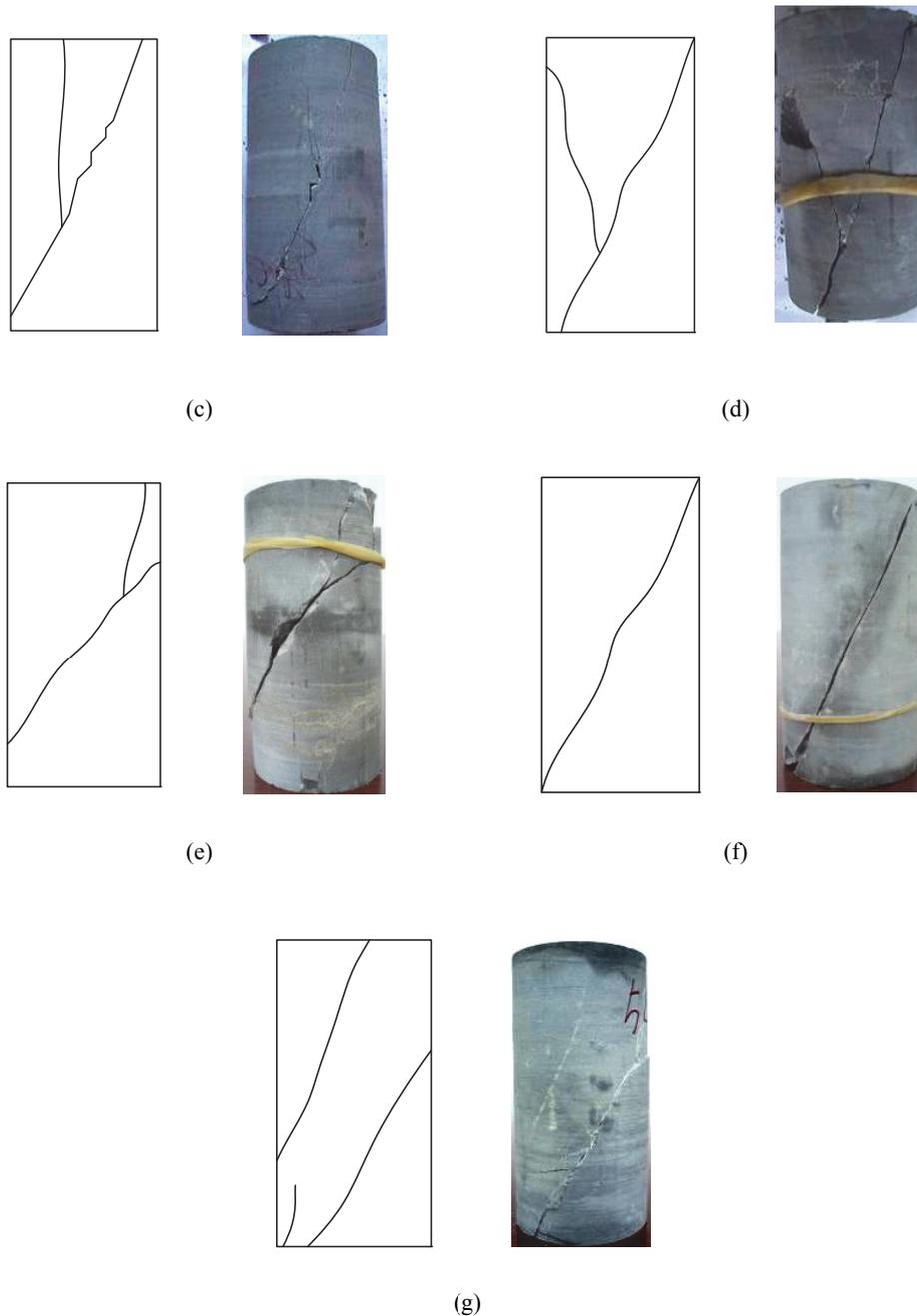


Figure 5 Schematic diagrams of cracks corresponding to photographs for specimens

(c) There is a shear crack throughout the specimen, which resulted in failure of the specimen, and there is a tension crack aligned along the axial direction at the same time. This also occurred in case of the specimens subject to slighting corrosion with confining pressure varying between 2.5 and 20MPa.

(d) There are two shear cracks similar to the shape of “V”. This occurred in case of the specimens subject to serious corrosion (such as specimens soaked in chemical solutions of pH 2) with confining pressure varying between 2.5 and 5MPa.

(e) There is a shear crack that stretches from the one side surface of the cylinder to another, which resulted in failure

of the sample. And there maybe shear cracks propagate in other local zones. This occurred in case of the specimens subject to confining pressure varying between 20 and 40MPa.

(f) There is only one shear crack that stretches from the one end surface of the cylinder to another. This occurred in case of the specimens subject to high confining pressure such as 40MPa.

(g) There are two shear cracks parallel to each other proximately. This also occurred in case of the specimens subject to high confining pressure such as 40MPa.

The failure modes are so complex under chemical corrosion and confining pressure. Based on the fracturing

mechanism, the fracture modes of samples can be divided into three groups: brittle tension model, tension and shear mixed model, shear model. In general, shear failures are more easily appear in case of serious corrosion and high confining pressure.

4 CONCLUSION

In the present study, chemical alterations of the deformation and strength of limestone have been investigated. The main conclusions are as follows:

(1) The deformation and failure behaviors of limestone are changed from brittle to ductile manner with different types of chemical solutions. Acidic solutions have the most distinct influence on deformation manners of limestone.

(2) The triaxial strength of the limestone varies significantly with pH value and ionic concentration of chemical solutions. In this study, the triaxial strengths of the specimens became the lowest in the solutions of pH 2; the corrosion effect of Na_2SO_4 solution with ionic concentration 0.1mol/l is more remarkable than CaCl_2 solutions with the same ionic concentration.

(3) Under confining pressure and chemical corrosion, the macroscopical crack forms have some differences in different specimens. According to crack inclination and quantity, the typical failure models can be classified 7 kinds. The chemical solutions and confining pressure have significant effects on the failure process and characteristics of rock.

ACKNOWLEDGMENTS

The work was supported by National Science Foundation of China under grant No. 50909053, and Key Laboratory of Geological Hazards on Three Gorges Reservoir Area (China Three Gorges University), Ministry of Education under grant No. 2008KDZ07.

REFERENCES

- [1]. FENG X T, CHEN S L, Li S J. Effects of Water Chemistry on Microcracking and Compressive Strength of Granite. *Int J Rock Mech Min Sci.* 2001, 38 (4): 557-568
- [2]. Feucht L J, Logan J M. Effects of Chemically Active Solutions on Shearing Behavior of a Sandstone. *Tectonophysics.* 1990, 175: 159-176
- [3]. Seto M, Vutukuri VS, Nag DK, et al. The effect of Chemical Additives on Strength of Rock. *Proc Civ Eng.* 1998, 603/III-44: 157-166
- [4]. Lajtai EZ, Schmidtke RH, Bielus LP. The Effect of Water on the Time-dependent Deformation and Fracture of a Granite. *Int J Rock Mech Min Sci and Geomech Abstr.* 1987, 24 (4): 247-255
- [5]. Seto M, Utagawa M, Feng X-T. Change of Chemical Environment to Change of Strength and Cracking Properties of Rocks. *Jpn Assoc Rock Mech.* 1998: 41-46
- [6]. FENG X T, Seto M. Micro-fracturing Characteristics of Rocks Under Chemical Erosion-Part I: Experiments. *Chin J Rock Mech Eng.* 2000, 19 (4): 403-407 (in Chinese)
- [7]. FENG X T, CHEN S L, ZHOU H. Real-time Computerized Tomography (CT) Experiments on Sandstone Damage Evolution During Triaxial Compression with Chemical Corrosion. *Int J Rock Mech Min Sci.* 2004, 41:181-192
- [8]. FENG X T, DING W X. Experimental study of Limestone Micro-Fracturing Under a Coupled Stress, Fluid Flow and Changing Chemical Environment. *Int J Rock Mech Min Sci.* 2007, 44 (3): 437-448
- [9]. LI N, ZHU Y M, SU B, et al. A Chemical Damage Model of Sandstone in Acid Solution. *Int J Rock Mech Min Sci.* 2003, 40 (2): 243-249
- [10]. FENG X-T, Seto M. Rock Fracturing Behaviors Under Chemical Corrosion-Part I: Experimental Study. *Chin J Rock Mech Eng.* 2000, 19 (4): 403-407 (in Chinese)
- [11]. YAO H Y, FENG X T, CUI Q, et al. Meso-mechanical Experimental Study of Meso-Fracturing Process of Limestone Under Coupled Chemical Corrosion and Water Pressure. *Rock Soil Mech.* 2009, 30 (1): 59-66 (in Chinese)
- [12]. YAO H Y, FENG X T, CUI Q, et al. Experimental Study of Effect of Chemical Corrosion on Strength and Deformation of Hard Brittle Limestone. *Rock Soil Mech.* 2009, 30 (2): 338-344 (in Chinese)
- [13]. LIU J, QIAO LP, LI P. Experimental studies and Constitutive Model of Elastoplastic Mechanical Behaviors of Sandstone With Hydro-Physicochemical Influencing Effects. *Chin J Rock Mech Eng.* 2009, 28 (1): 20-29 (in Chinese)
- [14]. ZHOU J, CHEN M, JIN Y, et al. Experimental Study on Strength Reduction Effects of Limestone Near Fracture Area During Acid Fracturing. *Chin J Rock Mech Eng.* 2007, 26 (1): 206-210 (in Chinese)
- [15]. CHEN G L, ZHOU R D. An Experimental Study Concerning the Macroscopic Effect of Water on the Deformation and Failure of Loaded Rocks. *Chin J Geophys.* 1991, 34 (3): 335-342 (in Chinese)
- [16]. ZHOU C Y, PENG Z Y, SHANG W, et al. On the Key Problem of the Water-Rock Interaction in Geoenvironment: Mechanical Variability of special weak rocks and Some Development Trends [J]. *Rock Soil Mech.* 2002, 23 (1): 124-128 (in Chinese)