Insights into the Effect of Spontaneous Fluid Imbibition on the Formation Mechanism of Fracture Networks in Brittle Shale: An Experimental Investigation

Daobing Wang,* Xiaoqiong Wang,* Hongkui Ge, Dongliang Sun, and Bo Yu

ABSTRACT: In this paper, the high-temperature/high-pressure triaxial testing system of rocks is used to study the effect of spontaneous fluid imbibition on the formation mechanism of fracture networks, by means of acoustic emission (AE) monitoring and ultrasound measurement. After the water–shale interaction, the rock mechanical parameters such as rock strength, elastic modulus, cohesion, and internal friction angle of shales significantly decrease as the imbibition time increases, indicating that the fluid has a strong influence on the mechanical properties of brittle shales. The stress–strain curves of the wet and dry shales and their AE characteristics are quite different: (i) the stress–strain curve of wet shale samples shows multiple fluctuations before macroscopic failure, and its cumulative AE number curve presents a step-like jump many times that corresponds to the local microcracking; (ii) the stress–strain curve of dry shale samples mainly shows the characteristic of linear elastic deformation during early loading, which has less AE event number, and the step-like jump is not observed in all the AE curves. The dry shale only has a large number of AE events until it is close to macroscopic failure. Nuclear magnetic resonance, mineral composition, and microstructure analysis show that Chengkou shale generally develops micro–nanoscale pores with a small pore throat, and thus strong capillary spontaneous absorption occurs. The shale–water interaction includes both chemical and physical effects, which affect the key parameters such as acoustic velocity, frictional force on the surfaces of artificial fracture, fracability, and other mechanical properties. This paper provides new insights to the investigation on the formation mechanism of artificial fracture networks in brittle shales.

1. INTRODUCTION

Shale gas resources of the main basins and regions in China occupy about 15–30 trillion cubic meters, which are roughly equivalent to that of the 28.3 trillion cubic meters of the United States. Therefore, China’s shale gas reservoir has huge potential economic values. However, because of the characteristics of ultralow porosity and ultralow permeability, it is extremely difficult for the shale gas reservoir to show commercial exploitation. Thanks to the advanced drilling and completion technology, such as horizontal well drilling and multistaged hydraulic fracturing (MSHF), shale gas is successfully developed for commercial development at present. The main goal of MSHF is to form complex artificial fracture networks by means of the stimulated reservoir volume (SRV) fracturing technology.

During the SRV fracturing of shale gas, 2–6 million gallons of fracturing fluids with proppants are injected into the formation to make the hydrofracture propagate forward with a high fracture conductivity. However, only 20–30% of fracturing fluids could be flowed back to the earth’s surface. Singh (2016) reported that the retained fracturing fluid in the subsurface was imbibed by the shale matrix, microfractures, and other fracture networks. Therefore, it is very important to
investigate the mechanism of spontaneous imbibition of fracturing fluid into shales, which could better explain the phenomenon of fracturing fluid loss and reservoir damage.\textsuperscript{12,14} The volume of spontaneous imbibition mainly depends on three aspects: the geometry of the porous media, the fluid–rock interaction, and the fluidity and capillary of the liquid.\textsuperscript{13} However, the mechanism of spontaneous imbibition in shales is more complex because of (i) the pore heterogeneity and connectivity and (ii) the electrochemical forces because of clay hydration and osmosis.\textsuperscript{11,13} The relationship between pore connectivity and wettability of a shale matrix is very critical to hydrocarbon production. By conducting the imbibition experiment on Barnett shale samples, Gao and Hu\textsuperscript{15} pointed out that the samples with a high carbonate content showed a more oil-wet property, whereas the shale samples with organic matter (OM)-hosted nanopores exhibited a mixed-wet characteristic. The lower value of water imbibition slopes was approximately equal to 0.28, which indicated that Barnett samples had a low pore connectivity to water.\textsuperscript{15} Yang et al.\textsuperscript{16} summarized the relationship between pore size distribution and imbibition curves in tight rocks. The slope of these imbibition curves varied from 0.1 to greater than 0.5. The results showed that the imbibition rate and the volume of liquid intake decreased with the decreasing pore connectivity. However, the induced microfractures by clay swelling are not considered in the above experiments, which may change the slope value of the imbibition curves.

The clay composition in the shale matrix is also very critical to the amount of water absorption. Some specific clays such as smectite and illite–smectite mixed-layer clays exhibited a strong swelling with water intake, and the clay with high specific surface area had a strong capacity of water absorption.\textsuperscript{13,16} Because of the high capillary force and the water-sensitive clay mineralogy, the spontaneous imbibition of water could induce an effective internal stress around a wellbore,\textsuperscript{16} which led to the generation of drilling induced fractures in shale gas wells. Strong water adsorption by shale samples could induce microfractures that improved the permeability nearby the artificial fracture surface or near the wellbore. This confirmed that an extended shut-in time for soaking could increase the initial production of shale gas wells.\textsuperscript{17} In addition, the rock bedding planes had a significant impact on water imbibition and salt diffusion in gas shales.\textsuperscript{18} The experimental results showed that the liquid imbibition rates along the lamination were higher than those against the lamination. The results also indicated that the confining pressure could decrease the imbibition rate which was parallel to the lamination. However, the effect of the direction which was perpendicular to the lamination on water imbibition could be negligible.\textsuperscript{18}

Because of the ultralow permeability of the shale matrix, the brittle shale has a significant capillary effect on hydraulic fracturing. After water absorption, a strong hydration effect could occur between the micropores and microfractures in a clay mineral matrix.\textsuperscript{19} Previous studies have mainly concentrated on the imbibition behavior of shales; however, the effect of the induced microfracture generation on fracture networks is not fully understood at present.

As a matter of fact, the injected slick water has a very low viscosity (only several mPa·s).\textsuperscript{20,21} The fracturing fluid is very easy to leak off into natural fractures in hydraulic fracturing. This may reduce the friction coefficient of artificial fracture surfaces. Thus, the cemented natural fractures are prone to shear slip and be activated. In addition, fluid pressure is easily transmitted into the crack tips, which may also reduce the fracture toughness of shales.

From the perspective of rock mechanics, this paper focuses on the formation mechanism of fracture networks induced by water intake in brittle shales. With the acoustic emission (AE) monitoring and acoustic velocity measurement, the mechanical behavior of brittle shales with spontaneous fluid imbibition is systematically investigated during triaxial loading. This study offers new insights into the formation mechanism of fracture networks in the effect of water intake.

2. RESULTS AND DISCUSSION

2.1. Acoustic Velocity Induced by Spontaneous Imbibition of Distilled Water. As shown in Figure 1, the longitudinal acoustic velocity $v_p$ of dry shales at each axial stress is first tested, and then each velocity of wet shale induced by spontaneous imbibition of distilled water for 24, 48, and 72 h is tested at each axial stress, respectively. Experimental results show that all the acoustic velocities of wet shale samples decrease after the spontaneous imbibition of distilled water, compared with those of dry shale samples. Furthermore, all of the acoustic velocity curves at each imbibition time (24, 48, and 72 h) are approximately parallel. This indicates that the result has a good repeatability.

2.2. Tensile Strength Induced by Spontaneous Imbibition of Distilled Water. Shale samples are processed...
into disk specimens with a 44 mm diameter and a 27 mm thickness; the specimens are naturally dried at room temperature; and then the tensile strength of each sample is respectively measured under dry and wet conditions. Under wet conditions, the shale samples experience spontaneous imbibition of different fluids for 7 days, and these fluids include distilled water, slick water, 15 wt % KCl, and kerosene. As shown in Figure 2, the experimental results show that the tensile strength of Chengkou shale significantly decreases after the spontaneous imbibition of distilled water, and the mass increment induced by the spontaneous imbibition of distilled water is the biggest among these fluids. The greater the mass increment is, the more the tensile strength decreases. The order of the tensile strength of shale samples is: dry > kerosene > 15% KCl > slick water > distilled water. It indicates that distilled water and slick water can greatly reduce the tensile strength of shales. The fracture surface characteristics of shale samples are shown in Figure 3. The fracture section is rough with caving fragments, and there are branching microcracks around the main section, which may be caused by the chemical microscopic effect during the shale-water interaction.

2.3. Fracture Toughness Induced by Spontaneous Imbibition of Distilled Water. According to the suggested standard of the International Society of Rock Mechanics (ISRM), the shale samples are processed into cracked chevron notched Brazilian disk specimens, with 50 mm diameter and 20 mm thickness. The calculated equation of mode-I fracture toughness is expressed as follows:

$$K_{IC} = \frac{P_{\text{max}}}{BD} \sqrt{\frac{a}{D}}$$

where $K_{IC}$ is the mode-I fracture toughness, $P_{\text{max}}$ is the maximum failure load, $D$ is the sample diameter, $B$ is the...
Figure 4. Results of the fracture toughness experiment: (a) parallel bedding plane; (b) normal to the bedding plane; (c) sample 03; and (d) sample 09.

Figure 5. Stress–strain curves of the dry shale samples in Lujiaping Formation: (a) sample P4h-6, $\sigma_3 = 0$ MPa; (b) sample cls-6-10, $\sigma_3 = 10$ MPa; (c) sample cls-6-16, $\sigma_3 = 15$ MPa; and (d) sample cls-6-18, $\sigma_3 = 20$ MPa.
thickness of the rock sample, and \( Y^{*}_{\text{min}} \) is the dimensionless critical stress intensity factor of the rock sample.

The shale samples are respectively cored along the parallel bedding planes and the vertical bedding planes, with five rock samples for each group. After a spontaneous imbibition of distilled water for 7 days, the fracture toughness is reduced, compared with the result under the dry condition. The fracture toughness in the parallel bedding direction is reduced by 32.8% on average, whereas the fracture toughness in the vertical bedding direction is reduced by 25.6% on average. Thus, the fracture toughness in the vertical bedding direction is higher than that in the parallel bedding direction. The fracture morphology is shown in Figure 4. We observe that the fracture surface is irregular, with caving fragments. This indicates that the rock strength decreases after the shale–fluid interaction, that is, the strength of the shale samples becomes weak. Thus, the wet sample is easy to fail along the weakness plane inside when loading.

2.4. Stress–Strain Response. The stress–strain curves of dry shale samples under different confining pressures are shown in Figure 5. We observe that, in all the cases of confining pressures, the shale is mainly subject to a linear

Figure 6. Stress–strain curves of the wet shale samples: (a) sample YC8-12, \( \sigma_3 = 0 \) MPa; (b) sample P4h-8, \( \sigma_3 = 5 \) MPa; and (c) sample S15-4, \( \sigma_3 = 15 \) MPa.

Figure 7. AE rate curves of shale samples: (a) sample cls-6-10 and (b) sample YC8-12.
elastic deformation before macroscopic failure, and the stress suddenly drops off beyond the peak value.\textsuperscript{24,25} The dilatancy point of the volumetric strain curve is not obvious. This is a typical characteristic of brittle failure. After the macroscopic failure, the stress–strain curve fluctuates many times, which shows the characteristics of the fracture networks.

The stress–strain curve of the wet shale sample under different confining pressures is shown in Figure 6. We observe that, regardless of being under the low confining pressure and high confining pressure, the stress–strain curve is characterized by many step-like jumps and fluctuates many times before the macroscopic failure. This corresponds to the formation of local microcracks in the wet shale samples. After spontaneous imbibition, the strength of the original bedding or microfractures is reduced, which easily promotes the friction sliding along the weak plane. This is quite different from the linear elastic deformation of the dry shale samples before the macroscopic failure.

2.5. Acoustic Emission Response. The curves of AE rate and cumulative AE events are respectively shown in Figures 7 and 8. We observe that, the AE curves of the wet shale have multiple step-like jumps, and these curves fluctuate many times. These fluctuations and jumps correspond to the formation of local microfractures. Instead, the dry shale sample has less AE events, and the above step-like jump cannot be seen in the AE curves. For the dry shale sample, a large number of AE events can only be observed until it is close to the macroscopic failure.

The mechanical parameters of the wet and dry shale samples under different confining pressures are shown in Table 1. With the increase of confining pressures, the rock strength, elastic modulus, and other mechanical parameters show an increasing trend. However, the strength of the wet shale sample is much lower than that of the dry shale sample. This is very consistent with the characteristics of the stress–strain and AE curves. After the shale–water interaction, the mechanical parameters of the shale samples are greatly reduced. This is conducive to the formation of fracture networks in hydraulic fracturing.

2.6. Discussion. 2.6.1. Failure Envelope. According to the failure criterion of Mohr–Coulomb (MC),\textsuperscript{24} the effect of the relationship between shear stress and normal stress on the slope is expressed as

\[ \tau = \sigma_n \tan(\phi) + c \]

where \( \tau \) denotes the shear stress along the slope, \( \sigma_n \) denotes the normal stress on the slope, \( \phi \) denotes the internal friction angle, and \( c \) denotes the cohesive strength of the rock mass.

The Hoek–Brown criterion (HB) is derived from the results of the research on the brittle failure of the intact rock by Hoek and the model studies of the jointed rock mass behavior by Brown.\textsuperscript{27,28} It is defined by the following equation

\[ \sigma_s = \sigma_3 + \frac{\sigma_3}{\text{UCS}} \left( \frac{m_b}{\text{UCS}} + s \right)^a \]

where UCS denotes uniaxial compressive strength; \( m_b \) is the value of the HB constant \( m \) for the rock mass; and \( s \) and \( a \) are constants which depend on the characteristics of the rock mass (\( s = 1 \) for intact rock, \( a = 0.5 \)). The equivalent MC criteria can also be obtained using the HB research.

Based on the lab data in Table 1, the MC and HB failure envelopes of the wet and dry shale samples are plotted in Figure 9, and the corresponding parameters such as frictional angle and cohesive strength are listed in Table 2. For each failure criterion, we observe that there is an approximate linear relationship between the rock strength and confining pressure. For the MC criterion, the cohesion of the dry shale samples is 19.89 MPa, and the internal friction angle is 46.28°. The cohesion of the wet shale samples is 19.89 MPa, and the internal friction angle is 46.28°. This indicates that both the cohesion and internal friction angle of Chengkou shale decrease after water absorption. For the HB criterion, the parameters \( m_b \) and \( s \) of wet shales are 2.90 and 0.10, respectively, whereas those of dry shales are 11.00 and 0.011, respectively. This also indicates that both the HB parameters of Chengkou shale also decrease after water absorption. This is consistent with the variation tendency of the rock mechanical parameters before and after the shale–water interaction.

![Figure 8. Cumulative AE curves of shale samples: (a) sample cls-6-10 and (b) sample YC-8-12.](https://dx.doi.org/10.1021/acsomega.0c00452)
2.6.2. Mineral Composition Analysis. By a powder X-ray diffraction (XRD) analyzer, the mineral and clay compositions of shale samples are tested, as shown in Figure 10. We observe that the clay mineral ingredients are: illite, mixed-layer illite−smectite, chlorite, and mixed-layer illite−chlorite. Among the clay mineral ingredients, the percentage of mixed-layer illite−smectite is as high as 25.04%. It indicates that Chengkou shale is easily prone to hydrate and swell after water absorption. This can lead to the shear slip along the weak plane, which corresponds to the characteristics of the stress−strain/AE curves. Thus, the wet shale samples can be easily damaged along the plane of weakness or internal microcracks. This can better explain the characteristic of multiple fluctuations in the stress−strain curves. It is caused by the shale friction failure after the water−shale interaction.

2.6.3. Microstructure Analysis. Using the technique of environmental scanning electron microscopy (SEM), the microstructure characteristics of dry and wet shale slices are tested at the same scale, as shown in Figure 11. In order to eliminate the effects of confining pressure, we select the shale samples under uniaxial stress conditions. Local natural microcracks, grain edge fractures, and mica foliation are widely distributed, and the mica sheets are oriented in directional distribution. The wet sample in Figure 11a corresponds to the sample YC8-12 in Figure 6a, and the dry sample in Figure 11b corresponds to the sample P4h-6 in Figure 5a. There are organic matter (OM) and pyrites in the shale samples. We observe that the wet shale samples contain large pores or microfractures, whereas the dry shale samples have relatively small pores or microfractures. This indicates that the shale−water interaction results in the generation of internal microcracks. This is also very consistent with the above experimental results. Water absorption has a significant impact on the acoustic velocity of shales, which greatly exceeds the expectation of the existing Gassmann equation. Conventional rock physical models cannot well explain the variation law of the acoustic velocity of shales, and the microscopic physical mechanism related to capillary force is in effect. The interaction between water and shale includes both chemical and physical effects, which have a wide influence on the mechanical properties of shale, such as acoustic velocity, permeability, fracability, and the formation of fracture networks.

2.6.4. Nuclear Magnetic Resonance Response Analysis. The nuclear magnetic resonance (NMR) response can reflect the characteristics of pore structure in shale samples. Before the test, the shale samples experience a spontaneous imbibition of distilled water, 15 wt% KCl, and kerosene for 7 days, respectively, and the results are shown in Figure 12. The samples correspond to the shale samples saturated with different fluids in Section 2.1. We observe that their transverse relaxation time is mainly distributed within a period of 0.1−10 ms. This indicates that the pore throat radius of the shale sample is very small and, in particular, the pores at the micro and nanoscale levels are well developed. Meanwhile, potassium chloride could inhibit the absorption of clay minerals because the amplitude is smaller than that of the sample saturated with distilled water. The Chengkou shale sample has stronger hydrophilicity because it absorbs more distilled water than kerosene. In addition, within a period of 10−10,000 ms, the amplitude of the T2 spectrum is very small. This indicates that the shale sample has ultralow water saturation, and micro and nanoscale pores, that leads to a strong swelling effect after water absorption. This may be one of the microscopic reasons for the change of rock mechanical properties after the shale−water interaction.

2.6.5. Experimental Analysis of Mercury Porosimetry at High Pressure. In order to analyze the characteristics of micro-

![Figure 9. Failure criterion of the dry and wet shale samples: (a) wet shale and (b) dry shale.](image)

![Table 2. Comparison of the MC and HB Failure Criteria for Wet and Dry Shale Samples](table)

| State   | Failure Criterion | Angle (deg) | Pressure (MPa) |
|---------|------------------|-------------|----------------|
| Wet     | MC criterion     | 34.12       | 13.53          |
|         | HB criterion     |             | 2.90 0.10 0.50 |
| Dry     | MC criterion     | 46.28       | 19.89          |
|         | HB criterion     |             | 11.00 0.11 0.50|

![Figure 10. Results of clay mineralogy by means of XRD.](image)
and nanopore throats of the shale samples, we conduct a high-pressure mercury injection experiment. The results are shown in Figure 13. It can be seen that the pore throat radius of shale samples is distributed in the range of $0.01\,\text{to}\,10\,\mu\text{m}$, and the pores are developed at the nanoscale. Both the displacement pressure $P_T$ and the median saturation pressure $P_{c50}$ are high. This indicates that the shale matrix is very tight. The saturation distribution curve shows that there is ultralow water saturation in the shale samples, and the developed micro- and nanopores strongly absorb water. This is very consistent with the experimental results in this paper. The mercury porosimetry experiment shows that the Chengkou shale sample has an extremely low matrix permeability, and the micro–nanometer pore fractures are well developed, with small pores/micro-

Figure 11. Experimental results of SEM: (a) wet shale sample and (b) dry shale sample.

Figure 12. T2 spectrum of NMR.

Figure 13. Results of mercury injection at high pressure: (a) mercury injection curve and (b) capillary radius distribution curve.
fractures and strong spontaneous imbibition, which are quite different from the conventional reservoirs.

2.6.6. Effects of Fluid Viscosity on the Formation of Fracture Networks. Besides the capillary effects and the high contents of the swelling clay, the viscosity of the injected fluid is another important factor that impacts the fracture complexity in hydraulic fracturing. In contrast to the gelled fracturing fluids, slick water has a lower viscosity and it is much easier to transmit to the crack tips in hydraulic fracturing. Wang et al. investigated the effect of three kinds of fluids, that is, water, viscous oil, and supercritical CO2 on the formation of fracture networks. They reported that supercritical CO2-based fracturing has a lower breakdown pressure than the other two kinds of fluids, and it is easier to develop complex fracture networks in hydraulic fracturing. This is consistent with our experimental observations because the viscosity of fluids in the experiment is close to 1 mPa·s, which is lower than that of the gel-based fracturing fluids.

3. CONCLUSIONS

In this paper, a servo-controlled high-temperature/high-pressure triaxial cell of rocks is used to investigate the effect of spontaneous water imbibition on the formation mechanism of fracture networks in shales, by means of AE monitoring and acoustic wave measurements. It provides a scientific guidance for the design of SRV fracturing in shale gas reservoirs. The main conclusions are as follows:

(1) After the shale samples experience a spontaneous imbibition of distilled water, the rock mechanical parameters such as rock strength, elastic modulus, cohesion, and internal friction angle are significantly reduced. This indicates that the fluid can exert an important impact on the mechanical properties of brittle shales.

(2) Although both the cohesion and friction coefficient of the wet shale samples decrease after the spontaneous imbibition of distilled water, they satisfy both the MC and HB failure criteria under different confining pressures.

(3) The wet shale and dry shale samples have different AE characteristics: the wet shale samples have multiple step-like jumps in the AE curves, and the steps are very steep that correspond to the local microfractures; however, the dry shale samples have less AE event during the early loading, and the step-like jumps disappear in the AE curves of the dry shale samples. The dry shale samples only have a large number of AE events until they are close to the microscopical failure.

(4) The stress–strain characteristics between the wet and dry shale samples are quite different: the strain–stress curve of the wet shale samples has multiple fluctuations before the microscopical failure that correspond to local microfractures, whereas the dry shale samples mainly show the characteristic of linear elastic deformation.

(5) NMR, XRD, and SEM analyses show that the water–shale interaction includes both chemical and physical effects. The ultralow water saturation of shales enhances the water absorption, which affects the acoustic velocity of shales, frictional failure, and fracability. It is necessary to further carry out a large number of experimental studies to analyze the mechanism of the formation of fracture networks induced by spontaneous imbibition in shales.

4. EXPERIMENTAL SETUP AND PROCEDURE

4.1. Sample Preparation. The black shale outcrops were deposited in the Lower Cambrian Lujiaping Formation, Sichuan Basin, China. As shown in Figure 14, the samples were drilled perpendicular to the bedding direction and cored into a standard cylindrical shape with 25 mm diameter and 50 mm height. To ensure a perfect parallelism of the ends of the samples, progressively finer grades of abrasive paper were used to polish the core samples while the samples were completely immersed in the formation fluid. This could provide less than 0.01 mm parallelism, which minimizes the friction on the end surface when loading. To ensure that these cored samples can represent the shale properties in the subsurface, the paraffin wax sealing method was adopted after drilling, and the core samples were stored in the formation fluid to avoid the weathering effect.

4.2. Description of Chengkou Shale. The study area is located in the Dabashan thrust belt. The belt is situated at the northern margin of the upper Yangtze block, which is in the transitional position between Sichuan Basin and Qinling Orogenic Belt. The total organic carbon content of Chengkou shale is between 1.79 and 10.40%, with an average value of 5.60%, which shows a high-quality source rock. The thermal evolutionary extent of Chengkou shale is very high because the vitrinite reflectance (Rv) is in the range of 3.01–3.70%, with an average value of 3.17%. Most of kerogen is of type I, and the others belong to kerogen type III. The porosity of Chengkou shale varies from 4 to 6%, with an average value of 4.37%, and the permeability is in the range of 0.0001–0.001 mD with an average value of 0.00056 mD. The main minerals are quartz, calcite, dolomite, potassium feldspar, plagioclase, pyrite, and clay minerals. Among these, the volume fraction of quartz is the highest, ranging from 9.0 to 89.0%, with an average of 49.7%, followed by calcite, with an average volume fraction of 21.1%; the average volume fraction of dolomite is 10.1% and that of clay mineral is 8.1%.

4.3. Experimental Equipment. In this experiment, the TAW series servo-controlled rock triaxial loading system is used to test the stress–strain curves, which is produced in China. Meanwhile, the PCI-II system produced by American Physical Acoustics Corporation is used for AE monitoring. The TAW series apparatus could provide a maximum axial force of 2000 kN, with a capacity of 70 MPa pore pressure and 140 MPa confining pressure, respectively. The circumferential and axial strains are measured by the strain gauges of linear variable differential transducer. The rock sample is confined

Figure 14. Cylindrical shale samples with about 25 mm diameter and 50 mm height.
within a heat-shrink Teflon tube in order to prevent silicone oil from migrating to the external surfaces of samples. The sampling rate of the PCI-II AE monitoring system is up to 40 MHz with an 18-bit A/D converter, which has the quality of continuous waveform recorded. This system can acquire a total of 20 characteristic parameters including AE events, energy, ring counts, and so forth. In addition, the stress–strain value of the rock samples can be simultaneously recorded in the AE acquisition system.\(^{26}\)

The acoustic velocity measurement system consists of an Olympus 5077PR electric pulse generator/receiver and an oscilloscope. To test the acoustic velocities of the rock samples at each stress level, a pair of nano-30 compressional wave transducers are clinged tightly to the end faces of the rock sample, and the cross-correlation technology of waveform is utilized to pick up the arrival time of acoustic velocities at each stress. To improve the contact area between the sample ends and transducers, a gelled couplant agent is painted on the surface of the rock samples.

4.4. Experimental Procedure and Methods. In the experiment, the strain rate is controlled at a strain rate of 2 \( \times 10^{-5} \) s\(^{-1} \) until the rock sample fails. The volumetric strain \( \varepsilon_r \) is calculated by the summation of the axial strain \( \varepsilon_a \) plus twice the radial strain \( \varepsilon_r \).\(^{24}\) In order to record and monitor the AE event waveform in real time, a nano-30 transducer is glued to the side of rock samples (position precision \( \pm 0.5 \) mm). The size of nano-30 is 8 mm \( \times \) 8 mm with a 50–750 kHz bandwidth. The preamplifier gain is set to 40 dB at a 10 MHz sampling rate. The filter bandwidth is chosen in the range of 100 kHz–2 MHz, and the length of single recording AE-data is up to 15 kB.

The procedure of the experiment in this investigation is conducted as follows:

(a) Under the condition of uniaxial compression, the acoustic velocities at each axial stress are tested at a constant stress rate of 2 MPa/min. Before loading, the samples are saturated with distilled water for 24, 48, and 72 h respectively, for comparison with the dry samples. In the process of loading, arrival times are determined using cross-correlation to a reference waveform; accordingly, the relative error of velocity measurement is limited to be lower than 1\%. A total of 100 waveforms are stacked at each stress level in order to increase the signal/noise ratio. The maximum axial stress is up to 50 MPa, which is about 25% of the uniaxial compressive strength of shale samples. This value can ensure that the samples are in the stage of elastic deformation when loading.

(b) The mechanical parameters including the tensile strength and fracture toughness are tested under the condition of uniaxial compression. Before loading, the shale samples are respectively saturated with different fluids such as distilled water, slick water, 15 wt% KCl solution, and kerosene for 7 days, for comparison with the dry samples. The maximum value of force on the displacement–force curves is selected to calculate the corresponding mechanical parameters according to the standards suggested by ISRM. To raise the experiment precision and effect, three samples are loaded for each group and then the average value is obtained to analyze the experimental results.

(c) The stress–strain curves of shale samples are obtained under triaxial compression conditions. The triaxial loading is conducted at a controlled strain rate of 2 \( \times 10^{-5} \) s\(^{-1} \). For each sample, the confining pressure is set as either 0, 5, 10, or 15 MPa in order to obtain the failure envelope. Meanwhile, the AE features between the wet and wet dry shale samples are also recorded during loading. Before loading, the samples are saturated with distilled water for 7 days, for comparison with the dry shale samples.

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\section*{Notes}

The authors declare no competing financial interest.

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\section*{NOMENCLATURE AND UNITS}

| Symbol | Description |
|--------|-------------|
| \( E \) | Young’s modulus, GPa |
| \( \nu \) | Poisson’s ratio, dimensionless |
| \( \tau \) | shear stress, MPa |
| \( \sigma_n \) | normal stress, MPa |

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\[ \phi \] internal friction angle, °

\[ c \] cohesive strength, MPa

\[ \sigma_1 \] the maximum principal stress, MPa

\[ \sigma_3 \] the minimum principal stress or confining pressure, MPa

\[ \epsilon_1 \] the axial strain, dimensionless

\[ \epsilon_2 \] the radial or circumferential strain, dimensionless

\[ \epsilon_v \] the volumetric strain, dimensionless

\[ v_p \] the longitudinal wave velocity, km/s

\[ S_1 \] deviatoric stress, MPa

\[ \rho \] rock density, g/cm³

\[ K_{IC} \] fracture toughness, MPa-m¹/²

\[ P_{max} \] the maximum failure load value, kN

\[ B \] the sample thickness, cm

\[ D \] the sample diameter, cm

\[ Y_{cr}^\phi \] the dimensionless critical stress intensity factor of the sample