Damage caused by freeze-thaw treatment with liquid nitrogen on pore and fracture structures in a water-bearing coal mass

Haifei Lin1,2 | Jinliang Li1,2 | Min Yan1,2 | Shugang Li1,2 | Lei Qin1,2 | Yizhen Zhang1,2

1College of Safety Science and Engineering, Xi'an University of Science and Technology, Xi'an, China
2Key Laboratory of Western Mine Exploitation and Hazard Prevention, Ministry of Education, Xi'an University of Science and Technology, Xi'an, China

Correspondence
Haifei Lin, College of Safety Science and Engineering, Xi'an University of Science and Technology, Xi'an 710054, China.
Email: lhaifei@163.com

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Abstract
The purpose of the study was to explore the microscopic and mesoscopic damage mechanisms of water-bearing coal mass in the freeze-thaw process with liquid nitrogen. For this purpose, by using a Zeiss microscope, a nonmetal ultrasonic velocity detector and a nuclear magnetic resonance (NMR) spectrometer, the changes in the structures of surface fractures and pores before and after coal masses with different moisture contents when subjected to freeze-thaw treatment with liquid nitrogen (FTTLN) were tested. The result showed that under the freeze-thaw effect of liquid nitrogen, the structures of surface fractures and internal pores of coal mass were both changed. Upon increasing the moisture content of the coal, the width of surface fractures increased; the surface fractures showed significant fractal characteristics: The higher the moisture content of the coal, the greater the rate of change of fractal dimension after FTTLN; the ultrasonic wave velocity in the coal mass gradually decreased after FTTLN, and the greater the moisture content, the more significant the decrease in ultrasonic wave velocity; under the freeze-thaw effect of liquid nitrogen, the pore structure in the coal mass was transformed from micropores and small pores to mesopores and macropores and certain new micropores and small pores were generated. Upon increasing the moisture content of the coal mass, the peak area of micropores and small pores and their proportion decreased at first and then increased (reaching the minimum at 9.12% of moisture content); the peak area, peak area proportion, and porosity of mesopores and macropores always increased. The damage factor attained based on the width of fractures, longitudinal wave velocity, fractal dimension of surface fractures, and porosity was favorably correlated with the moisture content: With increasing moisture content, the coal mass became more severely damaged.

KEYWORDS
damage, freeze-thaw treatment with liquid nitrogen, moisture content, pore structure
China’s coal reserve and associated gas content are abundant; however, owing to coal seams in a majority of mining areas having low permeability, the pre-extraction effect of gas is nonideal and coal mining and development of gas in coal seams are restricted. How to improve the permeability of coal seams has been key to preventing and controlling gas build-up in coal seams of low permeability and improving the extraction efficiency. At present, the commonly used technologies for increasing permeability of coal seams mainly involve hydraulic technologies (such as hydraulic fracturing, hydraulic flushing, hydraulic slotting, and increasing permeability through acidification) and blasting technologies (including powder blasting, electrical firing, and gas blasting); however, hydraulic technologies face the following problems: high water consumption, the water-lock effect, and environmental pollution from the fracturing liquid; blasting can cause secondary damage, and entails relatively complex operations. In recent years, to overcome the aforementioned drawbacks, waterless fracturing technology based on N2, CO2, liquid nitrogen, et al has been developed.

As a cryogenic fluid, liquid nitrogen has a gasification temperature of −195.8°C and it expands by 694 times after being gasified at room temperature. There are numerous factors influencing the effect of increasing permeability of coal-rock masses with liquid nitrogen, in which moisture content in coal-rock masses is key. By carrying out cold shock tests with liquid nitrogen on dry and water-saturated briquettes, Qin et al measured the change in wave velocity in coal briquettes before and after cold shocking with liquid nitrogen. Gao et al experimentally investigated the damage to pore structure in rocks under liquid nitrogen freezing conditions and they observed the damage condition of pore structure in rocks. Yang et al explored the influences of immersion in liquid nitrogen on the fracturing effect of coal samples with different moisture contents and initial temperatures. Yin et al conducted leaching experiments with liquid nitrogen on dry raw coal samples and observed the propagation of primary fractures and initiation of new fractures before and after leaching by using liquid nitrogen by applying a laser microscope. By conducting liquid nitrogen freezing tests on rocks with different lithologies and degrees of saturation, Zhang et al analyzed the changes in mechanical parameters after liquid nitrogen freezing. Wei et al analyzed the evolution of pore structure characteristics in a coal mass under the effect of freeze-thaw cycling by conducting freeze-thaw cycle tests on water-saturated coal samples. Using computerized tomography (CT) scanning, Jin et al found that the effect of cold shock with liquid nitrogen on the pore structure of coal-rock masses exhibits a certain correlation with the original porosity. Beier et al conducted semileaching tests on coal samples with different degrees of saturation and measured changes in wave velocity in coal samples and extension of the width of surface microfractures before and after liquid nitrogen treatment.

The aforementioned research mainly focusses on exploring the changes in pores and fractures in water-bearing coal masses under freeze-thaw cycling with liquid nitrogen. The evolution and characteristics of the damage to pores and fractures in a coal mass before and after freeze-thaw treatment with liquid nitrogen (FTTLN) warrant further exploration. Investigating the problem is important in that it could reveal microscopic and mesoscopic damage, deformation, and failure mechanisms in a coal mass during freeze-thaw cycling. By collecting coal samples from low-permeability mines with high gas contents, the development of surface fractures and pore distribution characteristics in coal masses with different moisture contents before and after FTTLN were explored. We used a Zeiss microscope, a nonmetal ultrasonic wave velocity detector, and nuclear magnetic resonance (NMR) technology. On this basis, different damage factors were defined and the evolution of damage via pores and fractures in a coal mass was analyzed, with a view to providing a certain theoretical basis for increasing the permeability of a coal mass based on FTTLN.

## 2 EXPERIMENTAL

### 2.1 Preparation of coal samples

Samples of blocky raw coal, taken from the same position in a coal seam of Hengyi Coal Mine in Shaanxi Province, China, were transported to the laboratory after being sealed and boxed. Cylindrical standard coal samples with a height of 100 mm and diameter of 50 mm were prepared by using a core-drilling machine and two end faces perpendicular to the axis were polished. Proximate analysis was carried out on the coal samples (Table 1). $M_{\text{ad}}$, $A_d$, $V_d$, and $FC_{\text{ad}}$ represent Moisture, Ash, Volatile, and Fixed carbon, respectively.

After the samples were dried to constant mass, 15 coal samples with insignificantly different appearances and approximately equal masses were selected for testing. These coal samples were then grouped according to their different moisture contents after saturation (Table 2).

### 2.2 Test scheme

1. The qualified coal samples through screening were divided into five groups, in each of which there were three

| Coal sample | $M_{\text{ad}}$ (%) | $A_d$ (%) | $V_d$ (%) | $FC_{\text{ad}}$ (%) |
|-------------|---------------------|-----------|-----------|---------------------|
| Proximate result | 4.04 | 10 | 31.96 | 55.69 |
coal samples. The five groups were designated groups: A (average moisture content of 11.67%), B (9.12%), C (6.06%), D (3.14%), and E (dried).

2. By using the Zeiss stemi 508 microscope produced by Beijing PRECISE Corporation (China), with a zoom ratio of 8:1 and maximum magnification of 100 times, the development of surface fractures on coal samples was observed and recorded.

3. By applying the NM-4B nonmetal ultrasonic velocity detector produced by Beijing CONCRETE Corporation in China, with an accuracy and range of transit time measurement of 0.1 μs and 0.1-210 000 μs, respectively, the changes in longitudinal wave velocity in coal samples were measured.

4. Using the ZYB-II vacuum saturator, the coal samples were saturated for 24 hours under a pressure of 30 MPa and then weighed until the mass of coal samples stabilized. The water-saturated coal samples were subjected to NMR test by applying a MacroMR12-150H-I NMR imaging analyser produced by Shanghai Niumag Science and Technology Corporation, China, under a magnetic field intensity of 0.3 ± 0.05 T and a magnet uniformity of 35 ppm. On this basis, the distribution curves of the $T_2$ spectra of coal samples were attained.
5. The tested samples were placed in a double-decked vacuum insulation bucket, to which liquid nitrogen was slowly added. After the liquid nitrogen completely flooded the coal samples, the bucket was covered. After being immersed for 1 hour, the coal samples were removed to recover to normal temperature.

6. By cyclically conducting steps (2), (3), and (4), the development of surface fractures, distribution of longitudinal wave velocity, and $T_2$ spectra of coal samples after FTTLN were measured. The results were compared with data measured before FTTLN. The experimental procedure is shown in Figure 1.

3 | RESULTS AND ANALYSIS

3.1 | Propagation and evolution of surface fractures in a coal mass

3.1.1 | Test results: surface fracture propagation in a coal mass

By observing the surfaces of each group of coal samples, visible fractures were selected as characteristic fractures and marked thus. The characteristic fractures of coal samples before and after FTTLN were observed by using an optical microscope, and the selected characteristic fractures are shown in Figure 2. Based on the ranging function using the scale in the microscope system, the widths of fractures at three selected positions (points A, B, and C) in Figure 2 were measured (Table 3).

The relationship between the crack expansion before and after freeze-thaw treatment of the average width of characteristic fractures and the moisture content is plotted (Figure 3): $d$ and $w$ refer to the propagation extent ($\mu$m) of fracture width and the moisture content (%), respectively. It can be seen from Table 3 and Figure 3 that the fracture width of dried coal samples (group E) after FTTLN increased from 138.87 to 169.82 $\mu$m, increasing by 30.95 $\mu$m, with the expansion rate of 15.08%; after conducting FTTLN, the fracture width of coal samples in group A with the moisture content of 11.67% increased from 319.26 to 478.01 $\mu$m, by 158.75 $\mu$m, showing a propagation rate of 49.72% (being 3.3 times that of dried coal samples). The fractures in coal samples after FTTLN all propagated, in which the rate of propagation of fracture width in water-bearing coal samples was greater than that of dried coal samples; with increasing moisture content, the rate of propagation of fracture width increased linearly. The reason was that the propagation of fractures in coal samples was related to the volumetric expansion of water caused by freezing in the coal samples. Moreover, the frost heave exceeded the tensile strength of these coal samples, thus leading to the propagation of fractures. (The thermal stress caused by temperature difference during FTTLN also affects fractures in the coal samples, other conditions being equal, the temperature difference remains the same, so the effect of thermal stress on the fractures in coal samples remains unchanged, the frost-heave effect played the dominant role in the damage to a water-bearing coal mass subjected to FTTLN). Under freeze-thaw conditions with liquid nitrogen, the moisture content influenced the level of damage: The higher the moisture content, the greater the frost heave and the greater the propagation and development of pores and thus more severe the damage to the coal samples.

**FIGURE 2** Surface fractures and damage to coal samples
3.1.2 | Evolution of propagation and fractal characteristics of surface fractures of a coal mass

To explore the changes in the fractures in coal samples before and after FTTLN, the images of fracture zones were partitioned and binarized by introducing box-correlation integration analysis software for calculation of fractal dimension. Limited by the length of the study, only two groups of images original and binarization are shown (Figure 4).

According to the definition of box dimension, the fractal dimension $D$ in a fracture zone is:

$$\ln N(L) = \ln A - D \ln L$$

(1)

where $L$, $A$, and $N(L)$ represent the side length of the grid, a constant, and the number of grids covering the damaged fracture zone, respectively.

According to Equation (1), the fractal dimensions after coal samples with different moisture contents were subjected to FTTLN can be calculated (Table 4).

The relationship between the increment of average fractal dimension and the moisture content is plotted (Figure 5, where $n$ denotes the fractal dimension). According to Table 4 and Figure 5, under the freeze-thaw effect of liquid nitrogen, the evolution of surface fractures of coal samples was fractal: The greater the fractal dimension, the more complex the development of fractures in the coal sample. The fractal dimensions of dried coal samples before and after FTTLN were 1.45 and 1.49, respectively, increasing by 0.04, a growth rate of 2.75%; the fractal dimensions of coal samples with a moisture content of 11.67% before and after FTTLN were 1.38 and 1.53, respectively, increasing by 0.15, a growth rate of 10.87% (being 5 times that of dried coal samples). As the moisture content increased, the fractal dimension positively exponentially increased. This trend was similar to that between the fracture width and the moisture content, which indicated that FTTLN can

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**TABLE 3** Fracture widths of coal samples with different moisture contents before and after FTTLN

| Group | Moisture content (%) | Before FTTLN fracture widths (μm) | After FTTLN fracture widths (μm) |
|-------|----------------------|-----------------------------------|-----------------------------------|
|       | A                    | B                   | C                   | Average | A                    | B                   | C                   | Average |
| A     | 11.67                | 297.45              | 377.68              | 282.65  | 319.26               | 455.49              | 592.33              | 386.21  | 478.01              |
| B     | 9.12                 | 445.87              | 686.24              | 153.26  | 428.46               | 617.23              | 833.25              | 224.56  | 558.35              |
| C     | 6.06                 | 338.69              | 423.63              | 301.25  | 354.52               | 402.55              | 568.24              | 352.10  | 440.96              |
| D     | 3.14                 | 296.77              | 201.12              | 133.60  | 210.49               | 335.31              | 228.57              | 191.23  | 251.70              |
| E     | 0                    | 113.12              | 205.98              | 97.52   | 138.77               | 131.55              | 234.70              | 113.23  | 159.82              |

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**F I G U R E 3** Relationship between fracture width and moisture content

**F I G U R E 4** Surface fractures on coal samples
effectively increase fracturing in a coal mass, thus further increasing its permeability.

### 3.2 | Changes in microscopic and mesoscopic fractures in a coal mass

#### 3.2.1 | Test results: wave velocity measurement in a coal mass

Ultrasonic wave velocity reflects the density of the propagating medium, and the propagation velocity of sound waves gradually reduces in solid, liquid, and gas phases successively. Therefore, through further inversion, the changes of microscopic and mesoscopic fractures in a coal mass were obtained. The wave velocities in coal samples before and after FTTLN measured by nonmetal ultrasonic velocity detector are shown (Table 5): The wave velocity of dried coal samples (group E) decreased from 1459 to 1384 m/s, a decrease of 75 m/s, with a rate of change of 5.11%; after FTTLN, the wave velocity of coal samples in group A with a moisture content of 11.76% decreased from 1633 to 822 m/s, a decrease of 811 m/s, with a rate of change of 49.68% (being 9.72 times that of dried coal samples).

#### 3.2.2 | Changes in wave velocity in a coal mass

Figure 6 shows the relationship between the increment (the difference of wave velocities before and after FTTLN) of average wave velocity of coal samples on freeze-thaw conditions and corresponding moisture content: \( v \) denotes the increment (m/s) in longitudinal wave velocity.

As shown in Figure 6, with increasing moisture content, the difference of wave velocities before and after FTTLN exponentially increased, the main reason for this was that sound waves propagate at different velocities in different media. Under identical conditions, the velocity of sound waves in a solid was faster than those in liquids and air. The cryogenic temperature effect of liquid nitrogen led to a change of internal structure of coal samples, thus influencing the mode and path of ultrasonic wave propagation. Freeze-thaw effects caused the primary fractures in coal samples to propagate and grow and new fractures were generated. As a result, the propagation of sound waves through air increased, decelerating the velocity of sound waves in more damaged coal samples (the higher the moisture content, the more significant this effect).

### 3.3 | Evolution of the pore structure in a coal mass

#### 3.3.1 | NMR results of a coal mass

During NMR, the pore-size distribution of pores in coal samples was detected mainly by testing the hydrogen ions in moisture in pores in water-bearing coal samples. On this basis, the relaxation time \( T_2 \) of pores in coal samples of different moisture contents and corresponding \( T_2 \) signal intensity could be attained. The relaxation time \( T_2 \) characterizes the size of pores in a coal mass and the \( T_2 \) signal intensity characterizes the number of pores of corresponding size. A representative coal sample was separately selected from five groups of coal samples: Figure 7 shows the \( T_2 \) spectra of coal samples from groups A to E before and after FTTLN. During NMR testing, the relationship between the transverse relaxation time \( T_2 \) and the specific surface area (SSA) of pores in coal samples can be expressed as follows:

\[
\frac{1}{T_2} = \rho \frac{S}{V}
\]

where \( T_2 \), \( \rho \), \( S \), and \( V \) refer to the transverse relaxation time (ms), intensity (m/ms) of transverse surface relaxation, SSA of pores (m\(^2\)/g), and pore volume (cm\(^3\)/g), respectively.

The radius of the pores is positively proportional to their SSA:
where \( r \) and \( F_s \) denote the radius of pores (μm) and the geometric shape factor, respectively.

From Equations (2) and (3), Equation (4) follows:

\[
\frac{1}{T_2} = \rho F_s \frac{1}{r}
\]  

(4)

According to Equation (4), it can be seen that \( T_2 \) distribution showed certain regularity consistent with that of the radius \( r \) of pores: The larger the \( T_2 \) value, the larger the radius of pores; the greater the \( T_2 \) peak area, the larger the number of pores. According to Equation (4), \( T_2 \) is converted. For columnar pores, \( F_s \) and \( \rho \) are 2 and 0.5 × 10^{-8} \text{ m/s}. In this condition, the pore sizes (as shown in Figure 7) corresponding to \( T_2 \) value could be attained.

By using the \( \chi_0 \) classification method for pores, the pores were divided into micropores (<10 nm), small pores (10-100 nm), mesopores (100-1000 nm), and macropores (>1000 nm).31

The \( T_2 \) spectral area characterizes the total porosity of the coal samples: The proportion of different wave crest areas in the total area corresponds to the ratio of pores with different sizes to the total pores in coal samples. Therefore, according to Figure 7, the spectral areas of micropores, small pores, mesopores, and macropores in coal samples with different moisture contents and their proportions before and after FTTLN were obtained (Table 6). Based on Figure 7 and Table 6, it can be seen that the number of micropores and small pores played a dominant role while there was a small number of mesopores and macropores. The number of micropores and small pores was much greater than that of mesopores and macropores. After FTTLN, the \( T_2 \) spectra were right-shifted, implying that the number of micropores and small pores decreased while that of mesopores and macropores increased after FTTLN.

### Table 5

| Group | Moisture content (%) | Before FTTLN (m s\(^{-1}\)) | After FTTLN (m s\(^{-1}\)) |
|-------|---------------------|-----------------------------|-----------------------------|
| A     | 11.67               | 1633                        | 822                         |
| B     | 9.12                | 1653                        | 1212                        |
| C     | 6.06                | 1486                        | 1282                        |
| D     | 3.14                | 1593                        | 1448                        |
| E     | 0                   | 1459                        | 1384                        |

*FIGURE 6*  Relationship between wave velocity and moisture content

![Wave velocity and moisture content](image)

\[
v = 57.3261 e^{0.2263w}
\]

\( R^2 = .9951 \)

The relationships of the increase in wave crest area and peak area proportion in \( T_2 \) spectra with moisture content are plotted (Figure 8). In the figure, \( S_m, S_s, P_m, P_s, \) and \( w \) represent the peak area of micropores and small pores, the peak area of mesopores and macropores, the proportion (%) of peak area of micropores and small pores, the proportion (%) of peak area of mesopores and macropores, and the moisture content (%), respectively.

It can be seen from Figure 8A that the increase in peak area of micropores and small pores first decreased, then increased: When the moisture content was increased from 0% to 9.12%, the increase in peak area of micropores and small pores always decreased, in which the minimum increase (6.63%) occurred at a moisture content of 9.12%. This indicated that micropores and small pores were transformed into mesopores and macropores, and the number of transformed micropores and small pores was greater than that of new micropores and small pores. At a completely water-saturated state of 11.67% moisture content, the peak area of micropores and small pores increased. This implied that the number of new micropores and small pores played a dominant role and was greater than the number transformed to mesopores and macropores. The peak area of mesopores and macropores increased linearly. At a moisture content of 11.67%, the increase in peak area of mesopores and macropores reached a maximum of 10.23%, which was 4.32 times that of dried coal samples.

With increasing moisture content, the proportion of pores with different sizes changed. According to Figures 7 and 8B, it can be seen that the peak area proportion of micropores and small pores was the largest, followed by that of mesopores and macropores. The increase in the peak area proportion of micropores and small pores first
decreased, then increased (reaching a maximum of 5.09% at a moisture content of 9.12%), which matched the trend in growth of the peak area of micropores and small pores. The increase in the peak area proportion of mesopores and macropores was linear with increasing moisture content, and it reached a maximum (5.03%) at the completely water-saturated state (a moisture content of 11.67%). The higher the moisture content, the more significant the effect of frost heave: There were many micropores and small pores while a smaller number of mesopores and macropores were present in the original coal samples; under the effects of frost-heave force, pores grew and interconnected. Eventually, some micropores and small pores were transformed into mesopores and macropores so that the number of mesopores and macropores increased. During the later stages of testing, some new micropores and small pores were generated and the number of new micropores and small pores was greater than that of transformed pores. The higher the moisture content, the greater the damage to the coal mass, which was conducive to increasing the gas permeability of the coal mass and improving the gas extraction efficiency.

3.3.3 | Changes in porosity

The ratio of the total pore space in coal mass to the volume of coal mass is its porosity. To some extent, total porosity can be used to characterize the potential for gas seepage from a coal mass. By using the NMR imaging analyser, the porosities of coal samples with different moisture contents before and after FTTLN can be measured (Table 7). Moreover, the relationship between the increment (the difference of porosities before and after FTTLN) of porosity and the moisture content
is plotted (Figure 9, where $\phi$ and $w$ denote the porosity and the moisture content, respectively): As the moisture content in the coal samples increased, the increment of porosity after FTTLN increased linearly. The increment in the porosity of dried coal samples was 0.276 while that of coal samples with the moisture content of 11.67% was 1.151, being 4.2 times that of dried coal samples: This indicated that the greater the moisture content, the more severe the internal damage to the coal mass after FTTLN, and the more developed the pores therein.

### 3.3.4 Changes in permeability

Permeability is a physical quantity that characterizes the ability of a porous medium to transport liquids, and it is able to intuitively represent the ability to allow gas seepage of coal
seams. The greater the permeability, the easier the gas flow and the better the extraction efficiency. Permeability varies with pore characteristics. According to the SDR model, the calculation formula for the growth rate of coal permeability can be obtained.

\[
\Delta k = \frac{k_1 - k_0}{k_0} = \frac{A\phi_{e1}^4T_{2e1}^2 - A\phi_{e0}^4T_{2e0}^2}{A\phi_{e0}^4T_{2e0}^2} = \frac{\phi_{e1}^4T_{2e1}^2}{\phi_{e0}^4T_{2e0}^2} - 1 \quad (5)
\]

where \( A \) refers to the constant and it is related to the porous medium; \( T_{2e0} \) and \( T_{2e1} \) separately refer to the geometric average of the \( T_2 \) spectral before and after FTTLN, which can be directly measured by NMR; \( \phi_{e0} \) and \( \phi_{e1} \) separately denote the effective porosity before and after FTTLN.

The relationship between the growth rate of permeability and the moisture content is plotted (Figure 10). As the moisture content increased, the growth rate of permeability positively exponentially increased. The growth rate in the porosity of dried coal samples was 18.5% while that of coal samples with the moisture content of 11.67% was 87.5%, being 4.7 times that of dried coal samples, which indicated that FTTLN can effectively increase permeability in a coal mass, enhance gas seepage ability, thus further improving the gas extraction efficiency.

![FIGURE 9](image-url)  
**FIGURE 9** Relationship between porosity and moisture content

\[ \phi = 0.2775 + 0.0722x \]
\[ R^2 = .9767 \]

![FIGURE 10](image-url)  
**FIGURE 10** Relationship between permeability and moisture content

\[ K = 18.716e^{0.132w} \]
\[ R^2 = .9919 \]

### DISCUSSION

#### 4.1 Quantitative analysis of damage to a water-bearing coal mass subjected to FTTLN

To describe the influence of moisture content on internal damage characteristics of coal mass on freeze-thaw condition with liquid nitrogen, the damage factor is separately defined by using longitudinal wave velocity, fracture width, fractal dimension of surface fractures, and porosity:

\[ D_V = 1 - \frac{V_0}{V_w} \quad (6) \]
\[ D_L = \frac{L_0}{L_w} - 1 \quad (7) \]
\[ D_F = \frac{F_0}{F_w} - 1 \quad (8) \]
\[ D_\phi = \frac{\phi_0}{\phi_w} - 1 \quad (9) \]

where \( D_V \) denotes the damage factor expressed by longitudinal wave velocity and \( V_w \) and \( V_0 \) separately refer to the

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### Table 7: Porosities of coal samples with different moisture contents before and after FTTLN

| Group | Moisture content (%) | Before FTTLN (%) | After FTTLN (%) |
|-------|----------------------|-----------------|----------------|
| A     | 11.67                | 12.583          | 13.734         |
| B     | 9.12                 | 12.520          | 13.367         |
| C     | 6.06                 | 12.945          | 13.698         |
| D     | 3.14                 | 12.863          | 13.337         |
| E     | 0                    | 12.470          | 12.746         |
longitudinal wave velocities before and after FTTLN; $D_L$ represents the damage factor expressed by fracture widths and $L_w$ and $L_0$ separately denote the fracture widths before and after FTTLN; $D_F$ denotes for the damage factor expressed by fractal dimension of surface fractures and $F_w$ and $F_0$ refer to the fractal dimensions of surface fractures before and after FTTLN, respectively; $D_\phi$ represents the damage factor defined by porosity and $\phi_w$ and $\phi_0$ separately refer to the porosities before and after FTTLN.

The relationships between various damage factors and moisture content under the freeze-thaw effect of liquid nitrogen are presented (Figure 11) and the fitting formulae listed (Table 8). According to Figure 11 and Table 8, it can be seen that the damage factors defined by fracture width, wave velocity, and fractal dimension of surface fractures all exhibited a positive exponential relationship with that of porosity, with a goodness of fit of greater than 0.9256. It can be seen that different damage factors measured produced different results; however, there were favorable quantitative correspondences of the change of microscopic structures of coal samples with various damage characteristics, that is, fracture width, wave velocity, and fractal dimension of surface fractures in freeze-thaw conditions with liquid nitrogen. The damage factor calculated based on porosity can be applied to characterize the damage factors expressed by fracture width, wave velocity, and fractal dimension of surface fractures to some extent.

### 4.2 Damage effect of FTTLN on a water-bearing coal mass

Other conditions being equal, the frost-heave effect played the dominant role in the damage to a water-bearing coal mass subjected to FTTLN. When frozen, the mineral particles in the coal mass were subjected to volumetric shrinkage. Due to the diversity of mineral compositions, the resulting shrinkage stress was nonuniform. Additionally, free water in the micropores and fractures in the coal mass froze to generate frost heave (9.1% volume expansion after water freezes in the coal mass). When the frost heave exceeded the bond strength, local failures occurred, which caused fractures in the coal samples to propagate. The higher the moisture content in the coal mass, the greater the frost heave and the more serious the local failure. When thawing, the frost heave abated and

| Independent variable | Damage factors | Fitting formulae | $R^2$ |
|----------------------|----------------|-----------------|-------|
| Moisture content ($W$) | $D_L$ | $D_L = 0.1282e^{0.111W}$ | .9693 |
|                      | $D_V$ | $D_V = 0.0387e^{0.2182W}$ | .9959 |
|                      | $D_F$ | $D_F = 0.0325 + 0.0069W$ | .9927 |
|                      | $D_\phi$ | $D_\phi = 0.0192e^{0.1484W}$ | .9942 |
| Porosity ($\phi_w$)  | $D_L$ | $D_L = 0.0853e^{19.0518\phi_w}$ | .9766 |
|                      | $D_V$ | $D_V = 0.0853e^{19.0518\phi_w}$ | .9256 |
|                      | $D_F$ | $D_F = 0.0235e^{25.264\phi_w}$ | .9867 |
moisture flowed inwards, thus continuously damaging the pores and fractures.

The evolution of the structures of pores and fractures in coal samples subjected to FTTLN is shown (Figure 13). Under freeze-thaw action, the internal pore structure of the water-bearing coal mass was changed (imaginary lines in $T_2$ spectra were all right-shifted; peak areas and peak area proportions of micropores and small pores as well as mesopores and macropores were all slightly different; the type, number, and connectivity of pores were changed): The porosity increased, which caused surface fractures to propagate (at a rate of propagation of 49.72% at most); thus, the fractal dimension increased and the propagation velocity of sound waves in coal samples was changed: The wave velocity decreased (with a maximum reduction of 49.68%). This confirmed the changes in damage factors characterized by the aforementioned various parameters.

5 | CONCLUSIONS

- The surface fractures on these coal samples all propagated after FTTLN: With increasing moisture content, the rate of expansion of the fracture width of coal samples linearly increased, in which the rate of expansion in water-saturated coal samples was 3.3 times that in dried coal samples; the evolution of surface fractures of coal samples exhibited significant fractal characteristics: With increasing moisture content, the fractal dimension of surface fractures exhibited positive exponential growth.
- Under the freeze-thaw effect of liquid nitrogen, the longitudinal wave velocity in coal samples gradually decreased and there was a positive exponential relationship between the reduction in wave velocity in coal samples and the moisture content; under the freeze-thaw effect of liquid nitrogen, micropores and small pores in coal mass were gradually transformed to mesopores and macropores and also new micropores and small pores were generated; with increasing moisture content, the peak area of micropores and small pores and its proportion first decreased, then increased while the peak area, peak area proportion and the increment of porosity of mesopores and macropores all increased linearly.
- With increasing moisture content, after FTTLN, the damage factors expressed by fracture width, longitudinal wave velocity, and porosity all increased exponentially while that defined by the fractal dimension of surface

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**FIGURE 12** Relationships of fracture width, wave velocity, and fractal dimension with porosity

**FIGURE 13** Change of pore and fracture features in coal samples subjected to FTTLN
fractures showed positive linear growth. The damage factors expressed by fracture width, wave velocity, and fractal dimension of surface fractures, all exhibited a positive exponential relationship with the porosity.

- The damage to a water-bearing coal mass subjected to FTTLN was mainly triggered by frost heave. That is, under freeze-thaw action, the internal pore structure of a water-bearing coal mass was changed (the type, number, and interconnectivity of pores varied): The porosity increased, thus leading to the propagation of surface fractures.

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ORCID
Jinliang Li https://orcid.org/0000-0002-8287-7951

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