Research Article

Changing Characteristics of Sandstone Pore Size under Cyclic Loading

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The size and distribution of pores in rocks are closely related to their physical and mechanical properties. It is important to study the structure and distribution of pore size inside the rock to assess the risk of damage to a given rock volume. These characteristics were studied under different pressures, pore diameters, and pore throat size distribution laws using a UTM5540 electronic universal testing machine, magnetic resonance imaging scanning, and low field nuclear magnetic resonance spectroscopy with cyclic loading on yellow sandstone. We found the following. (1) Under 0–10 MPa load, the peaks of the sandstone T2 spectrum move left as load increases, and the porosity of the sandstone decreases. The peak area of the middle relaxation spectrum increases as pressure increases from 10 to 20 MPa, and a peak for the long relaxation time spectrum appears. (2) Under 0–10 MPa load, the spectral peak associated with a large pore moves left and decreases in area as pressure increases. Under 10–20 MPa load, the large-pore spectral peak moves right and increases in area as pressure increases. (3) Under the applied 0–10 MPa load, the porosity of water-saturated sandstone gradually decreases, and the sandstone NMR images darken with increasing load. The porosity of saturated sandstone gradually increases under 10–20 MPa pressure, and its NMR image brightens. (4) The number of small pore throats increases with increasing load, but the number of large- and medium-sized pore throats decreases. From 0 to 15 MPa, crack (>1 micron) abundance decreases, and fractures are generated by compaction under a 20 MPa load. The pore interconnectivity is enhanced, as are the number and size of pores in the sandstone. With continuing increasing pressure, the numbers of pores and penetration of cracks increase, which damages the sandstone.

1. Introduction

The characteristics of pores in sandstone are closely related to the rock’s physical and mechanical properties [1–6]. The pores’ size, structure, and distribution not only determine the mechanical properties of the rock but are also the key factors affecting the permeability of the rock [7–12]. Therefore, to ensure the stability of rock formations in coal mining, it is important to study how the internal pore size and pore state in sandstone change under stress and to study the resulting damage to the body of rock.

Previous studies have used various methods to explore the relationship between the internal microstructural changes of sandstone and externally imposed stress. Common methods include numerical simulation, computerized tomography (CT) scans, acoustic emission tests, electron microscopy, and mercury intrusion. For example, Wu [13–15] studied the pore throat size and proportion of sandstone through mercury intrusion experiments. Ren and Yang [16–18] combined triaxial loading and CT scanning experiments through CT image analysis of the sandstone failure process under triaxial or uniaxial loading to reveal microscopic damage and the crack propagation law of the sandstone failure process. However, these experimental methods have the disadvantages of large errors, damage to the sample, and invisible static processes.

Low-field nuclear magnetic resonance (NMR) technology has the advantages of being nondestructive, using smaller
sample volume, having a rapid detection speed, and having dynamic visibility, and it has therefore been widely used to measure porosity, permeability, pore size distribution, and displacement among other physical properties of the medium being studied [19–24]. Zhang [25, 26] combined nuclear magnetic resonance and triaxial compression methodologies to study the crack propagation law of granite under different confining pressures and measured the stress-change curve and $T_2$ spectrum of granite under different confining pressure conditions. With increasing axial load, the $T_2$ spectrum peak, spectrum area, and porosity of granite gradually increased, and the degree of damage inside the rock continued to increase. Porosity increased exponentially with increasing axial load, which resulted in damage to the rock as cracks penetrated and increased in number. Zhou [27] studied the mechanism of damage to rock under triaxial compression using NMR technology to determine the functional relationship between porosity and damage and conducted "stress-strain," porosity, and relaxation time ($T_2$) measurements. Quantitative analysis of the damage spectrum and triaxial compression established a tensor relationship as the rock is damaged. Hu [28, 29] studied the meso-scale damage evolution characteristics of the internal pore structure of unloaded rock masses using NMR and triaxial unloading experiments on marble. They found that with increasing unloading confining pressure ratio, the elastic deformation of the rock gradually transformed into plastic deformation, the size of the small pores in the rock sample increased, and the size and number of the large pores increased. The porosity of the rock sample increased with increasing unloading confining pressure ratio, and the growth rate progressively increased.

In summary, there are insufficient studies on the structure and distribution of pores in sandstone under the influence of cyclic mining stress. Using low-field nuclear magnetic resonance spectroscopy, this study evaluates the evolution in pore size in fine yellow sandstone under cyclic loading and obtains the $T_2$ spectrum and characteristics of changes in pore throats in fine yellow sandstone under cyclic loading. It provides a reference for future research on the damage and destruction of sandstone and the stability of the surrounding rocks of mining roadways.

2. Materials and Methods

2.1. Experimental Sample. The selected samples are cylindrical fine yellow sandstone with a size of 25 mm × 60 mm. The measured uniaxial compressive strength of the sandstone is 26 MPa, and the maximum deformation is 0.9 mm. The stress and deformation curve of sandstone are shown in Figure 1.

2.2. Experimental Equipment. The equipment used in this experiment includes a low-temperature and high-pressure nuclear magnetic resonance spectrometer (Suzhou Newmai Analytical Instruments Co., Ltd.) and a UTM5540 electronic universal testing machine (Figure 2). Nuclear magnetic resonance is used to measure the $T_2$ spectrum, porosity, pore size, and pore throat distribution of the sample and to conduct NMR imaging of water-saturated sandstone under cyclic loading. Magnetic field strength was $0.3 \pm 0.05$ T, magnet frequency was $10.64–14.90$ MHz, pulse control accuracy was 0.1 Hz, and pulse accuracy was 100 ns.

ZYB–2 vacuum pressure saturation device is used as supporting equipment, sample chamber size: $\Phi$ 150 × 300 mm, withstand voltage: 50 MPa, power supply voltage: AC220 V/50 Hz, vacuum pumping rate: 4 L/s, and manual pump: 210 ml/50 MPa. The core is vacuumized with vacuum device and then soaked with liquid of certain specification under vacuum condition to make the core absorb the liquid fully. In order to speed up the absorption rate, a certain external pressure is applied to the core and liquid during the soaking process, and the external pressure can be determined according to the porosity and tightness of the core.

The electronic universal testing machine is used to apply different uniaxial compression forces to sandstone; the maximum test force is 50 kN, the test force measurement range is 2%–100% FS, the test force measurement range is within ±1%, the displacement resolution is 0.001 mm, and the displacement display value has relative error of ±0.5% and relative speed error ±1% of the set value.

2.3. Experimental Procedures. The UTM5540 electronic universal testing machine was used to apply cyclic loads to sandstone samples, and then, water-saturated sandstone was scanned using NMR to obtain $T_2$ spectra and porosity, pore size, and pore throat distribution measurements. NMR images were obtained under different loads. The complete methodology is as follows.

(1) The sandstone sample is placed in the core chamber of the vacuum pressure saturation device, and after vacuum treatment, the valve of the core chamber is opened to allow water to enter the core chamber such that the experimental sample reaches a state of complete water saturation

(2) When the temperature of the permanent magnet is $32 \pm 0.01^\circ$C, a coil suitable for the size of the experimental sample is installed, and a standard oil sample is used to calibrate the central frequency of the NMR instrument. By adjusting the appropriate parameters,
3. Results and Discussion

3.1. Characteristics of the Sandstone $T_2$ Spectrum under Cyclic Loading. Through the above experimental process, the $T_2$ spectrum of sandstone under different loads is shown in Figure 3.

The relaxation time of hydrogen atoms in the pore water is an indicator of the sample’s environment. The longer the relaxation time, the weaker the binding of the water molecules and the larger the pore size. We divide relaxation time into short (0.01–10 ms), medium (10–100 ms), and long (100–1000 ms) durations.

The peak distribution of sandstone $T_2$ spectrum under different loads is shown in Table 1. The relaxation time of sandstone in the untreated state is indicated by four spectral peaks. The short relaxation time is mainly concentrated in 0.5–4 ms. After pressure of 5 MPa is applied, short relaxation peak of 0.07–0.16 ms disappears, and the termination time of the medium and long relaxations decreases. Both sets of peaks move to the left, and the maximum relaxation time shortens. Applying a pressure of 10 MPa, the spectral peaks for a relaxation time of 0.1 ms disappear, which leaves three spectral peaks indicating relaxation time. The maximum relaxation time also decreases to 100 ms, the peak associated with long relaxation time eventually disappears, the sandstone continues to be compacted, and the size of the pores further decreases. When a pressure of 15 MPa is applied, a short relaxation time peak of 0.01–0.1 ms appears, and five relaxation time peaks are observed. The newly added short relaxation time peaks indicate the presence of tiny pores caused by small displacements and dislocations of the cemented particles inside the sandstone under the new pressure. The maximum relaxation time of the newly appearing long relaxation time peak is 174 ms, and a larger pore diameter is produced. After applying a pressure of 20 MPa, the spectral peaks for short relaxation times of 0.01 ms to 0.1 ms disappeared, and the relaxation time peaks all moved to the right relative to their position under 15 MPa, which indicates that the new micropores continued to expand after further compression. Under 20 MPa pressure, the penetration degree of the pore diameter increases, and more large pores are produced. As pressure increased, the formation of macroscopic cracks continued until the sandstone was destroyed.

3.2. Evolution of Pore Characteristics of Saturated Sandstone under Cyclic Loading. According to the NMR measurement results, the pore size distribution of the sandstone as obtained by inversion is shown in Figure 4.

According to their results, Yao [30] classified pore sizes in rock by radius ($r$) into small pores ($r < 0.1 \mu m$), mesopores ($0.1 \mu m < r < 1 \mu m$), and large pores ($r > 1 \mu m$). According to the analysis shown in Figure 4, in the original state of the sandstone, the micropores yielded two spectral peaks, and there were no pores in the size range of 0 to 0.01 $\mu m$. The large pores and mesopores all had one spectral peak distribution, but the large pores yielded smaller peak areas and were few in number. When a pressure of 5 MPa was applied, tiny pores measuring 0.001–0.01 $\mu m$ appeared, and the height and area of the peak associated with these pores in the original state decreased. The two spectral peaks resulting from the large and medium pores became connected under a pressure of 5 MPa. The peak associated with large pores moved to the left while the area of the peak decreased, which indicates that the sample was compacted under the new applied pressure.
When a pressure of 10 MPa was applied, the newly generated micropore spectrum peak under 5 MPa disappeared, and the micropore spectrum peak continued to move to the left, which indicates that the sandstone was further compacted, while the large-pore and mesopore spectrum peak area was generally unchanged and moved slightly to the left. When 15 MPa pressure was applied, a peak appeared at 0.001 μm, the micropore spectrum peak moved to the right and increased in area, and the 1–10 μm macropore spectrum peak appeared. This indicates that small pores begin to be generated inside the sandstone under 15 MPa pressure and that the sizes of large pores and mesopores increase under pressure. When a pressure of 20 MPa was applied, the peak at 0.001 μm disappeared, and the diameter of the small pores of about 0.001 μm size expanded. Although the peak associated with large pore size moved to the left, the peak area was greatly increased. This indicates that pore size inside the sandstone continued to expand. As the pressure was increased to the uniaxial compressive strength, the internal pore of the sandstone expanded and penetrated to form macroscopic cracks until the sandstone was destroyed.

### 3.3. Sandstone Porosity and NMR Imaging under Cyclic Loading

The unpressurized saturated rock sample was...
scanned by NMR, and then, different uniaxial compressive stresses were applied to the sandstone and maintained for 30 minutes. After unloading, the sample was scanned with NMR. The porosity of the sandstone under different loading conditions is shown in Figure 5.

According to the analysis in Figure 5, the porosity of all sandstones decreased under 5 MPa pressure, which indicates that the voids inside the sandstone were compacted and closed under that pressure. The porosity of sandstone decreased significantly because there were more large pores inside the sandstone, and the decreasing pore size under compression led to a larger decrease in porosity. When the pressure reached 10 MPa, the porosity decreased slowly, the internal fractures of the sandstone continued to be compacted and closed, and the porosity decreased. When pressure increased to 15 MPa, the porosity of the sandstone slowly increased, which indicates that the pores and pore throats inside the sandstone began to expand and that the speed of expansion was greater than the speed of compaction and closure. Cracks began to develop, and porosity increased. Under 20 MPa pressure, the porosity of the sandstone continued to increase, and the rate of increase was faster than that at 15 MPa, which indicates that the small pores in the sandstone continued to develop, and the existing pores began to penetrate the cracks and expand, and porosity increased.

Low-temperature and high-pressure nuclear magnetic resonance spectroscopy was used to perform NMR imaging of sandstone under cyclic loading at various pressure states. Niu-Spin Echo (soft pulse imaging) was selected for the imaging sequence, and the sandstone magnetic resonance imaging results are shown in Figure 6.
According to the NMR image analysis of sandstone:

(1) When no load was applied (0 MPa), the pores inside the sandstone were mainly distributed in the middle and lower parts. The upper part was darker, the particles were cemented well, and the diameter of the pores was small. At the edge of the lower part, the color was bright and evenly distributed, the diameter of the pores was larger, and the pores were unevenly distributed in the sandstone.

(2) After a load of 5 MPa, the left half and bottom of the sandstone image became brighter, and pores were mainly distributed in this region. The right half was darker than when no load was applied. Compared with the image when no load was applied, the number of blocks with uniform brightness had decreased, which indicates that some of the cracks inside the sandstone were compressed and closed under a load of 5 MPa and that the sandstone was initiating compaction.
(3) After a load of 10 MPa, the bright area at the bottom of the sandstone was less bright than that after 5 MPa, the area of uniform brightness was smaller, and the color of the upper left section was darker. The right half was darker than it was at 5 MPa, and the darker region was larger. The pores were mainly distributed in the lower left and bottom of the sample. Under 10 MPa, the voids inside the sandstone were compressed, closed, and further compacted. Under a load of 15 MPa, the upper right part of the sandstone image became brighter than it was at 10 MPa; the center-right, lower left, and bottom sections became brighter, and the total area of the brighter area increased. This indicates that under a load of 15 MPa, new pores began to form inside the sandstone.

(4) After a load of 20 MPa, the image tone of the left half of the sandstone was almost the same as that after 15 MPa, and the area of uniform brightness did not significantly change in size. The upper right section of the sandstone was brighter than it was under 15 MPa. Sections with brighter colors began to merge, which indicates that larger cracks began to penetrate the sandstone under this pressure. Furthermore, the rate of new pore generation increased, and the porosity of the sandstone increased.

### 3.4. Characteristics of Sandstone Pore Throats under Cyclic Loading

According to the NMR measurement results, the pore throat distribution of sandstone is determined and presented in Table 2. According to the size of pore throats, pore throats can be divided into small pore throats (0–0.25 μm), medium to large pore throats (0.25–1 μm), and cracks (>1 μm). According to this classification, the proportions of different types of pore throat in sandstone under cyclic loading can be established, as presented in Table 3.

As indicated in Table 3, the number of micropore throats increased with increasing pressure. Under the pressure of 0–10 MPa, the size of large and medium-sized pore throat decreases, and new micropore throat is formed, which leads to the increase of the number of micropore throat. The increase in the number of micropore throats under pressure of 10–20 MPa indicated newly generated micropore throats in the sandstone. Under this pressure, the internal pore of the sandstone begins to penetrate to form the micropore throats. At 0–5 MPa, the number of large and medium pore throats decreases as a result of compaction. Under pressure of 5–20 MPa, there was minimal change, and the number of pore throats only decreases at a pressure of 15 MPa. It may be that the sample was further compacted under this pressure, which led to a decrease in the number of pore throats. When pressure was increased to 20 MPa, the number of large, medium, and small pore throats all increases, which indicates that the degree of pore penetration inside the sandstone had increased.

Cracks (>1 μm) were gradually compacted and closed under a pressure of 0–15 MPa. Under 20 MPa pressure, the number of cracks increased sharply, the degree of penetration of the sandstone pores increased, and the number and size of pore throats increased. As pressure was further increased, the number of pore throats increased, and the cracks expanded until the sandstone was destroyed.

### 4. Conclusions

(1) Under increasing load from 0 to 10 MPa, the relaxation peak of the sandstone T2 spectrum moves to the left, and the porosity of sandstone decreases. Under a load of 10–20 MPa, the area of the medium relaxation time spectrum peak increases with pressure, and a long relaxation time spectrum peak appears. At 0–10 MPa, the large-pore spectrum peak moves to the left with increasing load, and the area under the spectrum peaks decreases, as does the porosity of the sandstone. The spectrum peaks of large and medium pores under pressures of 10–20 MPa move to the right with increasing area as load increases.

(2) Under increasing load from 0 to 10 MPa, water absorption and porosity of the saturated sandstone gradually decrease, and the sandstone NMR image becomes darker. Under a load of 10–20 MPa, water absorption and porosity of saturated sandstone gradually increase with increasing pressure, and its NMR image tone becomes brighter.

(3) With increasing load, the number of tiny pore throats increases, and the number of large and medium pore throats decreases. Under pressures of 0–15 MPa, the number of cracks (>1 μm) decreases, and these are gradually closed by compaction. Under a load of...
20 MPa, the number of cracks increases, and the degree of penetration of sandstone pores increases. The number and size of pore throats increase with pressure, until cracks expand to the point where the sandstone is destroyed.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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