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Study on Magnetic Resonance Characteristics of Coal Sample under Progressive Loads

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Received: 24 July 2020; Accepted: 16 September 2020; Published: 18 September 2020

Abstract: With the characteristics of gradual instability in the supporting pressure area of roadway as the engineering background, this paper aims to explore the evolution law of pore and fracture in the coal sample under progressive loads. The low-field nuclear magnetic resonance (NMR) test was designed and conducted with the coal sample under different axial loads (0, 3, 5, 7, 9, and 11 MPa). The characteristic parameters such as the porosity, the pore size distribution, the transverse relaxation time (T_2) distribution curve, and the magnetic resonance image (MRI) were obtained. As the test results show, significant difference in the NMR characteristics of the coal samples can be observed throughout the compaction stage and the elastic stage. In the compaction stage, the porosity of the coal samples decreases slightly; the T_2 distribution curve moves to the smaller value as a whole, and the percolation pore (PP) displays a tendency to transform to the adsorption pore (AP). In the elastic stage, the porosity of the coal samples rises gradually as the load increases; the T_2 distribution curve moves to the larger value as a whole, and the AP tends to transform to the PP. The MRI shows that some pores and fissures in the coal sample close up and disappear as the load increases gradually, while the main pores and fissures expand and perforate till the macro failure occurs. Compared with one-time loading, the progressive multiple loads can ensure the fracture of the coal sample to develop more fully and the damage degree higher. It indirectly reflects that the instability and failure of the coal under the progressive load has the stage characteristics, verifying that the coal in the supporting pressure area needs to be controlled in advance.

Keywords: coal sample; NMR; progressive load; porosity; fissure propagation; MRI

1. Introduction

In recent years, with the progress of coal mining technology and the improvement of mechanical equipment performance, highly efficient intensive coal mining has become the dominant trend of major coal mining countries in the world, such as China, the United States, and Australia [1–3]. Inevitably, the high-intensity coal mining activities would incur the enlargement of roadway cross-section and the drastic strata pressure behaviors. Under the complex disturbance of multiple stress, severe roof fall and floor heave are highly likely to occur in the roadway. Meanwhile, coal wall collapse is also highly probable in the roadway, which poses an extreme threat to the safety of production and miners [4,5]. When large-scale coal wall collapse occurs in the roadway with hard roof, it can trigger active movements in the top covers, which in turn are inductive to the overall rock burst [6,7]. However, coal wall instability does not happen overnight; instead, it is the consequence of gradual evolution

under progressive load. Therefore, it is of significance to consider the role of progressive load in analyzing the characteristics of pore and fracture evolution from a micro perspective.

Scholars, both at home and abroad, have conducted a large number of studies using a variety of testing methods. Coal and rock under the load would deform, and the crack would grow, so as to release energy rapidly and generate transient elastic waves. It is due to this property of rock that acoustic emission technique has been used to study the evolution law of pore and fracture. He et al. [8] studied the characteristics of acoustic emission distribution under different loads. Song et al. [9] analyzed the reasons for the difference in uniaxial compressive strength and acoustic emission energy caused by the anisotropy of coal. Li et al. [10] conducted uniaxial loading tests at different loading rates to explore the relationship between the cracks in coal sample, stress and energy, and acoustic emission. With the rapid development of ultrasonic technology, it has been widely used to study the mechanical property and structural characteristics of rock. Thill et al. [11] conducted a contrastive study of the anisotropy of ultrasonic velocity and fracture distribution in the coal. Zhao et al. [12] took the effect of axial compression on the velocity and attenuation of s-wave and longitudinal wave into consideration and analyzed the ultrasonic response characteristics of coal under uniaxial loading. However, acoustic emission and ultrasonic testing cannot directly illustrate the evolution law of pore and fracture on the microscopic level. The main characterization methods for pore and fracture structures on micro level are as follows: electron microscope method [13–15], mercury intrusion method [16–18], gas adsorption method [17,19], CT scanning method [20–22], and low-field nuclear magnetic resonance (NMR) method [23–25]. By electron microscope method, the distribution characteristics of pore and fracture can be observed directly. However, this method is applicable only in a relatively confined area, thus being unable to provide a panoramic view of the whole evolution process when the coal sample is under loading. Mercury intrusion method can be used to characterize the macro pores in the coal sample, but the relatively high pressure for mercury intrusion would damage the pore structure, thus being not applicable to measure the micro-pores in the coal sample. Gas adsorption method is commonly used to measure the specific surface area and pore structure of coal samples, but mainly for the micro-pore measurement. CT scanning method can be used for non-destructive pore structure testing within the coal sample, but the pores tested are mainly mesopores and macropores. The low-field NMR method can test the nuclei with odd numbers of nucleons, and then can be used to conduct non-destructive sophisticated characterization of the whole process of pore and fracture development in the coal sample [26,27]. The test has a wide range of applications and can measure small, medium, and large holes. It can effectively make up for the deficiencies of the above-mentioned testing methods.

To begin with, this paper analyzes the characteristics of, and reasons for, the gradual instability in the supporting pressure area in Zhangjiamao Coal Mine, then low-field NMR method is used to characterize the pore and fissure structure of the coal sample under progressive loads; through the tests, the porosity, the pore size distribution, the T_2 distribution curve and the magnetic resonance image (MRI) are obtained; and on this basis, the distribution characteristics of pore and fracture under different progressive loads can be analyzed; at last, suggestions are offered for the coal wall control in the supporting pressure area.

2. Engineering Geological Background

2.1. General Geological Situation

Zhangjiamao Coal Mine is located in Shenmu county, Yulin city, Shaanxi Province. Its landform is mainly featured with wind–sand regions and hilly–gully regions. Developed with adits, the coal mine has seven minable seams in total, with a designed production capacity of 10 million tons per year. The current main coal seam is 5-2 coal, with a burial depth of 91.20–302.72 m. The thickness of coal seam is 2.47–7.35 m, with the average depth being 5.8 m and average dip angle being 1.5° .

Currently, the main longwall coal face (LCF) of Zhangjiamao Coal Mine is LCF 15208. As the field investigation indicated, large-scale splitting failures occurred in the coal walls of the supporting pressure area within the range of 30 m in advance of the auxiliary haulage roadway, posing severe threats to the safety production of coal mining. The layout of LCF 15208 and its adjacent working faces is shown in Figure 1. As can be seen, the auxiliary haulage roadway (AHR) and belt haulage roadway (BHR) are located along the two sides of LCF 15208. The AHR is under the effect of strong dynamic pressure caused by two recoveries of the working face. This is the main cause for the coal wall instability. The burial depth of LCF 15208 is about 200 m, the ground stress of coal seam being relatively low. Above the coal seam is fine sandstone and siltstone while beneath the coal seam is siltstone and fine sandstone. The roof and floor of the coal seam are relatively hard. There are two aquifer regions above the coal seam: the siltstone aquifer in the roof, and the water-accumulating goafs of LCF 14207 and LCF 14208. Under the water seepage effect, the water content of the coal seam is relatively high, which has a certain weakening effect on the strength of coal.

Figure 1. Mining layout of the Zhangjiamao Coal Mine.

2.2. Characteristics of Gradual Coal Wall Failure in the Supporting Pressure Area

As can be known from Figure 2, the coal pillar wall in different positions of the supporting pressure area is analyzed. The upper part of the figure displays the in situ photos of coal wall and the lower part shows the corresponding sketches of coal wall failure. As the distance to the working face decreases gradually, the coal wall displays the characteristics of gradual failure. When the distance to the working face is 24 m (corresponding to Sketch ①), failure occurs firstly at the upper corner of the coal wall. The failure range is smaller than 1 m and the failure mode is mainly small pieces of coal peeling off. When the distance to the working face reduces to 19 m (corresponding to Sketch ②), the failure extends downward along the coal pillar wall. The failure range is about 2 m and more vertical coal lumps are peeled off. When the distance to the working face further shortens to 15 m (corresponding to Sketch ③), the failure range is further enlarged to 3 m, approximately two thirds of the total roadway height. When the distance to the working face is 8 m (corresponding to Sketch ④), the failure mode changes correspondingly. The failure range ceases to extend downward; instead it tends to bulge as a whole and appears to be a V-shape. In the working face (corresponding to Sketch ⑤), the coal wall failure is the most severe. The opening of the V-shape coal wall shrinks, and the maximum bulging depth reaches up to 1 m.

Figure 2. Failure diagram and sketch of the coal pillar wall in the supporting pressure area.

The recovery of the working face causes the stress readjustment in the roadway surrounding rock. The coal pillar bears greater supporting stress when it is close to the working face, thus leading to the severe failure in the coal pillar wall. By contrast, the coal pillars bear relatively smaller supporting stress when they are farther away from the working face, which in turn lessens the failure correspondingly. Numerical simulation was conducted to obtain the evolution law of the stress exerted on coal pillars. Based on the geological occurrence characteristics and mining environment of LCF 15208, the characteristics of vertical stress distribution in the coal pillar were simulated with FLAC3D numerical analytical software. The numerical model is $230 \times 180 \times 60$ m in length, width, and height. It is assumed that LCF 15207 has been recovered fully, and LCF 15208 has been recovered by half. In other words, there are still 90 m left unrecovered the vertical stress distribution at the position of 0 m on the roof of AHR 15208; meanwhile the vertical stress curve is drawn within the range of 90 m ahead of the working face and 0.5 m to the inner side of coal pillar. As can be known from Figure 3, the vertical stress of coal pillar displays a remarkable gradual evolution characteristic: the vertical stress increases gradually from the front side of the working face to its rear. Simultaneously, within the area of advance face, the vertical stress evolves from 3 MPa to 10 MPa, increasing first mildly and then rapidly as the measuring point gets closer and closer to the working face.

Figure 3. Vertical stress distribution.

As the working face advances constantly, the supporting pressure above the coal wall has a periodic evolution [28]. Suppose that one tiny unit of the coal wall is set as the research object, the vertical stress exerted on it would experience an evolution process of gradual increase, which in turn causes different failure modes in the coal wall. Therefore, it is of crucial significance for the control of coal wall instability to study the evolution of pore and fissures in the coal sample under progressive loads.

3. Materials and Methods

3.1. Test Equipment

As an extremely complicated medium with double pore structures, coal is composed of coal matrix and fissures. The former contains pores while the latter cuts the coal matrix [29–31]. Due to the coexistence of pores and fissures, coal is featured with inhomogeneity and anisotropy. Meanwhile, the strength of coal mass, mechanical property, and deformation characteristics all change correspondingly. Under the effect of progressive load, the evolution process of pores in coal is highly complex. To test the evolution mechanism of pore and fissures in the coal sample under progressive loads in a non-destructive way, low-field NMR is the best testing method [32].

The test equipment is mainly the MR 12-type low-field NMR analysis system (Suzhou Niumag Analytical Instrument Co., Ltd, Suzhou, Jiangsu, China). Its technical parameters are as follows: magnet strength 0.3 ± 0.05 T, magnet temperature 25–35 °C, Pulse Freq Range 1–30 MHz, the diameter of probe coil 60 mm. As is shown in Figure 4, MR12-type low-field NMR analysis system is composed of five parts, namely, ① control module, ② Imaging module, ③ display module, ④ NMR analyzer with large probe, and ⑤ probe coil. When testing, the coal sample to be tested should be put in the center of probe coil first, and then the probe coil is placed in the NMR analyzer. By means of control module, the porosity and pore size distribution can be measured, as is shown in Figure 4a. Then the imaging module is used to image the pore and fissure distribution inside the coal sample, which is shown in Figure 4b.

Figure 4. MR12-type low-field NMR analysis system: (a) the porosity and pore size distribution, (b) the pore and fissure imaging.

3.2. Test Method

The research object of low-field NMR testing method is hydrogen nucleus. To be specific, relatively low magnet strength is used to test the NMR signals of ^1H in the pore fluid, thus obtaining the T_2 of the fluid in pores and fissures. According to the theoretical analysis of NMR [33], the T_2 can be expressed as

$$1/T_2 = \rho (S/V) = F_S \rho / r, r = cT_2 \quad (1)$$

where ρ is the lateral surface relaxation rate, which is a parameter to characterize rock properties; S/V is the specific surface area; F_S is the porosity factor (spherical pores are 3, tubular pores are 2, and plate pores are 2); r denotes the pore radius; and c is a constant, referring to conversion coefficient. Therefore, the conversion relationship between T_2 and r is obtained.

As can be known from Equation (1), r is in direct proportion to T_2 . The longer T_2 is, the larger the pore radius is; the larger the spectral peak of T_2 is, the more fully the pore develops. Conversely, the larger the pore radius is, the T_2 of water in the pores is longer; the smaller the pore radius is, the T_2 of water in the pores is shorter since it is more confined [34,35].

When measuring T_2 with the NMR, the Carr-Purcell-Meiboom-Gill (CPMG) sequence will be used. Including the following specific parameters: the radio frequency (RF) center frequency is 12 MHz, the RF offset frequency is 696604 Hz, the echo time (TE) is 0.16 ms, the waiting time (TW) is 3.5 s, the number of scans accumulated (NS) is 4, and the length of the 90 and 180 degree pulses are 13.00 microseconds and 25.04 microseconds, respectively. Using these parameters, the T_2 distribution curve of the sample will be obtained, and the area of the T_2 distribution curve is the porosity of the sample [36].

After setting the NMR parameters, it is necessary to calibrate the relationship between the fixed porosity of the standard sample and the NMR signal amplitude measured under the current environment. Prepare five standard samples with different porosity (1%, 5%, 10%, 20%, and 30%), then test the NMR signal amplitude under the corresponding porosity, and fit the result to a straight line, as shown in Figure 5. The fitting relationship is $y = 1.394x - 0.212$, and the correlation coefficient R reaches 0.999996. Next, the NMR test of the coal sample can obtain the T_2 distribution curve of the coal sample. By integrating the T_2 distribution curve, the total NMR signal amount of the coal sample can be obtained. From the fitting relationship in Figure 5, calculate the volume of water in the coal sample, and finally the volume of water is divided by the total volume of the coal sample, and the value obtained is the porosity of the coal sample.

Figure 5. Calibration result fitting diagram of the standard samples.

3.3. Test Scheme

The coal samples are taken from 15,208 working face in Zhangjiamao Coal Mine. The coal briquettes are processed into standard samples ($\varphi 50 \times 100$ mm) whose non-parallelism between two end faces should be no larger than 0.01 mm and whose diameter deviation of the upper end and lower end should be no larger than 0.02 mm. This is to guarantee the uniform loading on the coal sample.

The test scheme is composed of the following steps:

- (1) Divide the standard coal samples into two groups and label them as Group S and Group C. Each group contains three samples, which are numbered as S-1, S-2, and S-3 and C-1, C-2, and C-3, respectively.
- (2) Place all samples into a drying oven to dry for 24 h. The temperature of the drying oven is set at 50 °C; then, the dried samples are put into water for water-saturation process which lasts for 12 h.
- (3) Conduct uniaxial loading to the water-saturated samples of Group S with a compression testing machine and obtain the mean peak strength.
- (4) Conduct low-field NMR test to the water-saturated samples of Group C to obtain the porosity, pore size distribution, and T_2 distribution curve; NMR imaging is performed to the coal samples along either transverse or longitudinal direction.
- (5) Conduct loading on the coal samples of Group C in three loading modes. For the coal sample C-1, uniaxial loading-holding test is conducted under five different loads in turn, namely, 3 MPa, 5 MPa, 7 MPa, 9 MPa, and 11 MPa. For the coal sample C-2, uniaxial loading-holding test is conducted under two different loads in turn, namely, 3 MPa, and 7 MPa. For the coal sample C-3, uniaxial loading-holding test is conducted under two different loads in turn, namely, 3 MPa and 11 MPa. The coal samples should hold the loading for 150 min under each loading condition. After each run of the test, step (2) and (4) should be repeated.

As is mentioned above, the vertical stress of coal wall within the range ahead of the working face evolves from 3 MPa to 10 MPa gradually. Accordingly, in the loading test, the load also starts from 3 MPa and increases gradually. Meanwhile, given that different recovery rates lead to the difference in stress increase, to better study the evolution characteristics of pores and fissures under different loads, the load exerted on coal samples C-2 and C-3 rises from 3 MPa to 7 MPa, and 11 MPa, respectively.

There are two reasons for holding the load on the coal sample for 150 min: one is to better reflect the actual stress situation of coal wall, because the supporting pressure above the coal wall may remain unchanged for some period of time due to the periodicity of recovery; the other is to let the coal sample fully adapt to the current load so that the pores and fissures in the sample could develop fully. Given that the primary fissures of coal samples are relatively developed, once the drying temperature is over 50 °C, the primary fracture will expand obviously, resulting in the change of the internal structure of the coal sample and the sudden drop of the compressive strength, which makes it impossible to further study the nuclear magnetic resonance characteristics. Therefore, the drying temperature can only be set at 50 °C.

Figure 6 shows the stress–strain curve of the coal samples of Group S. As is shown, the stress–strain curve is mainly divided into three stages: compaction stage, elastic stage, and failure stage. The splitting strength between the compaction stage and elastic stage is approximately 4.5 MPa, while that between elastic stage and failure stage is approximately 17.5 MPa. The peak stress of water-saturated coal samples of Group S is 19.14 MPa, 16.60 MPa, and 17.72 MPa, respectively, with the average peak stress being 17.82 MPa. The preset maximum loading (11 MPa) is positioned in the elastic stage of the coal sample. Coal sample C-1 is taken as an instance to elaborate the loading mode and NMR test, which is shown in Figure 7. As can be seen from this figure, the whole test of C-1 is divided into six stages. Stage 1: when coal sample C-1 is water-saturated, its pore and fissure distribution is tested by the low-field NMR analysis system and the data collected are recorded as NMR-0MPa. Stage 2: the coal sample C-1 is loaded to 3 MPa with a compression testing machine at a loading rate of 0.02 mm/s, the load is to be held for 150 min, then the sample is processed with drying and water-saturation, again it

is put into the low-field NMR analysis system to test its pore and fissure distribution, and the data collected are recorded as NMR-3 MPa. The following Stage 3, 4, 5, and 6 are similar to Stage 2, only differing in the load values; the data collected are recorded as NMR-5 MPa, NMR-7 MPa, NMR-9 MPa, and NMR-11 MPa.

Figure 6. Stress–strain curve of the Group S coal sample.

Figure 7. Test plan design of the coal sample C-1.

4. Testing Results and Analysis

4.1. Porosity

Table 1 and Figure 8 show the porosity and its evolution under progressive loads. As can be seen, before loading, the porosity of water-saturated coal samples C-1, C-2, and C-3 is 16.8470%, 16.8593%, and 17.5788%, respectively. When the load increases to 3 MPa and is held for 150 min, the porosity of C-1, C-2, and C-3 all decrease, though differing in their falling ranges. To be specific, the porosity of the three samples falls to 16.5783%, 16.5165%, and 17.2258%, respectively; and the decrease rate is 1.59%, 2.03%, and 2.01%, respectively. When the load is 3 MPa, the coal samples are in the compaction stage; the primary pores and fissures would close up after loading so that partial moisture content would

be squeezed out and the NMR signals of 1H would be attenuated, thus causing the slight decrease in porosity. As for coal sample C-1, when the load increases to 5 MPa, it evolves from compaction stage to elastic stage; the primary pores and fissures expand and secondary fissures begin to develop. Consequently, the porosity increases slightly to 16.6502%, with a growth rate of 0.43%. When the load increases to 7 MPa and 9 MPa, the porosity is 16.7930% and 17.0519%, respectively, and the growth rate is 0.86% and 1.54%, respectively. As the load increases gradually, the porosity growth rate of the coal sample rises correspondingly, indicating that the pores and fissures develop more and more drastically. When the load reaches to 11 MPa, macro-cracks appear in C-1; splitting occurs when the sample is water-saturated and its porosity is only 14.1662%. Under progressive loads, the bearing capacity of water-saturated coal samples reduces substantially, which further demonstrates that the strength of coal wall in the supporting pressure area would decline remarkably under the effect of long-term advance stress.

Table 1. Porosity of the coal samples in Group C under different loads.

Group C	Porosity (%)					
	NMR-0 MPa	NMR-3 MPa	NMR-5 MPa	NMR-7 MPa	NMR-9 MPa	NMR-11 MPa
C-1	16.8470	16.5783	16.6502	16.7930	17.0519	14.1662
C-2	16.8593	16.5165	/	16.6819	/	/
C-3	17.5788	17.2258	/	/	/	17.8763

Figure 8. Porosity evolution of the coal samples in Group C under progressive loads.

As for coal sample C-2, when the load increases from 3 MPa to 7 MPa, the porosity grows from 16.5165% to 16.6819%, the growth rate being 1.00%. As for coal sample C-3, when the load increases from 3 MPa to 11 MPa, the porosity grows from 17.5788% to 17.8763%, with a growth rate of 1.69%. When the coal sample is in the elastic stage, the pores and fissures expand under the effect of stress and the porosity increases accordingly. Meanwhile, it is discovered that when the load upon C-1 increases from 3 MPa to 7 MPa, the growth rate of its porosity is 1.30%, which is 0.30% larger than that of C-2 under the same load. The test results show that progressive loading exerts greater impact on coal samples than one-time loading does and will cause more drastic development of pores and fissures.

4.2. Pore Size Distribution

Pores and fissures of various sizes coexist in coal [37,38]. The evolution law of porosity under the loading is in essence the law of reciprocal transformation of pores and fissures. Yao et al. [39] observed that the pores in coal can be divided into three types: micro-pores ($<0.1 \mu\text{m}$), which are

also called adsorption pores (AP); mesopores and macropores ($>0.1 \mu\text{m}$, $<100 \mu\text{m}$), which are called percolation pores (PP); and crack pores ($>100 \mu\text{m}$) (CP). The pore volume ratio of different sizes is obtained by NMR, and then according to the classification standard of the reference [39], the volume ratio of corresponding pores (AP, PP, and CP) are respectively accumulated and obtained.

Figure 9 shows the evolution law of pores and fissures of different sizes under progressive loading. From Figure 9a it can be known that AP, PP, and CP all develop in coal sample C-1 under different loads. To be specific, AP develops much better than the other two while CP performs worst in terms of development. When there is no loading, the pore volume ratio of AP is 11.5996%, and that of PP is 5.2398%. When the load reaches 3 MPa, the coal sample is in the compaction stage; the rapid closing of some pores and fissures leads to the volume decline of PP and CP, which justifies the slight rise of pore volume ratio of AP. As the load further increases, the pore volume ratio of AP falls gradually while that of PP rises correspondingly. Since splitting occurs when the load on C-1 reaches 11 MPa, errors would be unavoidable in the data on porosity; in this case, the data collected are not qualified to be used for reference. When the load is 3 MPa, 5 MPa, 7 MPa, and 9 MPa, respectively, the change of pores and fissures of different sizes varies remarkably. To be specific, the pore volume of AP falls while that of PP rises; under progressive loading, AP tends to transform to PP. Meanwhile, PP changes much more noticeably than AP does, which further reveals the rapid expansion of fissures in the latter part of elastic stage.

Figure 9. Evolution law of different pores and fissures of Group C coal samples under progressive load: (a) Sample C-1, (b) Sample C-2, (c) Sample C-3.

Figure 9b shows the evolution characteristics of pores and fissures in C-2. Compared with that of C-1, AP in C-2 is more condensed and the pore volume ratio of PP is lower. Before loading, the pore volume ratio of AP is 13.5452% and that of PP is 3.3071%. AP and PP take up 80.3% and 19.6% of the total volume, respectively. When the load reaches 3 MPa, the coal sample is in the compaction stage; AP and PP take up 83.6% and 16.4% of the total volume, respectively. When the load reaches

7 MPa, the coal sample evolves into the elastic stage; AP and PP take up 76.3% and 23.2% of the total volume, respectively. The change of pores and fissures in coal sample C-2 is similar to that of C-1: the volume ratio of AP increases in the compaction stage, whereas that of PP increases in the elastic stage. Figure 9c displays the evolution characteristics of pores and fissures in C-3. When the load is 0 MPa, 3 MPa, and 11 MPa, AP takes up 73.8%, 80.2%, and 71.2% of the total volume, correspondingly, and PP takes up 18.8%, 15.7%, and 21.7% of the total volume, respectively. From Figure 9b,c it can be known that CP decreases in the compaction stage and increases significantly in the elastic stage; however, its proportion to the total volume of pores and fissures in the sample is still quite small. As a whole, the general distribution characteristics of pores and fissures in coal samples remain unchanged before and after loading, but the proportions of pores of different sizes change significantly.

When the load reaches 7 MPa, AP and PP take up 63.7% and 36.1% of the total volume in coal sample C-1, respectively. Compared with the case of C-2, PP changes more drastically, which indicates that progressive loading exerts greater impact on the mesopores and macropores in the sample than one-time loading does. This reveals the evolution law of pores and fissures under progressive loading.

4.3. T_2 Distribution Curve

Zhai et al. [36] and Li et al. [40] discovered that there were two or three peaks in the typical T_2 distribution curve. As can be obtained from Equation (1), the T_2 distribution curve reveals the distribution characteristics of pore radius. The longer T_2 is, the larger the pore radius is [41]. Figure 10 shows the T_2 distribution curves of coal samples in Group C. As is shown, there are two peaks in most of the curves. According to the study [37], when there are two typical peaks in the T_2 distribution curve, the left peak denotes AP while the right one stands for PP.

Figure 10. T_2 distribution curves of the coal samples in Group C: (a) Sample C-1, (b) Sample C-2, (c) Sample C-3.

As can be known from Figure 10, the T_2 distribution patterns of coal samples in Group C vary greatly from each other. The T_2 peaks of AP and PP hold the overwhelming advantages, whereas that of CP is virtually non-existent in the curve. Figure 10a shows that compared with the case before loading, when the load reaches 3 MPa, the T_2 peak value of AP witnesses a slight increase while that of PP declines remarkably. The T_2 distribution curve moves to the larger value as a whole, which indicates that when the coal sample is in the elastic stage, AP tends to transform to PP. From Figure 10b,c similar conclusions can be drawn in the cases of C-2 and C-3. In the compaction stage, the T_2 peak of AP displays an upward trend; on the contrary, that of PP tends to decline. Different from the case of progressive loading, when the load increases directly from 3 MPa to 7 MPa, the T_2 distribution curve of C-2 does not change as drastically as C-1. This demonstrates that progressive loading can promote the evolution of pores and fissures in the coal sample significantly. As for coal sample C-3, when the load increases directly from 3 MPa to 11 MPa, AP transforms to PP rapidly. As the load increases gradually, the relaxation time becomes longer gradually as the T_2 peak of AP begins to decrease, revealing that part of primary fissures close up when secondary fissures expand.

4.4. Magnetic Resonance Image (MRI)

Figure 11 shows the NMR images of the cross section and longitudinal section of the same position before and after uniaxial loading on the coal samples. These images are generated by the imaging module of the NMR, which mainly uses the proton density to create contrast. The sequence used in MRI is NiuSE, includes the following specific parameters: the TE is 500 ms, the TR is 5.89 ms, the averages are 4, the read size is 256, the phase size is 192, the echo position is 10%, the RF center frequency is 12 MHz, the RF offset frequency is 696643 Hz, the GxOffset is 0, the GyOffset is 15, the GzOffset is 20, the RFA90 is 1.8%, and the RFA180 is 3.8%. It is worth noting that the generated image is originally a gray image; the black area in the image is the background color, the white bright spot represents the area where the water molecules are located, and the brightness of the image reflects the water content in the sample. The higher the brightness of an area in the image, the higher the water content in that area, indicating that the pores and fissures in the area are larger. In order to improve the aesthetics of the image and facilitate the reader's reading, image processing software is used to convert the gray image into the color image, as shown in Figure 11.

Figure 11. Conversion between gray image and color image.

Figure 12a displays the NMR images of the cross section of the same position in C-1 under different loads. As is shown, before uniaxial loading, the pores are distributed evenly throughout the whole sample, intermingled with few fissures. When the load increases to 3 MPa, 5 MPa, and 7 MPa, respectively, the pore and fissure distribution changes greatly. The brightness of the image decreases gradually, indicating that most micro fissures close up after loading; meanwhile, the brightness congregates somewhere in the sample, which implies that some pores and fissures begin to expand, converge, and fuse with each other gradually. When the load reaches 11 MPa, the fissures feed through completely and form roughly Y-shaped macro-cracks. This verifies the characteristics of pore

size distribution: as the load increases, the micro pores and fissures tend to grow into macropores and cracks.

Figure 12. NMR images of the coal samples in Group C: (a) Sample C-1, (b) Sample C-2, (c) Sample C-3.

Figure 12b displays the NMR images of the cross section and longitudinal section of the same position in C-2 under different loads. As is shown, one cross section and five longitudinal sections are

selected to analyze the evolution law of pore and fissures. The five longitudinal sections are spaced at the interval of 15 mm; Sections 1 and 5 are 20 mm away to the two end faces, respectively; the cross section is vertical to the longitudinal sections. From the cross section image it can be known that before loading, pores and fissures are widely distributed in C-2 and there are some clear vertical micro-cracks. When the load reaches 3 MPa, part of pores and fissures in the sample close up; however, the distribution is relatively even and the macropores transform to micropores; when the load reaches 7 MPa, the distribution of pores and fissures is relatively concentrated, and some fissures close up. As can be seen from the five longitudinal sections, there are some clear striped fractures in the coal sample. This demonstrates that the pores and fissures are featured with centralized development in the elastic stage. With the closing of primary fissures and the opening of secondary fissures, the fissures form macro-cracks in the end.

Figure 12c shows the NMR images of the cross section of the same position in C-3 under different loads. To analyze more efficiently the evolution law of pores and fissures before and after uniaxial loading, the brightness observed are divided into two types. Before loading, the distribution of pores and fissures in C-3 is similar to that of C-2: the pores are distributed evenly throughout the whole sample, intermingled with some striped fractures in some areas. As can be seen from the images below, there are many bright short fractures on the right. When the load reaches 3 MPa, part of short fractures close up, but some fractures still congregate in the middle. When the load reaches 11 MPa, clear striped fractures appear in the middle part of the coal sample.

When the load increases from 3 MPa to 7 MPa, and then to 11 MPa, some laws can be observed in the NMR images of pores and fissures. Before uniaxial loading, the pores and fissures are distributed widely; when the load rises to 3 MPa, the sample is in the compaction stage, and the pores and fissures are distributed more evenly; when the load rises to 7 MPa, the sample is in the elastic stage, the pores and fissures are relatively congregated, and the new striped fissures appear and expand in this stage; when the load rises to 11 MPa, the sample is still in the elastic stage, but the pores and fissures are more congregated and the new fissures expand more drastically. Accordingly, it can be concluded that the evolution of pores and fissures under progressive loads experiences three phases: wide distribution—expansion in the concentration areas—macro perforation in the end.

5. Discussion

Coal sample C-1 is taken as an instance to draw the diagram of fracture evolution process according to the NMR images. As is shown in Figure 13, suppose that when loading, the splitting strength between the compaction stage and elastic stage is 4.5 MPa. When the sample is in the initial state, the fissures are distributed at random throughout the whole sample. When the sample enters the compaction stage, most fissures would close up completely or partially, with few exceptions of fissure expansion; meanwhile, some secondary fissures would appear. When the sample enters the elastic stage, the main fissures would expand and perforate each other while part of the primary fissures would close up and disappear. In other words, the fissures would expand along the main direction till the sample fails. Therefore, it is in the elastic stage that the expansion direction of fissures is determined.

Stress plays a crucial role in the evolution of pores and fissures in the coal sample. As the load increases gradually, the pores and fissures firstly close up, and then initiate the evolution of secondary fissures. In the end, the fissures perforate each other and cause the failure of coal samples. Therefore, it can be concluded that the failure of coal possesses some progressive and stage characteristics. Similarly, the instability of the coal wall also displays some stage characteristics. Given that the failure of the coal body lags behind the adjustment of vertical stress, reinforcing support should be provided before the full perforation of fissures occurs so as to give full play to the supporting stress and guarantee the long-term stability of roadway.

Figure 13. Fracture evolution process of coal sample.

6. Conclusions

Given that the vertical stress rises from small to large, the roadway in the supporting pressure area experiences different degrees of failure. The evolution law of pore and fissures in the coal sample under progressive loads is analyzed from the micro level, which is very important for how to control the coal wall and realize the safety of roadway. The low-field NMR can be used to finely characterize the evolution of pores and fissures in coal samples. The following conclusions are drawn specifically.

- (1) The porosity of coal samples evolves differently in the compaction stage and the elastic stage. During the compaction stage, the porosity declines mildly, indicating that part of the primary pores and fissures close up. During the elastic stage, the porosity increases correspondingly as the load rises gradually; and the growth rate of porosity tends to be larger and larger, indicating that secondary fissures develop more and more drastically.
- (2) Before loading, AP, PP, and CP all develop in the coal sample, with AP and PP taking the overwhelming advantage. When the load increases to 3 MPa, the volume ratio of PP declines while that of AP increases significantly. When the load increases to 11 MPa, the volume ratio of PP witnesses a significant rise while that of AP decreases remarkably, indicating that the elastic stage is a crucial phase for the rapid expansion of fissures. Meanwhile, it is also discovered that compared with one-time loading, progressive loading accelerates the evolution of pores and fissures.
- (3) Compared with the situation before loading, in the compaction stage, the T_2 distribution curve moves to the smaller value as a whole and PP displays some tendency to transform to AP; in the elastic stage, the T_2 distribution curve moves to the larger value as a whole and AP displays some tendency to transform to PP. As the load increases gradually, the relaxation time becomes longer correspondingly as the T_2 peak of AP starts to rise, revealing that part of the primary fissures close up when secondary fissures expand.
- (4) The NMR images show that as the load increases, the pores and fissures experiences three phases: wide distribution—expansion in the concentration areas—macro perforation in the end. This illustrates the processive and stage characteristics of fissure expansion on the one hand, while on the other hand, it also reflects indirectly that the instability of coal wall under the effect of progressive stress displays some stage characteristics. To guarantee the long-term roadway stability, reinforcing support should be provided to inhibit the full perforation of fissures in time.

Author Contributions: Data curation, Z.X. (Zhengzheng Xie), J.W., and Z.X. (Zhe Xiang); formal analysis, Z.X. (Zhengzheng Xie); funding acquisition, N.Z.; investigation, Z.X. (Zhengzheng Xie) and N.Z.; software, J.W. and C.Z.; writing—original draft, Z.X. (Zhengzheng Xie); writing—review and editing, Z.X. (Zhengzheng Xie), N.Z., J.W., Z.X. (Zhe Xiang), and C.Z. All authors have read and agreed to the published version of the manuscript.

Funding: This work is financially supported by the Future Scientists Program of China University of Mining and Technology (2020WLKXJ007) and the Postgraduate Research and Practice Innovation Program of Jiangsu Province (KYCX20_2013).

Acknowledgments: The authors are grateful to the staff at the Zhangjiamao Coal Mine for their assistance during the field measurements.

Conflicts of Interest: The authors declare no conflict of interest.

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