Effects of Microwave Heating Paths on Pores and Cracks in Bituminous Coal

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ABSTRACT: To follow the effect of the microwave heating path on the structure of coal, eight 50 mm diameter, 30 mm long dry coal cores from the same coal seam with similar pore structure characteristics were microwave-treated using 4 pathways. The T$_2$ spectrum, pore-volume, temperature, mass, and visual changes of coal samples were analyzed before and after microwave heating. The microwave heating path affected the macropores and microcracks and the crack development mode. When the same microwave energy was applied, microwave heating on the coal was mainly manifested by the opening of closed pores, before the pyrolysis temperature of the coal was reached. Increasing the energy density caused the water vapor to move from constant pressure expansion to constant volume expansion. This resulted in an exponential growth of the mesopore and macropore volumes. Meanwhile, the micropore volume increased due to the collapse of pore structures. As a result, high-power microwave heating could accelerate the vaporization rate of water. The rapid expansion of water vapor volume brought about a microwave heating effect similar to the “steam explosion”. The resulting local tensile stress enabled the cracks to develop, expand, and connect to others continuously. Thus, it formed a complex crack network leading to the outside of the coal sample.

1. INTRODUCTION

Xinjiang is abundant in low-rank coal with coalbed methane (CBM) reserves (approximately 4.5 × 10$^{10}$ m$^3$); however, they have not been explored or developed extensively. Reasonable exploitation of CBM in Xinjiang can meet the significant energy demand in China, which is of great strategic significance for sustainable development.

Most of the coalfields in Xinjiang are below their water saturation point and have a large dip angle. The conventional CBM mining methods available with the drainage and depressurization process are unsuitable for coal reservoirs in Xinjiang basins. Research showed that heat injection effectively improved gas production. When microwave heating coal was used, the heat was generated directly in the coal through microwave alternating electromagnetic fields and polar molecules. Compared with traditional heat injection methods, microwave heat injection possesses fast heating, small thermal inertia, intense penetration, and selective heating. Hence, improving reservoir quality by microwave preheating the formation was considered as a heat treatment method, especially suitable for coal reservoirs in Xinjiang basins.

The development and expansion of pores and cracks caused by selective heating of individual minerals in the coal by the microwave led to desorption, diffusion, and permeation of the adsorbed methane, with the evolution and development of pores and microcracks inside the coal. The past ten years have seen a proliferation of relevant research: Wang et al. used the N$_2$ adsorption and desorption experiments to investigate the variation of coal powder’s pore structure parameters when in a microwave field; Li et al. used nuclear magnetic resonance, P-wave tests, thermal imaging, and X-ray computed tomography scanner to evaluate the evolution of the pore and fracture structure of different ranked coals with microwave treatment; and Hong et al. evaluated the influence of microwave heating on pore size, quantity, volume, specific surface area, and connectivity between pores based on mercury injection testing and nuclear magnetic resonance (NMR) experiments. Cai et al. conducted NMR tests to investigate the petrophysical properties of coals (pore size distribution, pore structures, porosity, and permeability) at different temperatures subject to microwave heating. In the the papers mentioned above, the effect of a single microwave parameter (microwave power or heating time) on
the coal structure has been studied. Still, few have mentioned the differential impact of the microwave heating path on the coal structure. Additionally, the experimental results showed that the pore structure of coal samples affected the thermal cracking effect of the microwave,¹⁰,¹¹ so we can use coal samples with similar internal structural characteristics to compare the influence of microwave more accurately on the coal.

Accurate rock pore structure characterization provided the basis for analyzing the effect of microwaves on the coal structure. CT imaging, N₂ adsorption, mercury injection test, and NMR technology are the main techniques used for quantitative analysis of the pore structure of rocks. The NMR test used the low-frequency magnetic field and short pulse interval to calculate the relaxation time of the pore water with the high-frequency electromagnetic wave and obtains the relative information of the pore structure of the rock.¹² Therefore, compared with other pore testing techniques, the NMR test was characterized by a wide measuring range, high precision, and no damage to the rock structure and was significantly advanced in representing the pore structure at present.¹³

To examine the influence of microwave heating on the coal structure of high gas mines in Xinjiang, on the premise of the same output of microwave energy, four microwave heating paths were formed with combinations of different microwave powers and radiation times. The 60 coal samples were obtained from the same coal seam in Xinjiang. For microwave heating tests, eight coal samples with similar intrinsic characteristics were subsampled from the above-mentioned 60 samples. In this paper, we compared the T₂ curve, pore-volume, mass, temperature, and visual changes observed in the coal samples before and after heating. In addition, the mechanism of differential action of the microwave heating paths was analyzed. The research results provided a reference for applying microwave heating technology to high gas coal seams in Xinjiang.

2. EXPERIMENTAL SECTION

2.1. Materials and Equipment. The bituminous coal used was from the Wudong coal mine, a high gas mine in the southeast of Junggar Basin. The sampling location was specifically the B₁₂ coal seam in the south area. The dry density of the coal sample was 1.25 g/cm³. The processed coal sample’s integrity was ensured by coring them from a lump of large coal that had no cracks and water on the surface. We obtained sixty 50 mm diameter and 30 mm high, small cores of coal from this block.

A multisource and multimode microwave heating system with adjustable power in the range of 1−6 kW was used in the experiment. To better represent in situ conditions in a mining environment and normalize the water content, the coal samples were dried before microwave heating. The drying temperature was 40 °C for 24 h. Before NMR testing, the rock samples were saturated with water by a vacuum water saturation device. Then, the pore structure information of the samples was collected using the ANIMR-150 magnetic resonance imaging analysis system produced by Suzhou Niumag Corporation.

2.2. NMR Testing. In a low uniform magnetic field and short pulse environment, the distribution of NMR T₂ mainly focused on the surface relaxation at the interface of water and rock.¹⁴ Surface relaxation was a function of pore surface volume ratio

$$\frac{1}{T_2} = \rho S \frac{F_s}{V} = F_s \frac{\rho}{r_c}$$

(1)

where T₂ is the transverse relaxation time produced by surface interaction, ρ is the constant of transverse relaxation strength, S/V is the surface volume ratio inversely proportional to the hole’s radius (r), and F_s is the shape coefficient of the pore. F_s values of 1, 2, and 3, represented the strip pore, cylindrical pore, and spherical pore, respectively.

According to eq 1, the pore size was positively correlated with the transverse relaxation time, indicating that smaller pores correspond to shorter relaxation time and larger pores correspond to longer relaxation time. Hence, we used the relaxation time to distinguish pores and microcracks with different sizes.

The essence of the NMR T₂ spectrum was the normalized amplitude of the hydrogen proton in the external magnetic field, which was a dimensionless quantity. Previous research showed that the relaxation time of the T₂ spectrum gave the size of the coal pore, the ordinate of the T₂ spectrum was a measure of the number of coal pores, the integral area enclosed by the T₂ curve reflected the pore volume, and the continuity between wave crests signified the connectivity between pore systems in the coal samples.¹⁵,¹⁶ For the sake of simplicity, the pore radius less than 10 ms in the coal represented the micropore, 10−100 ms the mesopore, and greater than 100 ms the macropore and microcrack. Integrating the area enclosed by different T₂ curve segments, it helped to compare the volume changes (eqs 2 and 3) of micropores, mesopores, macropores, and microcracks in the coal samples before and after microwave heating.

$$S = \int A(T, dT)$$

(2)

$$\Delta S = S_1 - S_0$$

(3)

where S is the area enclosed by the T₂ curve segment and the abscissa; A represents the pore signal at T, and T₁ is the...
mass change of coal samples after heating accurately to normalize the mass change of coal samples to compare the heating to get the which indicated that the microwave heating did not change the on the left side was more signi
different masses, eq 4 was established to normalize the mass change of coal samples to compare the mass change of coal samples after heating accurately

\[ W = \frac{(M_D - M_w)}{M_D} \times 100\% \]  

(4)

where \( W \) is the percentage of mass reduction of the coal sample (%), \( M_D \) is the mass of the coal sample after initial drying (g), and \( M_w \) is the mass of the coal sample after microwave heating (g).

The preliminary screening results showed that the samples 11, 17, 18, 19, 20, 24, 30, and 58 had identical NMR porosity and \( T_2 \) curve distribution, so they have similar internal structural characteristics (see Table 1). The parameters of the microwave experiment are in Table 1, which presents the four different microwave heating paths used. In the table, microwave energy was the product of the output power and the heating time. The microwave energy of the four heating paths was all 150 J, and the position of the coal samples in the microwave cavity was in each case right under the antenna.

3. RESULTS

3.1. \( T_2 \) Spectrum. To compare the change in the pore structure of coal samples before and after microwave heating more clearly, the \( T_2 \) curves of one of the two samples from each path were selected and subtracted from the \( T_2 \) curve after heating to get the \( T_2 \) difference curve of the coal samples (see Figure 2).

As shown in Figure 2, \( T_2 \) curves of all coal samples showed typical bimodal distribution before and after heating. The crest on the left side was more significant than that on the right side, which indicated that the microwave heating did not change the pore distribution characteristics of the coal samples, and the pore number was still dominated by small pores, followed by large pores. The pores in the dry samples before and after heating were mainly micropores, followed by mesopores, macropores, and microcracks. Microwave heating did not improve the connectivity between micropores, mesopores, and macropores. The \( T_2 \) difference curve of the coal samples showed that the number of pores in coal samples generally increased after microwave heating. Still, the distribution of pores in the coal samples was different under different microwave heating paths: (1) according to the increasing degree of the number of macropores and microcracks, the distribution of pores in coal samples was different, path 1 < path 2 < path 3 < path 4; (2) compared with the heating paths 1 and 2, the \( T_2 \) curves of paths 3 and 4 extended obviously to the right, indicating the appearance of larger pores or microcracks; (3) the increasing of the number of microcracks in the four heating paths was the same, indicating that the number of microcracks was insensitive to the microwave heating path.

3.2. Pore Volumes. According to eqs 2 and 3, the change of pore volume under different heating paths is shown in Figure 3. Unfortunately, because of the accidental damage of the No. 19 coal sample, the data about the heating path 1 only came from the No. 30 coal sample. The observed volume changes in micropores and mesopores showed a linear relationship with the microwave heating path. However, the correlation between the volume change of micropores and the microwave heating path was weak. The volume change in macropores and microcracks was closely related to the microwave heating path, and the distribution was exponential. The changing trend of the total pore volume was consistent with that of macropore and microcrack volumes, exponential in distribution with good correlation, which indicated that macropores and microcracks contributed to increase in the total pore volume in coal samples.

3.3. Masses and Temperatures. Figure 4 shows the shift in mass and temperature of samples under different microwave heating paths, and the mass change was obtained by eq 4. There is a strong correlation between temperature and the percentage of mass reduction, and both increased with the increase of microwave power. Although the output microwave energy of four microwave heating paths was the same, the change of coal sample mass and the temperature was more evident under higher microwave intensity. This phenomenon was because the water in the coal could be divided into free water and bound water, and most of the free water can be removed at 110 °C, while it can only remove the bound water at 180 °C. In the experiment, the temperature in the drying process was only 40 °C, at which temperature a large amount of free water still existed in the pores of the coal. When the free water absorbed the microwave and transformed into water vapor to escape from

Table 1. Screening Results of Coal and the Microwave Heating Path

| heating path | sample | height (mm) | diameter (mm) | volume (cm³) | NMR porosity (%) | \( T_2 \) curve distribution | output power (kW) | heating duration (s) | microwave energy (J) |
|--------------|--------|-------------|--------------|--------------|-----------------|--------------------------|-----------------|-------------------|-------------------|
| 1            | 19     | 29.91       | 48.99        | 56.35        | 15.79           | bimodal distribution     | 1.5             | 360               | 150               |
| 1            | 30     | 30.01       | 49.05        | 56.54        | 13.51           |                          |                 |                   |                   |
| 2            | 17     | 29.82       | 48.96        | 56.11        | 14.54           |                          | 3               | 180               |                   |
| 2            | 24     | 29.97       | 49.04        | 56.38        | 16.61           |                          |                 |                   |                   |
| 3            | 11     | 30.04       | 49.19        | 57.06        | 15.35           |                          | 4.5             | 120               |                   |
| 3            | 58     | 30.14       | 49.35        | 57.62        | 15.34           |                          |                 |                   |                   |
| 4            | 18     | 29.95       | 49.04        | 56.54        | 15.99           |                          | 6               | 90                |                   |
| 4            | 30     | 29.75       | 48.95        | 55.96        | 16.23           |                          |                 |                   |                   |
the coal, the coal mass decreased. According to Li’s research on microwave drying of coal, when the microwave energy density was low, only a tiny amount of water vapor could break through the obstruction of pore structure and escape to the surface, while

Figure 2. Typical $T_2$ spectrum of water-saturated coal samples before and after heating. (a) An example from path 1, path 1 difference spectra; (b) an example from path 2, path 2 difference spectra; (c) an example from path 3, path 3 difference spectra; and (d) an example from path 4, path 4 difference spectra.
with high-power microwave applications, large amounts of water vapor in the pores could be forced out of the coal. Therefore, for reducing coal sample quality and the increase of temperature in the experiment, path 1 < path 2 < path 3 < path 4. In addition, the moisture in the rock during microwave heating could not only increase the surface temperature but also dominate the temperature distribution within the rock.16

3.4. Crack Propagation Mode. The above results showed that microwave heat treatment could effectively change the pore structure of the coal. The essence of this change was the microwave damage to the pore structure of the coal. In this process, microdamage accumulated continuously in the coal, resulting in macrodamage. How to find the formation law of macrodamage based on the image processing method is introduced in Section 2.3. The crack distribution on the surface of the coal sample can be obtained, as shown in Figure 5.

The effects from heating paths 1 and 2 were to expand the initially closed cracks in the coal samples; heating path 3 further made new cracks appear; the additional effect from heating path 4 progressed to develop secondary cracks and further expanded the primary cracks. Thus, the above phenomena demonstrated that the path adopted in injecting the same microwave energy into the coal resulted in significantly different modes of crack development. To reiterate, on adoption of heating path 1 (low power and long duration), the only effect visible was the increase in the original crack width; however, path 4 (high power and low duration) led to more complex crack development, with an increased number of cracks and an uncertain crack propagation pattern, facilitating an irregular network of crack formation in the coal.
4. DISCUSSION

Coal has a complex structure of pores and cracks, which meets the space needs for storage and migration of CBM. The development and change of the pore and crack structure will directly affect the desorption and migration law of CBM. CBM exists in the coal body in free and adsorption forms. The volume increase of the macropores and microcracks in the coal body will inevitably lead to a pressure decrease of CBM. When the free CBM pressure was lower than the adsorbed CBM pressure, the CBM adsorbed on the surface of pores and cracks will be desorbed and diffused and then transformed to the free CBM, entering the free space through larger pores and cracks.

According to Liu’s research,20 the evolution mechanisms of pore structures during microwave heating included the collapse of pore structures caused by shrinkage forces resulting from the removal of moisture, open and cross-linking of blind and closed pores, and thermal decomposition of organic macromolecular structures under high temperatures. Here, the highest surface temperature of the coal sample was 112 °C (see Figure 4), which was far lower than the temperature needed for coal pyrolysis (200 °C),20−22 so we could ignore the performance of the thermal decomposition, while there may be local hot spots where the minerals are located. Previously, scholars found through experimentation with microwave drying19 and centrifugation14 of coal samples that connectivity between macropores and microcracks in coal was the best, followed by mesopores. The connectivity between micropores ranked the worst. The pore throat connected the macropores in the coal body, and most of the water in the macropore was adsorbed on the surface of the pore while a small amount of free water was present inside the pore. When the microwave radiated the coal, the temperature of water in the pore increased. This achieves the desired target temperature using the required energy as calculated by adopting the following law in thermodynamics16

\[ P = \rho C_p \Delta T / \Delta t \]  

where \( P \) is the required energy (J), \( \rho \) is the density of the material (kg/m³), \( C_p \) is the specific heat capacity of the material (J/(kg·K)), \( \Delta T \) is the temperature difference of the material (K), and \( \Delta t \) is the heating time (s).

We ignored the change of material density and specific heat capacity during the heating process. When \( \Delta t \) is unit time, the

Figure 5. Crack’s distribution in coal samples. The black solid line circle indicated the expansion of the initially closed crack, the red solid line circle suggested that the central crack expanded and produced the secondary crack, and the yellow solid line circle indicated the appearance of the new crack.
temperature increase rate of the material is directly proportional to the microwave power. When the water in the coal pores was transformed into vapor, the relationship among pressure, volume, and temperature of the vapor followed the equation of the state of an ideal gas

\[ PV = nRT \]  

where \( P \) is the gas pressure (Pa), \( V \) is the gas volume (m\(^3\)), \( T \) is the temperature (K), \( n \) is the mass of substance of the gas (mol), and \( R \) is the molar gas constant (J/mol·K).

Equations 5 and 6 suggest that when a high-power microwave radiated the coal, the temperature of the adsorbed water and free water in the macropore increased rapidly. It vaporized in a short time, which accelerated the thermal movement of water molecules. Considering that the time required for this process was fast and water molecules have not yet had time to diffuse through the pore throat to the surrounding space, this process was a constant volume expansion process of water vapor. In this process, the pressure generated by the water vapor acts on the inner surface of the pore. When the pressure increased to a certain extent, the pore expanded immediately. After expansion, the pressure in the pore decreased rapidly, and then the water molecules in the pore moved toward the direction of the pressure gradient (outside the coal). When the coal was radiated by low microwave energy, with a low heating rate, a large amount of adsorbed water bound in the pores was not expelled in a short time. Thus, only the molecular thermal movement of a small amount of free water was lost.\(^{19}\)

With time, more and more pore water was heated and transformed into water vapor; meanwhile, water molecules also continuously diffused to the surrounding through the pore throat. This process can be regarded as the constant pressure expansion process of water vapor. In this process, the destructive effect of vapor pressure on pores was very minimal (see Figure 6a). For the pore system composed of micropores, although the pores were relatively independent and regarded as a constant volume expansion process, the micropores contained less water, which was adsorbed water. Furthermore, the pore wall thickness was more significant than that of micropores plus the collapse of pore structures caused by shrinkage forces, so the influence of microwave power on the micropore volume was small. For the above reasons, the volume of macropores and microcracks in the coal increased exponentially with high microwave energy. In contrast, the micropore volume only increased irregularly and slowly (see Figure 3). This suggested that when injected with the same microwave energy, the combination of high power and low duration was more conducive to the transformation of CBM from the adsorption state to the free state.

From the macroscopic view, cracks appeared on the surface of coal samples after microwave radiation (see Figure 5), caused by selective heating of the microwave and heterogeneity of the coal. The high-power microwave heating of coal significantly improved the vaporization rate of water in the coal, and increased the volume of water vapor by more than 1300 times in a short time. Local tensile stress was formed by the expanding volume in the pores and cracks of the coal, which promoted the formation and propagation of cracks.\(^{10,11}\) Some cracks were interconnected to form a crack network leading to contact with the external environment. Hence, the water vapor in the coal was ejected along the crack, resulting in a microwave heating effect that was similar to a "steam explosion" (see Figure 6b). This led to the exponential change of the coal sample mass with increased microwave intensity (see Figure 4).

Additionally, coal was a heterogeneous material, and the thermal expansion coefficient, elastic modulus, and dielectric constant of its mineral components were also significantly different. Therefore, the local thermal stress caused by uneven thermal expansion was also one reason for crack formation.\(^{24}\) The growth and development of cracks in the coal after microwave heating provided functional space for the migration
of free CBM, making it convenient for CBM to flow from the coal body to the free space.

5. CONCLUSIONS
To evaluate the influence of microwave heating paths on the coal structure, eight coal samples with similar porosity and pore structure were selected from 60 coal samples. The changes of T2 spectrum, pore-volume, mass, temperature, and visual of coal samples before and after microwave heating were compared and analyzed:

1. Although the microwave heating path did not change the pore distribution characteristics of the coal, connections between pores and pore volume enlargement caused by water removal led to a significant increase in the number and size of macropores and microcracks and the volume changed exponentially with the addition of microwave power.

2. The microwave heating path affected the crack development mode of the coal. The combination of high-power and low-duration path of microwave heating parameters made the crack development more complex, making it easier to form the crack network in the coal.

3. At the microlevel, the constant volume expansion and constant pressure expansion of water vapor produced by high- and low-power microwaves, respectively, were the main reasons for the different effects of microwave heating paths on the pore structure of the coal.

4. On the macrolevel, high-power microwave heating significantly improved the vaporization speed of water in the coal. The rapid expansion of water vapor produced a microwave heating effect similar to the steam explosion in the cracks of the coal samples, resulting in the development and expansion of coal cracks.

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