

Experimental Study of Adsorption Characteristics and Deformation of Coal for Different Gases

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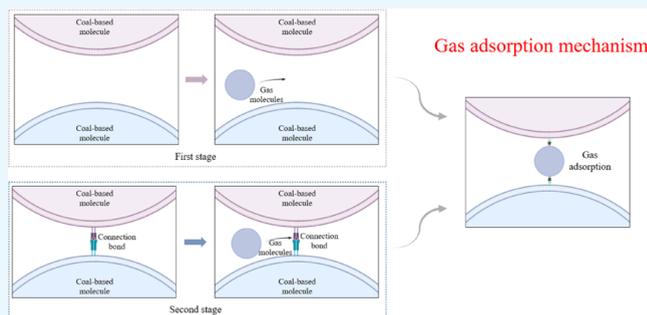
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ABSTRACT: Coal seam deformation due to gas adsorption affects the stability of the underground structure. Natural coal blocks of the Shanxi Formation were selected to study the dynamic adsorption characteristics of coal samples subjected to CO₂, CH₄, and N₂ gas injections under coaxial pressure and confining pressure (7 MPa), as well as the displacement of CH₄ with CO₂ and N₂ under the same conditions. The results show that, under the same conditions, the strain in the coal samples first increased, followed by a rapid increase along with the increase in pressure, with the transverse strain being always higher than the axial strain. The amount of gas adsorption varied from high to low as CO₂ > CH₄ > N₂, and the final adsorption strains and equilibrium times were different for each gas. Based on the increase in gas pressure, the gas adsorption strain curve can be divided into two stages. The displacement of N₂ only uses partial pressure to achieve the desorption of CH₄ in the coal sample, leading to shrinkage deformation of the coal sample. In contrast, the displacement of CO₂ has the dual effects of competitive adsorption and partial pressure reduction on CH₄, leading to the swelling deformation of the coal sample.



1. INTRODUCTION

Gas absorption in a coal seam will lead to volumetric deformation,^{1–3} which results in changes in the strength, stress state, and porosity of the coal^{4–7} and affects the stability of the underground structure.^{8–10} Therefore, studying the adsorption and deformation characteristics of coal samples with respect to various gases has important theoretical and engineering significance.

Researchers have done a lot of work on gas adsorption in coal.^{11–13} By analyzing the gas injection pressure,¹⁴ it was found that when the gas pressure reaches 15 MPa, the volumetric expansion of coal is 0.2–1.6%.¹⁵ When the gas pressure is 15–71 MPa, the volume will either decrease or remain unchanged. It was further revealed that during the adsorption deformation process, the presence of free methane will cause the coal base to shrink and deform,¹⁶ as per the control mechanism of the increase in coal seam permeability. There are significant differences in the gas absorption of coal at different stages.¹⁷ Some researchers analyzed the macroscopic and microscopic differences between medium- and high-grade coals^{18,19} and described their adsorption/desorption mechanisms.^{20–22}

Coal expands when gas is adsorbed, while it shrinks when the gas is desorbed.²³ This phenomenon has been recognized by researchers, and a prediction model for the expansion and contraction deformation of coal has been established.²⁴ A classical temperature-adsorption equation similar to that proposed by Langmuir²⁵ was put forward, and a volumetric

strain model suitable for the adsorption strain equilibrium conditions was gradually proposed and improved.²⁶ Combining with Bangham's law of adsorption-induced deformation, the relationship between the linear expansion and adsorption was obtained. Based on this linear adsorption deformation, a coal adsorption volume strain model was further deduced.^{27–29}

However, due to the difficulty in measuring the strains in the coal sample during the gas adsorption process, available data are hardly reliable. Also, during the exploitation of coalbed methane, the expansion of the coal is dynamic due to the influence of ground stress and gas displacement on the coal reservoir.³⁰ Existing literature on the dynamic behavior of coal seam adsorption is scant, and the analysis of gas displacements is not extensive. To this end, coal from the Shanxi group in the Wuyang Coal Mine of the Shanxi Coking Coal Group was selected to make cylindrical samples. The test device for coalbed gas-phase displacement simulation was combined with strain gauges and gas chromatographs to monitor the adsorption deformation and displacement deformation of the coal sample

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so as to explore the differential process that can provide a theoretical basis for underground coalbed methane extraction.

2. MATERIALS AND METHODS

2.1. Experimental Coal Samples.

- (1) Sample selection. The coal samples were selected from the 3# coal seam of the Shanxi group in the Wuyang Coal Mine under the Shanxi Formation. The sample is made of high-quality lean coal with an average mineral content of 9.3%, which is mainly composed of clay and calcite. It is classified as a medium-ash, extra-low-sulfur coal.
- (2) Preparation of coal samples. To obtain the deformation characteristics of the real coal during the gas adsorption process, the coal seam with a buried depth of 550 m was selected for sampling, from which cylindrical samples of $\Phi 50 \text{ mm} \times L100 \text{ mm}$ were prepared according to the "Standard for test methods of engineering rock mass GB/T 50266-99". In addition, the broken pieces of coal were ground and sorted using an 80-mesh screen for the analysis of the coal parameters and elements (Table 1).

Table 1. Coal Sample Parameters and Elemental Composition

parameter	value	parameter	value
moisture	1.32%	carbon	82.64
ash	9.32%	hydrogen	4.18
volatility	10.72%	nitrogen	1.71
firmness coefficient	0.63	oxygen	3.52
true density	1.53 g/cm ³	vitrinite	70.2
apparent density	1.38 g/cm ³	exinite	4.2
reflectivity	2.15%	inertinite	25.6

- (3) Grouping of samples. The experiment is divided into two stages: adsorption deformation and displacement deformation. Among them, the adsorption deformation stage considers the influence of different gas injection pressures for the same confining pressure and axial pressure, while the displacement deformation stage considers the

Table 2. Grouping of Experimental Coal Samples

confining pressure (MPa)	axial pressure (MPa)	gas injection pressure (MPa)	displacement pressure (MPa)
7	7	1	4
7	7	2	4
7	7	3	4
7	7	4	4
7	7	5	4

influence of different displacement gases under the same confining pressure and axial pressure (Table 2).

According to the existing research, it can be seen that moisture, firmness coefficient, and volatility have a great influence on the gas adsorption capacity; the increase of vitrinite or the decrease of inertinite will cause the adsorption capacity to increase; and reflectance and ash content reflect the metamorphism of coal degree, the greater the degree of metamorphism, the greater the adsorption capacity.

2.2. Experiment Apparatus. This experiment used a coal seam gas-phase displacement simulation test device, which can

simulate the environment where the confining pressure is 0–60 MPa and the boost pressure of the gas booster pump is 0–40 MPa, with real-time measurement of the gas flow at the outlet. The software system can realize the detection and automatic control and generate various charts and reports. The gas displacement pressure is controlled by a metering pump, and the component analysis in the gas displacement process is performed using a gas chromatograph (Figure 1).

Before the experiment, two groups of four resistance strain gauges each were attached to the middle of each coal sample to monitor the axial and transverse strains of the coal sample. CH₄, N₂, and CO₂ gases with a purity of 99.99% were used in the experiment. The steps in the experiment are:

- (1) Place the processed coal sample into a suitable holder and into the triaxial pressure chamber. Connect strain gauges and vent pipelines, inspect the tightness of the system, and complete the pipeline volume test. Then, vacuum pumps the system for 24 h.
- (2) Lower the system pressure headfirst to fix the coal sample, and then slowly apply axial and confining pressures until it reaches 7 MPa. During the loading process, the loading rate is controlled at 0.05 kN/s to avoid structural damage to the coal sample.
- (3) Set the metering pump to a constant pressure control mode. Set the initial gas injection pressure to 1 MPa to inject gas into the coal sample and allow the sample to fully adsorb the gas. The sample reaches the adsorption equilibrium state when the input volume of the air is the same as the output volume, the speeds of the metering pumps on both sides are equal, and the differential pressure and deformation no longer change.
- (4) Increase the gas injection pressure to 2 MPa. After the adsorption equilibrium is reached, the adsorption experiments at 3, 4, and 5 MPa are completed in turn. The experiments are also performed for different coal samples and CH₄, N₂, and CO₂ injection gases to complete the first stage of the adsorption deformation test.
- (5) Place the coal sample in the displacement group and steadily increase the confining pressure and axial pressure to 7 MPa. Inject CH₄ gas into the coal sample at a gas injection pressure of 3 MPa and wait until the coal sample reaches adsorption equilibrium.
- (6) Set the injection pressure to 4.5 MPa and continuously inject N₂ gas into the coal sample until the outlet pressure is 4 MPa. Ensure the fluidity of the gas during the replacement process and allow N₂ to fully replace the CH₄ in the sample. At the same time, collect the flow rate and composition data at the outlet.
- (7) The displacement will be completed when the CH₄ content at the outlet drops to approximately 20%. Now, replace the coal sample and repeat the CH₄ adsorption equilibrium process. Use CO₂ for displacement and complete the second stage of the displacement deformation test.

3. RESULTS AND DISCUSSION

3.1. Adsorption Strain Characteristics of the Coal Sample. Due to the small strains, the product of the strain in two or three directions can be ignored. Therefore, the volumetric strain can be expressed as

$$\varepsilon = 2\varepsilon_1 + \varepsilon_2 \quad (1)$$



Figure 1. Experiment process.

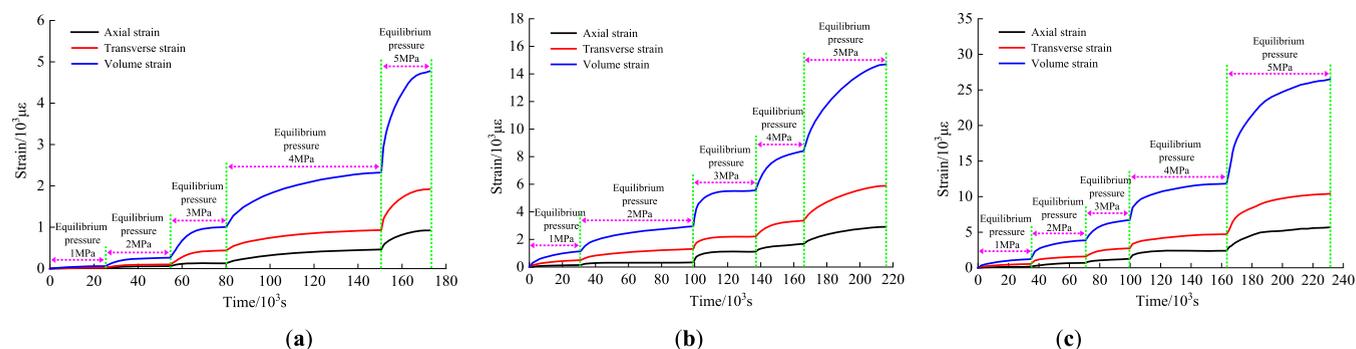


Figure 2. Relationship between coal sample strain and gas equilibrium pressure: (a) nitrogen adsorption curve; (b) methane adsorption curve; and (c) carbon dioxide adsorption curve.

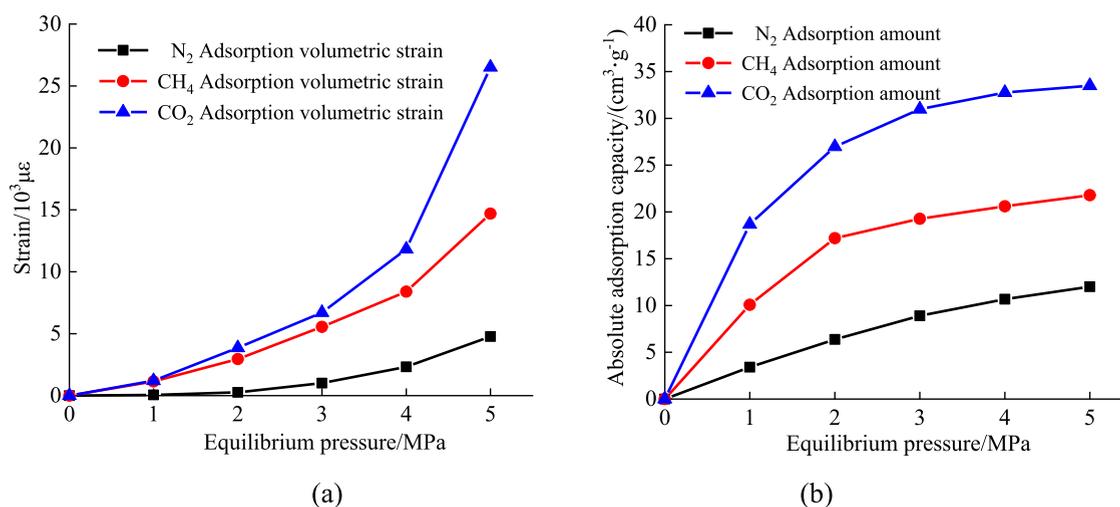


Figure 3. Adsorption deformation and adsorption amount under different equilibrium pressures: (a) equilibrium pressure and adsorption deformation; (b) equilibrium pressure and adsorption amount.

where ε is the volumetric strain, $\mu\varepsilon$; ε_1 is the transverse strain, $\mu\varepsilon$; and ε_2 is the axial strain, $\mu\varepsilon$.

According to eq 1, the linear strain at each pressure of the coal sample during the adsorption of different gases is converted to the volumetric strain, and the relationship curve of the strain with the equilibrium pressure of the gas after the adsorption of different gases is obtained as shown in Figure 2.

When the coal sample is exposed to a certain pressure of gas, the internal cracks and pores are filled with gas, and the coal sample begins to deform. From the point of view of molecular motion, gas adsorption can be instantaneous. However, in the laboratory, due to the slow diffusion of the gas from high concentration to low concentration in the coal sample, the deformation is slow until it finally reaches equilibrium stress.

It can be seen from Figure 2 that the molecules are adsorbed on the surface of the coal base during gas injection into the coal sample, reducing the surface tension of the coal sample and resulting in the expansion of the coal matrix. On the other hand, the increase in the internal pore pressure reduces the effective stress acting on the coal sample. The combined effect of the two causes the deformation of the coal sample at the macro-scale. At the same time, the variation trend of the axial and transverse strains with the increase in equilibrium pressure is basically the same. Both showed the pattern of an initial slow increase followed by a rapid increase, with the transverse strain being always higher than the axial strain.³¹ This is due to the anisotropy of the distribution direction and length and width of the cracks in the coal sample, which leads to the anisotropic

deformation of the coal sample. After the gas enters the cracks and the inside of the coal sample, the lateral cracks become more developed with a higher specific surface area and the transverse deformation is more obvious.³²

It can be seen from the curves that under the same conditions, the strain behaviors of the coal sample remain similar even when the gases differ. However, the final strain and adsorption equilibrium time are different for different gases, with $\text{CO}_2 > \text{CH}_4 > \text{N}_2$. The final volumetric strain associated with N_2 gas is $4.77 \times 10^3 \mu\epsilon$, and the total time taken to reach equilibrium is $172.50 \times 10^3 \text{ s}$. The final volumetric strain of CH_4 gas is $14.69 \times 10^3 \mu\epsilon$, and the total time taken to reach equilibrium is $215.68 \times 10^3 \text{ s}$. The final volumetric strain of CO_2 gas is $26.51 \times 10^3 \mu\epsilon$, and the total time taken to reach equilibrium is $231.22 \times 10^3 \text{ s}$. These values depend on the boiling points of the three gases at the same pressure. The higher the boiling point, the stronger the ability to be adsorbed and the greater the deformation caused.

3.2. Relationship between Adsorption and Equilibrium Pressure. As the gas equilibrium pressure increases, the strain of the coal sample increases, but the growth rates differ. The adsorption volumetric strain of the three gases rises sharply at a high pressure, and the growth rates of CO_2 , CH_4 , and N_2 in the final stage are 14.69, 6.30, and 2.45%, respectively. It can be found that all three curves can be roughly divided into two stages:

1. Slow deformation stage. Since there are many natural pores and fissures in the coal samples, gas molecules can enter the pores to adsorb on coal-based molecules. The gas gradually diffuses from the high-concentration area to the low-concentration area, resulting in slow deformation of the coal sample.
2. Accelerated deformation stage. The better the gas adsorption, the easier it is for the gas molecules in the coal base to be enriched and saturated, and the pores and fissures in the coal sample become filled with gas. In this state, the high pressure encourages the particles to continue to squeeze into the fissures of the coal base, causing microscopic damage and intensifying the deformation of the coal sample.³³

It can be seen from Figure 3 that under a constant axial pressure and confining pressure, the adsorption of different gases by the coal sample increases with the increase in equilibrium pressure. When the gas pressure is small, the quantity adsorbed by the coal sample increases rapidly with the equilibrium pressure. After the equilibrium pressure reaches a certain level, the growth rate of the adsorption by the coal sample gradually decreases, and the curve shows a slower trend. In addition, the poorer the gas adsorption, the greater the equilibrium pressure required for the curve to slow down.

The quantity of adsorption of CO_2 under axial pressure and confining pressure is greater than that of CH_4 and far greater than that of N_2 . In addition to the different adsorption strains of coal samples under different equilibrium pressures, the quantity of gas adsorption is also different. Therefore, there is a certain correlation between the strain in the coal sample and the amount of gas adsorption. The adsorption isotherm curves of the coal samples for the three gases satisfy the Langmuir equation. However, the deformation of coal samples is affected by many factors, and thus, the equation cannot be applied directly.

Figure 4 shows the variation of coal sample strain versus the quantity of adsorption. The ExpDec formula is used to fit the curves to compare the experiment data and fitted curves.

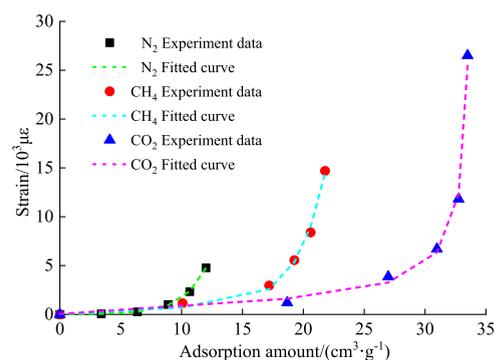


Figure 4. Relationship between adsorption amount and strain.

$$\epsilon = ae^{-Q/b} + c \quad (2)$$

where ϵ is the volumetric strain, $\mu\epsilon$; Q is the gas absorption amount, $\text{cm}^3 \cdot \text{g}^{-1}$; and a , b , and c are the calculation parameters (Table 3).

Table 3. Formula Parameters

gas	parameters			R^2
	a	b	c	
N_2	0.0091	-1.9188	0.0094	0.9993
CH_4	0.0014	-2.3604	0.6163	0.9872
CO_2	0.0082	-1.1512	1.9092	0.9599

The curve fitting correlation coefficients are all greater than 0.95. Therefore, this formula can describe the relationship between the adsorption and deformation of different gases at equilibrium pressure.

3.3. Displacement Strain Characteristics of Coal Samples. Due to the different adsorption behavior of each gas in the coal sample, the displacement mechanism of CH_4 is different, resulting in different strains in the coal sample during the displacement process. When using N_2 for displacement, the coal sample shrinks, while it expands when CO_2 is used for displacement (Figure 5).

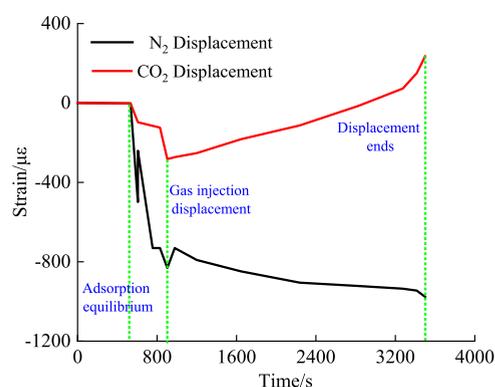


Figure 5. Deformation during displacement.

The adsorption capacity of coal samples for N_2 is less than the adsorption capacity of coal samples for CH_4 , and so the displacement of N_2 cannot achieve competitive adsorption. The displacement process mainly relies on N_2 forming a certain concentration gradient and pressure gradient on the surface of the coal seam. The resultant partial pressure is used to achieve the desorption of CH_4 in the coal sample, resulting in a shrinking

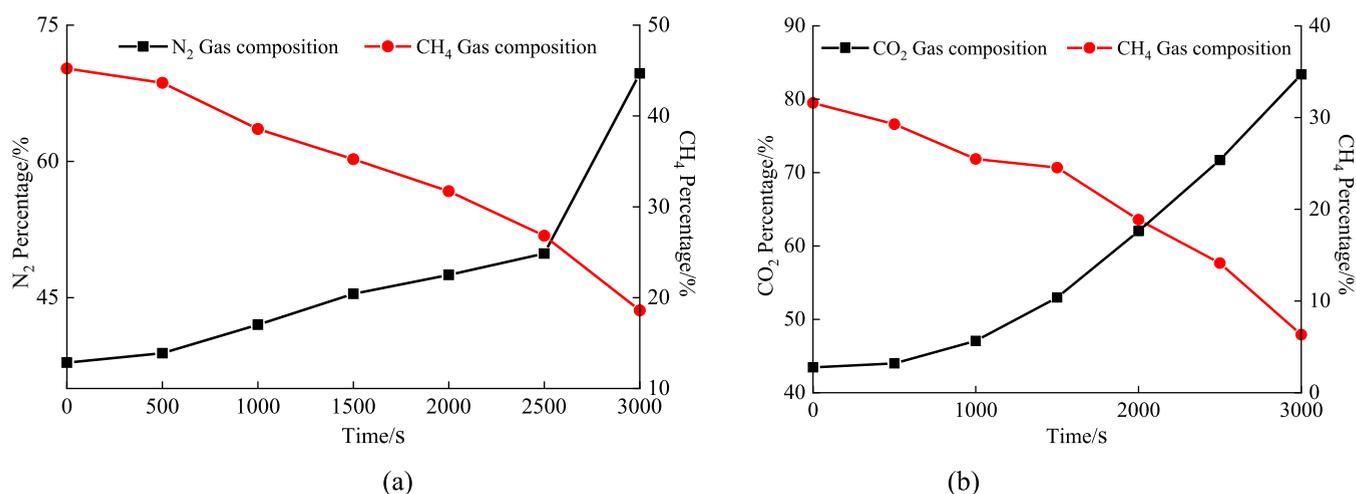


Figure 6. Gas composition in the displacement process: (a) N₂ displacement process; (b) CO₂ displacement process.

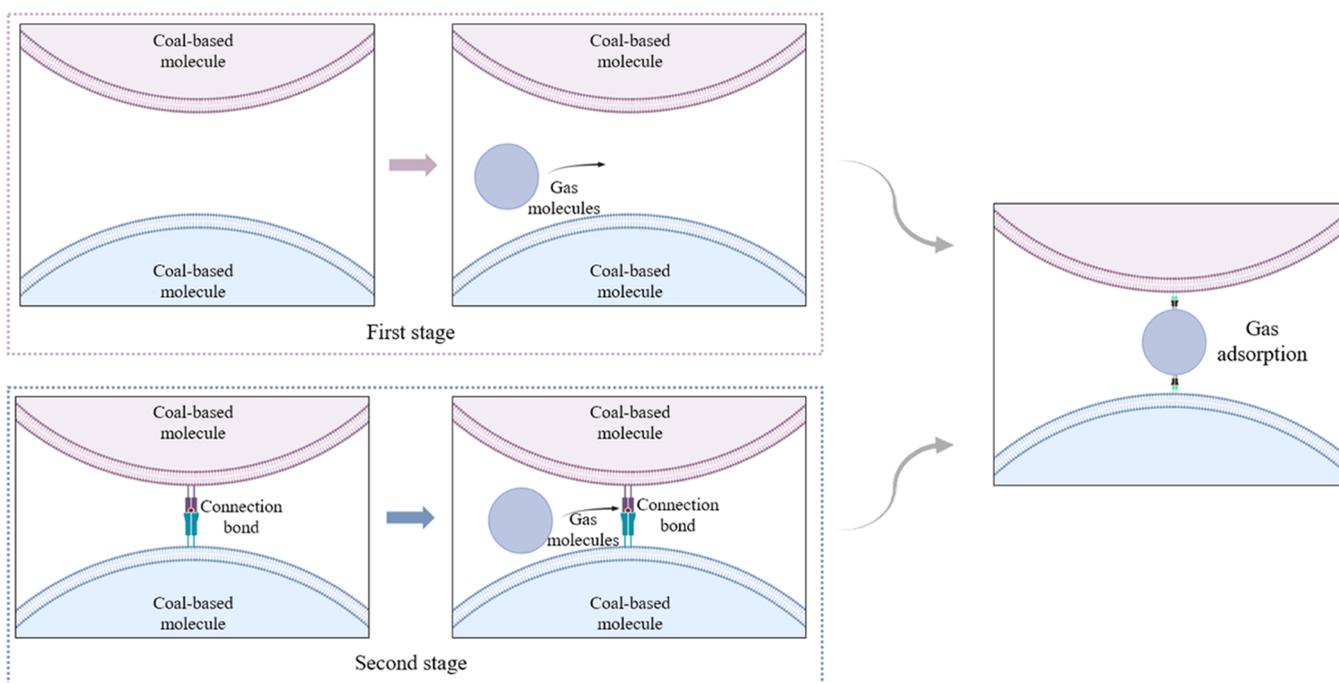


Figure 7. Gas adsorption mechanism.

state of deformation. The displacement process is mainly divided into two stages: The first stage completes the displacement of free CH₄. In the second stage, under the influence of gas injection pressure, a concentration gradient is formed from inside to the outside is formed to realize the desorption of CH₄ from coal matrix so that CH₄ is discharged from the coal sample.³⁴

The adsorption capacity of coal samples for CH₄ is less than the adsorption capacity of coal samples for CO₂, and so the use of CO₂ can achieve competitive adsorption and complete the displacement of CH₄. The displacement process is mainly divided into two aspects: on the one hand, after CO₂ comes into contact with the coal sample, it enters the micropores of the coal matrix through the pores and fissures to compete with the originally adsorbed CH₄, replacing the CH₄ molecules in the fissures, which increase the expansion deformation of the coal sample. This process also forces the adsorbed CH₄ to transform into free CH₄. On the other hand, the injection of CO₂ changes

the internal pressure structure of the coal sample. As a result, desorption occurs when the partial pressure of CH₄ is reduced, and a concentration gradient is formed, which continuously increases the displacement effect and realizes the discharge of CH₄.

3.4. Composition Change of Displacement Gas. A gas chromatograph is used to detect the components at the outlet. The collection interval is set to 500 s to record the changes of each component of the gas over time during the displacement process (Figure 6).

It can be seen from Figure 6 that as the displacement progresses, the percentage of CH₄ continues to decrease, while the N₂ and CO₂ curves both show an upward trend with two-stage characteristics. However, the trends in the curves are obviously different and are determined by the different displacement mechanisms of the two gases.

In the early stage of N₂ displacement, as time progresses, the free CH₄ is discharged first. Then, the gas partial pressure

continues to accumulate, and finally, the CH₄ in the coal sample is expelled explosively under the action of the pressure difference.

In contrast, the performance of CO₂ displacement is more stable, and it only grows slowly in the initial stage during which the free CH₄ is driven out. Then, the competitive adsorption makes CO₂ force the desorption of CH₄ in the coal sample, and finally completes the displacement of CH₄ under the condition of competitive adsorption and increasing pressure difference.

3.5. Adsorption Deformation Mechanism of Coal Samples. Studies have shown that the adsorption deformation of coal samples is caused by the combined action of the adsorbed gas and free gas. Among them, the adsorbed gas reduces the free energy of the surface of the coal matrix to cause expansion deformation, while the free gas has a compressive effect on the coal matrix through pore pressure.

The expansion of the coal matrix caused by gas adsorption is caused by the complex physical, chemical, and mechanical interactions between the gas and the solid. In addition to the adsorbed gas, there are other effects that can also cause the coal to expand. When the gas equilibrium pressure is low, the gas molecules can enter the pores or defects that are larger than or comparable in size to the gas molecules. This increases the volume of these micropores and microcracks, thereby promoting the expansion of the coal sample. When the gas equilibrium pressure is high and the energy of the gas is higher than the bond energy between the coal-based molecules, the gas molecules can intersperse between the coal-based macromolecules with the same diameter as the gas molecules. This indicates that the coal sample has developed microscopic fractures along these coal-based molecules, resulting in macroscopic expansion.

It can be seen that the free gas can also promote the expansion of coal samples by changing the coal-based structure. Therefore, the adsorption deformation of the coal sample is simultaneously affected by the expansion effect of the adsorbed gas as well as the compression and expansion effect of the free gas.³⁵

For experiments in the lab, due to the limitations of the chamber volume of the testing machine and the mechanism of action, the content of free gas is small, which cannot have a significant impact on the deformation of the coal sample. Hence, the adsorption deformation process of the coal sample can be divided into two stages (Figure 7).

In the first stage, when the gas pressure is low, the pores and fissures inside the coal sample continuously adsorb gas, leading to the expansion deformation of the coal sample caused by the adsorbed gas in the coal matrix. Also, the deformation rate decreases with the increase in pressure and increases with the increase in adsorption.

In the second stage, the quantity of adsorbed gas tends to be saturated when the gas pressure is high. However, the high pressure gives the gas molecules higher energy, which makes the gas molecules at the gas–solid interface invade the solid surface, causing microscopic fracture of the bond. Consequently, the gas continues to be adsorbed, which aggravates the expansion of the coal sample.

4. CONCLUSIONS

- (1) Under the same conditions, the trends of axial strain and transverse strain with increasing equilibrium pressure are basically the same, both of which increase slowly at first and then rapidly, with the transverse strain always higher

than the axial strain. The final volumetric strain of CO₂ gas reaches $26.51 \times 10^3 \mu\epsilon$. The final adsorption strain and equilibrium time of different gases are different, and the gas adsorption capacity is CO₂ > CH₄ > N₂ when arranged from high to low.

- (2) As the gas equilibrium pressure increases, the strain in the coal sample increases, but the growth rates are different; the growth rates of CO₂, CH₄, and N₂ are 14.69, 6.30, and 2.45%, respectively. The adsorption volumetric strain curve of the gas can be divided into two stages: slow deformation and accelerated deformation. The gas adsorption curve shows a rapid increase, which gradually reduces in slope. The lower the adsorption, the greater the equilibrium pressure required for the curve to slow down.
- (3) When considering the N₂ displacement, the coal sample shows shrinkage deformation, while it shows expansion deformation when CO₂ displacement is considered. The N₂ displacement cannot achieve competitive adsorption, and it only uses partial pressure to achieve the desorption of CH₄ in the coal samples. In contrast, CO₂ injection can achieve both competitive adsorption and partial pressure reduction of CH₄.
- (4) For indoor experiments, the adsorption deformation process of the coal samples can be divided into two stages. In the early stage, the gas adsorption in the pores and fissures is completed, while the connection bond is broken and damaged in the later stage.

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Notes

The authors declare no competing financial interest.

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