Study on the Equivalent Average Temperature Variation of the Coal Core during the Freeze Coring Process
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ABSTRACT: In the freeze coring process, the core tube is subjected to cutting heat, frictional heat with the coal wall, and refrigerant action, which causes the temperature of the coal core to be different at different positions and at different times. The equivalent average temperature is proposed to represent the change law of the whole temperature of the coal core and to provide the temperature boundary condition for calculating gas loss. Relying on the self-developed simulation platform for the freezing response characteristics of gas-containing coal, a temperature change simulation test of the freezing core under different external heat conditions was carried out, and the freezing core heat transfer model was constructed with the help of COMSOL to analyze the coal core radial temperature changes during the freeze coring process. Because the drilling sampling time of the freeze coring process is short and there is a thermal isolation device between the drill bit and the core tube, the influence of cutting heat is ignored when the model is established, and only the coal core diameter is studied. The results show that the law of equivalent average temperature of the coal core with time is consistent with the experimental law, which is divided into three stages: rapid decline, slow decline, and relative stability. The temperature drop amplitude and rate of the equivalent average temperature of the coal core decrease with increasing external heat temperature. For example, when the external temperature is 60, 70, 80, and 90 °C, the limit temperatures of the equivalent average temperature of the coal core are −36.301, −30.358, −23.956, and −18.899 °C, respectively.

1. INTRODUCTION

As an indispensable resource in people’s daily lives and production activities, coal has played a significant role in industrial production and has made significant contributions to the economic development of China.1,4 In the long run, the energy pattern of China will still be dominated by coal.2,3 The occurrence condition of coal is complicated in our country. Gas disasters frequently occur, especially with increasing mining depth. They have become the main disasters in mining.4 They have become the main disasters in mining.4 As such, it is imperative to measure gas content accurately in coal seams.5 However, because the friction heat and cutting heat generated by the coring tube and coal wall will accelerate the gas desorption of the coal seam,5−10 the gas in the coal body cannot be measured. There is a significant deviation between the gas desorption law obtained by these coal samples and the actual gas desorption law, so the gas content in the coal seam cannot be measured accurately.11

Temperature is one of the main factors affecting coal seam gas adsorption and desorption.12−16 Based on the inhibition effect of low temperature on gas diffusion, Wang17−19 and Yue20 proposed the method of freeze coring. The working principle of freeze coring is to add a refrigeration layer into the ordinary coring tube. On the one hand, the refrigeration layer serves as a cold source to cool the coal, and on the other hand, it overcomes the influence of friction heat between the coring tube wall and the coal wall during the coring process on the coal body. The reason why the cutting heat of the drill bit is neglected is because the sampling time for the freeze coring process is short and there is a heat isolation device between the drill bit and the coring tube, so the effect of cutting heat between the drill bit and the coal wall is ignored here. Li,21 Wang,22 Guo,23 Zhao,24 and Qi25 analyzed the refrigeration effects of different materials and compared the refrigeration effects of different refrigerants and catalysts. It was concluded that both methods of dry ice contact refrigeration and dry ice−ethanol refrigeration could keep the coal body in a low-temperature state for a long time and could meet the requirements of rapid and long-term low-temperature conditions of the coal cores in the process of coring. Han26

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studied the refrigerant dosage in the process of low-temperature coring and determined a reasonable refrigerant dosage corresponding to different coring depths. Ma\textsuperscript{27} studied the temperature change in the radial direction of the coal core during freeze coring and found that the cooling rate from the outside of the coal body to the center of the coal body is much slower, which is not a linear process.

The aim of freeze coring is to reduce the gas loss as much as possible so that more gas can be stored in coal to obtain a more accurate gas desorption law and to improve the accuracy of the gas loss calculation. However, the temperature boundary condition of the coal core is needed because the coal core is in a variable-temperature environment during the freezing process. Influenced by both cold and heat sources, the process of freeze coring belongs to a varying temperature process. The coal core temperature is different at different positions and at different times. The temperature of a certain point of the coal core can only represent the temperature at this point and not the temperature of the whole coal core. It cannot be used as a temperature boundary condition to determine the amount of gas leakage. Therefore, it is necessary to construct a temperature field of the coal core and use the whole temperature of the coal core as the temperature boundary condition to determine the amount of gas leakage.

The concept of equivalence is needed to determine the temperature boundary conditions. According to the definition of enthalpy, the sum of energy in an object multiplied by pressure and volume is called enthalpy. Enthalpy is not affected by the inhomogeneity of the temperature distribution of the object. Therefore, the average enthalpy of coal, that is, the equivalent average temperature, can be used to replace the whole internal energy of coal. Based on the heat conduction model and the relation between the temperature of the coal core and the radius of the coal core, the equivalent average temperature of the coal core is obtained by dividing the cross-sectional temperature of the coal core by area, which can be used to describe the overall temperature of the coal core at a certain time. After the equivalent average temperature and its change law with time are obtained, it can be used as the temperature boundary condition to accurately calculate the lost gas dosage during the coring process.

### 2. RESULTS AND DISCUSSION

**2.1. Experimental results.** Under the condition of a cold source temperature of $-40 \, ^\circ\text{C}$, the process of freeze coring with external heating conditions of 60, 70, 80, and 90 $\, ^\circ\text{C}$ was simulated, and the temperature change at the coal core and 1/2 radius of the coal are shown in Figure 1 and Figure 2.

The graphs show that the temperature changes at the core center and 1/2 radius of coal core mainly go through three stages: rapid descent, slow descent, and relative stability. In the rapid decline stage, the amount of cold source is abundant, and the refrigerating capacity is far greater than the heat provided by the external oil bath. In this stage, the cold source dominates. The temperature difference between the coal core and the refrigerant is significant so that the heat transfer efficiency is high, and the temperature drops rapidly at the coal core center and 1/2 radius. In the slow decline stage, as the heat transfer proceeds, the heat source continuously reduces the intensity of the cold source. The cold source not only cancels the heat provided by the heat source but also is used to reduce the temperature of the coal core, so the refrigerating capacity is greatly affected. Hence, the temperature drop trend of the coal core tends to be slow. At the same time, in the process of heat transfer, the temperature difference between the coal core and the refrigerant decreases continuously, which will lower the heat transfer efficiency; as the gas desorption time increases, the gas decay amount increases continuously, and the desorption rate decreases. The heat absorbed by gas desorption $Q$ decreases, and the decrease in $Q$ will, in turn, inhibit gas desorption, forming a cycle, which is manifested as a slow trend in the temperature drop of the coal core. In the stable phase, the coal core is balanced under the combined action of the inner cooling jacket and the outer oil bath jacket.

**2.2. Heat Transfer Model and Coal Core Temperature during Freeze Coring.** Because of the small size of the coal core, the number of temperature sensors installed in the experiment is limited, so only the core center temperature and the temperature at 1/2 radius can be obtained. As such, the core temperature field cannot be measured. Therefore, it is necessary to establish the thermal conduction model of the coal core during the process of freeze coring and obtain the temperature field of the coal core by numerical simulation. The equivalent

![Figure 1. Temperature curve at center of the coal core under different external thermal temperatures.](https://doi.org/10.1021/acsomega.1c06092)

![Figure 2. Temperature curve at 1/2 radius of the coal core under different external thermal temperatures.](https://doi.org/10.1021/acsomega.1c06092)
average temperature of the coal core is obtained by the surface integration method.

2.2.1. Establishment of a Heat Conduction Model. Through the on-site calculation of the sampling time of the core tube, it is found that, in the process of drilling and sampling, the cutting time of the coal wall is short, approximately 3 min. Therefore, the influence of cutting heat is not considered, and the refrigerant put into the coal core tube is much higher than required for freeze coring so that the freezing dose can be regarded as infinite. Thus, the simulation requirement for the freeze coring process is satisfied.

The short sampling time and the thermal isolation between the drill bit and the coal core tube are considered. Therefore, the heat conduction in the axial direction of the coal core is neglected in the modeling, and only the radial temperature of the coal core is studied. To calculate the equivalent average temperature of the coal core, the radial heat transfer characteristics of the experimental process are taken into account; a rectangular geometrical body is established, and five points, equally spaced at 0, 1/4R, 1/2R, 3/4R, and R of the coal core, are selected. The temperatures of these five points are extracted, and axisymmetric rotation is used to obtain the geometric model, as shown in Figure 3.

\[
\rho \frac{\partial \vec{u}}{\partial t} + \rho (\vec{u} \cdot \nabla) \vec{u} = \nabla \cdot [-p + \rho \nabla (\nabla \cdot \vec{u}) - \frac{2}{3} \nabla (\nabla \cdot \vec{u})] + \rho \vec{g} \tag{2}
\]

\[
\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \vec{u}) = 0 \tag{3}
\]

\[
\rho C_p \frac{\partial T}{\partial t} + \rho C_p \vec{u} \cdot \nabla T = \nabla \cdot (k \nabla T) + Q + Q_{sh} + W_p \tag{4}
\]

where \( \mu \) is the methane dynamic viscosity, Pa·s; \( p \) is the methane pressure, Pa; \( I \) is the tangential stress tensor; and \( g \) is acceleration of gravity, m/s².

The density of methane is given by the ideal gas equation of state, as shown in formula 5:

\[
\rho = \frac{M p}{RT} \tag{5}
\]

where \( M \) is the gas molar mass, kg/mol, and \( R \) is the universal gas constant, 8.314 J/(mol·K).

According to the model shown in Figure 3, as the model ignores the axial heat transfer of the coal core and only considers the radial heat transfer, the bottom surfaces of the upper and lower ends of the model are defined as adiabatic surfaces, satisfying formula 6:

\[
-\vec{n} \cdot (-k \nabla T) = 0 \tag{6}
\]

where \( \vec{n} \) is unit vector.

The initial and boundary conditions are \( T_i = 0 = T_0 \) and \( T = T_s \) (\( r > R, t \geq 0 \)), respectively.

The outer boundary of the model is defined as the heat source; the temperature boundary condition is set as \( T_{b1} \); the inner boundary of the model is defined as the cold source, and the temperature boundary condition is set as \( T_{c1} \). In the numerical simulation, the initial temperature of the coal body is \( T_0 = 303.15 \) K, and the boundary temperature conditions of cold and heat sources are the same as the temperature of the inner jacket and the outer jacket of the coal sample tank in the experiment.

2.2.3. Parameter Definition. The coal core is in a variable-temperature environment during the freeze coring process. The thermal conductivity of coal is one of the important parameters affecting the temperature change of the coal core. The thermal conductivity of coal is first assigned a value of \( K_1 \) and \( K_2 \) by dichotomy, and then the simulated temperature curves corresponding to the two thermal conductivities are obtained and compared with the measured temperature curve. If the measured curve is not between the two simulated curves, the thermal conductivity is re-evaluated. Otherwise, the new thermal conductivity \( K_1 = (K_1 + K_2)/2 \) is taken to obtain a new temperature simulation curve of coal core and compared with the measured curve to find the simulated curves on both sides of the measured curve again, and the new thermal conductivity is evaluated, and the above operation is repeated. The thermal conductivity \( K_n \) corresponding to the curve with the highest coincidence degree is determined as the thermal conductivity of coal by observing the intervals of the measured curves. The thermal conductivity of coal in this paper is \( K = 0.088 \) W/(m·K). The simulated and measured temperature curves corresponding to different thermal conductivities are shown in Figure 4.

![Coal core model diagram](https://doi.org/10.1021/acsomega.1c06092)
The parameters and properties of the remaining materials in the process of freeze coring shall be determined by reference to the valuing method of Ma.30 The results are shown in Table 1.

### Table 1. Coal Core Heat Transfer Model Parameters

| parameter name | parameter value | unit | parameter description |
|----------------|-----------------|------|-----------------------|
| Cp_coal        | 0.746           | kJ/(kg·k) | specific heat capacity of coal sample |
| eps_coal       | 0.93            | 1    | surface emissivity of coal sample |
| rho_coal       | 1.39            | g/cm³ | density of coal sample |
| Mw_ch4         | 16.04           | g/mol | molar mass of methane |
| mu_ch4         | 11.067          | Pa·s  | dynamic viscosity of methane |
| Cp_ch4         | 2.06            | kJ/(kg·k) | atmospheric heat capacity of methane |
| k_ch4          | 0.029           | W/(m·K) | thermal conductivity of methane |
| p0             | 2               | MPa  | free gas pressure |
| rho_steel      | 7900            | kg/m³ | density of stainless steel |
| k_steel        | 16.28           | W/(m·K) | thermal conductivity of stainless steel |
| Cp_steel       | 0.46            | kJ/(kg·k) | atmospheric heat capacity of stainless steel |
| eps_steel      | 0.16            | 1    | surface emissivity of stainless steel |

### 2.3. Simulation Results and Analysis of the Core Temperature Field

#### 2.3.1. Validation of Thermal Conduction Model of Freezing Coring

The reliability of the model is verified by comparing the experimental data with the data obtained from the model simulation. For example, the measured core center temperature, 1/2 radius temperature, and simulated core center temperature, 1/2 radius temperature are compared when the external heat is 60 °C and the cold source temperature is −40 °C, as shown in Figure 5a,b.

Figure 5 shows that the coincidence degree between the simulated temperature curve and the measured temperature curve is high, which indicates that the established model is reliable and that the simulated temperature has practical significance.

#### 2.3.2. Analysis of the Coal Core Temperature Field during Freeze Coring

Considering that the sampling time is short and the thermal isolation device is installed between the bit and the sampling tube, this paper ignores the axial heat transfer of the coal core and sets the upper and lower boundaries of the coal as thermal insulation. Only the radial temperature change of the coal core is studied during the freeze coring process. To visually express the radial temperature change of the coal core, a graph of the radial temperature distribution of the coal core at a certain time is intercepted to reflect the radial temperature field of the coal core. Taking 60 °C of external heat and −40 °C of cold source as examples, the radial section of the coal core is taken at different times during freeze coring, as shown in Figure 6a–h.

As can be seen from Figure 6, the coal core radial temperature decreases gradually from the outside to the inside during the process of freeze coring, and with the passage of time, the cold source gradually affects the inside temperature of the coal core. Macroscopically, the low-temperature area diffuses along the radial direction of the coal core from outside to inside, and the temperature difference at different positions of the coal core decreases gradually. Finally, the coal core radial temperature tends to the cold source temperature.
To calculate the equivalent average temperature of the coal core, the temperature curves at different positions of the coal core radial direction were obtained using COMSOL to take five equidistant points in the radial direction of the heat conduction.
model and extract the temperatures of these points, which are the coal core radial 0, 1/4R, 1/2R, 3/4R, and R. The temperature curves at different positions in the radial direction of the coal core during the freeze coring process are obtained and shown in Figure 7.

![Figure 7. Temperature curves of the coal core at different radial positions at 60 °C external heat and -40 °C cold source.](image)

As can be seen from the Figure 7, with the passage of freeze coring time, the overall temperature of the coal core shows a downward trend. In the radial direction, from the center of the coal core to the edge of the coal core, the coal core temperature decreases faster and faster. The closer to the edge of the coal core, the faster the temperature of the coal core decreases. At 30 min, the temperature difference of the coal core is 2.45304, 6.64157, 8.07803, and 7.48473 °C for every 0.25R from inside to outside. This is because, affected by the temperature difference, the closer to the edge of the coal core, the closer to the coal source, the greater the temperature difference, and the higher the heat conduction efficiency, indicating that the heat conduction during the freeze coring process is not linear.

According to the coal core temperature change curve, the coal core radial temperature distribution chart is drawn under the same cold source with different external heat temperatures during the freeze coring process. When the characteristics of a large temperature difference between the coal core and the cold source, high heat transfer efficiency, and quick cooling at the initial stage of freeze coring are taken into account, a group of data are extracted every 5 min at the initial freezing stage. After 20 min, a group of data is extracted every 20 min. In other words, the coal core radial temperature values after 5, 10, 15, 20, 40, 60, 80, 100, 120, and 140 min are extracted altogether and shown in Figure 8.

Because the axial heat transfer is neglected in this model, the temperature at the same radial position of the core is the same, and the equation of the core temperature about the radial position of the core can be obtained. Based on this, the equivalent average temperature of the coal core at different times can be calculated by dividing the area of the radial section of the coal core according to formula 7. The equivalent average
Table 3. Equivalent Average Temperature of Coal Core at Different Times at 70 °C External Heat and −40 °C Cold Source

| time (min) | fitting equation | $R^2$ | equivalent average temperature (°C) |
|------------|------------------|-------|------------------------------------|
| 5          | $T = 29.34542 \pm 0.155378 - 0.00397 - 0.001623^3$ | 0.99  | 15.412                             |
| 10         | $T = 26.95197 + 0.21218 - 0.009097 + 0.000860327^3$ | 0.99  | 7.459                              |
| 15         | $T = 21.21939 + 0.11788 - 0.016943^2 + 0.0013183^3$ | 0.99  | 4.289                              |
| 20         | $T = 13.54212 + 0.06246 - 0.093953^2 + 0.001823^3$ | 0.99  | 0.089991                           |
| 40         | $T = -9.46891 + 0.16646 - 0.05050^3 + 0.000964673^3$ | 0.99  | -5.089                             |
| 60         | $T = -20.9441 + 0.007666 - 0.02376^2 + 0.000458704^3$ | 0.99  | -18.803                            |
| 80         | $T = -26.16558 + 0.00362 - 0.01125^2 + 0.00021375^3$ | 0.99  | -25.375                            |
| 100        | $T = -28.71029 + 0.00172 - 0.005333^2 + 0.000102929^3$ | 0.99  | -28.263                            |
| 120        | $T = -29.91548 + 0.000814895r - 0.002533^2 + 0.0000487561^3$ | 0.99  | -29.704                            |
| 140        | $T = -30.48633 + 0.000385314r - 0.00112 - 0.0000230912r^2$ | 0.99  | -30.388                            |

where $\bar{T}$ is the equivalent average temperature of the coal core, °C; $T$ is the coal core temperature, °C; $\Delta C$ is the corresponding perimeter of different coal core radius, $m$; $\Delta r$ is the increment of coal core radius, $m$; $R$ is the coal core radius, $m$; $n$ is number; $a$ is $0$; $b$ is $R$.

Considering the characteristics of the coal core radial heat transfer, formula 7 divides the coal core radial section using the coal core radial section perimeter as the unit and combines the radial temperature of the coal core with the radial position fitting equation to establishes the differential equation of the coal core radial temperature in the process of freeze coring to obtain the equivalent average temperature of the coal core at different times.

Considering that the initial temperature of the coal core is the same at each point, the equivalent average temperature of the
The equivalent average temperature of the coal core decreases continuously with time. The rate of decrease decreases continuously until it approaches 0, which means that the equivalent average temperature of the coal core has an extreme value; the equivalent average temperature of the coal core is fitted by formula 8.

\[ T = T_0 \exp(-\alpha t) + h \]  

where \( h \) is the amount of shift down the curve, that is, the low limit temperature of the equivalent average temperature of the coal core, °C; \( T_0 + h \) is the initial temperature of the coal core, °C; and \( \alpha \) is the cooling coefficient of the equivalent average temperature of coal core.

The fitting result obtained from formula 8 is shown in Figure 9.

As shown in Figure 9, the equivalent average temperature of the coal core obviously decreases with time. It is divided into three stages: fast decline, slow decline, and relative stability. The rapid decline stage: the temperature difference between coal core and cold source is large, so the heat transfer efficiency is high, the cold source occupies the leading position of heat transfer, and it is manifested as a rapid decrease in the equivalent average temperature of the coal core; the slow decline stage: on the one hand, the coal core temperature decreases continuously, so the temperature difference between coal core and cold source decreases continuously, and the heat transfer efficiency decreases; on the other hand, the influence of heat source on the heat transfer process expands continuously, which affects the cooling of coal core by cold source, and makes the equivalent average temperature of coal core decrease slowly; the relatively stable stage: as the heat transfer proceeds, the coal core temperature decreases continuously, and finally the coal core reaches heat balance through the joint action of refrigerant and external heat, and the equivalent average temperature of the coal core tends to be relatively stable. The parameters of the equivalent average temperature of the coal core at each stage are shown in Table 7.

The equivalent average temperature drop rate of the coal core decreases with increasing external heat temperature. For example, in the stage of slow decline, the equivalent average temperature drop rates of the coal core at 60, 70, 80, and 90 °C are 0.425, 0.386, 0.347, and 0.269 °C/min, respectively. The equivalent average temperature of the coal core increases with increasing external heat temperature. For example, the equivalent average temperatures of the coal core at 60, 70, 80, and 90 °C are −36.301, −30.358, −23.956, and −18.899 °C, respectively.

### 3. CONCLUSION

The temperature field of the coal core under different external thermal conditions (60 °C, 70 °C, 80 °C, 90 °C) was studied using the response device of coal cores containing gas. Based on COMSOL software, a heat conduction model is established to solve the equivalent average temperature change characteristics of the coal core during freeze coring. The conclusions are as follows:

1. The temperature change laws at the core center and the 1/2 radius of the coal core are the same and can be divided into three stages: rapid decline, slow decline, and relative stability.
2. The heat conduction model of freeze coring is established. The data obtained by the model are consistent with the experimental data. The feasibility of the model is verified.
3. Based on the experimental data, the temperature of the coal core at different positions in the radial direction is extracted from the heat conduction model. The coal core radial section temperature field is constructed, and the

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**Table 6. Time Fitting Equation of the Equivalent Average Temperature of the Coal Core at Different External Thermal Temperatures**

| External thermal temperature (°C) | Fitting equation | R² |
|-----------------------------------|-----------------|----|
| 60                                | \( T = 65.29952\exp(-0.04292t) - 36.30103 \) | 0.99 |
| 70                                | \( T = 58.874\exp(-0.04289t) - 30.35838 \) | 0.99 |
| 80                                | \( T = 53.88989\exp(-0.04296t) - 23.95625 \) | 0.99 |
| 90                                | \( T = 47.94504\exp(-0.04399t) - 18.89854 \) | 0.99 |

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**Table 7. Parameters of Equivalent Average Temperature Variation of Coal Cores at Different Stages during Freezing and Coring**

| External thermal temperature (°C) | Rapid descent stage (0–20 min) | Slow descent stage (20–80 min) | Relatively stable stage (80–140 min) | Limit equivalent average temperature |
|-----------------------------------|---------------------------------|---------------------------------|-------------------------------------|-------------------------------------|
|                                   | Temperature drop (°C) | Rate of temperature drop (°C/min) | Temperature drop (°C) | Rate of temperature drop (°C/min) | Temperature drop (°C) | Rate of temperature drop (°C/min) | Temperature drop (°C) | Rate of temperature drop (°C/min) |
| 60                                | 38.518              | 1.926                           | 25.474                   | 0.425                             | 2.687                    | 0.045                          | −36.301                     |                                    |
| 70                                | 35.089              | 1.754                           | 23.174                   | 0.386                             | 2.448                    | 0.041                          | −30.358                     |                                    |
| 80                                | 31.218              | 1.561                           | 20.824                   | 0.347                             | 2.196                    | 0.037                          | −23.956                     |                                    |
| 90                                | 31.173              | 1.559                           | 16.147                   | 0.269                             | 1.948                    | 0.032                          | −18.899                     |                                    |
equivalent average temperature of the coal core is obtained. The variation law of the equivalent average temperature is consistent with that measured at the core center and 1/2 radius of the coal core.

4 The equivalent average temperature can express the whole temperature of the coal core at a certain time during the process of freeze coring and can be used as the temperature boundary condition for estimating the lost gas amount.

4. EXPERIMENTAL SYSTEM AND EXPERIMENTAL METHOD

4.1. Experimental Process. The aim of this experiment was to study the equivalent average temperature of the coal core during the freeze coring process. First, the temperature change of the coal core was measured under different external heating conditions, and the temperature change law of the coal center and 1/2 the coal core radial radius was studied. Then, the heat conduction model was established according to the technical principle of freeze coring, and the equation of coal core temperature about the radial position at different times was obtained. Finally, the equivalent average temperature of the coal core at different times was obtained using a surface integral. The equation of the equivalent average temperature of the coal core over time was established.

4.2. Experimental System. During the process of freeze coring, the coal core will be affected by dual effects, which are the friction heat between the coal core wall and the coal wall and the influence of refrigerant. Therefore, we built a methane coal-freezing response device that can simulate the process of freeze coring and arranged the cold source and heat source outside the coal sample tank to simulate the variable-temperature environment of the coal core during the freeze coring process. The cooling control system is arranged in the jacket outside the coal core, and the temperature is set to \(-40^\circ C\) with ethanol as the coolant. The heating control system is arranged in the jacket on the outside of the coal core, and the phenyl silicone oil is set to 60, 70, 80, and 90°C to simulate the frictional heat between the coal sample tank and the coal wall during the freeze coring process. The experimental setup is shown in Figure 10.

The device mainly comprises a vacuum degassing system, a special coal sample tank, an adsorption balance system, a pneumatic lifting and rotating mechanism, a freezing control system, a heating control system, a constant temperature water bath system, a gas metering system, and a data monitoring, collecting, and processing system.

4.3. Test Methods. The temperature data at the center and 1/2 radius of the coal body in the process of freeze coring can be obtained by presetting the temperature sensor by punching holes in the coal sample, and the gas desorption can be recorded by the gas metering device. The above parameters can be recorded every 5 s.

4.4. Coal Sample Preparation and Parameter Determination. The experimental coal sample was selected from the Guhanshan coal mine. The destruction degree of the Guhanshan coal sample is high, and its strength and particle size composition are similar to those of the briquette. Therefore, the briquette is used to simulate the coal sample collected by the core tube. Coal with a particle size of 0.25–0.5 and 0.25 mm or less was crushed by the pulverizer and was uniformly mixed at a 1:2 ratio. The coal was uniformly stirred by adding 15% distilled water, and the coal samples were uniformly stirred into a homemade mold, put on a pressure loader, and pressed for 30 min at 60 kN pressure. Finally, the briquette needed for the experiment is made, as shown in Figure 11.

4.5. Experimental Steps.

1. The briquette is dried and kept in a 105°C incubator for 8 h.
2. The coal sample is vacuum degassed to below 10 Pa.
3. The coal sample tank is placed in a 30°C constant temperature water bath, and the coal sample tank is filled with methane until its pressure reaches 2 MPa. When the pressure remained unchanged for 3 h, the coal sample is deemed to have reached adsorption equilibrium.
4. The freezing control system is turned on to lower the temperature of the cold and heat exchange tank to \(-40^\circ C\).

Figure 10. Structural sketch of freezing response characteristics of coal containing gas.

Figure 11. Briquette coal sample.
°C, and the intelligent heating thermostat of the heating control system is raised to the set temperature (the circulation with the outer oil bath jacket is closed temporarily).

(5) The coal sample tank is converted to a cold and heat exchange tank, the circulation between the outer oil bath jacket and the outer part is opened, the valve of the coal sample tank is opened, the coring process is simulated, the free gas is released, the automatic gas metering device is connected, and the coal sample is determined for gas desorption.

(6) When desorption begins, the automatic gas metering device automatically records the gas desorption amount in real time every 5 s. Also, the temperature collection device automatically records the center temperature of the coal core and the 1/2 radius temperature of the coal core in real time every 5 s. The temperature, atmospheric pressure, and other laboratory data are manually recorded every hour.

(7) The temperature of the external heating source is set to 60, 70, 80, and 90 °C, in turn, and steps 4–6 are repeated until the end of the experiment.

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Notes
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■ REFERENCES

(1) Lin, B. Q.; Chang, J. H.; Zhai, C. Analysis on coal mine safety situation in China and its countermeasures. China Saf. Sci. J. 2006, 42–46.
(2) Li, B. Dilemmas in achieving the objectives of the coal resource tax reform and the countermeasures. China Popul. Resour. Environ. 2013, 23, 69–74.
(3) Ye, J.; Lu, X. Development Status and Technical Progress of China Coalbed Methane Industry. Coal Sci. Technol. 2016, 44, 24–28.
(4) Wang, Y. K.; Zhao, X. C.; You, B. Study on seam gas deposit law as well as gas prevention and control technology in tunliu mine. Coal Sci. Technol. 2010, 38, 50–54.
(5) Zhang, J. G. Construction of gas prevention and control system in China pingmeishenma group. Coal Sci. Technol. 2017, 45, 13–18.
(6) Lian, H.; Chen, D. K.; Mao, D. B. Current status of deep mining and disaster prevention in China. Coal Sci. Technol. 2016, 44, 39–46.
(7) Diamond, W. P.; Schatzel, S. J. Measuring the gas content of coal: A review. Int. J. Coal Geol. 1998, 35, 311–331.
(8) Sun, J.; Wei, Q.; Yan, B. Full desorption process of coalbed adsorbed gas and changes of components and carbon isotopes: Results based on thermal simulation experiments. J. China Coal Soc. 2018, 43, 2848–2856.
(9) Liu, Y. W.; Liu, M. J. Effect of particle size on the difference of gas desorption and diffusion of soft and hard coal particles. J. China Soc. 2015, 40, 579–587.
(10) Wang, L.; Wang, Z. F.; Li, X. J.; Yang, Y. H. Molecular dynamics mechanism of CH4 diffusion inhibition by low temperature in anthracite microcrystallites. ACS Omega. 2020, 5, 23420–23428.
(11) Zhang, H. T.; Wei, J. P.; Wang, Y. G.; Li, H. Sampling methods for coalbed gas content direct determination. J. Saf. Technol. 2016, 12, 186–192.
(12) Tang, X.; Li, Z.; Ripepi, N.; Louk, A. K.; Wang, Z.; Song, D. Temperature-dependent diffusion process of methane through dry crushed coal. J. Nat. Gas. Sci. Eng. 2015, 22, 609–617.
(13) Yue, G.; Wang, Z.; Tang, X.; Li, H.; Xie, C. Physical simulation of temperature influence on methane sorption and kinetics in coal (II): temperature evolution during methane desorption in coal measurement and modeling. Energy Fuels. 2015, 29, 6355–6362.
(14) Yue, G. W.; Wang, Z. F.; Xie, C. Experimental study of coal surface adsorption uniformity in low temperature environment. J. Sci. Technol. Rev. 2014, 32, 71–74.
(15) Qin, L.; Wang, P.; Li, S. G.; Lin, H. F.; Wang, R. Z.; Wang, P.; Ma, C. Gas adsorption capacity changes in coals of different ranks after liquid nitrogen freezing. Fuel. 2021, 292, 120404.
(16) Qin, L.; Ma, C.; Li, S. G.; Lin, H. F.; Wang, P.; Long, H.; Yan, D. J. Mechanical damage mechanism of frozen coal subjected to liquid nitrogen freezing. Fuel. 2022, 309, 122124.
(17) Wang, Z. F.; Tang, X.; Yue, G. W.; Kang, B.; Xie, C.; Li, X. J. Physical simulation of temperature influence on methane sorption and kinetics in coal: Benefits of temperature under 273. K Fuel. 2015, 158, 207–216.
(18) Wang, Z. F.; Xie, C.; Qi, C. J.; Yue, G. W.; Li, X. J. Inhibitory effect of low temperature on methane diffusion in Coal. Saf. Coal Mines. 2016, 47, 16–19.
(19) Wang, Z. F.; Yue, G. W.; Kang, B.; Xie, C. Gas desorption inhibitory effect of coal in low temperature environment. J. Chongqing Univ. 2014, 37, 106–112.
(20) Yue, G. W.; Wang, Z. F.; Tang, X.; Li, H. J.; Xie, C. Physical Simulation of Temperature Influence on Methane Sorption and Kinetics in Coal (II): Temperature Evolution during Methane Adsorption in Coal Measurement and Modeling. Energy Fuels 2015, 29, 6355–6362.
(21) Li, X. J.; Wang, Z. F.; Qi, C. J.; Yue, G. W. Freezing experiments on molded coal with methane using dry ice as cold source. J. China Coal Soc. 2017, 42, 160–165.
(22) Wang, L.; Wang, Z. F.; Qi, C. J.; Ma, S. J.; Yue, J. W. Physical simulation of temperature and pressure evolution in coal by different refrigeration modes for freezing coring. ACS Omega. 2019, 4, 20178–20187.
(23) Guo, W. Research on simulation of heat transfer and indoor experiment of freezing method for sampling gas hydrates. Ph.D. Thesis, Jilin University, China, 2013.
(24) Zhao, J. G. Research on indoor experiment and design of wire line freezing coring tool for natural gas hydrates. Ph.D. Thesis, Jilin University, China, 2010.
(25) Qi, C. J. Simulation research on temperature changing regularity of coal core during freezing process. Ph.D. Thesis, Henan Polytechnic University, China, 2016.
(26) Han, E. G. Study on research freezing dose in the freezing core sampling process. Ph.D. Thesis, Henan Polytechnic University, 2019.
(27) Ma, S. J. Study on the temperature variation characteristics of coal core during freezing coring. Ph.D. Thesis, Henan Polytechnic University, 2020.
(28) Deng, J.; Ren, S. J.; Xiao, Y.; Li, Q. W.; Wang, C. P. Comparative study on heat transfer characteristics of coal during low temperature oxidation and pyrolysis. *J. China Coal Soc.* **2019**, *44*, 171–177.
(29) Yue, G. W.; Li, H. J.; Wang, Z. F.; Li, X. J. Effect of temperature and particle size of thermal conductivity in loose coal. *J. Saf. Sci. Technol.* **2015**, *11*, 17–22.
(30) Ma, X. P. Study on gas desorption characteristics of coal core during low temperature coring. Ph.D. Thesis, Henan Polytechnic University, 2017.