Experimental Investigation on the Abnormal Measurement of Gas Pressure during Canister Desorption of Coal Cuttings

Xianshang Zhang\textsuperscript{1,2,3,*}

\textsuperscript{1} Coal Technology and Engineering Group Chongqing Research Institute, Chongqing, 400037, China
\textsuperscript{2} State Key Laboratory of the Gas Disaster Detecting, Preventing and Emergency Controlling, Chongqing, 400037, China
\textsuperscript{3} School of Architecture and Environment, Sichuan University, Chengdu, Sichuan, 610065, China

*Corresponding author’s e-mail: zhangxianshang@sina.com

Abstract. Accurate measurement of gas pressure during canister desorption of coal cuttings is an important foundation to prevent coal gas dynamic hazards and determine gas content of coal by volumetric method. It was a new finding that gas pressure in the insulation canister increased and then decreased during the initial desorption stage in the field test, which was different with common sense about gas desorption law. We took a series of experiments to explore the possible reason for this in the laboratory. The experimental results have shown the abnormal measurement of gas pressure was caused by the temperature decrease induced by adsorbing the heat and less external energy during canister desorption. The test condition was a key factor to accurate measurement of gas pressure in the canister. These findings offered insight into the temperature variation and also changed our understanding of the temperature effect on the gas pressure measurement during canister desorption of coal cuttings. The temperature variations induced by the adsorbed gas desorption should be taken into account in engineering application, especially the accurate measurement was applied for the initial stage of gas desorption.

1. Introduction

Coal from freshly drilled cores is typically placed in desorption canisters from which desorbed gas is subsequently determined to the gas content to evaluate the coal bed methane potential [1-3]. Pressure data is part and parcel of understanding gas adsorption behavior and desorption properties; hence high-quality pressure data and reliable device continue to be highly regarded in the coal mines [4].

The influencing factors of gas adsorption/ desorption quantity of coal cuttings with emphasis on coal nature has been reported by many investigators, such as coal ranks [5], moisture content [6], coal particle size [7]. At the same time, the temperature that is subject to variation also plays a significant role in the gas desorption of coal cuttings [8, 9].

In the recent years, more and more attraction is applied to the temperature variation in the gas desorption of coal cuttings. As a contrary process of adsorption, gas molecules break away on the surface and assimilate heat during gas desorption, leading to a temperature drop [10, 11]. Many scholars [12-13] have observed the temperature decreasing phenomenon in the underground coal mine working face when the coal and gas outburst disaster occurred. Nodzeriski [14] studied a temperature drop during the desorption down to 1 bar pressure by calculating the non-isothermal desorption equilibrium.
Liu[15] analyzed the temperature variation during canister desorption of coal with an infrared thermal imager. Yang [10] provided that the temperature drop is caused by adsorbed gas desorption, free gas expansion and gas diffusion and seepage in the desorption. Xu [16] thought that the gas adsorb stronger and desorbs more rapidly with the increase of gas pressure equilibrium, the temperature decreases more drastically. Ma[17, 18] discovered that the water temperature decreased dramatically after producing 2000m³ in the coalbed gas production wells, the field phenomenon showed that the temperature variations in the desorption process had an obvious impact on the gas production. Barker[19] found that a canister had an air and desorbed gas mixture backflow into the canister during a measurement. The view was quite clear of temperature variations in the gas desorption/adsorption of coal to us.

Usually, the pressure in the canister elevates as gas desorption goes on. However, the decrease of gas pressure was occasionally found in the mine working of the Zhaozhuang coal mine which was out of step with the fundamental law about desorption, as shown in Fig.1. After the exclusion of other factors, possible explanations for this abnormal phenomenon were that either the temperature changes affect the pressure more than gas desorption, or the measuring device leakage led to the pressure drop. To investigate the first possibility, the experiments described in this paper were designed to demonstrate the pressure drop due to the temperature decrease which was caused by the endothermic reaction of gas desorption.

![Fig.1 Gas pressure curves with time during canister desorption of coal cuttings underground the coal mine](image)

2. Experimental studies

In laboratory, the PCTPro-evo volumetric sorption instrument was developed to measure temperature variations and gas desorption pressure, see Fig.2. To certify the effect of temperature reduction on the gas pressure measurement, the comparison test under non-isothermal and isothermal conditions was undertaken in the laboratory. The coal cuttings of 1mm-3mm were applied in the experiments to measure the gas pressure during canister desorption.

2.1. Gas desorption measurement in the laboratory

The coal samples used in the laboratory experiment were obtained by drilling in the same coal seam, and the results of the proximate analysis and basic adsorption parameters of the coal were listed in Table 1.
Table 1. Industrial analysis and adsorption constant of coal

| Parameters             | Values       |
|------------------------|--------------|
| Moisture content Ma    | 0.95 %       |
| Ash content Aa         | 14.63 %      |
| Volatile matter Vdaf   | 11.68 %      |
| Porosity F             | 7.24 %       |
| Apparent density ARD   | 1.41 g/cm³   |
| Adsorption constant a  | 33.8197 cm³/g|
| Adsorption constant b  | 1.2057 MPa⁻¹|

2.1.1. Measuring gas pressure and temperature of coal cuttings under isothermal condition. Kinetics and pressure measurements were evaluated by a fully automated manometric sievert type apparatus (PCTPro-E&E, Hy-Energ & Setaram, pressure measurement accuracy: 1% of reading) using ultra-high purity methane gas (BOC, 99.999%), see Fig. 2. To obtain an accurate measurement of the temperature change during canister desorption, a sensor was inserted into the canister to acquire the temperature within the coal sample. The prepared about 70g of coal particles with sizes of 1-3mm was chosen and loaded into the canister. Secondly, Helium gas was used to check the device for air tightness and calibrate the free volume of the sample canister. After the check pass of the device, the coal sample was started to be vacuumized for 12h by the vacuum pump. Next, high-pressure CH4 was injected into the canister to allow to adsorb on the coal sample and reach to the adsorption equilibrium.

Fig. 2 PCTPro-evo volumetric sorption instrument (1 The instrument typed PCTPro-evo 2 Test gas cabinet 3 Vacuum pump 4 Thermostatic water bath 5 Canister 5-1 sample tank 5-2 coal cuttings sample 5-3 venthole 5-4 temperature sensor 5-5 sampler cover)

2.1.2. Gas pressure measurement during canister desorption under non-isothermal condition. In order to explore the effect of temperature decrease, the non-isothermal experiments about the gas desorption were designed and carried out under the ambient temperature, which contained two processes: firstly measuring gas pressure during canister desorption in the air and then in a thermostatic water bath. In addition, the thermostatic water bath for isothermal experiment was set to the same temperature for the comparison. Certainly, it needs 30g from 1mm to 3mm in particles size. And the canister was monolayer and made of brass which has a good thermal conductivity.
3. Results and discussion

3.1. Gas desorption and temperature change under isothermal condition

These were the situ temperature (14℃) and temperature (30℃) of determining the adsorption constants according to Chinese standards GB/T 19560-2004, respectively, and the gas desorption quantity and coal temperature variation were determined under isothermal condition, as shown in Fig.3.

From Fig.3(a), the gas pressure increases continuously during canister desorption and achieves a new balance with the increase in time because of the isothermal condition. The gas content of coal is as a function of adsorption equilibrium pressure and temperature referring to the Langmuir equation, which is the critical factor to decide the result of gas desorption speed. The higher is the equilibrium pressure and the lower is the temperature, the more gas adsorbed and desorbed faster. From Fig.3(b), it can be seen that the temperature of coal cuttings does not keep constant as the thermostatic water bath. It decreases sharply to the lowest level when the gas desorbs rapidly after the coal sample exposure and then increases gradually with time due to the heating from the thermostatic water bath, and finally goes up to the thermostatic water bath temperature prior to reaching desorption equilibrium. This is because that gas adsorption/desorption on coal surface can be regarded as a physical adsorption/desorption process of gas on porous solids which is accompanied by exothermic/endothermic phenomena[20]. Desorption is an endothermic process, which means that in the course of gas desorption the temperature of the coal goes down if the heat transfer is hindered. The heat of desorption decreases the temperature of the coal cuttings, which in turn decreases the gas phase temperature due to convective heat transfer.

![Fig.3 the variation curves about gas pressure and temperature with time under isothermal condition](image)

Compared with Fig.3(a), the gas desorption law shown in Fig.1 preforms anomalous. That is because the canister has a good thermal conductivity and it is always placed in the thermostatic water bath to be warmed up for reducing the temperature variation gradient in the whole process. Thus, the effect of temperature variation is less than desorption on the gas pressure change. In addition, the temperature variation is often coupled with coal gas desorption. As the liberation of the gas from the coal proceeds, the temperature drop affected by this desorption process results in hindering further desorption and a retention of some gas in the coal. So, temperature influence on gas pressure measurement induced by the heat of adsorption cannot be neglected in the initial desorption stage. Inspired by this, we designed the comparison experiment with the combination of the two test conditions in the laboratory.

3.2. Gas pressure measurement under isothermal and non-isothermal experiment

The gas pressure curves are divided into two segments in Fig.4. At first, gas pressure curves increase and then decrease when the canister placed in the ambient air, which is analogous to the situ test underground the coal mine. Due to the endothermic nature of desorption, the gas phase temperature is
much lower than the initial room temperature. As the elapsed time lasts longer, desorption adsorbs more heat and therefore the gas pressure is lower. Secondly, put the canister into the thermostatic water bath which keeps the initial temperature constant, and the gas pressure curves increase rapidly. That is because canister started to be warmed up to room temperature again by the heat transfer from the thermostatic water bath.

The points 1, 2 in Fig.4 are inflection point. The point 1 shows that the gas pressure starts to decrease after about 5 minute exposure because the effect on the gas pressure of temperature starts to be more than desorption. And the time is in line with each other in different experiments, as shown in Figs.1 and 4, which further expresses the effect on the pressure measurement of temperature variations during canister desorption of coal cuttings. The point 2 appears because gas pressure starts to increase drastically when the canister is put in the thermostatic water bath during canister desorption measurement, and it indicates that the water bath warms up the coal canister.

The gas adsorption in coal is not an isothermal process in the initial sorption stage. For the adsorption experiment, the amount of gas adsorbed is often determined instead of gas adsorption kinetics. Temperature influence on the sorption equilibrium state induced by the heat of adsorption can be neglected because the temperature variation stage only lasts a short time in the initial sorption stage. But desorption is the reverse process of adsorption, and useful information can be obtained by determining gas desorption kinetics in the initial stage for coal. When the external energy cannot meet the need of the desorption consumption, the canister does not keep the initial temperature constant. It will lead to a huge error in the measurement of gas pressure and the calculation of several important parameters to prevent the coal and gas outburst. The isothermal condition is applied to weaken the temperature effect during the process of gas adsorption and desorption in the laboratory, such as thermostatic water bath. In addition, the temperature variation has a relationship with the thermal characters of coal, such as specific heat, enthalpy and heat transfer coefficient.

![Fig.4 Gas pressure variation when canister was placed firstly in the air and second in a thermostatic water bath under the temperature of 20℃](image)

**4. Conclusions**

(1) It was a new finding that abnormal measurement of gas pressure increases and then decreases with time occurs during canister desorption of coal cuttings underground the Zhaozhuang coal mine, Shanxi province in China, which was led by temperature decrease induced by endothermic reaction with adsorbed gas desorption based on the situ and laboratory experiments.

(2) The higher was the gas content of coal, the gas desorbed more rapidly, and the temperature decreased more larger. A thermostatic water bath can weaken the temperature effect on the gas pressure measurement during canister desorption of coal cuttings.
(3) The temperature variation should not be ignored during canister desorption because it has a huge effect on the gas pressure measurement and easily leads to the error in determining the parameters to prevent the coal and gas outburst and the gas content of coal seams. We should explore the principle to adsorb the heat and the method to avoid the abnormal measurement further.

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