Evolution features of coal matrix porosity with the variation in temperature and stress

Jianlou Li1,2, Buqing Li3

1School of Resources and Civil Engineering, Suzhou University, Anhui Suzhou 234000, China
2Coal Mine Exploration Engineering Technology Center of Anhui province, Anhui Suzhou 234000, China
3School of Information Engineering, Suzhou University, Anhui Suzhou 234000, China
E-mail: jianlouli1973@126.com

Abstract. To clarify the differentiated effect of temperature and stress on coal matrix pores, the coals from No. 8 coal bed in Taoyuan coal mine of Huaibei Coalfield, Anhui Province, were chosen as experimental samples. Cryogenic nitrogen adsorption methods were used in laboratories to test the matrix pore parameters of the dried coal samples treated under the temperature of 60 °C, 70 °C, 80 °C, 90 °C and 100 °C. Also efforts were made to test the parameters of matrix pore structure of dried coals after being treated under the stress of 10 MPa, 15 MPa, 20 MPa and 25 MPa respectively. The results show that: the specific surface area of coal matrix pore decreases, the average pore diameter and median pore diameter increases with the increase of temperature. As the stress goes up, the coal matrix pore first experiences increase in specific surface area, average pore diameter and median pore diameter, followed by a slight decrease after rising to a certain point. Therefore, the change in specific surface area of coal from the same coal bed can, to some extent, reflect the variation in temperature and ground stress.

1. Introduction
The Coal-mass pores can be regarded as a dual-pore structure system comprising fracture and coal matrix pores [1]. Also it can be seen as a tri-element structure system consisting of cleat, microscopic fracture and coal matrix pores [2]. Based on the pore genesis, coal pore is classified into gas pore, residual plant tissue pore, corrosion pore, inter-granular pore, etc [3]. There are three types of pore shape [4, 5], which is identified based on mercury injection curve characteristics in mercury injection experiment or the loop curve characteristics in liquid nitrogen adsorption experiment. The coal pore size varies from nanoscale to millimeter [6]. With the downward extension of coal mining, the exploited coal bed will experience constant growth in temperature and stress. What effect will temperature and stress have on coal matrix pores which are the major storage space for coal bed gas? Studies focusing on this topic are of great significance to clarifying the laws of storage and migration of coal bed gas.

According to research literatures concerning coal matrix pores [7-11], the specific pore volume and specific surface area of coal matrix, during the low and medium coal rank, will gradually decrease with progress in coal rank. For the same coal rank, the specific pore volume and specific surface area of coal matrix will rise with the growth in stress. The changing characteristics of microscopic pores from high-rank coal samples, with the variation in temperature and stress, were studied in literature.
research [12]. However, the changing characteristics of microscopic pores from low-rank coal samples were not mentioned. Above all, previous research literatures failed to make in-depth analysis of the affecting mechanisms of ground stress and temperature on coal matrix pores.

In this study, coal samples were taken from No.8 coal bed in Taoyuan coal mine of Huaibei Coalfield. The coal samples were heated in labs under relatively low temperature (\(\leq 100^\circ C\)) and underwent stress-elevation simulation experiment respectively. With the application of cryogenic nitrogen adsorption methods, structural features of coal matrix pores were ascertained. And meanwhile, analysis was made of the evolutionary characteristics of coal matrix pores during the increase in temperature and stress.

2. Equipment, samples and methods involved in the study

2.1. Experimental equipment
The Gemini VII 2390t full automatic and quick analyzer for specific surface area and pores, manufactured by U.S. Micromeritics, was used in the study. The equipment consists of refrigeration system, vacuum-pumping system, gas injection system, measuring system and data analysis system. Accessories include heating dehydration pre-treatment system, electronic scale, etc. The equipment is shown in Figure 1.

![Figure 1. Experimental equipment of cryogenic nitrogen adsorption.](image)

2.2. Experimental samples
The coal samples are taken from No. 82 coal bed in Taoyuan coal mine of Huaibei Coalfield, which is located in the southeast of Huaibei Coalfield. Taoyuan coal field features a mono-inclined structure, which strikes north-south and inclines toward the east. The position of the field in regional geological structure is shown in Figure 2.

The coal-bearing formation in Taoyuan coal field belongs to permo-carboniferous system. Among the major minable seams, 82 coal bed lies beneath the Shihezi formation and varies from 0 to 4.10 meters in thickness, with the average thickness of 2.01m. 82 coal bed has simple structure, sandstone roof and mudstone baseboard. Its rank is gas coal.

2.3. Experimental methods
(1) Pre-treatment of samples: Seal the newly-collected samples before taking them to the labs, where they are broken into small granules with a rubber hammer. Screen them with 60-mesh and 80-mesh sieves, followed by picking out around 50 grams of coal granules with 60-80 mesh in diameter as
back-up samples. Then, take 10 experiment-dedicated glass tubes and fill each with around 1.5 grams of experimental samples. Weigh and record the actual mass of each sample. Put the tubes filled with samples into the heating devices where the heating temperature is set to be 50 °C. Meanwhile, isolate the samples with nitrogen from the external air to prevent coal oxidation during the heating process. Dry the coal samples until no water left. Reweigh the mass of each dried coal sample. Finally, store the 10 tubes filled with samples in room temperature for further treatment.

(2) Heating steps: Randomly take 5 tubes filled with coal samples and heat them for 2 hours under the temperature of 60 °C, 70 °C, 80 °C, 90 °C and 100 °C respectively. Meanwhile, isolate the samples with nitrogen from the external air to prevent coal oxidation. Then, cool the samples to room temperature before cryogenic nitrogen adsorption tests one by one.

(3) Steps for stress-elevating experiment under room temperature: Take 5 tubes filled with coal samples. Elevate the stress on them with 769YP-15A type tablet presser to 10MPa, 15MPa, 20MPa and 25MPa respectively. Every stress-elevating stage last for two hours. Then, conduct cryogenic nitrogen adsorption tests one by one.

Figure 2. Geological structure background of sampling area [13].

3. Experimental results and analysis

3.1. Results and analysis of the heating experiment

Distribution features of specific surface area of 5 coal samples in Figure 3.

According to Figure 3, under the temperature of 60 °C, the maximal peak of specific surface area comes where the pore width ranges from 3.0nm to 2.7 nm. Under the temperature of 70 °C, the maximal peak of specific surface area also comes where the pore width ranges from 3.0nm to 2.7 nm. Under the temperature of 80 °C, the maximal peak of specific surface area comes where the pore width ranges from 2.7 nm to 2.4 nm. When the temperature rises to 90 °C, shrink in the specific surface area takes place where the pore width ranges from 3.4 nm to 2.4 nm. When the temperature goes up to 100 °C, there is obvious rise in specific surface area where the pore width ranges from 2.1 nm to 1.6 nm. On
the whole, with the rise in temperature, the obvious change of specific surface area tends to take place in smaller micro-pores rather than in larger pores.

The BET specific surface area, pore volume, average pore diameter and median pore diameter of five coal samples under different temperature are shown in Table 1.

![Figure 3. Specific surface area distribution in different pores under different temperature.](image)

**Table 1.** Parameter values of coal matrix pore under different temperature.

| Tem/℃ | BET specific surface area (m²/g) | Pore volume (cm³/g) | Average pore diameter /nm | Median pore diameter /nm |
|-------|---------------------------------|--------------------|---------------------------|--------------------------|
| 60    | 1.6700                          | 0.002900           | 6.94667                   | 10.4670                  |
| 70    | 1.5512                          | 0.002873           | 7.40713                   | 10.9979                  |
| 80    | 1.5200                          | 0.002910           | 7.65765                   | 11.6139                  |
| 90    | 1.4828                          | 0.002883           | 7.77639                   | 12.0830                  |
| 100   | 1.4758                          | 0.002877           | 7.79836                   | 12.0485                  |

According to Table 1, the BET specific surface area of coal matrix pore decrease with the rise in temperature. There is no obvious change in pore volume. There is increase in average pore width and median pore width. This is because, on conditions where the heating temperature is low and the heating time is short, the effect of coal metamorphism can be ignored and considerations should be given to the influence of thermal expansion and extraction on coal matrix pores only. When the temperature rises, intermolecular distance of coal matrix grows and the coal matrix will expand inward and outward. As a result, the pore-shrinking effect arises during the inward expansion. This results in the evolution of larger pores into smaller ones. Micro-pores with small diameter are more likely to be blocked by liquid organic matters as the pores shrink. Besides, as micro-pores contribute significantly to specific surface area, large quantities of blocked and sealed micro-pores after the heating process will impair the connectivity. Therefore, the number of effective pores decreases, the specific surface area shrinks, and the average pore diameter and median pore diameter grow accordingly.

It should be noted that the study focused on inner pores of coal matrix rather than cleat and fracture. Therefore, outward expansion of coal matrix was not involved herein. Outward expansion of coal matrix will result in compressed fracture between coal matrixes and weakened coal permeability [14].

### 3.2. Results and analysis of the stress-elevating experiment

The change in specific surface area of five stress-elevated coal samples with the variation of pore diameter is shown Figure 4.
According to Figure 4, the pores no more than 200nm in diameter grow rapidly in specific surface area when the stress goes up from 0 MPa to 10 MPa. Later, the specific surface area of all pores grows continually until the stress goes up to 15 MPa. However, it grows at a relatively slow pace. When the stress goes up to 20 MPa and 25 MPa, the growth in specific surface area of all pores is not obvious.

The BET specific surface area, pore volume, average pore diameter and median pore diameter of five coal samples under different stress are shown in Table 2.

![Figure 4. Specific surface area distribution in different pores under different stress.](image)

| Stress (MPa) | BET specific surface area (m²/g) | Pore volume (cm³/g) | Average pore diameter (nm) | Median pore diameter (nm) |
|--------------|---------------------------------|--------------------|---------------------------|--------------------------|
| 0            | 1.2635                          | 0.002749           | 8.70238                   | 13.9422                  |
| 10           | 2.2177                          | 0.005165           | 9.31576                   | 20.8997                  |
| 15           | 2.7719                          | 0.006627           | 9.56263                   | 22.9060                  |
| 20           | 2.7929                          | 0.006933           | 9.92930                   | 24.8504                  |
| 25           | 2.8377                          | 0.006474           | 9.12514                   | 20.5297                  |

According to Table 2, the coal matrix pores grow rapidly in pore volume, specific surface area, average pore diameter and median pore diameter. When the stress goes up to a certain level, there is a decrease to some extent in pore volume, specific surface area, average pore diameter and median pore diameter. This is because majorities of organic matters in coal belong to brittle organic component, e.g. Vitrinite and Inertinite. Brittle organic matters are easy to fracture under destructive stress. The fracture gives rise to more effective micro-pores and fracture. Newly generated fracture will form more absorption space. With the increase in the proportion of absorption space resulting from the fracture, the average pore diameter and median pore diameter increase accordingly. When the stress goes up to a certain level, e.g. 25 MPa in this experiment, the increase in fracture will be no longer obvious. Meanwhile, the volume of the formed absorption space will be further compressed. This leads to the decrease in pore volume, specific surface area, average pore diameter and median pore diameter of coal matrix pores.

4. Conclusions
After heating and stress-elevating experiments on coal gas from No. 8 coal bed in Taoyuan coal mine, the following conclusions can be drawn:

1. The specific surface area decreases, but the average pore diameter and the median pore size of coal matrix increase with the rise of the geo-therm.
The specific surface area, pore volume, the average pore diameter and the median pore diameter of coal matrix increases firstly with the increase of ground stress, followed by the decrease to some extent after the peak.

(3) Specific surface area does a better job in making a distinction between the effect of terrestrial heat and ground stress.

Acknowledgements
The study was supported by the National Nature Science Foundation of China (41302274), Doctoral research project in Suzhou University (2011JB05) and the opening scientific research platform in Suzhou University (2014YKF02).

References
[1] Close J C 1993 Natural fracture in coal in Hydrocarbons from coal AAPG 38 119-32.
[2] Gamson P, Beamish B and Johnson D 1998 Effect of coal microstructure and secondary mineralization on methane recovery Geol. Spec. Publ. 199 165-79.
[3] Yiwen J, Bo J, Guiliang W and Quanlin H 2005 Tectonic Coal Structure and Physical Properties of Coal Reservoirs (Xuzhou: China Mining University Press).
[4] Ping C and Xiuyi T 2001 The research on the adsorption of nitrogen in low temperature and micro-pore properties in coal J. China Coal Soc. 26 552-6.
[5] Yanbin Y, Dameng L, Wenhui H, Dazhen T and Shuheng T 2006 Research on the pore-fractures system properties of coalbed methane reservoirs and recovery in Huainan and Huaibei coal-field J. China Coal Soc. 31 163-8.
[6] Xuehai F, Yong Q, Wanghong Z, Chong T and Rongfu Z 2005 Fractal classification and natural classification of coal pore structure based on migration of coalbed methane Chin. Sci. Bull. 50 51-5.
[7] Shuheng T, Chao C, Baocun Z, Lijiang D and Jiazan Z 2008 Control effect of coal metamorphic degree on physical properties of coal reservoirs Nat. Gas Ind. 28 30-3.
[8] Yiwen J and Xiaoshi L 2009 New progress in studying on ultra microstructure of tectonic coal Prog. Nat. Sci. 19 131-141.
[9] Xianghao W, Yanbin W, Shasha G, Pengfei H and Meijuan Z 2012 Differences in pore structures and absorptivity between tectonically deformed and undeformed coals Geol. J. China Univ. 18 528-32.
[10] Guangwu X, Hongfu L, Huifang Y and Wei L 2011 The types of tectonic coals and pore characters in Hancheng J. China Coal Soc. 36 1845-51.
[11] Wenping J, Xiaozhong S, Lingwen Z 2011 Research on the pore properties of different coal body structure coals and the effects on gas outburst based on the low-temperature nitrogen adsorption method J. China Coal Soc. 36 609-14.
[12] Zhaoping M, Shanshan L, Baoyu W, Yongdong T and Jie W 2015 Adsorption capacity and its pore structure of coals with different coal body structure J. China Coal Soc. 40 1865-70.
[13] No.3 exploration team of coalfield geology bureau in Anhui province 2011 Geological report of Taoyuan coal mine (Huaibei: Huaibei Mining Co. LTD).
[14] Jianlou L 2010 Study of Desorption and Emission of Coal Gas under Sonic Waves (Huainan: Anhui University of Science and Technology).