Research Article

Experimental Study of 3D Micro-CT on Meso-Structure Evolution of Coal Samples with Different Coal Grades under the Action of Temperature

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In the three-dimensional micro-CT experiment system, the room temperature is set to 300 °C when different coal samples (lignite, anthracite, lean coal and gas coal) are observed for mesoscopic observation. The evolution regularity of mesoscopic structure is analyzed according to the CT scan of coal samples under different temperatures and three sections of scanning images, and by ImageJ image processing software, image processing, and analysis of the characteristics of the profile, the following conclusions are obtained: (1) Coal specimen will have an overall expansion deformation along with the rise of temperature. The sample expansion can be divided into two types: outward expansion and inward expansion. Outward expansion means that the expansion of the skeleton extends outward from the adjacent pores, while inward expansion means that the solid skeleton intrudes into the adjacent pores. When the temperature rises, the outward expansion and inward expansion occur simultaneously. The dominant expansion mode is influenced by the type of coal sample and the temperature value. (2) With the increase of temperature, coal and anthracite coal specimen pore fissure structure shows an expansion tendency before contraction, while gas coal and lean coal show reverse patterns; in addition to the above the reason of the difference vitrinite differences, one must also consider selected specimen original porosity and mechanical physical properties, such as a combination of other factors. (3) In the temperature range of 100–200 °C, when the temperature increases at the same rate, lignite porosity increases the most, followed by gas coal, lean coal, and anthracite. (4) There are certain differences in the variation trends of the three sections of the coal specimen, and the temperature values of the three curves at the maximum pixel point are also different, which indicates that the expansion of each point in the coal specimen with the change of temperature is not completely synchronous, and the physical and mechanical properties of the sample are heterogeneous.

1. Introduction

The pore and fissure structure of the coal body plays a vital role in the permeability of the coal body. The fissure is the main channel for seepage and connects countless pores around it. In coal, the fissure degree is several to tens of times smaller than the porosity, while the fissure permeability is several orders of magnitude larger than the pore permeability. Therefore, increasing the coal fissure degree can greatly increase the coal permeability, thereby increasing coal seam gas drainage efficiency [1–6]. Existing studies have shown that temperature has a great influence on the generation, development, and expansion of pores and cracks in coal. The effect of temperature on coal and rock is related to coal type, physical structure of the section, components involved, and heating temperature of coal. Many scholars have carried out a large number of detailed experiments and obtained valuable results from the genesis and classification of coal pores and fissures based on the influence of temperature on the pore structure and permeability of coal and rock masses [7–12].

Gan et al. [8] classifies the pores of coal body by their genesis and divides pores into intermolecular pores, coal plant pores, crack pores, and thermogenic pores. Thermal factors are regarded as a type of pores. Qi [9] divided coal pores into: dissolution pores, mold pores, plant tissue
pores, intergranular pores, and intercrystalline pores. Zhang [10] studied a large number of coal samples by means of scanning electron microscopy and divided the coal pores into primary pores (a few to tens of microns), metamorphic pores, exogenous pores, and mineral pores. Primary pores refer to pores that have been formed when coal is deposited, mainly including cell pores and inter-chip pores. Metamorphic pores are the pores formed by various physical and chemical processes during the metamorphic process of coal. There are mainly interchain pores (the pore diameter is between 0.01 and 0.13 μm) and pores (the pore diameter is between 0.1 and 3 μm). Exogenous pores refer to the pores formed by the influence of external factors after coal is consolidated and formed into rock. It mainly includes horn-grain pores (aperture diameter of 2 ~ 103 μm), granular pores (aperture diameter between 0.5 and 5 μm), and friction pores. Mineral pores refer to the pores created by the existence of minerals. The size of the pores is in the order of micrometers, including dissolution pores, mold pores, and intercrystalline pores. Yan et al. [11] applied the μCT225kVFCB high-precision CT experimental system to study the influence of coal rank, ash content, and coal microscopic components on the pore structure of coal through micro-CT experiments. The relationship between the porosity, permeability, and fractal dimension of the coal sample is obtained. It is pointed out that the coal type minerals will affect the porosity and average particle size of the coal body. Indicators of sexual evaluation. Zhang et al. [12] analyzed the influence of structural deformation on coal pore and fissure structure through CT scanning experiments of four types of coal samples, and pointed out that compared with primary coal samples, a large number of exogenous pores and micro-fractures are easily formed in the fragmentation stage of coal, and the average pore size is. The mixed surface porosity is also the largest; the mylonitic coal is prone to plastic deformation at the stage of mylonitic coal, and the mylonitic material develops and fills the pores, and the average pore size and surface porosity are the smallest. Wang et al. [13] established a computational digital model that simulates the pore structure of coal based on CT three-dimensional reconstruction technology and put forward many intuitive research methods in the use of CT three-dimensional digital technology, which broadened the application of CT technology. Chen Tonggang et al. used X-CT technology to reconstruct the pore and fissure structure of the experimental coal sample, including minerals, and pointed out that the CT number and porosity have a good correlation, which can be used to analyze and evaluate the pores, fissures, and spaces in the coal form. Yuet al. [14, 15] used CT technology to study the law of lean coal pores and fissures with temperature and pointed out that the small pores of lean coal connect and expand into large pore clusters at 300°C, from 180°C ~. At 600°C, the number of pores decreases first and then increases. Song et al. [16] used micro-CT technology to test the seepage pores of coal samples for mesoscopic characterization and pointed out that the number of coal pores, surface porosity, etc. increase with the increase of structural deformation, and the local powdery mylonite of coal particles will be filled. Part of the coal body's pores causes the average pore size of the coal body to decrease.

To sum up, the evolution characteristics of mesostructure of coal samples of different coal grades under the action of temperature need to be further studied. In this article, the microstructure evolution characteristics of lignite, anthracite, lean coal, and gas coal during real-time heating in the range of room temperature~300°C were studied by micro-CT technique, aiming to reveal the microstructure evolution law of different coal grade samples under the effect of temperature.

2. Experimental System and Process

2.1. Experimental Method. CT scanning can effectively distinguish pores and solid skeleton. Even after the CT image of the specimen is processed by gray level, it can be further quantitatively analyzed with gray level 0~255 to quantitatively express the ratio of pores and solid skeleton on the corresponding point of the specimen represented by a single pixel point. That is, for the heating of the specimen, the change of the shape of the internal pores and skeleton of the tested object can be studied by the change of the sum of pixels in different color scales of the image, and the change of permeability can be studied indirectly.

The amount of data scanned by CT are relatively large. Three fixed sections of the specimen are selected for analysis, namely the section 5 mm from both ends of the specimen in the radial direction and the midpoint section. The experiments are carried out at room temperature, 100°C, 200°C, and 300°C. The scanned pattern is grayed, and the influence process of temperature change on the formation, connection, and expansion of pores and cracks of the specimen is analyzed.

2.2. Experimental System. The test system adopts μCT225kVFCB high-precision micro-CT system of Institute of Mining Technology, Taiyuan University of Technology. As shown in Figure 1, the scanning magnification of the system is 1~400 times, the maximum sample size is 50 mm and the spatial resolution is 0.5 μm. In addition, a self-made atmosphere furnace is used to heat the sample slowly, and the furnace is heated by 300 W furnace wire with temperature control accuracy of ±1°C.

2.3. Sample Preparation. Among the experimental samples, lignite coal samples are selected from Ulanqab mining area in Inner Mongolia, and gas coal, lean coal, and anthracite are selected from Shansi Coking Coal Zhengli, Ximing, and Lutaishan mines, respectively. After the sample is sent to the laboratory, it is processed into a cylindrical specimen with a bottom diameter of 7 mm and a height of 20 mm by rough specimen and manual fine grinding.

2.4. Experimentation. In the experiment, the specimen was first placed on the micro-CT turntable for CT scanning observation at room temperature. Then the sample was
placed in an atmosphere furnace and heated. When the heating temperature was shown to a predetermined temperature, the sample was kept at constant temperature for 30 min, and then cooled to room temperature. The atmosphere furnace was removed to carry out the scanning experiment of the sample at this experimental temperature. During heating, constant temperature, and cooling process, the sample was always under the protection of argon environment. The first temperature point of heating was set as 100°C, then 200°C, and finally 300°C. The change of mesostructure of specimen with the increase of temperature was observed by CT pattern. This experiment focuses on the analysis of three specific faults scanned and studies the changes of pores and fissures of the section under the condition of temperature rise. By combining the changes of its characteristic pores and fissures, the law of permeability changing with temperature is reflected.

3. Meso-Statistical Analysis of Each Section of Coal Sample under Temperature

3.1. Statistical Analysis Principle. For the analysis of CT images, the image of the specimen is theoretically regarded as composed of many very small square grids (pixels). In this way, the different color order of each small grid can reflect the characteristics and differences of solid particles and pore distribution of coal and rock samples. By counting the number of pixels of all color orders in the circle of the specimen image and analyzing the percentage of pixels under the fixed color order, the change of pore cracks in the specimen can be reflected.

With the color level as abscissa and the percentage of pixel number of different color levels as ordinate, it can be obtained from the graph analysis that all pores and the critical part of pores and skeleton can be summarized by describing a pixel point with 0–50 color level value. That is, pixels with a color level value of 0 represent pores, pixels with a color level value of 255 represent solid skeleton, and pixels with a color level value of 0–50 represent areas where porosity and skeleton change critically, and pores are in the majority in this area. The above experimental principles are the basis of statistical calculations.

3.2. Statistical Analysis of Lignite Samples. The three sections of the sample were selected for postprocessing of CT scanning images, and the images of the three sections varying with temperature (room temperature, 100°C, 200°C, and 300°C) are shown in Figure 2.

It can be seen from Figure 2 that the formation, development, and expansion of pores and fractures of lignite coal samples are very obvious in the range of room temperature–300°C, and some fractures even cross the coal samples between 200 and 300°C.

Before 100°C, the coal sample is mainly formed by cracks, and the shape of coal sample hardly changes; After 100°C, the pores and fractures are further expanded and connected, and the expansion develops towards bedding, forming many large channels. These large channels increase the permeability of the coal body. The continuous expansion of the large fractures drives the surrounding pores to further aggravate the intensity of deformation, so that the small fractures directly become pure pores from the transition area between the pores and the coal skeleton. No solid particles will be mixed during this period.

In addition, in the temperature range from room temperature to 100°C, the shape of the coal sample has hardly changed. After 100°C, the shape change of the sample intensifies, but the deformation of the sample is still dominated by internal pore expansion. Figure 2 qualitatively discusses the pore fracture evolution process of lignite samples at room temperature–300°C and carries out color scale quantitative statistics and analysis on the above three sections. The results are shown in Figures 3–5.

Figures 3–5 show the proportion of pixels of each color scale in the three sections of lignite sample at different temperatures. Figure 4(a) shows the cumulative percentage of 0–255 color scale pixels in each section image at room temperature–300°C, and Figure 4(b) shows the percentage of pixels at room temperature–300°C when the color scale values are 20, 30, 40, and 50. It should be noted that when the color scale is 0, it means that the study area is all pores or fractures; when the color scale is 255, it reflects that the research object is coal skeleton (continuum); if the color scale is between 0 and 255, the analysis scope covers both pore and skeleton structures. For the same specimen, the...
smaller the color order in the regional image range, the larger the proportion of pore structure, and the more serious the coal damage. On the contrary, the better the continuity of the specimen.

According to the above analysis, it can be seen that in Figures 3(a)∼5(a), although the CT scanning sections of the test piece are different, the cumulative proportion of pixels in each color scale is generally the same, that is, for normal temperature, the percentage change of pixel value in the range of 0∼50 and 200∼255 color scale is not large, which means that the color scale values below 50 and above 150 are relatively constant at this time. Correspondingly, when the color scale is between 50 and 200, the pixel cumulative curve rises faster and the percentage ratio changes greatly. The reason is that when the color scale is above 50, the research scope is dominated by pore and fracture structure, while when the color scale is 200, the coal skeleton structure (continuum) has a great advantage, which makes it possible to take the color scale below 50 and above 200 as the measurement standard of pore (fracture) structure and skeleton (continuum) structure at room temperature. In addition, by comparing the cumulative distribution curves of pixels with different temperature scales, it can be found that when the temperature is higher, the proportion of pixels with color scales below 50 also increases. It reflects that choosing 50 as the critical level value is appropriate. Based on the above analysis, it can be concluded that the proportion of pore and fracture structures in the sample area below 50 color level is relatively large, and the pore structure of the sample changes rapidly with the increase of temperature in this area. This phenomenon corresponds to the drastic change of
permeability of coal samples in macroscopic state. Obviously, this phenomenon corresponds to the drastic change of coal sample permeability in the macro state.

Next, the change trend of 50 and its nearby color order under different temperature conditions is taken as an example to illustrate the evolution law of pore and fracture structure (Figures 3(b)∼5(b)). Similarly, the proportion of color scales in different CT scanning sections is basically the same. Even in the same section, the change trend of each color scale below 50 is almost the same. Before 200°C, the percentage of pixels within the color scale increases gradually with the increase of temperature, and when the temperature exceeds 200°C, the percentage of pixels with color scales of 50 and below shows a negative correlation with the temperature. The reason is that the lignite specimen expands and deforms with the increase of temperature, resulting in the change of proportion. The sample expansion involved here includes the increase of the overall porosity of the specimen with the increase of the pores between some skeletons with the increase of temperature, and the decrease of the overall porosity of the specimen due to the decrease of the pore area due to the intrusion of the solid skeleton into the adjacent pores. With the increase of temperature, these two phenomena occur at the same time. The dominant mode

Figure 4: The statistical figure for the section 2 of the lignite sample. (a) Cumulative proportion of pixels with 0 ~ 255 color levels at different temperatures. (b) Pixel ratio graph under specific color gradation with temperature change.

Figure 5: The statistical figure for the section 2 of the lignite sample. (a) Cumulative proportion of pixels with 0 ~ 255 color levels at different temperatures. (b) Pixel ratio graph under specific color gradation with temperature change.
is affected by coal type, physical structure of section, and temperature factors. Between normal temperature and 200°C, the accumulation of temperature makes the pore and fracture structure of lignite specimen produce, expand, and connect, and the permeability of lignite specimen also increases. When the temperature exceeds 200°C, the lignite specimen skeleton invades the adjacent pores, the expanded pores and fracture structures are squeezed, and the permeability of the sample decreases gradually with the increase of temperature.

In the above analysis process, the cumulative proportion of color scale pixels and temperature change trend of the three sections are basically the same. It should be noted that there are still some differences in the sum of pixels of each section. According to the statistics of the total pixel size of the three sections, as shown in Figure 6, it can be seen that the overall pixel values of the three sections show a trend of increasing first and then decreasing, which verifies the inference that the porosity change is caused by the interaction between the framework and pores of lignite specimen. In addition, the temperature values of the three curves are different when they are located at the pixel maximum point, which reflects that the expansion of temperature changes at each point in the lignite specimen used in the test is not completely synchronous, which is caused by the nonuniformity of physical and mechanical properties of coal.

3.3. Statistical Analysis of Anthracite, Lean Coal, and Gas Coal Samples. Due to the limitation of space, only the change of the sum of pixels in different sections of the coal samples with three coal grades with the increase of temperature is analyzed. As shown in Figure 7, in the range of room temperature to 300°C of anthracite, the total pixels of the three sections are decreasing, indicating that the three sections have a tendency to shrink with the increase of temperature. In the temperature range of 100–200°C, the section pixels have a slight rise, that is, the section area has a slight increase in this temperature range. These phenomena also reflect the law of sample deformation with temperature. Obviously, this phenomenon is obviously different from that of lignite specimens. In addition to the vitrinite difference, the influence of other factors such as the original porosity and mechanical physical properties of the selected specimens should be considered.

As shown in Figure 8, in the range of room temperature–200°C of lean coal, the total pixels of the three sections almost have no change, indicating that with the increase of temperature profile almost has no deformation, at 200°C to 300°C, the total number of pixels decreased sharply, indicating that at this temperature range, the three sections of the coal sample had a shrinkage effect, and the contour shape changed greatly. That is to say, the three sections have a tendency to shrink with the increase of temperature, and the situation of the solid framework in the specimen invading the pores is dominant.

As shown in Figure 9. In the range of room temperature to 100°C, the total pixels of the three sections almost have no change, indicating that the section almost has no deformation with the increase of temperature. In the range of 100°C to 300°C, the sum of pixels decreases sharply, indicating that the three sections of the coal sample have a shrinkage effect in this temperature range. The evolution curves of the gas coal specimen and the lean coal specimen are similar.

4. Variation Patterns of Pore Clusters of Coal Samples of Different Coal Ranks under Temperature

4.1. Analysis Principle. After the CT image is grayed, the image within the range of 0 color order is extracted by the graphics processing software. The analysis of the pixel change can directly reflect the change of the area size. The 0 color order in the sample image is the extraction index, and
the score of the 0 color order is the background. All of them are set to 255 color order, so the counting of the 0 color order pixels can reflect the scale change of the pores. The images of lignite, anthracite, lean coal, and gas coal samples were processed according to the standard, and the variation law with temperature was analyzed.

4.2. Change Rule of Lignite Porosity. The change of 0 color pixel of lignite with temperature is shown in Figure 10. From the figure, it can be seen that the number of original and new cracks in sections 1, 2, 3 increased from 7154, 4160, and 7492 at room temperature to 242561, 213543, and 228129 at 300°C, respectively, which increased by 30 times. It can be seen that the temperature has an impact on it. The change of fractures is positively correlated with the change of permeability. The formation, expansion, and connection of fractures merge into large fractures, which will inevitably lead to the increase of coal permeability. From the figure, this situation is very obvious at 200–300°C, so the increase of coal permeability should be tens to millions of times between 200 and 300°C. The change law of pore fracture under the 0 color order of lignite is consistent with that obtained by statistical method in Section 2.2 above.

4.3. Variation of Pore Mass of Anthracite, Lean Coal, and Gas Coal. As shown in Figure 11, there is a change of 0 color pixels of low-value anthracite with temperature. From the experimental data, the number of original and new fracture pixels of sections 1, 2, 3 increases from 134, 606, and 254 at room temperature to 12438, 16842, and 12135 at 300°C, respectively. The pixel increases by 95%∼98%, resulting in great changes in macroscopic permeability. Analysis of the reasons, coal samples at room temperature∼100°C coal solid particles due to the original cementation, pore fissure change is not obvious, after 100°C, the temperature destroyed its internal cementation structure, resulting in the generation, development, and expansion of cracks, study the law of temperature change is the key to study the anthracite permeability change with temperature.

As shown in Figure 12, the number of original and new fracture pixels in sections 1, 2, 3 increased from 534, 245, and 968 at room temperature to 14783, 30182, and 72731 at 300°C, which increased by 50 times. On the one hand, with the increase of temperature, a large number of micro cracks are generated, developed, and connected, which leads to the increase of the number of fracture pixels. On the other hand, coal pores expand and increase in situ. The increase of permeability at room temperature and 100°C is dominated by the development and expansion of primary fractures. Many new pore cracks were added to the specimens at 200–300°C, and the number of pore pixels was further increased.

As shown in Figure 13, the number of 0 color pixels of gas coal varies with temperature. From the experimental data, it can be seen that coal pixels are large at room temperature∼100°C.
Discussion on the Evolution of Coal Pore Structure under Temperature

The coal matrix has thermal expansion characteristics. If the initial porosity of the coal is large under the effect of temperature, the thermal expansion of the coal skeleton will increase the porosity, which further increase the pore pixels of the coal in the CT image. If the initial porosity is small, then too many solid particles per unit volume will be heated and expanded, and will quickly invade and squeeze the adjacent pores, resulting in the decrease in porosity of the specimen. With the increase of temperature, the pore and fissure structure of lignite specimens and anthracite specimens showed a trend of expansion first and then compression, while gas coal and lean coal showed the opposite law. The reason for the above differences except the difference of vitrinite. It is also necessary to consider the combined effect of other factors, such as the original porosity and mechanical and physical properties of the selected sample, that means the change of the pore structure of the coal sample under the action of temperature is related to the coal rank.

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Jianlin and Yangsheng [17] used polarized light microscope to observe the evolution of coal and rock mass pore structure under the action of temperature and found that the pore structure changes obviously under the coexistence of solid matrix and pores, which is consistent with the conclusions of this article. Yaoqing et al. [18] adopted the experimental study of the influence of temperature on the permeability characteristics of lignite and found that before 50°C, when the volumetric stress and pore pressure remain unchanged, the permeability of coal samples will decreases with the increase of temperature. The opposite pattern will appear after 50°C. In addition, it is also found that the permeability fluctuates at very low and very high points with the increase of temperature. It is believed that this fluctuation of permeability is the result of the comparison between the thermal stress and the effective stress of the coal.

Liet et al. [19] adopted the permeability of the coal body changes with temperature and stress in the experimental study, found that the permeability changes with the temperature fluctuation, and explained the effect of internal and external expansion caused by the heating of the coal body. The conclusion is consistent with this article.

6. Conclusion

In micro-CT scanning, the three sections of coal samples were scanned at three different temperatures, and then images were processed and analyzed with the help of image processing software. From the experimental results, it is feasible to characterize the development and change of pores and cracks by different color levels of pixel values, and the change of pixels in pores and cracks can also reflect the change of coal permeability. Experimental results show:

The solid skeleton of the coal sample will undergo expansion and deformation with the increase in temperature. On the one hand, this inflated deformation will increase the pores between some skeletons, which further increase the overall porosity of the sample. On the other hand, the pore area will reduce when the solid framework invades into the adjacent pores, leading to the decrease in the overall porosity
of the specimen. These two phenomena occur at the same
time as the temperature rises. Which method is dominant
depends on the combined effect of coal type, physical
structure of the section, and temperature. Ct scanning and
image analysis is an effective method to quantitatively de-
scribe these phenomena, which can quantitatively analyze
the law of porosity change caused by the expansion of solid
particles or the change of solid skeleton shape.

As the temperature rises, the pore and fissure structure of
the lignite and anthracite specimens appear expands first
and then shrinks, while gas coal and lean coal show the
opposite pattern. The reason for the above difference except
the difference in vitrinite group. The combined effect of the
original porosity, mechanical and physical properties, and
other factors of the selected specimen should be considered.

In the temperature range of 100–200°C, when the same
temperature is increased, the porosity of lignite increases the
most in four specimens, followed by gas coal, lean coal, and
anthracite.

There is a certain difference in the color-level pixel ratio
of the three sections of the coal sample. The temperature
values are different when the three curves are located at the
pixel maximum point, which explains that the various inside
points of coal sample expanded with temperature changes
are not completely synchronized, and the physical and
mechanical properties of the samples are heterogeneous.

Data Availability
The raw/processed data required to reproduce these findings
cannot be shared at this time as the data also form part of an
ongoing study.

Conflicts of Interest
The authors declare that they have no conflicts of interest to
this work.

Authors’ Contributions
Dong Zhao was involved in data curation. Pengwei Li
performed investigation. Jianlin Xie reviewed and edited the
original draft.

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