Experimental study of influence regularity of pyrite-bearing filling joints on mechanical properties of coal-rock

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Abstract. The acoustic emission (AE) monitoring technique, scanning electron microscope (SEM) system and uniaxial compression test were carried out to study the effect of pyrite-bearing filling joints on the mechanical properties of coal samples. The results show that the pyrite-bearing filling joints have adverse effect on strength, elastic modulus and peak strain of coal samples. Compared with the relatively complete samples, the post peak failure was ductile of samples with pyrite-bearing filling joints, the stress-strain curve was ladder-shaped after the peak and the AE energy of each drop appeared a peak. Many inclined and slender filling joints on the coal surface had a significant effect on the mechanical properties of the samples. Under the action of axial compression stress, the internal cracks of coal-rock with pyrite-bearing filling joints had enough time to expand and connect, and induced failure of surrounding structures, thus reducing the strengths of the samples. When fine pyrite-bearing filling joints on the coal surface, the distribution of macro-cracks were less, and SEM image showed that the fracture surface was relatively flat. While many inclined and slender filling joints on the coal surface, the sample failure accompanied by more macro-cracks, and SEM image showed that there were more holes and cracks on the fracture surface.

1. Introduction

Coal-rock mass is a kind of natural heterogeneous material, and its strength is closely related to the composition and structure [1-3]. Under the long-term geological process, there are various joints, fractures and weak structural planes in the coal-rock. In addition, the joints can often fill with materials. The filling joints cause the external deformation of coal-rock to inhomogeneity and discontinuity [4-6], thus affecting the long-term stability of coal-rock.

At present, scholars mainly studied the mechanical properties of coal-rock with filling joints by means of similar materials and numerical simulation. Shrivastava et al. [7] studied the mechanical properties of filling joints under constant normal load and constant normal stiffness by using the mixture of gypsum, fine sand and mica powder as the filling medium. Meng et al. [8] used acoustic emission to study the influence of normal pressure and filling material type on the mechanical characteristics of mortar with joints under cyclic shear. Jiao et al. [9] conducted direct shear test on marble joint samples filled with different grain sizes, and they found that the shear stress of the samples with joints decreased.
Liu et al. [10] studied the relationship between shear properties and shear rate of natural river sand filled granite joint. Zhong et al. [11] studied the influence of initial fracture length on fracture characteristics of filled jointed rock mass. Xu et al. [12, 13] studied the influence of filling thickness and other factors on shear strength of filling joint through numerical simulation. Zhang et al. [14] studied the influence of joint length, dip angle and column spacing on uniaxial compression properties of non-across filling jointed rock mass by FLAC$^3$D numerical simulation. Zhang et al. [15] studied the influence of joint thickness, stiffness ratio and other factors on fracture initiation stress and failure characteristics of jointed rock mass under uniaxial compression by PFC numerical simulation.

Most of the researches focused on the shear mechanical properties of rock with filling joints, while the compression strength and failure characteristics of samples were seldom studied. And there exists distinct difference between the simulated filling joints and the natural filling joints [16]. Moreover, the effect of different types of filling joints on the mechanical properties of the samples are different. Therefore, the uniaxial compression tests were carried out on coal samples with natural pyrite-bearing filling joints in this paper, and the effect of pyrite-bearing filling joints on the mechanical properties of samples were studied by acoustic emission and scanning electron microscope.

2. Test conditions

2.1. Sample preparation

The coal masses were taken from 11609 working face of 16 coal seam in Daizhuang coal mine of Jining, Shandong Province. After the coal transported to the laboratory, the coal samples were processed into standard samples of $\phi 50 \text{ mm} \times 100 \text{ mm}$ through the core drilling machine and stone sawing machine. By observing whether there were filling joints on coal samples surface, the coal samples were divided into two groups. In addition, each group contained three coal samples. Some processed samples are shown in Fig 1. The cyan line in the figure indicates that the filling joints on samples surface, and the distribution characteristics of pyrite-bearing filling joints on coal samples surface are shown in Table 1.

![Fig 1. Some processed coal samples](image)

Table 1. Distribution characteristics of pyrite-bearing filling joints on coal surface

| Sample number | Distribution characteristics of filling joints on coal surface |
|---------------|-------------------------------------------------------------|
| A-1, A-2, A-3 | No filling joint distributes on the sample surface           |
| B-1           | Several fine filling joints near the bottom of sample surface|
| B-2           | One vertical and two inclined slender filling joints are distributed at the upper part of sample surface, and two fine filling joints are distributed at the bottom of sample surface |
| B-3           | Many inclined and slender filling joints are distributed on the sample surface, and a horizontal and fine filling joint is distributed near the bottom of sample surface |

After took the samples, the samples were ground to less than 250 mesh through a mortar. Then, the samples were tested by infrared spectrum. And the test results are shown in Fig 2. It was found that the absorption peak appeared at $420 \text{ cm}^{-1}$ in group B sample, group A sample did not appear. While the
absorption peak at 420 cm\(^{-1}\) was the characteristic absorption peak of FeS\(_2\) [17], indicating that pyrite distributed in group B samples.

![FTIR spectrum of coal samples](image)

**Fig 2.** FTIR spectrum of coal samples

2.2. Test instruments

In this study, an electronic (AG-X250, Shimadzu, Japan) universal testing machine was selected as the loading system. In addition, PCI-2 acoustic emission equipment of MISTRAS series was used to monitor the test process. The acoustic emission sensor was fixed on the sample by tape, and the system was calibrated by pencil lead fracture method [4]. A high and low-vacuum SEM (JSM-6510LV, Jeol, Japan) was used to study the micro morphology of the samples. After spraying gold on pyrite-bearing filling joints samples, the micro morphology was observed when the samples were magnified 500 times.

3. Results and discussion

3.1. Uniaxial compression characteristics

A displacement loading method was adopted in the test at a loading rate of 0.01 mm/s. And the sampling interval was 10 ms. The uniaxial compression stress-strain curves of samples are shown in Fig 3.

![Uniaxial compression stress-strain curves](image)

**Fig 3.** The uniaxial compression stress-strain curves of coal samples

It can be seen from Fig 3 that the stress-strain curves of the relatively complete samples presented ladder like fluctuation in the pre-peak stage, and instantaneous strength change occurred after the peak stage. This was mainly due to the fact that the deformation of the relatively complete samples were still increasing after the stress reached the peak value. With the internal cracks extended and connected with each other, the samples damaged instantaneously when the internal structure reached the limit state. The post peak failure of coal-rock with pyrite-bearing filling joints samples were ductile, the stress-strain curves were ladder-shaped after the peak. Compared with the B-1 sample, the stress drop of the B-2 and
B-3 samples were more obvious. In addition, the internal structure had enough time to adjust to the axial stress after reaching the peak strength. Therefore, it adjusted to reach a new peak point after the stress drops.

Table 2. The uniaxial compression test results of coal samples

| Sample number | Uniaxial compression strength/MPa | Elastic modulus/GPa | Peak strain |
|---------------|----------------------------------|--------------------|-------------|
| A-1           | 9.11                             | 1.03               | 0.012       |
| A-2           | 9.62                             | 1.04               | 0.011       |
| A-3           | 10.35                            | 1.04               | 0.012       |
| Average value | 9.69                             | 1.04               | 0.012       |
| B-1           | 7.51                             | 0.98               | 0.011       |
| B-2           | 6.17                             | 0.77               | 0.010       |
| B-3           | 5.43                             | 0.66               | 0.010       |
| Average value | 6.37                             | 0.80               | 0.010       |

The uniaxial compression test results of coal samples are shown in Table 2, and the comparison of uniaxial compression strength, elastic modulus and peak strain of group A, B samples are shown in Figure 4. It can be seen from table 2 and Figure 4 that the uniaxial compression strength, elastic modulus and peak strain of pyrite-bearing filling joints samples are lower than relatively complete samples. Compared with the average values of compression strength, elastic modulus and peak strain of relatively complete samples, these parameters of B-1 sample are reduced by 22.50%, 5.77%, and 8.33%, respectively. In addition, the uniaxial compression strength, elastic modulus and peak strain of B-2 sample are reduced by 36.33%, 25.96% and 16.67% respectively. And the uniaxial compression strength, elastic modulus and peak strain of B-3 sample are reduced by 43.96%, 36.54% and 16.67%, respectively.

3.2. Acoustic emission characteristics

The variation of axial stress and AE energy of samples are shown in Fig 5. The AE energy fluctuation of relatively complete sample was relatively small in the pre-peak stage, only a small amount of acoustic emission information was generated. However, the AE energy fluctuated significantly when the stress was close to the peak strength. Moreover, the AE energy increased suddenly when the sample was suddenly destroyed. The AE energy of pyrite-bearing filling joints samples fluctuated before the peak strength, which was mainly caused by the crack initiation and propagation under continuous load increase. The stress-strain curves of pyrite-bearing filling joints samples were ladder-shaped after the peak and the AE energy of each drop appeared a peak. Nevertheless, the acoustic emission information gradually decreased with the decrease of stress. It indicated that there were enough time for internal

Fig 4. Comparison of uniaxial compression strength, elastic modulus and peak strain of group A, B samples

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cracks of pyrite-bearing filling joints samples to develop and expand, and recombined to form a certain resistance, which aggravated the sample damage.

Fig 5. Acoustic emission characteristics of coal samples

3.3. Failure characteristics

The internal stress of rock is mainly composed of elastic stress and additional stress caused by defects [18, 19]. The tensile stress concentration will occur at the joint under compression, which leads to the initiation and expansion of tensile cracks near the joint [20]. However, the local stress does not lead to the overall failure of the samples, and the overall failure of the sample caused when a certain number of bearing structures are damaged [21].

The failure characteristics of the samples are shown in Figure 6. The cyan line represents the macroscopic fracture surface, and the yellow area indicates the pyrite-bearing filling joints. It can be seen from the figure that the relatively complete samples were mainly in the form of splitting failure, and the crack width was large. The stable propagation of crack was along the direction parallel to the pressure axis. With the continuous increase of load, the density and length of cracks increased. After a few axial cracks connected, the samples were eventually destroyed. The tensile cracks occurred when B-1 sample was damaged, and the macro-cracks on the surface were relatively few, but some spalling occurred. The cracks were concentrated near the pyrite-bearing filling joints, propagated along the extension line of the pyrite-bearing filling joints and the tensile failure occurred along the connecting line when B-2 sample was damaged. In addition, two inclined pyrite-bearing filling joints participated in the final penetration of the sample. When B-3 sample was damaged, there were many macro-cracks and relatively wide cracks, and most of them were distributed along the pyrite-bearing filling joint. Moreover, the deformation of the sample was relatively large.
To further analyze the influence of pyrite-bearing filling joints on the failure characteristics of the samples, the fracture surface of A-1, B-1, B-2 and B-3 samples were selected for scanning electron microscope (SEM). And the SEM pictures are shown in Fig 7. When the fracture surface of relatively complete sample is magnified by 500 times, it can be seen that the fracture surface is uneven and there are more small clastic grains. It indicated that the failure of the sample was sudden, and the internal crack did not have enough time to develop and expand. Moreover, two slender cracks were distributed on the fracture surface of B-1 sample. And the fracture surface was relatively flat, and the clastic grains were larger. In addition, the fracture surface of B-2 sample was vein like with large clastic grains, and the fracture surface was smooth and distributed with micro-cracks. The fracture surface of B-3 sample was distributed with more holes and cracks, and more clastic grains were distributed.

The existence of pyrite-bearing filling joints in coal samples would change the distribution of internal stress field, and the stress concentration would occur near the end of pyrite-bearing filling joints under axial compression stress. When the loading stress is greater than the damage stress of coal sample, a large number of micro-cracks in the coal samples have enough time to expand and connect, and cause the damage of surrounding structure, thus reducing the strength of coal samples.
4. Conclusion
In this paper, the mechanical properties of pyrite-bearing filling joint samples were studied by acoustic emission (AE) monitoring technique, scanning electron microscope (SEM) system and uniaxial compression test. And the results are as follows.

1) The pyrite-bearing filling joints had adverse effect on mechanical properties of coal samples. The uniaxial compression strength, elastic modulus and peak strain of pyrite-bearing filling joint samples were smaller than those of relatively complete samples. Many inclined and slender filling joints on the coal surface had significant effect on the mechanical properties of the samples, the uniaxial compression strength, elastic modulus and peak strain decreased by 43.96%, 36.54% and 16.67%, respectively.

2) The stress-strain curves of the relatively complete samples presented ladder like fluctuation in the pre-peak stage, and the AE energy increased when the instantaneous strength change occurred after the peak stage. The AE energy fluctuated significantly when the stress was close to the peak strength. The stress-strain curves of pyrite-bearing filling joints samples were ladder-shaped after the peak and the AE energy of each drop appeared a peak. Nevertheless, the acoustic emission information gradually decreased with the decrease of stress. When the surface of the sample was distribute with many inclined and slender pyrite-bearing filling joint, the stress drop was obvious.

3) The fracture surface of relatively complete samples were mainly in the form of splitting failure, and the fracture surface was uneven. When the surface of the sample was distributed with fine pyrite-bearing filling joint, the macro-cracks were less distributed. And the SEM images showed that the fracture surface was relatively flat. Moreover, the macro-cracks were more and the deformation of the sample was relatively large when the surface of the sample was distributed with many inclined and slender pyrite-bearing filling joint. And the SEM pictures showed that the fracture surface of sample was distributed with more holes and cracks.

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