EFFECT OF LOW TEMPERATURE DAMAGE ON TENSILE STRENGTH OF COAL UNDER THE LIQUID NITROGEN FREEZING

by

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To explore the influence of low temperature damage on the tensile strength of coal under the liquid nitrogen freezing, the Brazilian splitting tests are conducted on the bedding coal in this paper. The tensile strength, macro fracture surface morphology, failure path and micro-structures of the coal samples before and after liquid nitrogen treatment were analyzed systematically. The results show that the tensile strength of coal after liquid nitrogen freezing is reduced, the macro fracture surfaces with greater roughness or fractal dimension were induced in the coal treated by liquid nitrogen freezing during the Brazilian splitting tests, the opening and number of the failure paths of coal after liquid nitrogen freezing increase significantly, and the microscopically, the bedding and matrix structures of coal are destroyed due to the temperature stress caused by large temperature difference between the liquid nitrogen and coal matrix.

Key words: liquid nitrogen, roughness, fractal dimension, coal, tensile strength

Introduction

The coal bed methane (CBM) is regarded as a plentiful and environment friendly energy resource, which has attracted a great attention around the world. Its efficient extraction and utilization can not only reduce the gas outburst disaster, but also improve the energy structure [1]. At present, hydraulic fracturing is a common method in increasing the production of CBM [2]. However, hydraulic fracturing has caused many problems, such as the environmental pollution, waste of water resources and clay swelling in the gas reservoir [3-8]. Liquid nitrogen (LN₂) fracturing, as a waterless fracturing technology, can overcome the above problems. Besides, LN₂ with -196 °C low temperature, the 696 times expansion capacity under gasification and 207 MPa frost-heave stress under normal temperature and pressure is beneficial to fracturing the gas reservoir [9]. Among them, the ultra-low temperature effect of LN₂ on rocks cannot be ignored, which can reduce the temperature around reservoirs leading to the open of natural fractures or the generation of new fractures [10].

So far, many scholars have focused research on the low temperature damage of rock under the LN_2 freezing. Cha *et al.* [11] investigated the feasibility of fracture stimulation by

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using LN₂ based on the fracturing experiment conducted on the concrete block. The results showed that LN₂ can cause a local tensile stress in the rocks surrounding a borehole due to a strong thermal gradient, which can make the rock skeleton shrink sharply leading to the initiation and propagation of crack. McDaniel et al. [12] studied the propagation behaviors of hydraulic fracture induced by injecting LN_2 , and discovered that many thermal microfractures were created orthogonal to the hydraulic cracks as a result of the huge temperature difference caused by LN₂. Cai et al. [13] investigated the effect of low temperature caused by LN₂ freezing on the porosity, mechanical strength and permeability of coal with water content by using the nuclear magnetic resonance method. Tran et al. [14] studied the initiation and propagation of secondary cracks due to the thermal stresses caused by LN₂. Guo [15] investigated the laws of the crack growth and permeability evolution of coal under the LN_2 freezing. Qin *et al.* [16] investigated the effects of LN₂ freezing on mechanical and physical properties of coal. The results showed that LN₂ freezing has an obvious weakening effect on the wave velocity and compressive strength of coal. However, there are few reports on the effect of LN₂ freezing on the tensile strength and fracture degree of coal. As we known, the tensile strength and fracture degree of coal are so important to the fracturing scheme optimization and productivity evaluation in CBM extraction engineering that it is necessary to study the effect of LN_2 freezing on the tensile strength and failure characteristics of coal in depth.

The main target of this paper is to show that by the Brazilian splitting tests, the fracture surface scanning tests and the SEM tests are conducted on pre- and post-treated coal samples by the LN_2 freezing. The changes of the tensile strength, the fracture surface roughness, the failure path and microstructure characteristics of the coal samples are analyzed before and after LN_2 treatment, and the influence mechanism of the LN_2 freezing on the failure characteristics of coal is discussed in details.

Experimental material and procedures

Experimental material

The coal samples used in this paper were collected from Yulin coal mine, Shanxi Province, China, which are of obvious bedding. They were firstly cored parallel to the coal bedding direction into the cylinders with the diameter in 50 mm and then processed into the Brazilian disc samples (BDS) with the thickness in 25 mm by cutting and polishing. The BDS were divided into four groups with the serial numbers of V, VC, P, and PC. There were tree specimens for each group. Among them, the groups V and P were the control groups without LN_2 freezing, the groups VC and PC were the test groups subjected to the LN_2 treatment. It is worth noting that the groups V and VC were set to carried out the Brazilian splitting tests by a load vertical to the bedding direction of coal, while the groups P and group PC were set to carried out that by a load parallel to the bedding direction of coal. In addition, all samples were dried at 60 °C for 6 hours before the tests, and the test samples after freezing for 1 hour by LN_2 were stored a sealed bag to recover to the room temperature (25 °C).

Experimental procedures

The whole experimental procedures is as follows:

- Firstly, the microstructures of coal before and after LN₂ treatment were obtained by a high vacuum SEM (FEI Quanta TM 250).
- The Brazilian splitting tests were carried out by using a universal testing machine (CSS-88020) with a loading rate of 0.05 mm/min.
- Thirdly, the failure paths of all specimens were recorded by a camera.

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 Finally, the fracture surface morphology of all specimens was scanned by a 3-D Profilometer (VR-5000) produced by KEYENCE, and a series of 3-D planes with a length in 24 mm and a width in 18 mm were captured from the fracture surface at 12 times magnification.

Results

Changes in the tensile strength of coal

Figure 1(a) shows the load-displacement curves of coal before and after LN_2 treatment under Brazilian splitting tests. Obviously, compared with the results of control groups, the load-displacement curves of coal treated by LN_2 freezing become rougher. A longer compaction stage and more obvious yield stage were found on the load-displacement curves of coal after LN_2 treatment under the condition of the loading vertical to the bedding direction, and the stress relaxation was shown in the elastic stage of the load-displacement curves of coal after LN_2 treatment under the condition of the loading parallel to the bedding direction. In addition, the peak loads of the treated samples decrease significantly while the corresponding peak displacements increase obviously. Further, the histogram of coal tensile strength based on the load-displacement curves is shown in fig. 1(b).



Figure 1. (a) The load-displacement curves of the BDS before and after the LN_2 treatment (b) the tensile strength of BDS before and after the LN_2 treatment

The tensile strength of coal decreases after the LN_2 treatment. At the same time, it can be found that the decrease rate of the tensile strength under the parallel bedding loading is obviously larger than that under the vertical bedding loading. In detail, they are 23.23% and 14.68%, respectively. The reason for the above results may be that the coal bedding interface with weak cementation strength is easier than the coal matrix to be weaken or damaged by the LN_2 freezing, which leads to the tensile failure of coal is easier to occur when the loading direction is parallel to the coal bedding.

Fracture surface morphology

Figure 2 shows the fracture surface morphology of coal under the Brazilian splitting tests. In order to quantitatively characterize the roughness of the fracture surface, five roughness parameters were analyzed, *i. e.* the average value of absolute height of each point in the



Figure 2. The 3-D profile scanning of fracture surface of BDS before and after the $LN_{\rm 2}$ treatment

measured area, S_a , the root mean square of height, S_q , the sum of maximum peak and maximum valley depth, S_z , the number of peak vertices per unit area, *Spd*, and the fractal dimension, *D*, of fracture surface [17]. Among them, the S_a is calculated:

$$S_a = \frac{1}{A} \iint_A |z(x, y)| dx dy$$
(1)

where x and y represent the plane projection co-ordinates of measuring points in the fracture surface, z(x, y) is the height function of the measuring points and A is the projected area of the measurement area. The paremater S_q is calculated:

$$S_q = \sqrt{\frac{1}{A} \iint_A z^2(x, y) \,\mathrm{d}x \,\mathrm{d}y} \tag{2}$$

The paremater S_z is calculated:

$$S_z = S_p + S_v \tag{3}$$

where S_v and S_p represent the absolute height of the lowest point and the height of the highest point in the defined area, respectively. The fractal dimension D is calculated by the box-counting method, given [18]:

$$D = -\lim_{\delta \to 0} \frac{\ln N_{\delta}(\Sigma)}{\ln \delta}$$
(4)

where δ is the side length of a cube box and $N(\delta)$ is the number of the cube boxes.

The calculated results for the roughness parameters of fracture surface are shown in fig. 3. It can be found that, compared with the untreated coal samples, the average S_a , S_q , S_z , and *Spd* of BDS after LN₂ treatment increased by 69.2%, 100.5%, 69.7%, and 15.1% under the loading vertical to the coal bedding while that increased by 34.8%, 36.8%, 39.3%, and 22.8% under the loading parallel to the coal bedding. In addition, the average *D* of the fracture surface of BDS increased from 2.157 to 2.189 under the loading vertical to the coal bedding while that increased from 2.133 to 2.139 under the loading parallel to the coal bedding.

Typically, the fracture surface characteristics of the failure coal samples can reflect the damage degree in their internal structures. For the coal samples with serious damage in their





Figure 3. The roughness and fractal dimension of fracture surface of BDS before and after the $\rm LN_2$ treatment

internal structures, more complex fracture surfaces will be induced in the Brazilian splitting experiments. The reason is that the more serious damage in the internal structure of coal, the smaller the cohesion between the coal particles, which increases the probability of micro-crack propagation randomly, and leads to more complex macro fracture surface finally. Therefore, the increased roughness or fractal dimension of the fracture surface on the coal samples treated by LN_2 under the Brazilian splitting experiments indicates that the LN_2 freezing can improve the low temperature damage to the coal, which may be beneficial for increasing the destruction degree of coal.

Failure paths

Figure 4 shows the crack propagation paths of coal under the Brazilian splitting tests. It can be seen that due to the LN_2 freezing action, the propagation paths of the induced cracks became more complex and multi branched when the BDS are destroyed in Brazilian splitting experiment under whether the loading vertical to the bedding or parallel to the bedding. Concretely, under the loading vertical to the bedding, the main cracks on the BDS treated by LN_2 were wider and the multi branch cracks extended from them. Under the loading parallel to the bedding, multiple main cracks were observed on in the BDS treated by LN_2 , and they propagated along the bedding plane. Simultaneously, they were connected



Figure 4. The failure paths of BDS before and after the LN₂ treatment

with each other by the transverse branch paths. The above results indicate that there is much damage on the micro-structures of coal by LN_2 freezing, which results in significant effect on the propagation paths of the induced crack. Therefore, it can be inferred that the LN_2 may be beneficial for improving the failure paths of coal and increasing their permeability in the CBM exploitation.

Discussions

According to the experimental results in this paper, the LN₂ has a significant influence on the tensile strength, fracture surface morphology and the propagation paths of induced crack of coal under the Brazilian splitting tests. Essentially, the internal factor affecting the aforementioned properties coal under the LN₂ treatment is related to the damage process of internal structure of coal. On one hand, the matrix and pores of coal under the LN_2 freezing would shrink severely resulting in the thermal tensile stress. When the thermal tensile stress exceeds the tensile limit of coal, the matrix and some pores of coal would be damaged to generate the micro-cracks. On the other hand, the strong bonding in the bedding interface of original coal is weaken due to the low temperature effect of LN2 freezing, which also leads to generate the micro-cracks. As shown in figs. 5A1 and 5A2, the original pores, micro-cracks and matrix in coal are without development. However, the density, length and width of the micro fracture in the coal after LN_2 freezing are improved significantly, figs. 5B1 and 5B2. It is worth noting that the micro-cracks mainly propagate along the bedding direction of coal and rarely extend into the matrix, fig. 5 B1, due to the larger tensile limit of coal matrix than coal bedding. This is in agreement with the aforementioned experiment results that the coal samples treated by LN₂ are easier to be destroyed under the loading parallel to coal bedding. Further, the induced micro-cracks by LN₂ freezing at a high magnification can be clearly observed in the coal matrix, fig. 5B2. In conclusion, the LN₂ treatment can effectively stimulate the damage of coal, improve the fracture development and weakening the tensile strength of coal.



Figure 5. The SEM images of coal before and after the LN_2 treatment; A1 and A2 represent the SEM images of original coal, B1 and B2 represent the SEM images of coal treated by LN_2

Conclusion

In the present work, the load-displacement curves of coal specimens treated by LN_2 are more tortuous under the Brazilian splitting tests. The LN_2 freezing has obvious weaken effect on the tensile strength of coal. In detail, the tensile strength of coal samples under the loading vertical and parallel to the coal bedding direction decreased by 14.68% and 23.23%, respectively. After LN_2 treatment, more complex fractures are easier to be induced in the coal samples under the Brazilian splitting tests. The induced fracture surface is of larger roughness

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and fractal dimension. In addition, the opening and density of the failure paths are also larger. The LN_2 freezing may cause the thermal stress resulting in the damage on the microstructures of coal. The SEM results show that multi microcracks are developed from the matrix, original pores and microcracks of coal under the LN_2 freezing. It indicates that the LN_2 treatment may improve the permeability of coal seam.

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Nomenclature

D	 fractal dimension, [-] 	S_{q}	- root mean square of height, [µm]
S_{a}	 roughness parameter, [um] 	S_{-}^{2}	 roughness parameter, [um]

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