Petroleum Science 20 (2023) 407–423

Contents lists available at ScienceDirect

Petroleum Science

journal homepage: www.keaipublishing.com/en/journals/petroleum-science

Original Paper

Rock mechanical properties of coal in cryogenic condition

Hai-Tao Wen, Rui-Yue Yang*, Mei-Yang Jing, Zhong-Wei Huang**, Chun-Yang Hong, Jian-Xiang Chen, Ri-Chao Cong

State Key Laboratory of Petroleum Resources and Prospecting, China University of Petroleum (Beijing), Beijing, 102249, China

ARTICLE INFO

Article history:
Received 25 March 2022
Received in revised form 27 November 2022
Accepted 29 November 2022
Available online 1 December 2022

Edited by Yan-Hua Sun

Keywords:
Liquid nitrogen freezing
Mechanical properties
Damage and strengthening
Coal

ABSTRACT

Liquid nitrogen (LN_2) fracturing is a kind of non-aqueous fracturing technology, which is expected to provide a new and efficient way for coalbed methane (CBM) development. The mechanical properties of coal under LN_2 freezing are very important for studying the mechanism of LN_2 fracturing. However, most of the current research is limited to studying mechanical properties of rocks after being frozen by LN_2 and returned to room temperature. In this paper, the effect of LN_2 freezing on the mechanical properties of coal was studied. Uniaxial strength tests and Brazil tests were carried out for dry and water-saturated coal samples with different types and bedding directions. In addition, standard electron microscopy (standard SEM) and cryo-electron microscopy (Cryo-SEM) were used to compare the fracture morphology of coal samples at room temperature and LN_2 temperature. The results showed that LN_2 freezing can damage and improve the mechanical properties of coal simultaneously. The strength of saturated coal under freezing is higher than that of dry coal, and the filling of ice can enhance the mechanical strength of coal. In addition, the mechanical properties of coal with higher porosity are enhanced more than that of coal with lower porosity under LN_2 freezing. The main findings of this study are the keys to the research of LN_2 fracturing mechanisms in CBM reservoirs.

1. Introduction

LN_2 fracturing is a non-aqueous fracturing technology, which has been successfully applied in CBM stimulation (McDaniel et al., 1997). Compared with hydraulic fracturing, LN_2 fracturing will not cause clay swelling, formation damage, waste of water resources, or groundwater pollution (Wen et al., 2020). And it is more likely to form complex fracture networks. It is a non-aqueous, formation damage-free, environmentally friendly CBM stimulation technology (Huang et al., 2020; Shao et al., 2021; Wen et al., 2020; Yan et al., 2020). In addition, the temperature of LN_2 is extremely low (−195.8 °C). The extremely high thermal stress can be formed in reservoir rock when it contacts with the LN_2. This thermal stress can cause significant damage to the rock, which can effectively improve the permeability of the reservoir and improve the fracturing effect (Yang et al., 2021).

In 1997, McDaniel et al. conducted the first field test of LN_2 fracturing (McDaniel et al., 1997). Four CBM wells and one tight gas well were fractured using LN_2 as fracturing fluid. The stimulation effect is significant in the early stage after LN_2 fracturing, and the daily production after fracturing is 1.22–6.48 times that before fracturing (McDaniel et al., 1997). In 1998, LN_2 was used as fracturing fluid in the Devonian shale, and the gas production was 8% higher than the offset well that had been completed with nitrogen gas fracturing (Grundmann et al., 1998). Since then, cryogenic fracturing with LN_2 in different rocks has been studied extensively.

Cryogenic fracturing with LN_2 can effectively generate fractures in shale, sandstone, coal, and geothermal reservoir rocks (Hong et al., 2022; Huang et al., 2020; Wang et al., 2016; Wen et al., 2022; Yang et al., 2019; Zhao et al., 2022). Due to the high thermal stress, the internal stress field of the rock will be disturbed during the fracturing, complex fractures will be generated after fracturing (Cha et al., 2018; Yang et al., 2021). In addition, a thermally cracked region around the borehole will be formed (Zhao et al., 2022). As a result, permeability enhancement can be achieved after LN_2 stimulation (Cha et al., 2018), and the breakdown pressure of rock after cryogenic stimulation can be significantly reduced (Wang et al., 2016; Yang et al., 2021). Cryogenic fracturing

* Corresponding author.
** Corresponding author.
E-mail addresses: yangruiyue@cup.edu.cn (R.-Y. Yang), huangzw@cup.edu.cn (Z.-W. Huang).

https://doi.org/10.1016/j.petsci.2022.11.025
1995-8226/

using LN_2 also can be combined with other fracturing technologies to cost-effectively enhance the production of unconventional reservoirs (Wang et al., 2016).

Besides, LN_2 cryogenic stimulation can considerably enhance the permeability of water-bearing coal (Zhao et al., 2022). And repeated stimulations can further increase the permeability (Yao et al., 2017). The circulation of LN_2 in wellbore at low pressure can be applied as a near-wellbore formation permeability enhancement technique with low cost (Yao et al., 2017). Based on the effect of LN_2 cryogenic stimulation in water-bearing coal, Qin et al. proposed a method for extracting CBM through increasing the permeability of the coal seams using the freeze-thaw cycle of LN_2 (Qin et al., 2016. 2018a). Under the effect of frost heaving force by water phase change, gasification swelling force by N_2, and the damage caused by low-temperature LN_2, macro and micro fractures are expanded and connected, forming fracture network and increasing the permeability of coal seam (Qin et al., 2017, 2018b, 2020, 2022).

To further study the mechanism of LN_2 fracturing, rock physical properties and morphologies including porosity, permeability, and cracks after LN_2 stimulation are researched. Research on low-temperature LN_2 damaging the coal is mainly through soaking the coal into LN_2 to observe the change after the freeze-thaw (Akhondzadeh et al., 2020; Cai et al., 2018; Hou et al., 2022b; Qin et al., 2017; Shao et al., 2021). These studies show that the LN_2 has a significant degradation ability for low permeability rocks. The porosity, fracture volume, fracture aperture, and fracture connectivity increase with the extension of the LN_2 quenching period (Qin et al., 2017; Yan et al., 2020). LN_2 freezing not only causes thermal fractures on the coal surface, but also causes fractures along the macroscopic fractures on the surface of the sample (Cai et al., 2015). After freezing with LN_2, new cleat network was formed in the coal, which connected the originally isolated pores and microcleats, thus increasing the connectivity of the pore network (Akhondzadeh et al., 2020; Su et al., 2020). The damage of saturated coal is higher than that of dry coal, and the damage fractures of dry coal occur more on the surface of coal than in the interior (Du et al., 2020; Shao et al., 2021). The strength of coal is reduced after being frozen by LN_2. And the decreasing degree of coal strength is positively correlated with the number of freezing-thawing cycles (Qin et al., 2017; Zhao et al., 2018).

The above studies have proved that LN_2 has significant damage to coal. However, they are limited to studying the physical properties of coal samples after being frozen by LN_2 and then restored to room temperature. Through these studies, the changes in reservoir physical properties after freezing and restoring formation temperature during LN_2 fracturing can be obtained. However, during the LN_2 fracturing, the rock around the primary fracture will continue to be frozen with the injection of LN_2 (Wen et al., 2022), and the frozen rock will be fractured to form secondary fractures (Zhao et al., 2022). The physical properties of the frozen rock are different from those of the rock after being frozen by LN_2 and then returned to the formation temperature. In addition, pre-cooling is required in LN_2 fracturing (Hong et al., 2022; Yang et al., 2021; Zhao et al., 2022). The pre-cooled rock is in frozen state, which physical properties are crucial to study the characteristics of LN_2 fracturing initiation. Therefore, to study the LN_2 fracturing mechanism, the mechanical properties of coal under LN_2 freezing should be studied first, but there are few reports at present. Hou et al. investigate the mechanical properties and fracture behaviors of coal under the frozen state (Hou et al., 2022a). However, the effects of different moisture conditions and bedding directions were not considered. The moisture conditions and bedding directions can strongly affect the failure strength and the failure pattern of coal (Hou et al., 2022a; Shao et al., 2021).

In this paper, dry and water-saturated coal with different bedding directions were selected for LN_2 treatment, and the strength of coal samples under room temperature and LN_2 freezing was tested and compared to obtain the influence of ultra-low temperature of LN_2 on mechanical properties of coal and to provide the theoretical and experimental basis for the study of LN_2 fracturing mechanism of CBM wells.

2. Method and materials

2.1. Material preparation

To make the results more convincing, two types of coals were selected for test. The one is from the Dabaodang Coal Mine in Shaanxi. The other is from Guangsheng Coal Mine in Inner Mongolia. The vitrinite reflectance (R_o) of the two coals is similar, but the porosity and permeability are quite different. The coal industry analysis of the coal used in the experiment is shown in Table 1. And the mineral composition analysis is shown in Table 2.

The coal was cut into two shapes: cylinders with a length of 50 mm and a diameter of 25 mm and discs with a thickness of 13 mm and a diameter of 25 mm, as shown in Fig. 1. Cylindrical samples were used for permeability and uniaxial compression tests, while disc samples were used for Brazil tests. The ends of these samples were grinded and polished to meet the standard suggested by ISRM (Bieniawski and Hawkes, 1978).

The coal samples were put into an air-ventilated oven for drying treatment. To avoid damage caused by high temperature, the drying temperature was set at 45 °C. The sample was taken out when the weight change is not more than 0.2% within 3 h and cooled to room temperature naturally. The P-wave velocity of dry coal samples was tested, and the coal samples with large difference in wave velocity were removed. Then, the coal samples were grouped according to wave velocity to reduce the dispersion of coal samples in physical properties. And the porosity and permeability of two types of coal samples were tested, which average values are shown in Table 3.

2.2. Experimental design

The coal samples used in the experiment are divided into two categories: Dabaodang coal samples and Guangsheng coal samples. Each category is divided into dry coal and water-saturated coal. The water-saturated coal samples were vacuum-saturated at room temperature and taken out when the rock weight change was no more than 0.2% within 24 h. The moisture content of Guangsheng coal under the saturated condition is 7.87%. And the moisture content of Dabaodang coal is 5.67%. In addition, according to the loading direction and bedding direction, the samples were divided into vertical bedding (VB) samples and parallel bedding (PB) samples, as shown in Fig. 2.

The compressive strength and tensile strength of each type of coal sample were tested using cylindrical and disc samples at room temperature (RT) and LN_2 temperature, respectively. The experimental process is shown in Fig. 3. Generally, three rock samples were used in each group. When the error of the three experimental

Table 1
Industry analysis of coal samples.

Core sample	FC_{ad}, %	M_{ad}, %	A_d, %	V_{daf}, %	R_o
Guangsheng	39.18	8.05	11.4	41.37	0.81
Dabaodang	40.14	8.16	13.1	38.6	0.90

Note: FC_{ad}: Fixed carbon (air-dried); M_{ad}: Moisture content (air-dried); A_d: Ash content (dried); V_{daf}: Volatile matter (dry-ash-free).

Table 2
XRD analysis of coal samples.

Core sample	Mineral composition, %					
	Quartz	Calcite	Barite	Siderite	Pyrite	Clay mineral
Guangsheng	3.5	84.6	3.3	/	/	8.6
Dabaodang	4.4	40.5	/	35.5	11.1	8.5

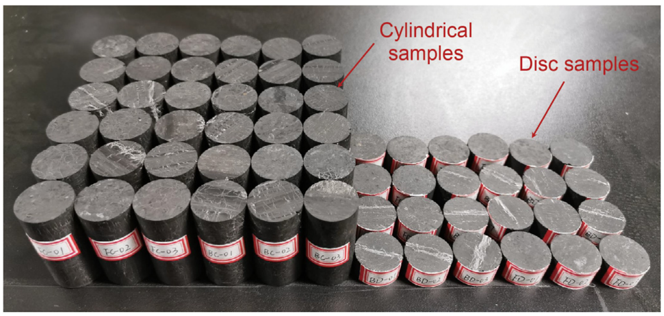


Fig. 1. Coal samples used in the experiment.

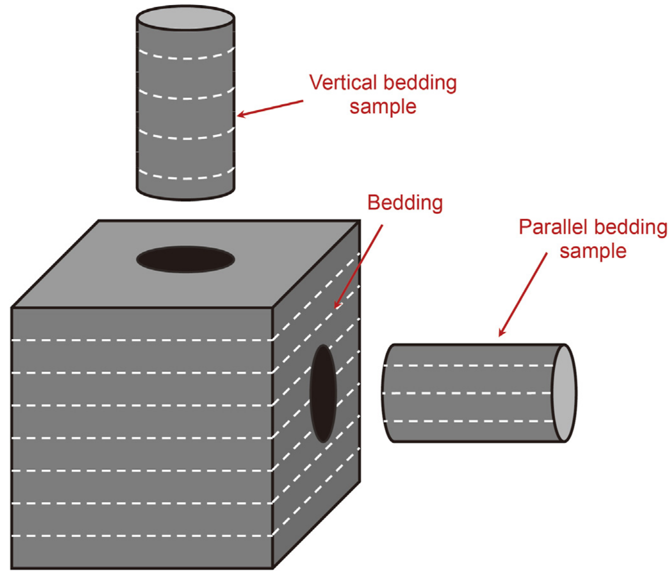


Fig. 2. Vertical bedding sample and parallel bedding sample.

results was large, six rock samples were used in the group. In this experiment, a total of 108 coal samples were used. The number of rock samples used in each group is shown in Table 4. The *z*-score method is adopted to remove the values with large differences, and then the average value is taken to represent the coal strength for a treatment method. The *z*-score is a statistical measurement of a value's relationship to the mean in a group of values. The *z*-score can be calculated by

$$z = \frac{x - m}{s} \tag{1}$$

where z is the value of the *z*-score; x is the raw score; m is the population mean; and s is the population standard deviation.

2.3. Instruments and test methods

The universal testing machine used in the experiment can test the strength of the material at room temperature and LN_2 temperature, as shown in Fig. 4. When testing the strength of coal at LN_2 temperature, the sample was completely immersed in LN_2 and frozen for 30 min before real-time freezing loading. The loading speed was 0.05 mm/min in the compressive strength test and 1 mm/min in the Brazil test. The Brazil test is a laboratory test conducted in rock mechanics to indirectly determine the tensile strength of rocks. The tensile strength of the coal samples is calculated by

$$\sigma_t = \frac{2P}{\pi D t} \tag{2}$$

where σ_t is the tensile strength of the specimen; P is the maximum loading; D is the diameter of the specimen; t is the width of the specimen.

In addition, standard SEM and Cryo-SEM were used. The former can observe the fracture morphology of rocks at room temperature, and the latter can observe the fracture morphology of rocks at LN_2 temperature. The testing methods can be found in the relevant literature (Yang et al., 2021). The combination of the two kinds of electron microscopy can observe and compare the changes of the same fracture at room temperature and LN_2 temperature.

3. Results and analysis

3.1. Effect of LN_2 freezing on the mechanical properties of dry rocks

The tensile strength of dry Dabaodang and Guangsheng coals at room temperature and LN_2 temperature are shown in Fig. 5. It can be found that the tensile strength of dry coal with different bedding directions under LN_2 freezing is lower than that at room temperature. This indicates that the LN_2 freezing can reduce the tensile strength of the dry coal.

The compressive strength of dry Dabaodang and Guangsheng coals at room temperature and LN_2 temperature are shown in Fig. 6. For dry coal with vertical bedding, the compressive strength of Dabaodang coal under LN_2 freezing is smaller than that under room temperature, while the Guangsheng coal under LN_2 freezing is larger than that under room temperature. For dry coal with parallel bedding, the compressive strength of Dabaodang and Guangsheng coals under LN_2 freezing is higher than that under room temperature. But the amplitude of Dabaodang coals is smaller than that of Guangsheng coals. The results indicate that LN_2 freezing can greatly enhance the compressive strength of dry Guangsheng coal, and can also enhance the compressive strength of dry Dabaodong coal in the parallel bedding direction, and reduce the compressive strength of dry Dabaodong coal in the vertical bedding direction. In

Table 3
Porosity and permeability of coal samples.

Coal sample	Porosity, %	Permeability, 10^{-3} μm^2	
		In vertical bedding direction	In parallel bedding direction
Guangsheng	17.20	0.1063	0.2759
Dabaodang	10.66	0.0547	0.1659

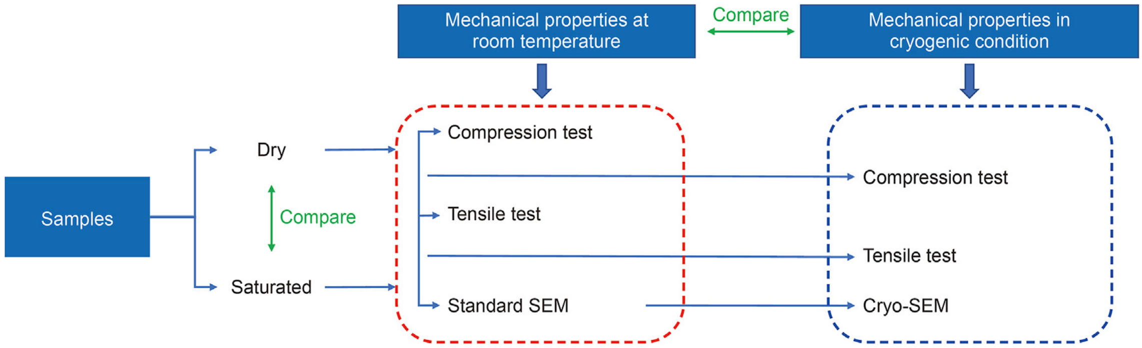


Fig. 3. Experimental process.

Table 4
Number of coal samples in each group.

Core sample shape	Group	Number of Guangsheng coal samples	Number of Dabaodang coal samples
Cylindrical	Dry-PB-RT	3	3
	Saturated-PB-RT	3	3
	Dry-PB-LN_2	6	3
	Saturated-PB-LN_2	6	3
	Dry-VB-RT	3	3
	Saturated-VB-RT	3	3
	Dry-VB-LN_2	6	3
	Saturated-VB-LN_2	6	3
Disc	Dry-PB-RT	3	3
	Saturated-PB-RT	3	3
	Dry-PB-LN_2	3	3
	Saturated-PB-LN_2	3	3
	Dry-VB-RT	3	3
	Saturated-VB-RT	3	3
	Dry-VB-LN_2	3	3
	Saturated-VB-LN_2	3	3

Note: * PB is parallel bedding samples, VB is vertical bedding samples, RT is room temperature.

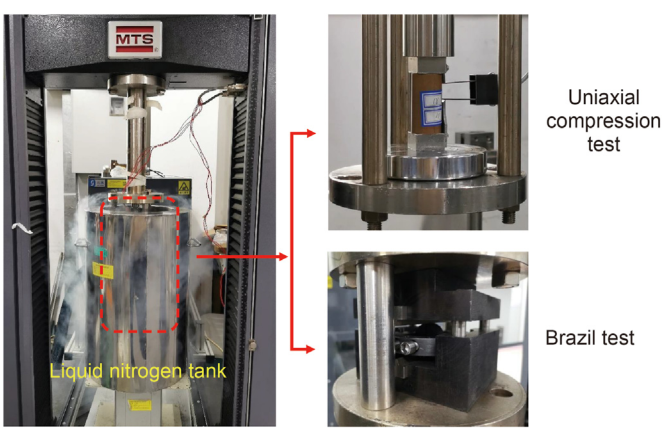


Fig. 4. Mechanical tests at room temperature and LN_2 temperature.

addition, the influence of LN_2 freezing on the compressive strength of Dabaodong coal is smaller than that of Guangsheng coal.

Elastic modulus can measure the ability of rock to resist deformation in the elastic deformation stage. For the tight rock, the pore and fracture volume are small, and the ability to resist deformation is strong, so the elastic modulus is large. On the contrary, rocks with higher porosity have smaller elastic modulus. The elastic modulus of dry Dabaodang and Guangsheng coals at room temperature and LN_2 temperature are shown in Fig. 7. It can be found that the elastic modulus of dry Dabaodang and Guangsheng coals with different bedding directions under freezing of LN_2 are significantly higher than those under room temperature. This indicates that the pores of rock shrink and the deformation resistance are enhanced under the ultra-low temperature of LN_2. The LN_2 freezing can greatly improve the elastic modulus of coal. In addition, the increased amplitude of elastic modulus in parallel bedding direction of Dabaodang and Guangsheng coals under LN_2 freezing is significantly higher than that in vertical bedding direction, indicating that LN_2 freezing has a greater impact on the elastic modulus of coals in parallel bedding direction, which significantly enhances the deformation resistance of coals in parallel bedding direction.

The brittleness index can measure the brittleness of rock and is an important parameter to evaluate the fracturing performance of the reservoir. The brittleness index model proposed by Tarasov and Randolph is adopted (Tarasov and Randolph, 2011), which can consider the post-peak energy balance of elastic and rupture energy

$$k = \frac{dW_r}{dW_e} = \frac{E - M}{M} \tag{3}$$

where k is the brittleness index of rock; dW_r is the post-peak rupture energy absorbed by the rupture process at stress degradation; dW_e is the elastic energy withdrawn from the specimen due to stress degradation; E is the elastic modulus before the peak value of rock stress-strain curve; and M is the post-peak modulus.

It can be seen from the formula that k is a negative value. In order to more conveniently compare the brittleness index of coal in different states using the coordinate diagram, K is used in this paper

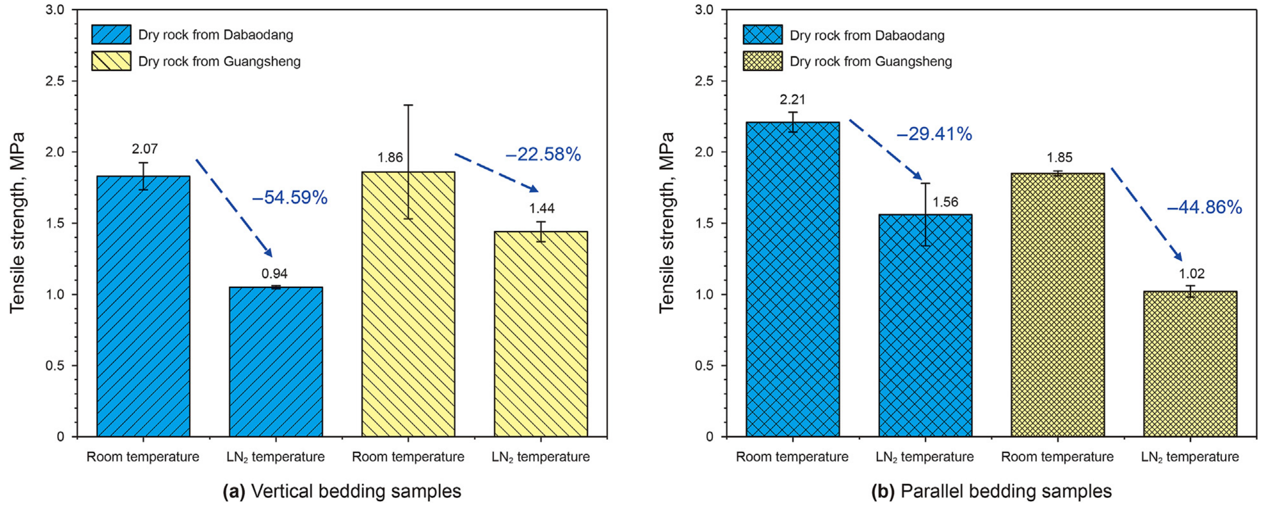


Fig. 5. Tensile strength of dry coal samples.

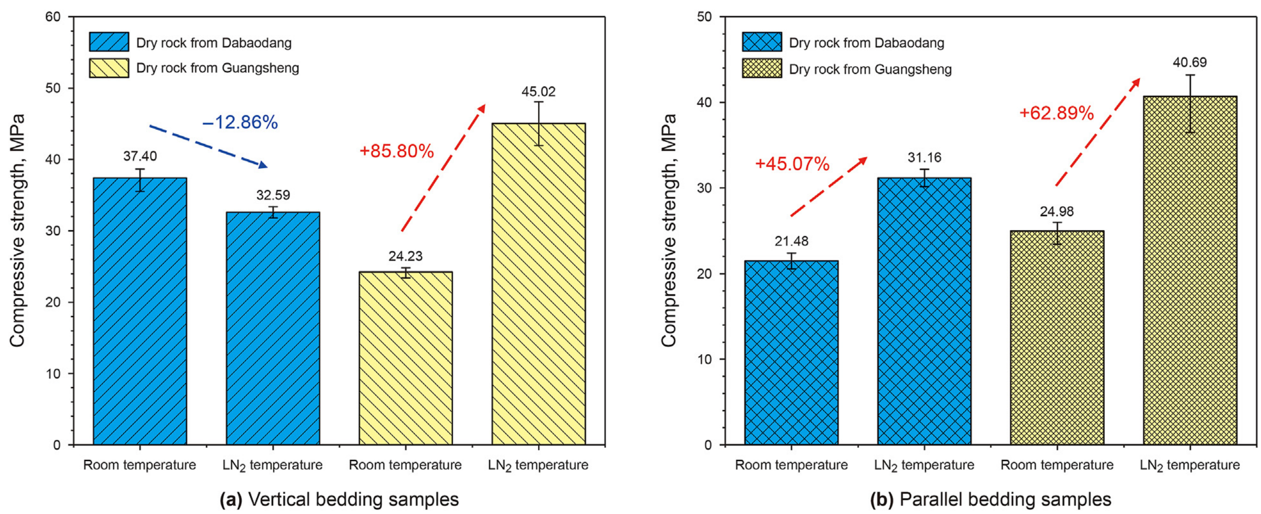


Fig. 6. Compressive strength of dry coal samples.

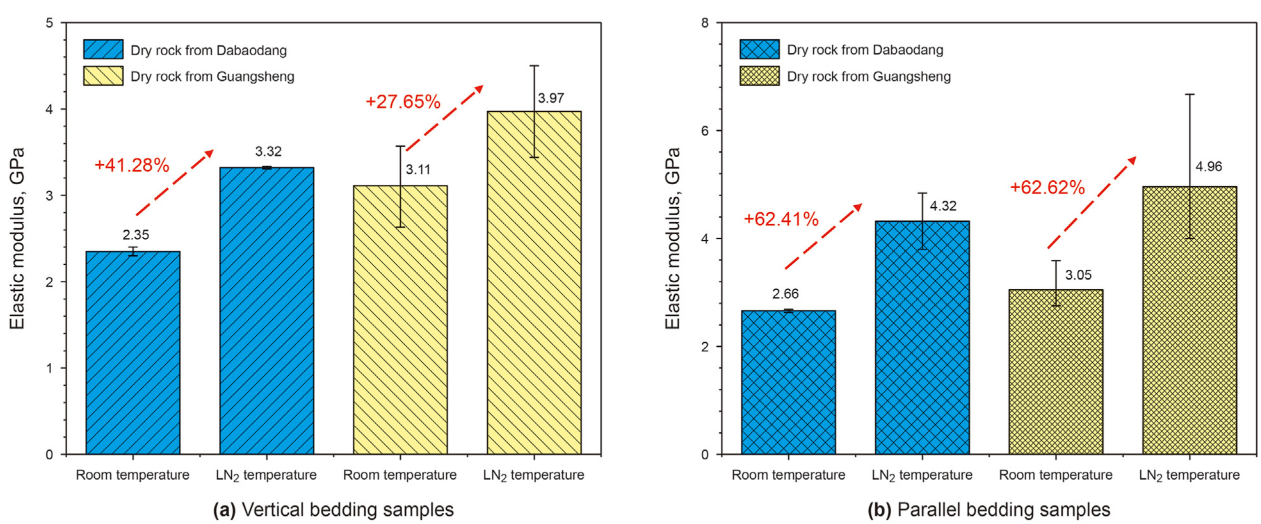


Fig. 7. Elastic modulus of dry coal samples.

to represent the brittleness index of rock

$$K = -k = \frac{M - E}{M} \tag{4}$$

The higher the value of K, the lower the brittleness of rock, and the higher the plasticity.

Fig. 8 shows the brittleness index of dry Dabaodang and Guangsheng coals under room temperature and LN_2 freezing. It can be found that the brittleness index of dry Dabaodang and Guangsheng coals with different bedding directions under LN_2 freezing is lower than that under room temperature obviously, indicating that LN_2 freezing can significantly improve the brittleness of dry coals.

The uniaxial compression loading curves of dry Dabaodang and Guangsheng coals under room temperature and LN_2 freezing are shown in Fig. 9. Coal generally goes through four stages of compaction, elasticity, plasticity, and failure during the uniaxial compression test. The Dabaodang coal with vertical bedding at room temperature can be taken as an example. O-A is the compaction stage, during which the micro-fractures in the axial direction of coal are gradually pressed and closed, and the curve presents a downward convex shape. A-B is the elasticity stage, during which the original micro-fractures and pores are completely closed. The coal matrix begins to undergo elastic deformation under uniaxial pressure, and the stress-strain curve increases linearly. B-C is the deformation stage, during which a large number of micro-fractures began to develop, expand and connect, and local weak areas began to break. When micro-fractures develop to a certain degree, sudden fracture failure occurs in the coal sample. The stress peak at point C is the uniaxial compressive strength of coal. The stress–strain curve is convex at this stage. C-D is the failure stage. In this stage, fractures rapidly run through to form macro fault surfaces. The friction and sliding between the fault surfaces make the coal still able to bear axial stress, but the bearing capacity decreases gradually.

It can be seen from the figure that the coal under LN_2 freezing does not have an obvious plastic stage before failure, and it rapidly fails after entering the plastic stage. In addition, the rock sample at room temperature still has supporting capacity due to the friction of shear planes in the failure stage, and its failure is not instantaneous. While the stress curve of the coal frozen by LN_2 drops sharply after entering the failure stage, showing strong brittle failure characteristics. It indicates that the internal structure of the coal is rapidly destroyed and loses its supporting ability.

Fig. 10 shows the failure patterns of part of Dabaodang and Guangsheng dry coals after uniaxial compression. It can be seen that the fragmentation degree of coal samples under LN_2 freezing is higher than that under room temperature. It indicates that a large number of fractures develop and run through during the failure stage. The break of coal under LN_2 freezing has obvious brittle failure characteristics.

Fig. 11 shows the failure patterns of part of Dabaodang and Guangsheng dry coals after the Brazil test. It can be seen that the number of fractures formed after splitting under LN_2 freezing is more than that under room temperature for Dabaodang coal. And the tortuosity degree of fractures formed after splitting under LN_2 freezing is higher than that under room temperature for Guangsheng coal. The results indicate that the coal under cryogenic freezing tends to form complex fractures after Brazil split test.

3.2. Effect of LN_2 freezing on the mechanical properties of water-saturated rocks

The tensile strength of water-saturated Dabaodang and Guangsheng coals at room temperature and LN_2 temperature are shown in Fig. 12. It can be found that the tensile strength of saturated Dabaodang coal under LN_2 freezing is lower than that under room temperature. While the Guangsheng coal under LN_2 freezing is higher than that under room temperature. The results indicate that the ultra-low temperature freezing of LN_2 degrades the tensile strength of Dabaodang coal and strengthens the tensile strength of Guangsheng coal.

The compressive strength of water-saturated Dabaodang and Guangsheng coals under room temperature and LN_2 temperature are shown in Fig. 13. It can be found that the compressive strength of the two water-saturated coals in different bedding directions under LN_2 freezing is higher than that under room temperature, which indicates that the cryogenic freezing of LN_2 can strengthen the compressive strength of saturated coal. In addition, the increase of compressive strength in the parallel bedding direction of water-saturated coal under LN_2 freezing is significantly higher than that in vertical bedding direction, indicating that LN_2 freezing has a greater influence on the compressive strength of water-saturated coal in parallel bedding direction. The increased amplitude of compressive strength of water-saturated Dabaodong coal under LN_2 freezing is less than that of Guangsheng coal, and the compressive strength of the latter is twice higher than that under room temperature. It

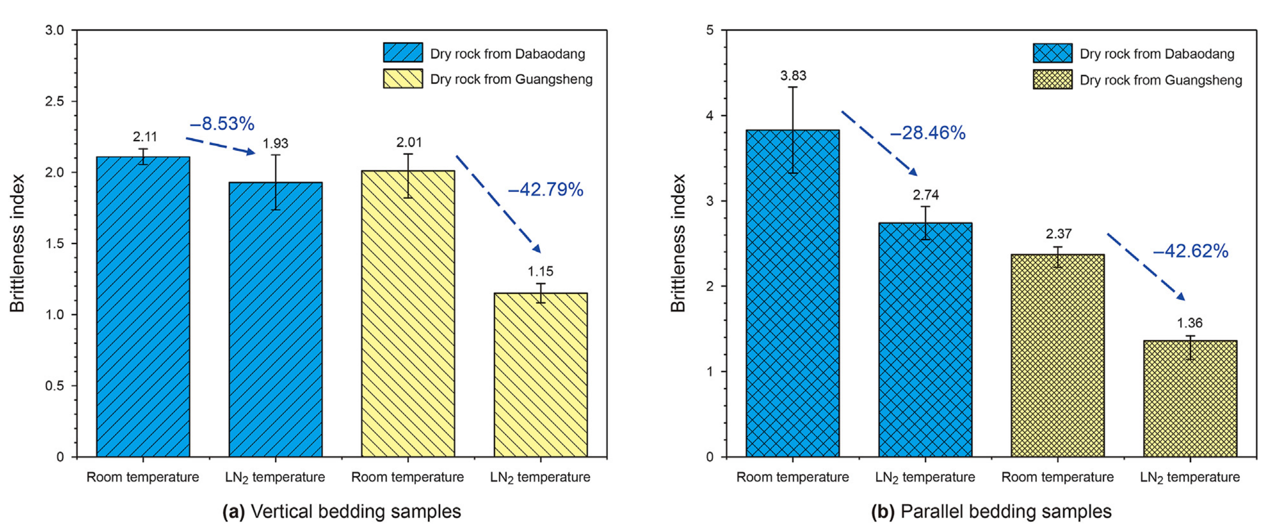


Fig. 8. Brittleness index of dry coal samples.

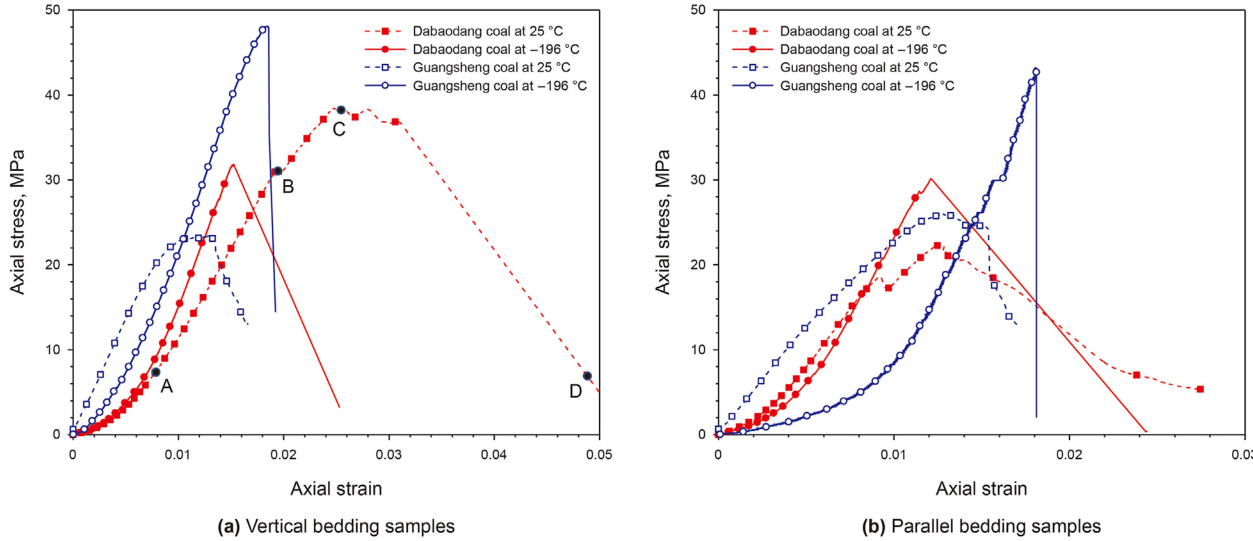


Fig. 9. Uniaxial compression loading curve of dry coal samples.

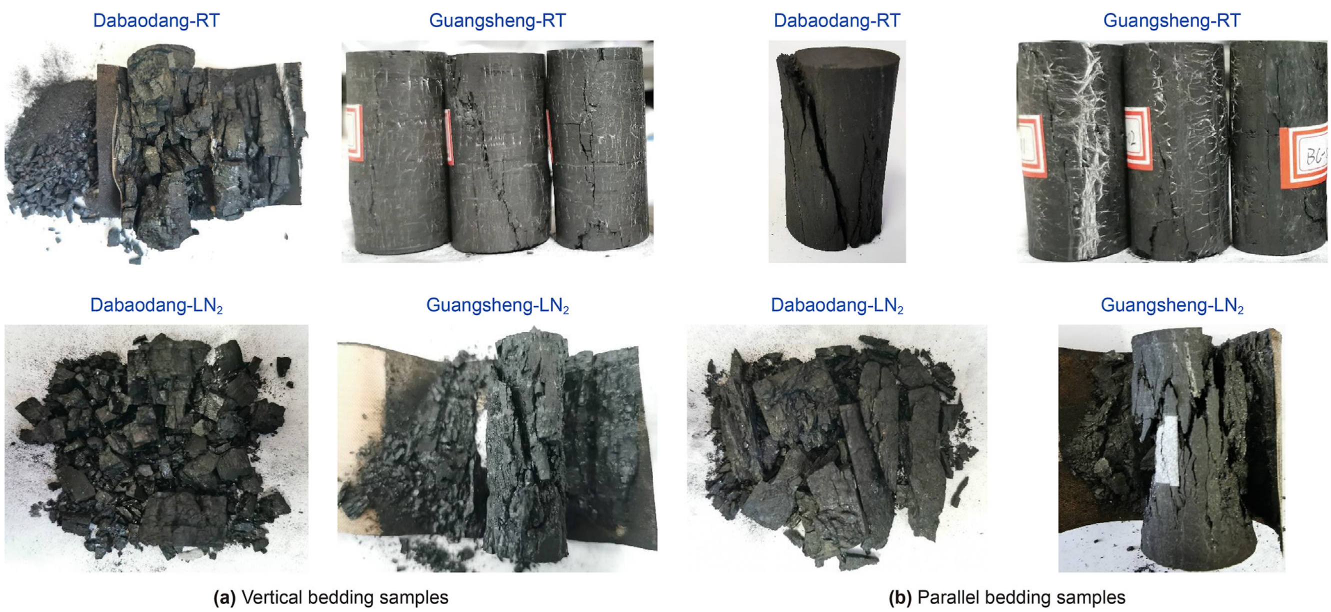


Fig. 10. Uniaxial compression failure pattern of dry coal samples.

indicates that LN_2 freezing has a more significant effect on strengthening the compressive strength of Guangsheng coal. In addition, compared with the dry coal in Fig. 6, the average compressive strength of water-saturated Dabaodang and Guangsheng coals under LN_2 freezing is increased by 27.48% and 35.35% respectively, indicating that pore water can significantly enhance the compressive strength of rock under freezing.

Fig. 14 shows the elastic modulus of Dabaodang and Guangsheng water-saturated coals at room temperature and LN_2 temperature. It can be found that the elastic modulus of Dabaodang and Guangsheng saturated coals with different bedding directions under freezing of LN_2 more than one time that at room temperature, which indicates that LN_2 freezing can greatly enhance the elastic modulus of saturated coal. In addition, compared with Fig. 7, the increased amplitude of elastic modulus of water-saturated coal under LN_2 freezing is significantly higher than that of dry coal, indicating that the filling of ice in the pores significantly improves the resistance to deformation of coal.

The brittleness index of water-saturated Dabaodang and Guangsheng coals under room temperature and LN_2 temperature are shown in Fig. 15. It can be seen that the brittleness index of water-saturated Dabaodang and Guangsheng coals with different bedding directions under LN_2 freezing are lower than that under room temperature, indicating that LN_2 freezing can significantly improve the brittleness of saturated coal. In addition, the decrease of brittleness index of saturated coal with vertical bedding is lower than that with parallel bedding under LN_2 freezing, indicating that the brittleness of saturated coal in vertical bedding direction is less affected by LN_2 freezing than that in parallel bedding direction.

The uniaxial compression loading curves of water-saturated Dabaodang and Guangsheng coals at room temperature and LN_2 temperature are shown in Fig. 16. It can be seen that, compared

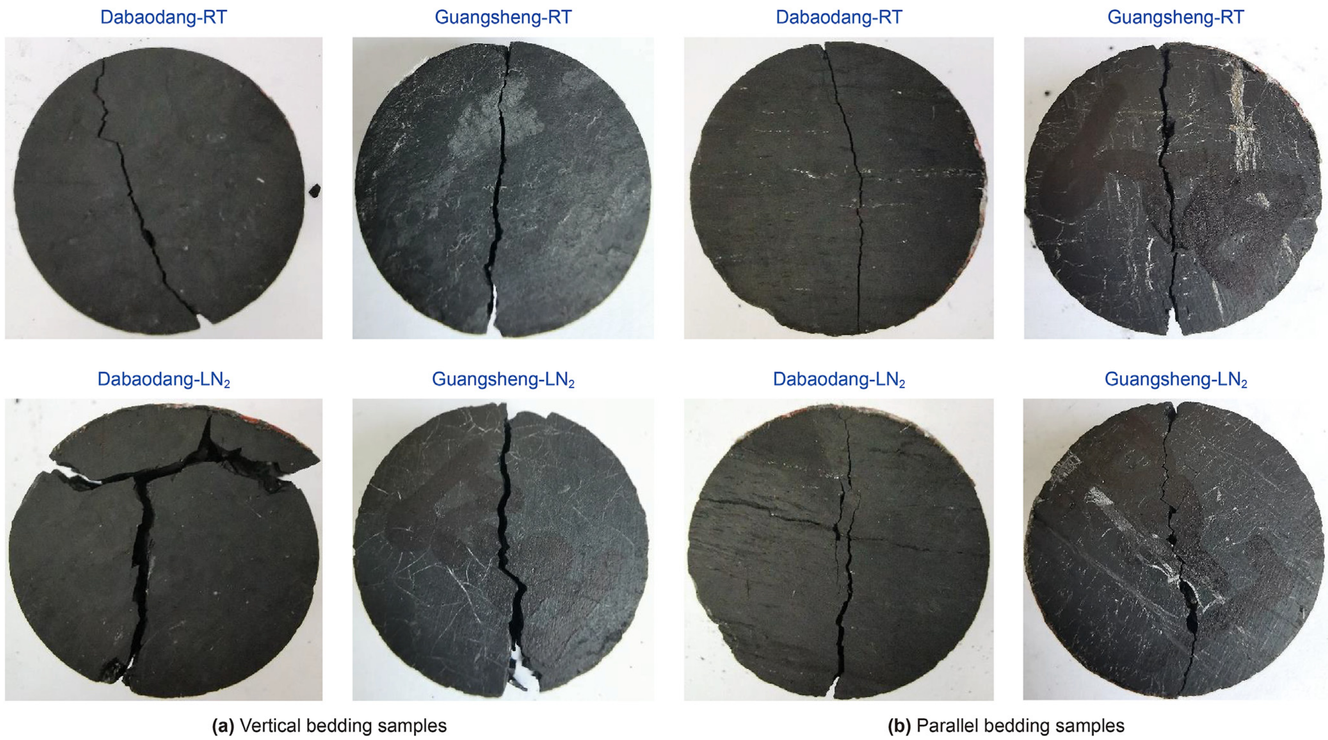


Fig. 11. Brazil split failure pattern of dry coal samples.

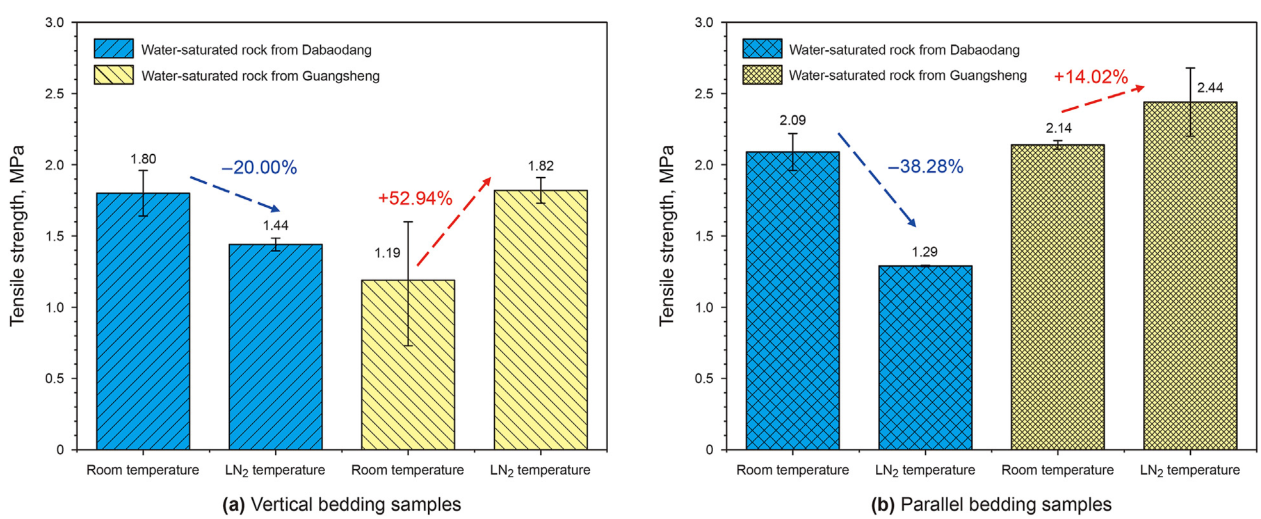


Fig. 12. Tensile strength of water-saturated coal samples.

with the coal at room temperature, there is no obvious plastic stage before the failure under LN_2 freezing, and the coal rapidly breaks after entering the plastic stage, which is the same as the loading characteristics of dry coal at LN_2 temperature. In addition, the stress curve drops sharply after entering the failure stage, showing strong brittle failure characteristic. Compared with Dabaodang coal, the stress curve of Guangsheng coal shows a nearly vertical downward trend after entering the failure stage under LN_2 freezing, indicating that the failure speed of Guangsheng coal is faster than Dabaodang coal.

Fig. 17 shows the failure patterns of part of Dabaodang and Guangsheng water-saturated coals after uniaxial compression. It can be seen that the crushing degree of coal samples in cryogenic condition is higher than that at room temperature, which is similar to dry coal. It indicates that a large number of fractures develop and run through in the failure stage, and the rock is broken with obvious brittle failure characteristics.

The failure patterns of part of Dabaodang and Guangsheng water-saturated coals after the Brazil test are shown in Fig. 18. The phenomenon is similar to dry coal. The number of fractures formed after splitting under LN_2 freezing is more than that under room temperature for Dabaodang coal. And the tortuosity degree of fractures formed after splitting under LN_2 freezing is higher than that under room temperature for Guangsheng coal. It indicates that the saturated coal under low temperature tends to form complex fractures after Brazil split test.

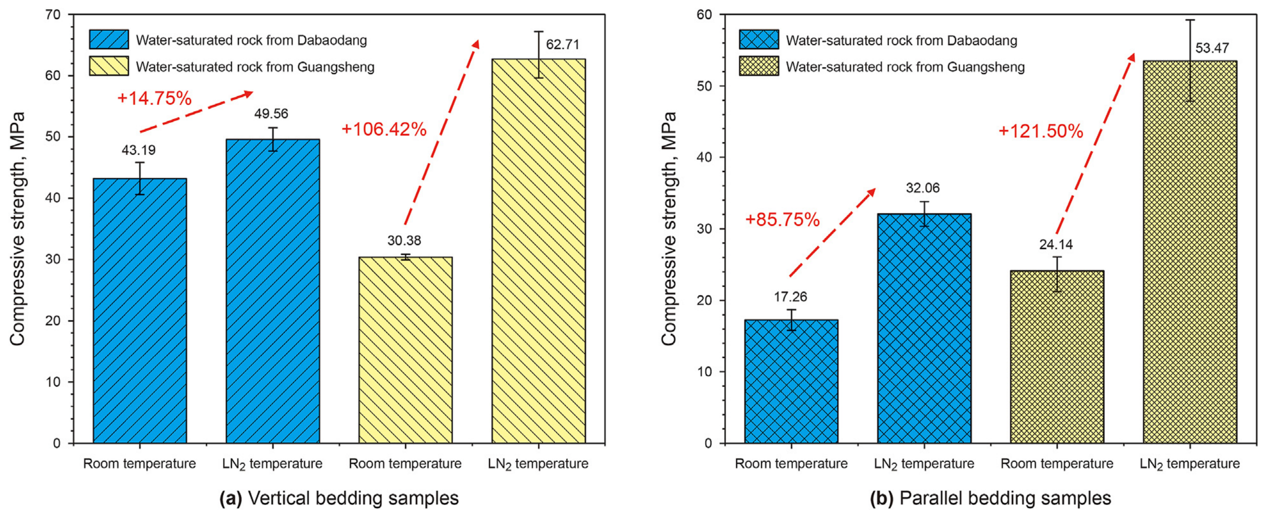


Fig. 13. Compressive strength of water-saturated coal samples.

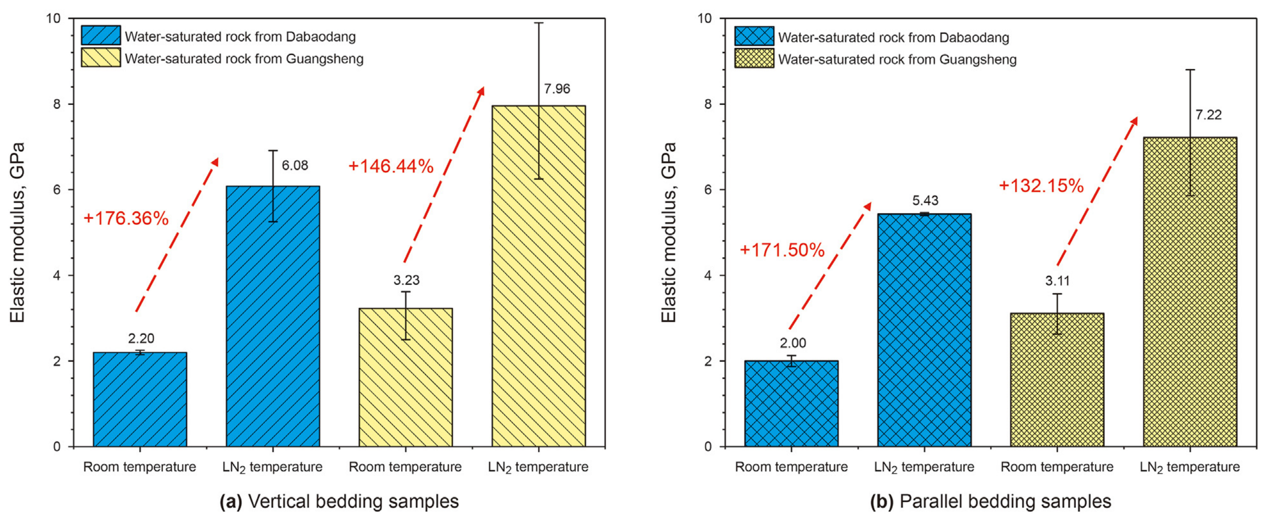


Fig. 14. Elastic modulus of water-saturated coal samples.

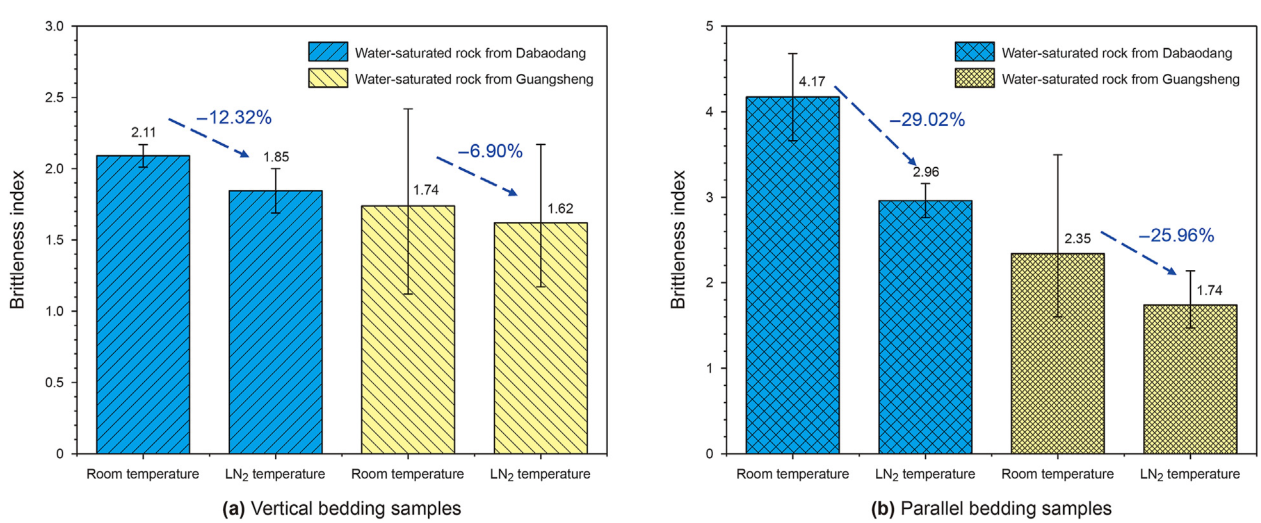


Fig. 15. Brittleness index of water-saturated coal samples.

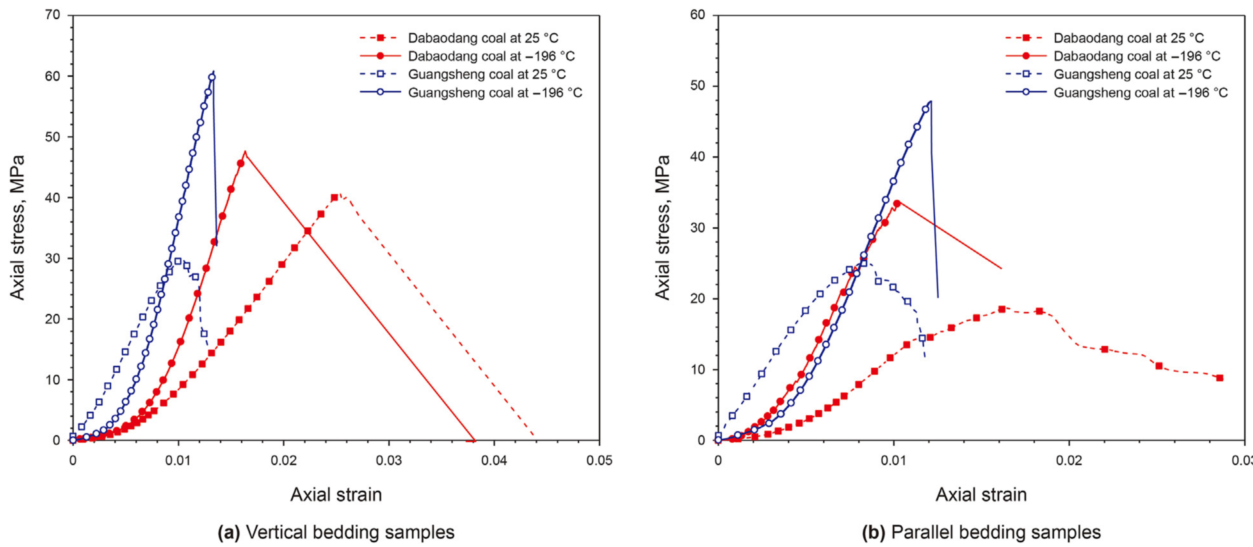


Fig. 16. Uniaxial compression loading curves of water-saturated coal samples.

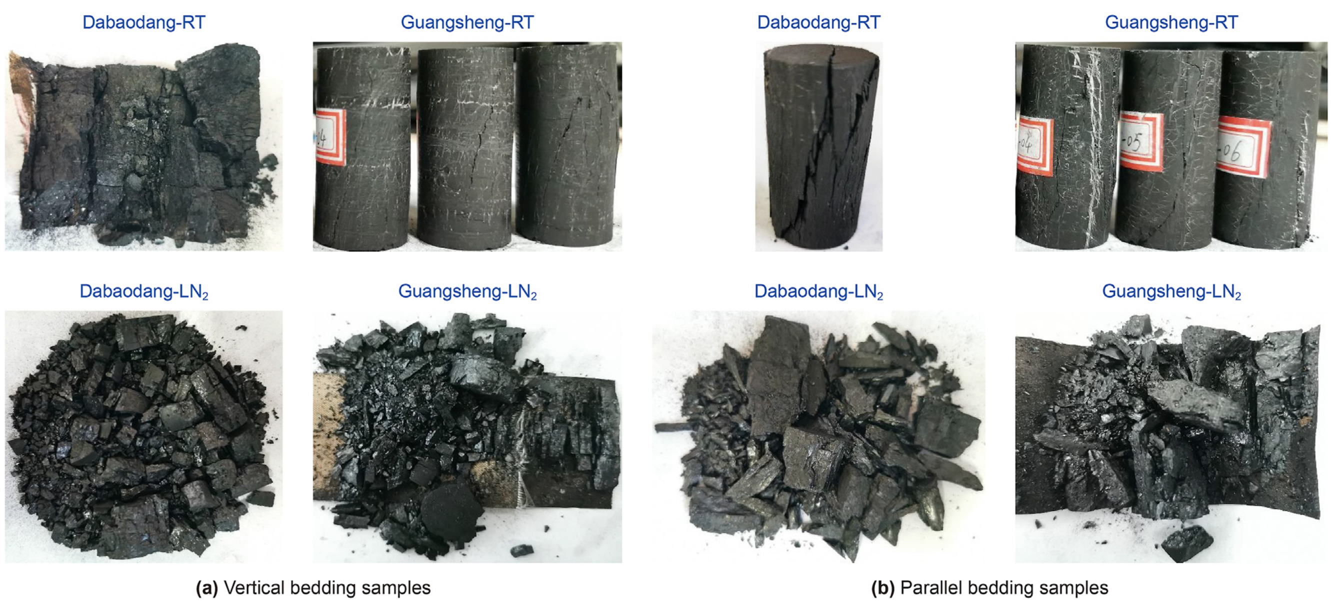


Fig. 17. Uniaxial compression failure pattern of water-saturated coal samples.

4. Discussion

4.1. Damage and strengthening of dry coal under freezing

The freezing of LN_2 can damage and strengthen coal simultaneously. On the one hand, the high thermal stress generated by the temperature difference will damage the coal and reduce the mechanical strength. On the other hand, the shrinkage of the rock at low temperature will increase the stiffness and the mechanical strength. For the same type of coal sample, the phenomenon of compressive strength increase and tensile strength decrease may occur under LN_2 freezing. The reason is that the stiffness of the rock increases, but thermal damage cracks are formed, and the effect of damage on the tensile strength is greater than that of strengthening, which is consistent with the results of previous studies by Hou et al. on the coal (Hou et al., 2022a). It is also possible that the tensile strength and compressive strength increase simultaneously, which is consistent with previous research results on sandstone, limestone, and granite (Mellor, 1971), indicating that the effect of damage on rock strength is less than that of strengthening. In addition, the mechanical parameters of different bedding directions may change inconsistently under LN_2 freezing. The reason is that the rock matrix shrinks and its mechanical properties are strengthened under LN_2 freezing, but the LN_2 freezing also weakens the cementation between the bedding interfaces and even promotes the growth of cracks due to inconsistent deformation between the coal matrix and weak layer (Hou et al., 2022a). All these lead to the inconsistent change of mechanical properties of the same type of coal. For different types of coal samples, the change of mechanical properties under LN_2 freezing is different. Different pore structures and mineral composition are the main reasons that affect the variation of mechanical properties of different types of coals. For rocks with high porosity, the strengthening effect caused by LN_2 freezing is higher than that of rocks with low porosity. And

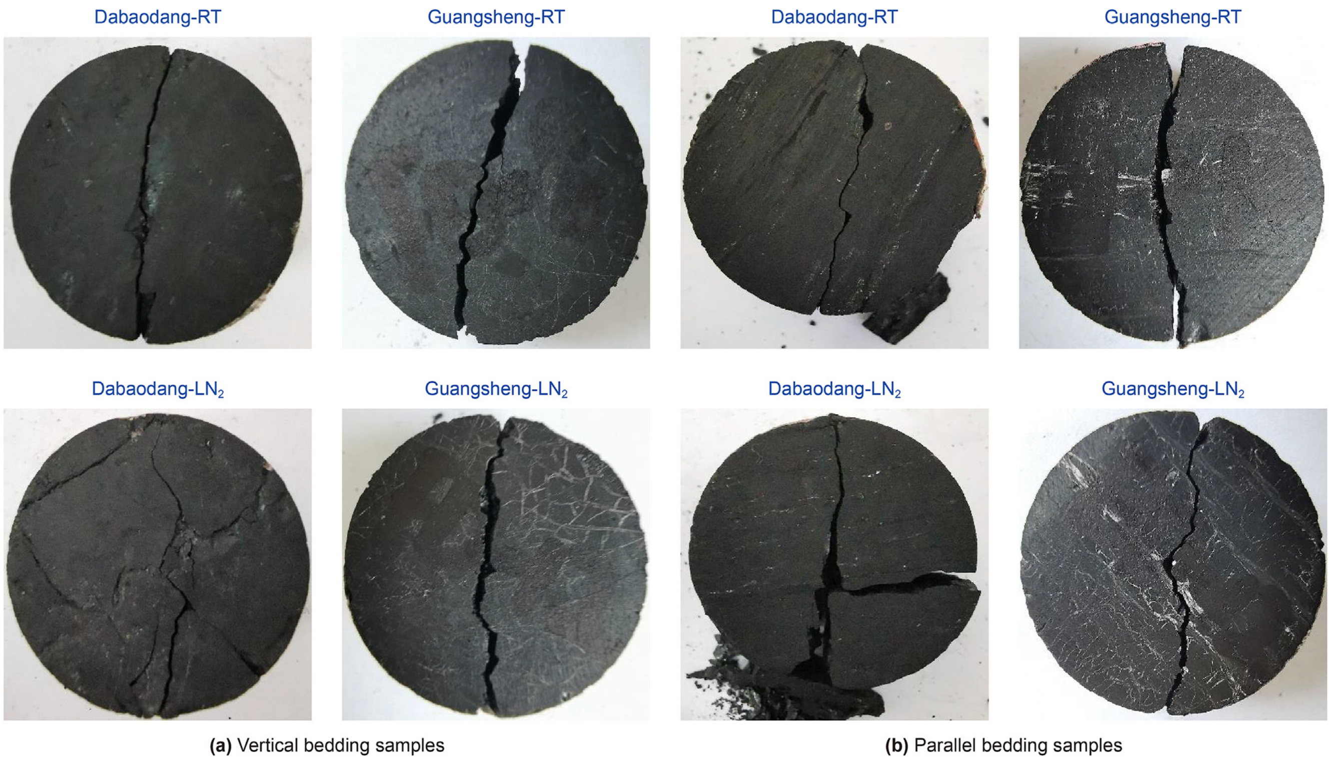


Fig. 18. Brazil split failure pattern of water-saturated coal samples.

rocks with complex mineral composition may suffer higher damage due to heterogeneous deformation of mineral particles under freezing. These lead to the inconsistent change of mechanical properties of the different types of coal.

4.1.1. Damage of dry coal by LN_2 freezing

Coal is damaged by thermal stress in the following two ways during LN_2 freezing. The first is caused by the temperature gradient inside the rock. The thermal conductivity of coal is very low, which value is only 0.28 W/(m K). When LN_2 freezes the rock, the temperature of the outer layer drops rapidly, while the temperature of the inner layer drops slowly. The temperature of the inner and outer layers of rock is different, so the degree of shrinkage of the inner and outer layers of the matrix is not the same, and uneven matrix shrinkage induces thermal stress (Wu et al., 2019). The inner layer of coal is subjected to compressive stress and the surface layer is subjected to tensile stress when the coal is immersed in LN_2. When the tensile stress is greater than the tensile strength of rock, thermal stress damage fractures occur in the outer layer of rock. The second type of damage is caused by the mineral heterogeneity inside the rock. In addition to more than 90% of the matrix, there are also a small amount of minerals in the coal. The arrangement of these internal mineral particles is disordered, so the coal is heterogeneous and anisotropic. When the temperature around coal decreases, the internal mineral particles inside the coal will shrink. And the shrinkage deformations of different mineral particles are different during the LN_2 freezing due to the different thermal expansion coefficients of different mineral particles (Wu et al., 2019). In addition, the rock is a continuous body and its deformation is continuous, so each mineral particle cannot be deformation free with the change of temperature according to its thermal expansion coefficient (Mellor, 1971). As a result, particles inside the rock with a high coefficient of thermal expansion are stretched and those with a low coefficient of thermal expansion are compressed, which induces thermal stress. When the thermal stress exceeds the tensile strength of rock, the internal structure of the rock will be destroyed, resulting in damage fractures. The carbonate minerals are the main mineral components of these two types of coals. The mineral composition of Dabaodang coal is relatively complex. Calcite, siderite, and pyrite are the main minerals, accounting for 40.5%, 35.5%, and 11.1% respectively. The main mineral of Guangsheng coal is calcite, which accounts for 84.6%. The existence of siderite and pyrite is an important reason that the mechanical strength of Dabaodang coal is higher than Guangsheng coal at room temperature. However, the complex mineral composition will lead to enhanced thermal damage caused by the heterogeneous shrinkage of mineral particles at low temperature, which may be an important reason for the higher damage effect of Dabaodang coal under LN_2 freezing.

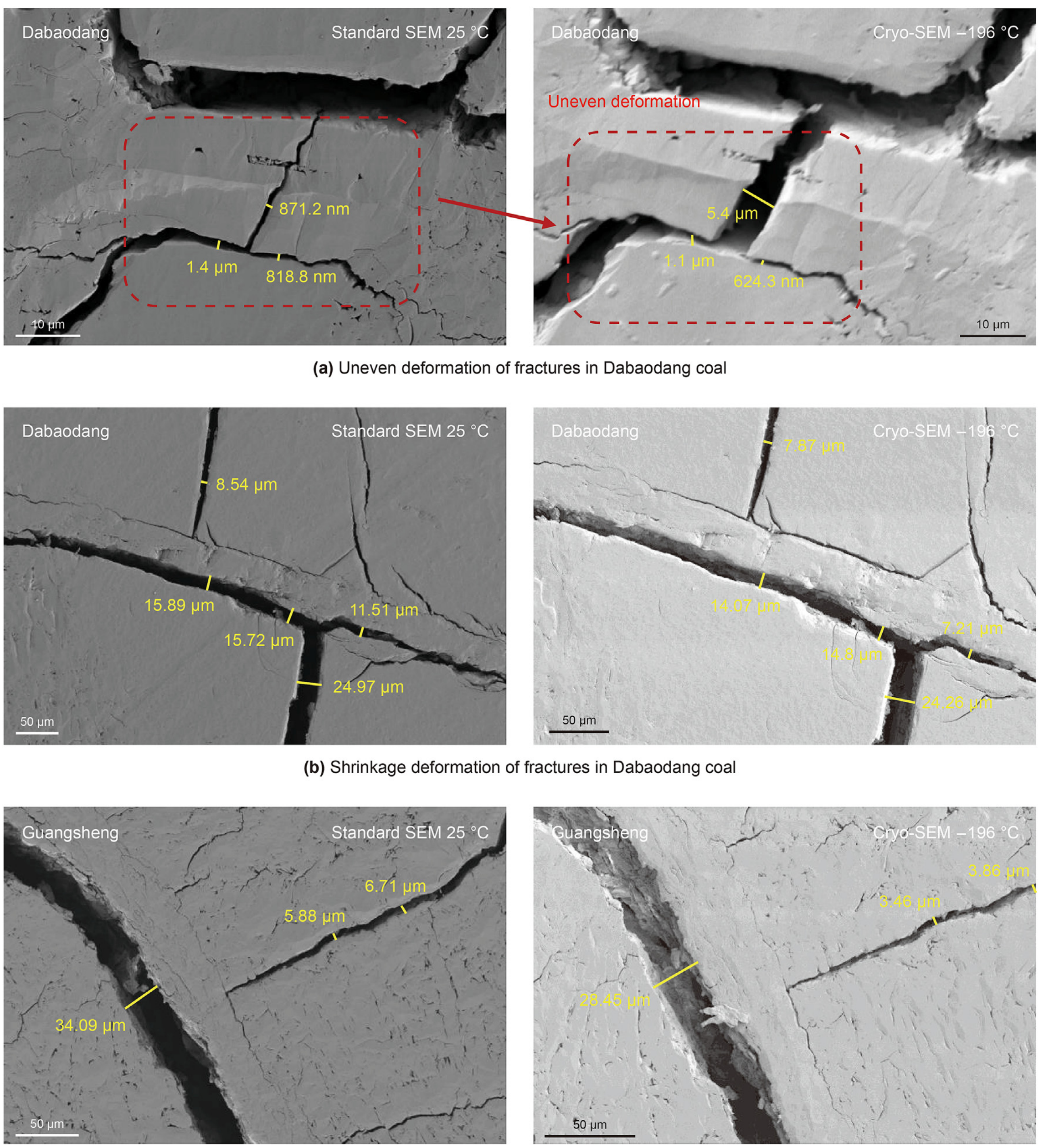
4.1.2. Strengthening of dry coal by LN_2 freezing

When the coal is frozen to ultra-low temperature, the matrix and pore of the coal will shrink, the contact between the particles will be closer, and the connection force will be enhanced due to the influence of cooling shrinkage. The coal under freezing is tighter than that under room temperature, and its internal particles are not easy to slip each other under external forces, so its strength and resistance to deformation are higher. According to the experimental test, the average linear expansion coefficient of coal in the vertical bedding direction and horizontal bedding direction is 3.68×10^{-5} and 2.83×10^{-5} 1/°C respectively in the range of 25 to −195.8 °C. It means that the vertical bedding direction will shrink by 0.407 mm and the parallel bedding direction will shrink by 0.313 mm for a 50 mm cubic coal that was frozen from room temperature to LN_2 temperature. This shrinkage will enhance the strength of the coal greatly.

Fig. 19 shows the fracture morphology of two types of dry coals observed by standard SEM and Cryo-SEM at room temperature and

LN_2 temperature. The effect of LN_2 freezing can be explained by comparing the morphology of the same fracture at room temperature and LN_2 temperature. As can be seen from the figure, no obvious thermal damage fractures caused by internal mineral heterogeneity can be found. But the fractures in Dabaodang coal have uneven shrinkage and compression deformation, as shown in Fig. 19a. It can be seen from Figs. 19b and c that the primary fractures of dry coals in Dabaodang and Guangsheng have different degrees of shrinkage deformation under LN_2 freezing. The fracture width of Dabaodang coal is smaller than that of Guangsheng coal. According to statistics, the fracture width of Dabaodang coal and Guangsheng coal has shrunk by 13.07% and 33.39% on average respectively.

The main reason is that the porosity of Guangsheng coal is higher than that of Dabaodang coal. When the rock shrinks, the Guangsheng coal with high porosity has a large space for shrinkage and deformation, so it is relatively easy to shrink after freezing, and the shrinkage degree is larger. This results in the difference in the physical properties of the two types of coals under LN_2 freezing. Firstly, the rock with higher temperature in the inner layer of Guangsheng coal is easier to shrink under compression stress during freezing, and the difference in the degree of shrinkage deformation between the inner and outer layers is small. Therefore, the thermal stress damage caused by the internal temperature gradient is smaller than that of Dabaodang coal. It also can be observed from Cryo-SEM that there is no uneven shrinkage deformation in the primary fractures of Guangsheng coal. Secondly, the overall shrinkage degree of Guangsheng coal is more significant, indicating the more strengthening effect of frozen shrinkage on the rock. Therefore, the positive impact of the strengthening effect of



(a) Uneven deformation of fractures in Dabaodang coal

(b) Shrinkage deformation of fractures in Dabaodang coal

(c) Shrinkage deformation of fractures in Guangsheng coal

Fig. 19. Fracture morphology of dry coals at room temperature and LN_2 temperature.

LN_2 freezing on the internal structure is higher than the negative impact of the damaging effect on Guangsheng coal, and its compressive strength and elastic modulus are both greatly enhanced. However, for the tight Dabaodang coal, the degree of cold shrinkage is relatively small, and the thermal stress damage caused by LN_2 freezing is greater. As a result, the compressive strength of its vertical bedding even decreases under freezing.

The freezing experiments of granite and sandstone carried out by researchers can also explain the experimental results in this paper (Mellor, 1971). The freezing results of granite and sandstone show that the compressive strength of sandstone and granite increases firstly and then decreases with the decrease of temperature (compressive strength increases from 23 to −120 °C, decreases from −120 to −195 °C) (Mellor, 1971). When the cooling temperature is high, the thermal stress is small. The effect of thermal damage is not significant, and the strengthening effect of frozen shrinkage is greater than the deterioration effect of thermal damage. When the temperature is very low, the temperature difference between inside and outside is huge during the freezing, the thermal stress is high, and the effect of thermal damage is significant. So, the degradation of thermal damage is greater than the strengthening effect of frozen shrinkage. In addition, the enhanced amplitude of compressive strength of sandstone samples with higher porosity is significantly higher than that of granite samples with lower porosity, indicating that the strengthening effect of LN_2 freezing is more significant in higher porosity rock (Mellor, 1971).

The tensile strength of dry coal decreases under LN_2 freezing, indicating that the strengthening effect of LN_2 freezing on the tensile strength is less than the negative effect of thermal damage on the tensile strength for the coal used in this experiment. The result is similar to the previous study (Hou et al., 2022a). And the granite freezing experiments conducted by previous researchers showed that the tensile strength of granite increases first and then decreases with the decrease of temperature (Mellor, 1971). It illustrates that the tensile strength of rock under cryogenic freezing is enhanced by thermal shrinkage and damaged by thermal stress. When the influence of the latter is higher than that of the former, the tensile strength of the rock under freezing decreases.

In addition, the brittleness of dry coal is enhanced under LN_2 freezing. Compared with the coal at room temperature, the brittleness index of Guangsheng and Dabodang coals under LN_2 freezing decreased by 42.71% and 18.50%, respectively. The main reason is that the coal macromolecule arrangement is affected by the ultra-low temperature. More than 90% of coal is organic matter, which is composed of large molecules. When the temperature decreases, the gaps between the molecules decrease. The external force cannot be absorbed through plastic deformation in the failure stage, and the brittle fracture directly occurs. Therefore, the brittleness of dry coal in Dabaodang and Guangsheng is enhanced under LN_2 freezing, and the failure degree is much higher than that at room temperature.

Mechanical properties in vertical and parallel bedding under freezing are different. There are three main reasons. First, coal has strong anisotropy with face cleats and bull cleats. The mechanical properties of vertical and parallel bedding directions are quite different at room temperature. Second, the primary fractures and pores in coal extend along the bedding direction, and the width of fractures perpendicular to the bedding direction decrease under the freezing of LN_2. Therefore, the shrinkage deformation of rock in this direction is mostly the contraction of micro-fractures, while the shrinkage of the matrix is relatively small. While the parallel bedding direction is parallel to the micro-fracture extension direction, the matrix has a relatively greater degree of shrinkage. Therefore, the strengthening degree of coal matrix mechanical properties in parallel bedding direction is higher than that in the vertical bedding direction. Finally, LN_2 freezing will weaken the cementation between the bedding interfaces and reduce the mechanical strength of the bedding interfaces (Hou et al., 2022a). However, bedding cracks will shrink under LN_2 freezing, and the shrinkage degree of bedding cracks in vertical bedding direction is higher than that in parallel bedding direction. As a result, the change of mechanical properties of the vertical bedding coal sample and the horizontal bedding coal sample has different amplitudes under LN_2 freezing. According to the experimental results, the strength and weakness of bedding interface and matrix are also different for different rocks under freezing. Therefore, the increase and decrease of compressive and tensile strength in different bedding directions under freezing are also related to the type of coal.

4.2. Effect of pore water

When pore water freezes in saturated coal, it can also damage and strengthen the coal. The damage caused by pore water freezing in coal is mainly reflected in two aspects. First, when the water-saturated coal is frozen by LN_2, the water in the pores turns into ice and its temperature gradually drops to LN_2 temperature (−195.8 °C). The volume of ice increases first and then decreases in this process, and the volume of ice at LN_2 temperature is about 1.678 times the volume of water at room temperature, as can be seen in Fig. 20. Ice in rock can expand the pores and fractures, so the volume of primary fracture will be increased, some new fractures will be induced, and the rock will be damaged. Second, the ice can be thought of as a mineral embedded in the matrix. The coefficient of expansion for ice (linear coefficient $\approx 5.0 \times 10^{-5}$ 1/°C) is much greater than the coefficient for coal. Therefore, the uneven shrinkage of ice and the matrix will induce thermal damage during the freezing (Harvey, 2019).

The freezing of pore water can also strengthen the structure of coal. The reason for the dramatic increase of strength under LN_2 freezing is that pore water freezes and fills fractures and pores of the rock with solid material, effectively grouting the rock (Mellor, 1971). The compressive strength of ice is 2.35–5.88 MPa. The increase of modulus under LN_2 freezing is almost due to the freezing of pore water, which has the effect of replacing pore fluid with viscoelastic solid (Mellor, 1971).

The fracture morphology of Dabaodang and Guangsheng water-

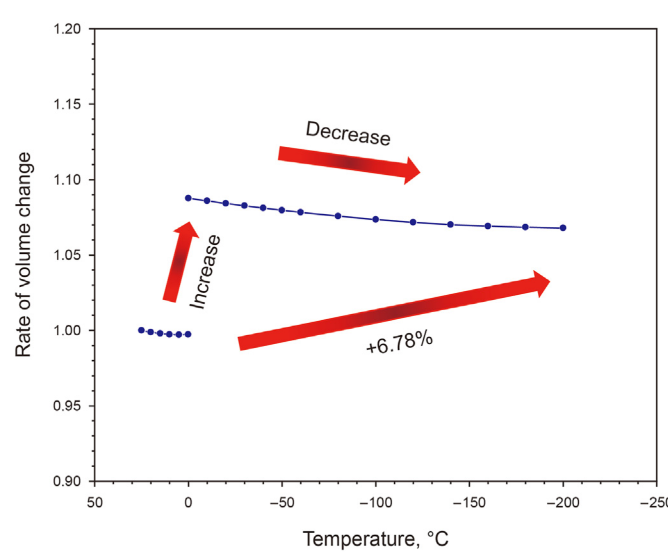


Fig. 20. Rate of volume change of ice at low temperatures (Harvey, 2019).

saturated coals at room temperature and LN_2 temperature are shown in Fig. 21. It can be seen from Fig. 21a that the width of narrow fractures increases and the width of wider fractures decreases under LN_2 freezing, resulting in the reduction of the average fracture width. There may be two reasons for this. First, the fractures of water-saturated coal are not filled with water, so the ice particles cannot fill the fractures and enhance the width of the fractures. The fracture width is reduced due to matrix shrinkage. Second, the frozen rock was sublimated during Cryo-SEM testing, which may lead to the vaporization of ice particles inside the surface fractures. And the ice particles were unable to support these fractures effectively. In this test, the fractures of the water-saturated coal in Dabaodang and Guangsheng have different degrees of shrinkage deformation under LN_2 freezing, which is similar to the dry coal. The deformation degree of fracture width of Dabaodang coal is smaller than that of Guangsheng coal. According to statistics, the fracture width of Dabaodang coal and Guangsheng coal has shrunk by 6.31% and 31.45% on average respectively.

The main reason is that the porosity of Guangsheng coal is higher than that of Dabaobang coal. Guangsheng coal with high porosity has a large space for shrinkage deformation and its shrinkage is relatively easy during LN_2 freezing.

According to the experimental results, the modulus of water-saturated coal under LN_2 freezing is significantly higher than that of dry coal, indicating that water-saturated coal under LN_2 freezing has stronger resistance to deformation. The filling effect of ice on coal structure is higher than its damage effect. In addition, the strength of Guangsheng coal is significantly higher than that of Dabaodang coal. The reason is that the filling volume of ice in Guangsheng coal with high porosity is larger and the strengthening effect on coal structure is more significant.

4.3. Effects on LN_2 fracturing

The breakdown pressure of the reservoir is related to *in-situ* stress, pore pressure, and tensile strength of rock. When the effective tangential stress around the hole reaches or exceeds the tensile strength of the rock, the surrounding rock breaks. The formula of breakdown pressure is as follows (Hubbert and Willis, 1957; Liu et al., 2007):

$$P_b - P_0 = \frac{3(\sigma_h - P_0) - (\sigma_H - P_0) + T}{C} \tag{5}$$

where P_b is the breakdown pressure; P_0 is the pore pressure; σ_h is the minimum horizontal principal stress; σ_H is the maximum horizontal principal stress; T is the tensile strength.

The higher the pore pressure, the lower the formation effective stress $\sigma - P_0$, and the lower the breakdown pressure. The greater the tensile strength, the greater the breakdown pressure. C is the elastic parameter of pore permeability, $1 \le C \le 2$, and its value is related to the permeability of the rock. The larger the permeability, the larger the value of C, and the lower the fracture pressure. For rock without leak-off, the value of C is 1.

The pore pressure, tensile strength, and permeability of rock are affected by LN_2 freezing directly. The pore pressure will decrease when water in formation pores is frozen into ice, leading to an increase of breakdown pressure. The matrix and fractures will shrink under freezing, and the porosity and permeability will be decreased. Therefore, the value of C decreases under LN_2 freezing, leading to an increase in breakdown pressure. In addition, the tensile strength of coal will change under LN_2 freezing, and the change is different for different types of rock, which may increase

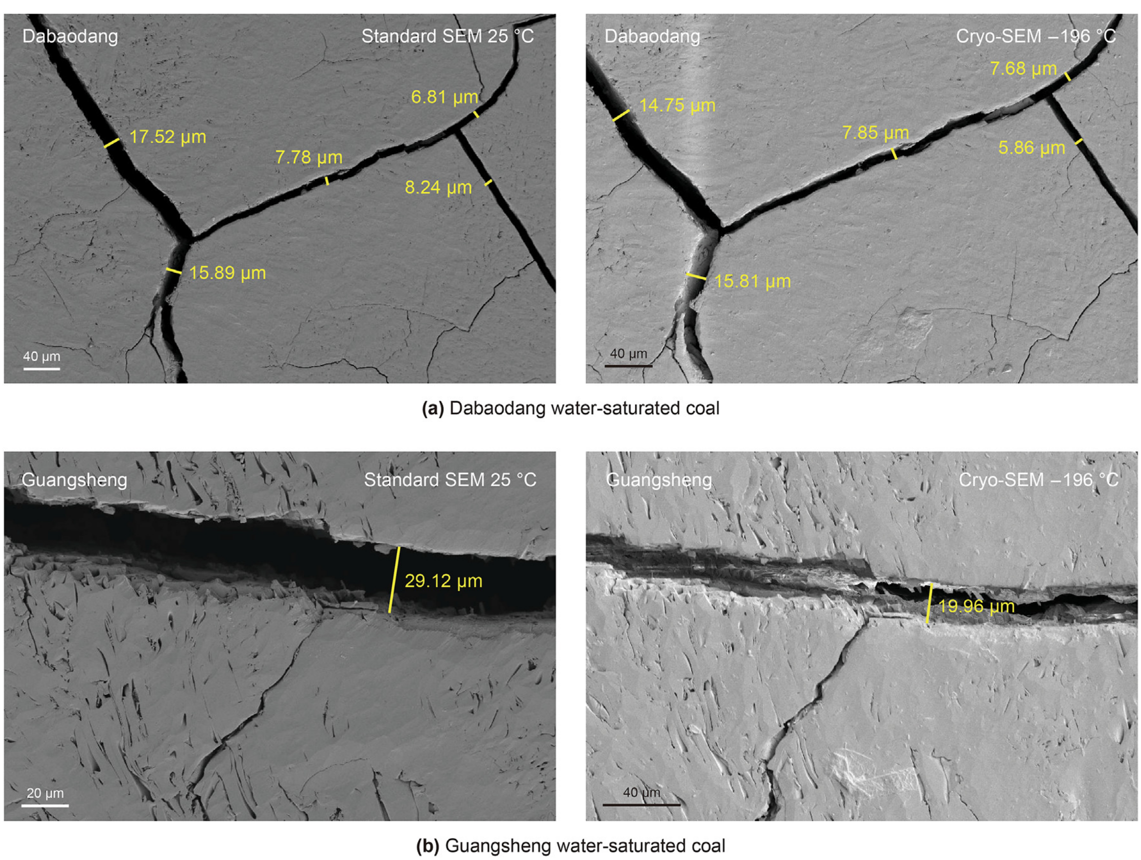


Fig. 21. Fracture morphology of saturated coals at room temperature and LN_2 temperature.

or decrease. If the tensile strength increases, the breakdown pressure will increase; otherwise, the breakdown pressure will decrease. In conclusion, the changes in pore pressure and permeability under LN_2 freezing will increase the breakdown pressure, and the uncertainty of tensile strength under LN_2 freezing will make the change of breakdown pressure uncertain. For example, the tensile strength of Guangsheng coal increases under LN_2 freezing, so its breakdown pressure under freezing will also increase. When the tensile strength of coal decreases under freezing, such as the Dabaodang coal, the increase or decrease of breakdown pressure under freezing should be considered by the influence of the three factors comprehensively.

Fractures formed in coal under LN_2 freezing will be more complicated. There are two main reasons. On the one hand, the brittleness of rock is enhanced by LN_2 freezing, which makes the fracture of rock more complicated during fracturing. On the other hand, some thermal damage fractures are induced under LN_2 freezing, which will provide more paths for fracture propagation and form complex fracture networks (Huang et al., 2020; Yang et al., 2021).

If water is used as the sand-carrying fluid after LN_2 freezing, the effect of proppant carrying may be improved. Water is often used as the sand-carrying fluid in coal reservoir fracturing. Due to the development of cleat fractures in coal, the leak-off of water is large during hydraulic fracturing, so the proppant migration distance is short, and the effective fractures are also short. If the coal seam is first frozen with LN_2, the fractures and matrix will shrink. And the ice filling the coal will seal the fractures. Previous experiments show that the breakthrough pressure gradient of frozen coal is higher than 457 MPa/m (Zhou, 2013). If water is then used to carry proppant, the leak-off will be greatly reduced, the migration distance of proppant will increase, and the effect of sand carrying may be better.

After LN_2 freezing, the formation permeability will increase when the coal seam temperature is restored from the ultra-low temperature to the original reservoir temperature. Fig. 22 shows

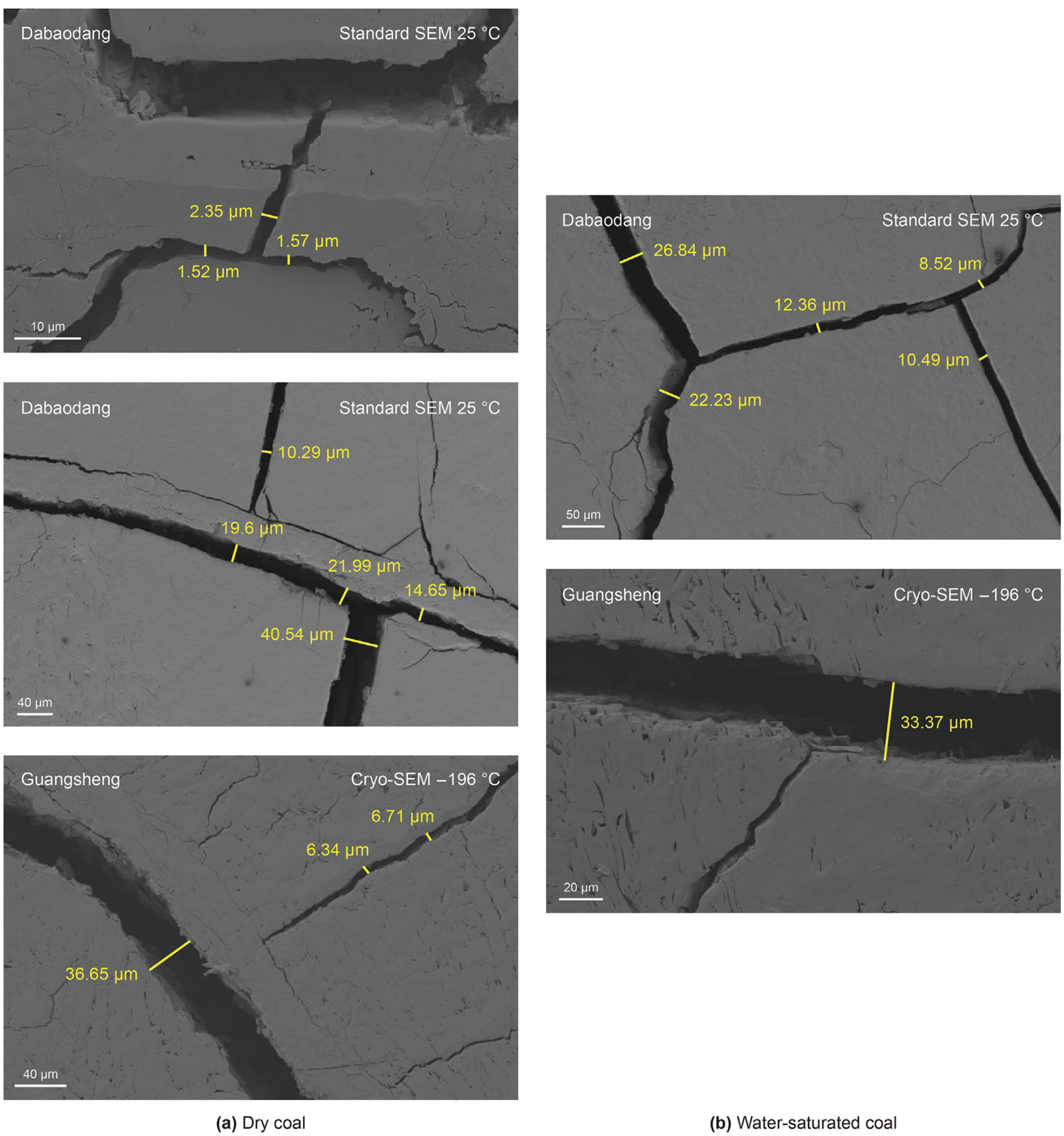


Fig. 22. Fracture morphology of coal at room temperature after being frozen by LN_2.

the fracture morphology of coal at room temperature after being frozen by LN_2, which corresponds to the fractures in Figs. 19 and 21. Combining these three figures, it can be found that the width of fractures at room temperature after freezing is larger than that of fractures without LN_2 treatment. According to statistics, the fracture width of dry Dabaodang coal and Guangsheng coal has increased by 34.24% and 5.11% on average respectively. And the fracture width of water-saturated Dabaodang coal and Guangsheng coals has increased by 40.88% and 14.59% on average respectively. It indicates that the porosity and permeability of rocks will increase after freezing, which is consistent with the previous studies (Cai et al., 2015; Qin et al., 2020). The increase in porosity and permeability will greatly enhance the migration rate of gas and water in the coal seam and increase production.

So, the method of cryogenic fracturing with LN_2 can reduce the leak-off of the coal seam, improve proppant carrying efficiency, form complex fractures, improve the permeability of the frozen zone, and finally increase the production of CBM.

5. Conclusions

The effects of LN_2 ultra-low temperature on the mechanical properties of coal were studied in this paper. Uniaxial strength tests and Brazilian splitting tests were carried out for dry and saturated coals with different types and bedding directions. In addition, standard SEM and Cryo-SEM were used to compare the fracture morphology of coal samples at room temperature and ultra-low temperature. The major findings in our study are concluded as follows:

(1) LN_2 freezing can damage and improve the mechanical properties of coal simultaneously.
(2) The compressive strength of water-saturated Guangsheng and Dabaodang coals under freezing is 35.35% and 27.48% higher than that of dry coals respectively, and the filling of ice can enhance the mechanical strength of coals.
(3) Ultra-low temperature freezing of LN_2 can enhance the brittleness of coal. The brittleness index of Guangsheng and Dabaodang coals under LN_2 freezing is 42.71% and 18.50% lower than the coal at room temperature respectively. And the degree of coal breakage at low temperature is more significant than that at room temperature under loading.
(4) The mechanical properties of coal with different porosity change in different amplitude under LN_2 freezing. The mechanical properties of coal with higher porosity are enhanced more than that of coal with lower porosity under LN_2 freezing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This work is supported by the Youth Program of National Natural Science Foundation of China (No. 52004299), the National Key Scientific Research Instrument Research Project of National Natural Science Foundation of China (No. 51827804), and Beijing Outstanding Young Scientist Program (No. BJJWZYJH012 01911414038). The authors would like to thank the Peking University for our cryo-SEM work, and we are also grateful to Yiqun Liu, and Yingying Guo for their help in making the Cryo-SEM sample and taking Cryo-SEM images.

References

Akhondzadeh, H., Keshavarz, A., Al-Yaseri, A.Z., et al., 2020. Pore-scale analysis of coal cleat network evolution through liquid nitrogen treatment: A Micro-Computed Tomography investigation. Int. J. Coal Geol. 219, 103370. https://doi.org/10.1016/j.coal.2019.103370.

Bieniawski, Z., Hawkes, I., 1978. Suggested methods for determining tensile strength of rock materials. Int. J. Rock Mech. Min. Sci. Geomech. Abstracts 15 (3), 99–103. https://doi.org/10.1016/0148-9062(78)91494-8.

Cai, C., Gao, F., Yang, Y., 2018. The effect of liquid nitrogen cooling on coal cracking and mechanical properties. Energy Explor. Exploit. 36 (6), 1609–1628. https://doi.org/10.1177/0144598718766630.

Cai, C., Li, G., Huang, Z., et al., 2015. Experiment of coal damage due to super-cooling with liquid nitrogen. J. Nat. Gas Sci. Eng. 22, 42–48. https://doi.org/10.1016/j.jngse.2014.11.016.

Cha, M., Alqahtani, N.B., Yao, B., et al., 2018. Cryogenic fracturing of wellbores under true triaxial-confining stresses: experimental investigation. SPE J. 23 (4), 1271–1289. https://doi.org/10.2118/180071-PA.

Du, M., Gao, F., Cai, C., et al., 2020. Study on the surface crack propagation mechanism of coal and sandstone subjected to cryogenic cooling with liquid nitrogen. J. Nat. Gas Sci. Eng. 81, 103436. https://doi.org/10.1016/j.jngse.2020.103436.

Grundmann, S.R., Rodvelt, G.D., Dials, G.A., et al., 1998. Cryogenic nitrogen as a hydraulic fracturing fluid in the devonian shale. In: SPE Eastern Regional Meeting. Society of Petroleum Engineers. https://10.2118/51067-MS.

Harvey, A., 2019. Properties of ice and supercooled water. In: CRC Handbook of Chemistry and Physics. CRC Press, Boca Raton, FL. https://tsapps.nist.gov/publication/get_pdf.cfm?pub_id=926353.

Hong, C., Yang, R., Huang, Z., et al., 2022. Fracture initiation and morphology of tight sandstone by liquid nitrogen fracturing. Rock Mech. Rock Eng. 55 (3), 1285–1301. https://doi.org/10.1007/s00603-021-02755-x.

Hou, P., Su, S., Gao, F., et al., 2022a. Influence of liquid nitrogen cooling state on mechanical properties and fracture characteristics of coal. Rock Mech. Rock Eng. 55 (7), 3817–3836. https://doi.org/10.1007/s00603-022-02851-6.

Hou, P., Xue, Y., Gao, F., et al., 2022b. Effect of liquid nitrogen cooling on mechanical characteristics and fracture morphology of layer coal under Brazilian splitting test. Int. J. Rock Mech. Min. Sci. 151, 105026. https://doi.org/10.1016/j.ijrmms.2021.105026.

Huang, Z., Zhang, S., Yang, R., et al., 2020. A review of liquid nitrogen fracturing technology. Fuel 266, 117040. https://doi.org/10.1016/j.fuel.2020.117040.

Hubbert, M.K., Willis, D.G., 1957. Mechanics of hydraulic fracturing. Transact. AIME 210 (1), 153–168. https://doi.org/10.2118/686-G.

Liu, Q., Li, W., Zeng, X., et al., 2007. Indoor hydraulic pressure crackingmethod to test and measure critical break ing pressure and water resistance coeffic ient of rock mass. Coal Sci. Technol. 35 (1), 85–87. https://10.13199/j.cst.2007.01.89.liuqm.025.

McDaniel, B., Grundmann, S.R., Kendrick, W.D., et al., 1997. Field applications of cryogenic nitrogen as a hydraulic fracturing fluid. In: SPE Annual Technical Conference and Exhibition. Society of Petroleum Engineers. https://10.2118/38623-MS.

Mellor, M., 1971. Strength and Deformability of Rocks at Low Temperatures. Corps of Engineers, US Army, Cold Regions Research and Engineering Laboratory.

Qin, L., Li, S., Zhai, C., et al., 2020. Changes in the pore structure of lignite after repeated cycles of liquid nitrogen freezing as determined by nitrogen adsorption and mercury intrusion. Fuel 267, 117214. https://doi.org/10.1016/j.fuel.2020.117214.

Qin, L., Zhai, C., Liu, S., et al., 2016. Failure mechanism of coal after cryogenic freezing with cyclic liquid nitrogen and its influences on coalbed methane exploitation. Energy Fuel. 30 (10), 8567–8578. https://doi.org/10.1021/acs.energyfuels.6b01576.

Qin, L., Zhai, C., Liu, S., et al., 2017. Changes in the petrophysical properties of coal subjected to liquid nitrogen freeze-thaw–a nuclear magnetic resonance investigation. Fuel 194, 102–114. https://doi.org/10.1016/j.fuel.2017.01.005.

Qin, L., Zhai, C., Liu, S., et al., 2018a. Mechanical behavior and fracture spatial propagation of coal injected with liquid nitrogen under triaxial stress applied for coalbed methane recovery. Eng. Geol. 233, 1–10. https://doi.org/10.1016/j.enggeo.2017.11.019.

Qin, L., Zhai, C., Liu, S., et al., 2018b. Fractal dimensions of low rank coal subjected to liquid nitrogen freeze-thaw based on nuclear magnetic resonance applied for coalbed methane recovery. Powder Technol. 325, 11–20. https://doi.org/10.1016/j.powtec.2017.11.027.

Qin, L., Zhang, X., Li, S., et al., 2022. Fluid space reformation law of liquid nitrogen fracturing coal based on NMR T_1–T_2 spectra and inspiration for coalbed methane production. Fuel 324, 124811. https://doi.org/10.1016/j.fuel.2022.124811.

Shao, Z., Ye, S., Tao, S., et al., 2021. Experimental study of the effect of liquid nitrogen penetration on damage and fracture characteristics in dry and saturated coals. J. Petrol. Sci. Eng. 201, 108374. https://doi.org/10.1016/j.petrol.2021.108374.

Su, S., Gao, F., Cai, C., et al., 2020. Experimental study on coal permeability and

cracking characteristics under LN_2 freeze-thaw cycles. J. Nat. Gas Sci. Eng. 83, 103526. https://doi.org/10.1016/j.jngse.2020.103526.
Tarasov, B., Randolph, M., 2011. Superbrittleness of rocks and earthquake activity. Int. J. Rock Mech. Min. Sci. 48 (6), 888–898. https://doi.org/10.1016/j.ijrmms.2011.06.013.
Wang, L., Yao, B., Cha, M., et al., 2016. Waterless fracturing technologies for unconventional reservoirs-opportunities for liquid nitrogen. J. Nat. Gas Sci. Eng. 35, 160–174. https://doi.org/10.1016/j.jngse.2016.08.052.
Wen, H., Yang, R., Huang, Z., et al., 2022. Flow and heat transfer of nitrogen during liquid nitrogen fracturing in coalbed methane reservoirs. J. Petrol. Sci. Eng. 209, 109900. https://doi.org/10.1016/j.petrol.2021.109900.
Wen, H., Yang, R., Huang, Z., et al., 2020. Numerical simulation of proppant transport in liquid nitrogen fracturing. J. Nat. Gas Sci. Eng. 84, 103657. https://doi.org/10.1016/j.jngse.2020.103657.
Wu, X., Huang, Z., Zhang, S., et al., 2019. Damage analysis of high-temperature rocks subjected to LN_2 thermal shock. Rock Mech. Rock Eng. 52 (8), 2585–2603. https://doi.org/10.1007/s00603-018-1711-y.
Yan, H., Tian, L., Feng, R., et al., 2020. Liquid nitrogen waterless fracking for the environmental protection of arid areas during unconventional resource extraction. Sci. Total Environ. 721, 137719. https://doi.org/10.1016/j.scitotenv.2020.137719.
Yang, R., Hong, C., Huang, Z., et al., 2021. Liquid nitrogen fracturing in boreholes under true triaxial stresses: Laboratory investigation on fractures initiation and morphology. SPE J. 26 (1), 135–154. https://doi.org/10.2118/201224-PA.
Yang, R., Huang, Z., Shi, Y., et al., 2019. Laboratory investigation on cryogenic fracturing of hot dry rock under triaxial-confining stresses. Geothermics 79, 46–60. https://doi.org/10.1016/j.geothermics.2019.01.008.
Yao, B., Wang, L., Yin, X., et al., 2017. Numerical modeling of cryogenic fracturing process on laboratory-scale Niobrara shale samples. J. Nat. Gas Sci. Eng. 48, 169–177. https://doi.org/10.1016/j.jngse.2016.10.041.
Zhao, D., Wang, Q., Li, D., et al., 2018. Experimental study on infiltration and freeze–thaw damage of water-bearing coal samples with cryogenic liquid nitrogen. J. Nat. Gas Sci. Eng. 60, 24–31. https://doi.org/10.1016/j.jngse.2018.09.027.
Zhao, X., Wang, L., Yao, B., et al., 2022. Cryogenic fracturing of synthetic coal specimens under true-triaxial loadings-An experimental study. Fuel 324, 124530. https://doi.org/10.1016/j.fuel.2022.124530.
Zhou, N., 2013. Experimental and Theoretical Studies on the Feasibility of the Ice Temporary Plugging Fracturing of CBM Well. Ph.D Dissertation. Southwest Petroleum University.