

Study on the Influencing Factors on the Effectiveness of Liquid Nitrogen Fracturing Technology

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Abstract: Liquid nitrogen fracturing technology is a new fracturing and permeability enhancement technology for reservoirs unsuitable for hydraulic fracturing, and its ultralow temperature, high compression ratio, high freezing and expansion force, and low viscosity characteristics are expected to provide a program for fracturing and permeability enhancement in low-permeability reservoirs. The paper systematically analyzes the influencing factors restricting the effect of liquid nitrogen fracturing, aiming to provide theoretical basis and technical support for the efficient and environmentally friendly exploitation of low-permeability oil and gas fields.

Keywords: Liquid Nitrogen Fracturing; Low-permeability Reservoirs; Fracturing Effect; Influencing Factors.

1. Introduction

China is rich in coalbed methane (CBM) resources, according to the statistics, the CBM resources in the shallow 2000 m in China is 36.8 trillion m³, of which the deep (1500 m deep) resources account for 1/3, according to the world's third, which provides a guarantee for the national energy security [1-2]. In 2023, China's surface CBM production is only 13.94 billion m³, accounting for 3.48% of the natural gas production in that year, and the average production of single well is lower than 800 m³/d, and the average production of single well is lower than 800 m³/d, and the average production of single well is lower than 1,000 m³ /d. The average production of a single well is lower than 800 m³/d, which is mainly due to the low permeability, low gas saturation, and extensive development of tectonic coal in coal beds, in which more than 60% of the mining areas have permeability lower than 0.1 mD, which leads to more than 90% of the CBM gas wells needing artificial fracturing technology to form a network of fissure seams to communicate with the wellbore, and how to use appropriate technology to enhance the permeability and increase the production is still an important problem faced by the industry at present [3-4].

In fact, as early as in the 1990s, some scholars proposed the idea of injecting high-pressure, large-displacement liquid nitrogen (LN₂) into coal seams to replace conventional water-based fracturing fluids for fracturing reservoirs. The ultra-low temperature (-196 °C), high compression ratio (1:696), high freezing and expansion force (up to 207 MPa) and low viscosity of LN₂ make it a perfect fit for the coal seams and it does not react with the minerals in the coal, and there is no liquid residue after vaporization. It is expected to provide a new method for the transformation of low-permeability reservoirs because of its perfect adaptability to the coal bed and no reaction with coal minerals, no liquid residue after vaporization, no reservoir damage such as water sensitivity and water lock, and low rock-breaking pressure, complex fracture network, and synergistic effect of pressurization-excavation [5]. Since then, McDaniel et al. [6] and Grundman et al. [7] attempted to use low-temperature-resistant fiberglass tubing and stainless steel wellhead devices to press LN₂ into coal and shale formations to improve the reservoir

permeability and daily production, which proved its feasibility as a fracturing fluid for engineering implementation.

Currently, liquid nitrogen fracturing (LNF) methods for low-permeability coal reservoirs are mostly carried out in laboratories, and large-scale field implementation has not yet been carried out, and the current focus is on the low-temperature fracturing and permeability enhancement effect that occurs after LN₂ is immersed into the coal body. Laboratory studies have found that LN₂ immersed in the coal body degrades the mechanical properties of the rock and produces a dense fracture network, and the diffusion capacity and permeability of the gas are greatly improved (10-20 times), and at the same time, it has a dual role of pressurization-excavation, which has a promising application [8-9]. Further, some studies have pointed out that the number of fracturing, fracturing time, water content, reservoir temperature, etc. in the process of LNF can significantly affect the effect of reservoir reforming [10]. This paper summarizes the control factors of the effect of liquid nitrogen fracturing of low-permeability reservoirs, which provides reference and ideas for the engineering of LNF technology in low-permeability reservoirs.

2. Influencing Factors of Low Temperature Fluid Fracturing Effect

The factors affecting the effect of low temperature fluid fracturing coal are complex and diverse, which are generally divided into external factors (Temperature difference, fracturing times, time) [11-17] and internal factors of coal itself (coal rank, water content, pore structure and mechanical properties of coal body itself, etc.) [18-19]. However, studies have shown that low temperature damage and expansion stress are the main factors affecting the effect of LNF. The former can increase the matrix damage around the pore wall, provide the initial seepage path for fracturing, increase the randomness of fracture path expansion, and improve the complexity of fracture network. The latter promotes fracture initiation and continuous expansion by increasing pore pressure. In the LNF experiment, freeze-thaw can give full

play to the advantages of the former, and higher temperature gradient can expand the advantages of the latter [20].

2.1. Effect of Temperature Difference on Fracturing Effect of LN₂

The rate of rock frost heave and fragmentation is primarily influenced by temperature difference. Low temperatures can exacerbate rock damage and deteriorate its mechanical properties. A larger temperature gradient leads to increased anisotropic deformation between minerals, making the rock more prone to breakage. Numerous scholars have conducted studies on the impact of initial rock temperature on fracturing effects through simulations. (Table 1). Liu et al. [21] found that the thermal stress increased by 17% when the temperature gradient of coal increased by 150 °C, which caused the initiation, expansion, extension and expansion of the fracture network, and significantly changed the fracture structure of coal; Zhang et al. [22] found that with the increase of initial rock temperature, the volume and depth of rock breaking increase, and there is a key temperature range that promotes the sudden failure of granite. When the temperature difference is 450 °C, only weak damage occurs. When the temperature difference reaches 670 °C, the rock mass will quickly break into two halves; Cai et al. [23] analyzed the breakdown pressure and fracture morphology of high temperature granite at different heating temperatures. The results showed that the breakdown pressure of granite decreased with the increase of temperature gradient, and the failure mode was mainly tensile fracture; Based on the theory of trace elements and damage mechanics, Lin et al. [24] established a thermodynamic damage model of coal; Li [25]

found that the higher the initial temperature of the coal sample, the greater the deterioration of the physical and mechanical properties of the coal sample, and the shorter the macroscopic failure cycle; Yan et al. [26] found that the prefabrication temperature of coal is positively correlated with its surface fracture propagation and internal damage; Some scholars have found that the thermal shock effect of LN₂ is selective in coal with different metamorphic degrees, and the fracturing effect of low metamorphic and medium metamorphic coal is higher than that of high metamorphic coal [27].

Compared to the warm region, weathering and crushing occur at a faster rate in the alpine area. During this period, the rock may not experience extreme low temperatures (such as -196 °C of LN₂), but rather undergoes repeated temperature fluctuations within a certain range (from 0 °C to -30 °C). This phenomenon suggests that low-temperature fracturing does not necessarily require the same temperature as LN₂, but only needs to surpass the critical fracturing temperature at a specific frequency to induce rock damage. Additionally, it has been observed that numerous fractures can be generated in tuff between -0.92 °C and -2.77 °C, sandstone between -5 °C and -10 °C, and granite between 0°C and -1°C. Below this temperature range, there is no significant acceleration in the fracture process. [28], indicating that absolute low temperature should not be a necessary condition for rock fracturing, and repeated crossing of critical fracturing temperature is the key to low temperature fracturing [29]. How does the temperature distribution and conduction occur during in-situ LN₂ injection? In which temperature range does the fracturing efficiency of LN₂ reach its peak? Is this the next crucial issue to be investigated?"

Table 1. Effect of temperature difference on fracturing effect of LN₂

Sample information	Coal rank	Temperature difference (°C)	Fracturing times (Cycles)	Test content change rate	
Zhao Gu, Henan	Anthracite	30→180	1	Thermal stress: ↑17% Small, medium and large pore specific surface area: ↑8%/↑70%/↓15%	[21]
Xu Tuan, Jiangsu	Bituminous coal	216→296	2	Damage depth: 0 mm→85 mm Permeability: ↑740%	[22]
Fu Xin, Liaoning	long-flame coal	216→391	3	Thermal stress: ↑23% Damage depth: 16.84μm→Crushing	[24]
Heng Yi, Shanxi	long-flame coal	221→261	1	Thermal stress: ↑18% Porosity: ↑9.2% Damage depth: ↑14%	[25]
Shandong	Granite	221→496	1	Thermal stress: ↑139% Breakdown pressure: ↓94%	[23]
Numerical simulation	/	221→291	1	Thermal stress: ↑14% Damage depth: ↑44% Breakdown pressure: ↓58%	[26]

Note: “/” represents no data.

2.2. Effect of Moisture on LNF

The study found that the ice wedge expansion of saturated water under low temperature conditions has the greatest impact on the destruction of coal and rock joint structure. This is because the pore water becomes ice during the freezing process of LN₂, and the volume expansion, pore pressure increase, and the internal pore structure of the rock is destroyed, resulting in stress concentration and fracture propagation [30]. For dry and low water content coal, low temperature freezing mainly plays a role in weakening pore structure. However, when the water saturation of coal exceeds

a certain critical value (such as more than 60%), the number of micropores in coal will show a significant growth trend [31]. It is particularly noteworthy that the size and number of micro-fractures in LN₂-treated hydrous coal samples are significantly increased compared to dry coal samples. This is because the low temperature treatment of LN₂ not only aggravates the cold shrinkage effect of coal, but also promotes the migration and redistribution of water in coal, thus inducing more microfractures. The formation of these microfractures not only enhances the permeability of coal, but also provides more favorable conditions for the exploitation of

CBM.

Many researchers have studied the effect of LNF under different water conditions (Table 2). It is found that the effect of LNF coal has a great correlation with the initial water content of the sample. The larger the initial water content, the greater the frost heave force formed by water freezing and expansion, and the more significant the fracturing effect. Wang et al. [32] found that in the process of LNF, the water content of coal was positively correlated with its surface frost heaving force and porosity, and the permeability of coal showed stage characteristics; Li et al. [33] found that in the process of LNF, the permeability of coal increases exponentially with the increase of water content. Dry coal samples mainly germinate new fractures on the basis of primary pores, while water-bearing coal samples germinate new fractures. At the same time, the primary fractures will be spatially connected to each other to form a dense fracture network; Similarly, Li [34] also put forward a similar point of view after LNF of anthracite with different water content, believing that the water in coal can promote the fracturing effect of LN₂; In addition, Qi et al. [35] also found that the mechanical properties such as compressive density and

elasticity of coal after LNF were weakened to varying degrees; Lin et al. [36] found that after LNF, with the increase of water content of coal, the width of surface fractures increased, and the ultrasonic wave velocity also decreased significantly; Li et al. [37] determined the pore structure of dry and saturated coal samples after freeze-thaw, and considered that the existence of water-ice phase transition in saturated water coal samples accelerated the conduction of temperature in coal, which was easier to fracture than dry samples; Cai et al. [38] pointed out that compared with only macroscopic fractures formed after freezing and thawing of sandstone, coal samples will form a more complex fracture network under the action of LNF, which is more suitable for the transformation of low permeability coal seams. Our research group carefully designed the LNF test, in-depth study of the pore size distribution of coal before and after LNF, confirmed that the water content of coal can promote the fracturing effect of LN₂ [39]. However, although we have made some progress in the field of LNF coal, there are still many problems that need to be further studied. For example, the response mechanism of coal with different saturation degrees to LN₂, and how the water content of the reservoir adapts to the fracturing scheme.

Table 2. Study on the influence of water content of coal on LNF effect

Sample information	Coal rank	Moisture content (%)	Fracturing times (Cycles)	Test content change rate	
Wang Zhuang, Shanxi	coking coal	0→100	1	Porosity: ↑8.1% Permeability: ↑32% Characteristic fracture width: ↑380%	[32]
Zhu Jiao	Anthracite	0→100	2	Porosity: ↑230.5% Permeability: ↑607.8%	[33]
Qian Jin, Guizhou	Anthracite	0.23→5.12	1	Porosity: ↑100% Permeability: ↑293%	[34]
Wang Zhuang, Shanxi	coking coal	0→9	2	Permeability: ↑108.24% Characteristic fracture width: ↑1909% Mechanical strength: ↓8.18%	[35]
Heng Yi, Shanxi	coking coal	0→11.67	2	Porosity: ↑6.94% Permeability: ↑71.9% Characteristic fracture width: ↑34.64%	[36]
Jiu Lishan, Henan	Anthracite	0→100	1	Porosity: ↑157% Characteristic fracture width: ↑115%	[37]

2.3. Rock Mechanical Property

In the process of LNF, the physical and mechanical properties of rock play an important role in the formation and expansion of fractures, among which the strength parameters are particularly critical. The strength parameters include compressive strength, tensile strength and shear strength of rock. When LN₂ is injected into the rock, its low temperature effect will form a temperature gradient inside the rock and generate thermal stress, which will have a significant impact on the coal fracturing process. Cai et al. [40] showed that the elastic modulus of dry coal samples decreased by 25% and the compressive strength decreased by 34% after soaking in LN₂. In other studies, it was also observed that the Young's modulus and compressive strength of coal samples freeze-thawed in LN₂ decreased significantly. Among them, different rock types show obvious differences in the initiation point after LN₂ injection. Cui et al. [41] found that the strength and stiffness of marine clay decreased by about 48.5% and 22.7% respectively through low temperature freeze-thaw experiments; Liu et al. [42] studied the effect of freeze-thaw

cycles on the consolidated soil matrix; Quan et al. [43] found that the resilient modulus of Qinghai-Tibet clay decreased with the increase of freeze-thaw cycles; Chang et al. [44] evaluated the shear strength of coarse-grained soil according to the freeze-thaw cycle; Memon [45] discovered that the nanoindentation modulus of Mancos shale samples was significantly affected by low temperature LN₂. The application of 50 mN and 200 mN resulted in a decrease in the nanoindentation modulus of Mancos samples from 24.6 GPa and 16.8 GPa to 15.6 GPa, respectively.; Jin [46] conducted experiments on anthracite and coking coal using LNF, which led to a reduction in elastic modulus, compressive strength, and brittleness index of coal samples after thawing; consequently changing their properties from brittleness to ductility.

In addition, the pore structure of the rock will also affect the fracture initiation point of the coal freeze-thawed by LN₂. The pore structure of the rock will affect the physical properties such as permeability and elastic modulus of the rock, and then affect the effect of LNF. When LN₂ is injected into the rock, the rock with complex pore structure will lead

to the uneven distribution of LN₂ in the rock, which will cause the difference of thermal stress at different positions, and then affect the formation and expansion of fractures. Qin et al. [47] studied the effect of LNF on the physical pore and fracture structure of coal. The results showed that the elastic modulus of coal samples decreased after freezing and thawing, while the porosity and Poisson's ratio showed an upward trend, and the mechanical strength also decreased accordingly. In addition, the damage effect of cyclic freezing and thawing on coal is continuous, especially after 20 freeze-thaw cycles, the failure rate is significantly increased.

2.4. Effects of Other Factors

The effect of LN₂ on coal fracturing is influenced by various factors, including internal coal characteristics such as coal rank, mineral composition, and microstructure. The mineral composition and organic matter content of coal samples can impact the thermal expansion coefficient, thermal conductivity, and thermal shock resistance of coal. Consequently, different degrees of damage and fractures may occur in distinct mineral particles during LNF, thereby affecting the growth rate and fracture area of internal fractures in coal. [48]. The reaction of different types of coal to LNF is also different. Generally, the fracturing effect of low rank coal is higher than that of high rank coal [49]. Qin et al. [50] used nuclear magnetic resonance (NMR) technology to observe the effect of lignite, bituminous coal and anthracite before and after freezing and thawing with LN₂ for 60 min. The analysis found that the degree of LN₂ transformation of different coal rank coal bodies was the most significant for lignite, followed by anthracite, and finally bituminous coal; The permeability improvement effect of lignite under the influence of LN₂ is found to be the most pronounced, while anthracite shows insignificant changes, as concluded by Lu [51]. Furthermore, our research reveals that the fracturing effect of LN₂ on coal exhibits selectivity, with a more favorable outcome for lignite compared to bituminous coal and anthracite. The key factor in forming a fissure network lies in the extension and expansion of original fissures during the fracturing process.

The external factors that affect the effect of LNF coal also include the number of LNF, freeze-thaw time, etc. Compared with the single freeze-thaw treatment, the multiple fracturing process can gradually reduce the pressure required for reservoir fracturing, and significantly improve the fracturing effect through the superposition effect [52-53]; Qin et al. [54] pointed out that compared with a single injection of LN₂, the cyclic injection method performs better in fracturing efficiency; the research of Zhai [55-56] shows that the freezing of LN₂ will cause damage to the coal body, which will promote the formation of fractures, and the degree of damage is positively correlated with the water saturation of the coal; Zhang et al. [22] found a single cycle of fractures with a length of 80 mm after LNF on coal samples with no obvious fractures on the surface. After three cycles of treatment, the fracture length expanded to 85 mm, and the permeability increased by 1129.79% compared with the original; Ghobadi et al. [57-58] found that cyclic freezing and thawing would aggravate rock damage, resulting in more significant mechanical degradation and permeability improvement through cyclic freezing and thawing experiments. However, different scholars have different understandings of the effect of LNF. Some scholars have pointed out that with the progress of nitrogen cycle fracturing process, the effect of LNF on the transformation of pore

fractures in coal is gradually weakened [59], which implies that there is an upper limit for the transformation of coal structure in the process of cyclic fracturing. For example, Zhang et al. [60] found that after 240 h of LN₂ action, increasing the action time of LN₂ had no effect on the fracturing effect.

3. Conclusion

This paper provides an in-depth discussion of the factors influencing the effectiveness of LN₂ fracturing of low-permeability reservoirs, and draws the following conclusions: the influencing factors on the effectiveness of low temperature LNF can be classified into internal factors and external factors, among which coal moisture content and preset temperature have significant impacts. Notably, water-bearing coal samples exhibit different fracturing mechanisms compared to dry coals. Water-bearing coal samples generate substantial thermal stresses during LNF processes. Moreover, large temperature differences can deteriorate mechanical properties of coal while reducing fracturing pressure accordingly. Therefore, higher water content levels combined with elevated preset temperatures favor more effective LNF outcomes.

References

- [1] Xu F, Hou W, Xiong X, et al. The status and development strategy of coalbed methane industry in China[J]. Petroleum Exploration and Development. 2023, 50(4): 669-682.
- [2] Liu C, Zhu J, Che C, et al. Methodologies and results of the latest assessment of coalbed methane resources in China [J]. Natural Gas Industry. 2009, 29(11): 130-132.
- [3] Zhang Q, Feng S, Yang X. Basic reservoir characteristics and development strategy of coalbed methane resource in China [J]. Journal of China Coal Society. 2001, 26(3): 230-235.
- [4] Mu F, Zhong W, Zhao X, et al. Strategies for the development of CBM gas industry in China[J]. Natural Gas Industry. 2015, 35(6): 110-116.
- [5] Huang Z, Wei J, Li G, et al. An experimental study of tensile and compressive strength of rocks under cryogenic nitrogen freezing[J]. Rock and Soil Mechanics. 2016, 37(3): 694-700, 834.
- [6] McDaniel B W, Grundmann S, Kendrick W, et al. Field applications of cryogenic nitrogen as a hydraulic fracturing fluid. Jpt Journal of Petroleum Technology 50.3 (1998): 38-39.
- [7] Grundmann S R, Rodvelt G D, Dials G A, et al. Cryogenic nitrogen as a hydraulic fracturing fluid in the devonian shale. Society of Petroleum Engineers 1998.
- [8] Li H, Liu J, Gao X, et al. Effect of cold loading by liquid nitrogen on damage of coal samples with varied joint angles and water saturation levels[J]. Journal of Mining & Safety Engineering. 2021, 39(2): 413.
- [9] Tian M, Zhang L, Xue J, et al. Study and prospection of liquid nitrogen fracturing coal technology[J]. Coal Science and Technology. 2022, 50(7): 191-198.
- [10] Yan M, Fan Y, Yue M, et al. Heat-mass transfer coupling effects in water-ice phase transformation of water-bearing coal frozen with liquid nitrogen[J]. Applied Thermal Engineering. 2022, 215: 118902.
- [11] Wang Q. Experimental Study on liquid nitrogen Cracking of Coal[D]. Taiyuan University of Technology. 2018.

- [12] Zhai C, Wu S, Liu S, et al. Experimental study on coal pore structure deterioration under freeze–thaw cycles[J]. *Environmental Earth Sciences*. 2017, 76(15): 1.
- [13] Li H, Wang L, Niu F, et al. Study on effect of freeze-thaw cycle with liquid nitrogen on crack extension of coal at different initial temperatures [J]. *China Safety Science Journal*. 2015, 25(10): 121-126.
- [14] Zhang C, Xu G, Yu Y, et al. Study on permeability-enhancing mathematical model of coal fracturing with borehole water injection and liquid nitrogen injection[J]. *Coal Science and Technology*. 2019, 47(1): 139-144.
- [15] Li H, Wang L, Zhang H, et al. Investigation on damage laws of loading coal samples under cyclic cooling treatment[J]. *Journal of China Coal Society*. 2017, 42(09): 2345-2352.
- [16] Cha M, Yin X, Kneafsey T, et al. Cryogenic fracturing for reservoir stimulation – Laboratory studies[J]. *Journal of Petroleum Science and Engineering*. 2014, 124: 436-450.
- [17] Wang Q, Zhao D, Feng Z, et al. Experimental study on fracturing of coal by injection liquid nitrogen in drill based on CT scanning[J]. *Coal Science and Technology*. 2017, 45(04): 149-154.
- [18] Yan D. Experimental Study on the Effects of liquid nitrogen Freezing and Thawing Damage in Coal with Different Water Content[D]. *China University of Mining and Technology*. 2019.
- [19] Zhang C, Zhang H, Yu Yong, et al. Effects of saturation and re-submersion on coal fracturing subjected to liquid nitrogen shock[J]. *Journal of China Coal Society*. 2016, 41(z2): 400-406.
- [20] Wang S, Su S, Wang D, et al. Experimental study on fracture characteristics of coal due to liquid nitrogen fracturing [J]. *Geomechanics for Energy and the Environment*. 2023, 33: 100438.
- [21] Liu S, Li X, Wang D, et al. Mechanical and Acoustic Emission Characteristics of Coal at Temperature Impact[J]. *Natural Resources Research*. 2020, 29(3): 1755-1772.
- [22] Zhang L, Lu S, Zhang C, et al. Effect of cyclic hot/cold shock treatment on the permeability characteristics of bituminous coal under different temperature gradients[J]. *Journal of Natural Gas Science and Engineering*. 2020, 75: 103121.
- [23] Cai C, Zou Z, Ren K, et al. Experimental study on the breakdown mechanism of high temperature granite induced by liquid nitrogen fracturing: An implication to geothermal reservoirs[J]. *Heliyon*. 2023, 9(8): e19257.
- [24] Lin H, Li B, Li S, et al. Numerical investigation of temperature distribution and thermal damage of heterogeneous coal under liquid nitrogen freezing[J]. *Energy*. 2023, 267: 126592.
- [25] Li H, Liu J, Wang L, et al. Effect of freeze-thaw cycles in liquid nitrogen on damage of coal samples with different initial temperatures[J]. *Coal Engineering*. 2023, 55(11): 142-147.
- [26] Yan M, Zhang Y, Lin H, et al. Effect on liquid nitrogen impregnation of pore damage characteristics of coal at different temperatures[J]. *Journal of China Coal Society*. 2020, 45(8): 2813-2823.
- [27] Li Y, Ren Z, Song D, et al. Selection Effect of liquid nitrogen Freeze–Thaw Cycles on Full Pore Size Distribution of Different Rank Coals[J]. *ACS omega*. 2023, 8(10): 9526-9538.
- [28] Walder J, Hallet B. A theoretical model of the fracture of rock during freezing[J]. *Geological Society of America Bulletin*. 1985, 96(3): 336-346.
- [29] Haeberli W, Hallet B, Arenson L, et al. Mountain permafrost-research[J]. *Permafrost and Periglacial Processes*. 2006, 17: 189-214.
- [30] Lai X, Zhang S, Dai J, et al. Multi-scale damage evolution characteristics of coal and rock under hydraulic coupling[J]. *Chinese Journal of Rock Mechanics and Engineering*. 2020, 39(S2): 3217-3228.
- [31] Zhang L, Tian M, Lu S, et al. Analysis of permeability variation and stress sensitivity of liquid nitrogen fracturing coal with different water contents[J]. *Rock and Soil Mechanics*. 2022, 43(S1): 107-116.
- [32] Wang X, Qi X, Ma H, et al. Experimental study on freeze–thaw damage characteristics of coal samples of different moisture contents in liquid nitrogen[J]. *Scientific reports*. 2022, 12(1): 18543.
- [33] Li B, Zong C, Huang L, et al. Study on the Influence of liquid nitrogen Cold Soaking on the Temperature Variations and Seepage Characteristics of Coal Samples with Different Moisture Contents[J]. *Geofluids*. 2021, 2021: 1-10.
- [34] Li C, Nie B, Feng Z, et al. Experimental Study of the Influence of Moisture Content on the Pore Structure and Permeability of Anthracite Treated by liquid nitrogen Freeze–Thaw[J]. *ACS Omega*. 2022, 7(9): 7777-7790.
- [35] Qi X, Hou S, Ma H, et al. A Study of the Effect of Freeze–Thawing by liquid nitrogen on the Mechanical and Seepage Characteristics of Coal with Different Moisture Content Values[J]. *Processes*. 2023, 11(6): 1822.
- [36] Lin H, Li J, Yan M, et al. Damage caused by freeze-thaw treatment with liquid nitrogen on pore and fracture structures in a water-bearing coal mass[J]. *Energy Science & Engineering*. 2020, 8(5): 1667-1680.
- [37] Li B, Huang L, Lv X, et al. Study on temperature variation and pore structure evolution within coal under the effect of liquid nitrogen mass transfer[J]. *ACS omega*. 2021, 6(30): 19685-19694.
- [38] Cai C, Li G, Huang Z, et al. Rock pore structure damage due to freeze during liquid nitrogen fracturing [J]. *Arabian Journal for Science and Engineering*. 2014, 39(12): 9249-9257.
- [39] Cai C, Li G, Huang Z, et al. Experimental study of the effect of liquid nitrogen cooling on rock pore structure[J]. *Journal of natural gas science and engineering*. 2014, 21: 507-517.
- [40] Choi H, Lee D, Won J, et al. Influence of in-situ cryogenic freezing on thermal and mechanical characteristics of korean marine clay[J]. *KSCE Journal of Civil Engineering*. 2020, 24(11): 3501-3515.
- [41] Liu Y, Wang Q, Liu S, et al. Experimental investigation of the geotechnical properties and microstructure of lime-stabilized saline soils under freeze-thaw cycling[J]. *Cold regions science and technology*. 2019, 161: 32-42.
- [42] Quan X, Gong Y, Wang B, et al. Experimental study on the shear strength of Qinghai-Tibet clay under freeze-thaw cycles[J]. *Journal of Glaciology and Geocryology*. 2023, 45(3): 1016-1025.
- [43] Chang D, Liu J, Li X. A constitutive model with double yielding surfaces for silty sand after freeze-thaw cycles[J]. *Chinese Journal of Rock Mechanics and Engineering*. 2016, 35(3): 623-630.
- [44] Memon K R, Mahesar A A, Ali M, et al. Influence of Cryogenic liquid nitrogen on Petro-Physical Characteristics of Mancos Shale: An Experimental Investigation[J]. *Energy & fuels*. 2020, 34(2): 2160-2168.
- [45] Jin X, Gao J, Su C, et al. Influence of liquid nitrogen cryotherapy on mechanic properties of coal and constitutive model study[J]. *Energy sources*. Part A, Recovery, utilization, and environmental effects. 2019, 41(19): 2364-2376.
- [46] Qin L, Zhai C, Liu S, et al. Failure Mechanism of Coal after Cryogenic Freezing with Cyclic liquid nitrogen and Its Influences on Coalbed Methane Exploitation[J]. *Energy & fuels*. 2016, 30(10): 8567-8578.

- [47] Davidson G P, Nye J F. A photoelastic study of ice pressure in rock cracks[J]. *Cold regions science and technology*. 1985, 11(2): 141-153.
- [48] Li S, Tang D, Pan Z, et al. Characterization of the stress sensitivity of pores for different rank coals by nuclear magnetic resonance[J]. *Fuel*. 2013, 111: 746-754.
- [49] Cai Y, Xue Y, Dang F, et al. Effect of liquid nitrogen Cooling and Heating on Mechanical Properties and Acoustic Emission Characteristics of Coal[J]. *Geofluids*. 2023, 2023: 1-21.
- [50] Lu S. Experimental study on seepage characteristics and influencing factors of liquid nitrogen fracturing coal [D]. *Journal of China University of Mining & Technology*. 2021.
- [51] Zhai C, Qin L, Liu S, et al. Pore Structure in Coal: Pore Evolution after Cryogenic Freezing with Cyclic liquid nitrogen Injection and Its Implication on Coalbed Methane Extraction[J]. *Energy & Fuels*. 2016, 30(7): 6009-6020.
- [52] Chen S, Dou L, Zhang L, et al. Mechanism of Reducing the Bursting Liability of Coal using liquid nitrogen Cyclic Fracturing[J]. *Natural Resources Research*. 2023, 32(3): 1415-1433.
- [53] Qin L, Zhai C, Liu S, et al. Mechanical behavior and fracture spatial propagation of coal injected with liquid nitrogen under triaxial stress applied for coalbed methane recovery[J]. *Engineering Geology*. 2018, 233: 1-10.
- [54] Qin L, Zhai C, Liu S, et al. Changes in the petrophysical properties of coal subjected to liquid nitrogen freeze-thaw – A nuclear magnetic resonance investigation[J]. *Fuel (Guildford)*. 2017, 194: 102-114.
- [55] Qin L, Li S, Zhai C, et al. Changes in the pore structure of lignite after repeated cycles of liquid nitrogen freezing as determined by nitrogen adsorption and mercury intrusion[J]. *Fuel (Guildford)*. 2020, 267: 117214.
- [56] Ghobadi M H, Babazadeh R. Experimental Studies on the Effects of Cyclic Freezing–Thawing, Salt Crystallization, and Thermal Shock on the Physical and Mechanical Characteristics of Selected Sandstones[J]. *Rock Mechanics and Rock Engineering*. 2015, 48(3): 1001-1016.
- [57] Wang P, Xu J, Fang X, et al. Dynamic splitting tensile behaviors of red-sandstone subjected to repeated thermal shocks: Deterioration and micro-mechanism[J]. *Engineering Geology*. 2017, 223: 1-10.
- [58] Xu J, Zhai C, Liu S, et al. Feasibility investigation of cryogenic effect from liquid carbon dioxide multi cycle fracturing technology in coalbed methane recovery[J]. *Fuel*. 2017, 206: 371-380.
- [59] Xu J, Zhai C, Liu S, et al. Pore variation of three different metamorphic coals by multiple freezing-thawing cycles of liquid CO₂ injection for coalbed methane recovery[J]. *Fuel*. 2017, 208: 41-51.
- [60] Wang H, Fu X, Jian K, et al. Changes in coal pore structure and permeability during N₂ injection[J]. *Journal of Natural Gas Science and Engineering*. 2015, 27: 1234-1241.