EFFECT OF LIQUID NITROGEN PRE-TREATMENT ON THE FRACTURE BEHAVIOUR OF SHALE

by

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Liquid nitrogen enhancement has been successfully applied in petroleum extraction engineering. In order to explore its feasibility in shale gas production, the three-point bending experiments of shale were conducted before and after liquid nitrogen treatment in this paper. The results showed that the fracture toughness of shale decreased by about 18.8%, the wave velocity decreased by about 6%, and CT scans showed transgranular fracture occurring inside the shale. After destruction, the tortuosity of crack path significantly increases, and the fracture area increases, but applied energy demand reduced by 35%. It can be seen that the application of liquid nitrogen in shale reservoir transformation is beneficial for increasing the fracture volume of the reservoir, increasing the complexity of the fracture network, and reducing the design parameter requirements of fracturing equipment, thus saving costs.

Key word: *LN_2, fracture toughness, applied energy, shale, CT*

Introduction

Shale gas is mostly adsorbed and adsorbed in a free state in tight shale and its interlayers with extremely low permeability [1]. Therefore, the vast majority of shale reservoirs need to undergo hydraulic fracturing before they can be put into production [2]. Shale reservoirs have nanometer sized pore sizes and are rich in clay minerals, which can easily cause water lock and water sensitivity damage when in contact with water. This greatly reduces the effectiveness of hydraulic fracturing in reservoir modification. At the same time, hydraulic fracturing requires a large amount of water, which limits its large-scale application and promotion in water scarce areas. In addition, water-based fracturing fluids contain a large amount of chemical agents, which can easily cause water pollution [3]. Given the aforementioned issues, dehydration induced fracturing and enhanced permeability have become the inevitable path for shale gas extraction.

In recent years, scholars at home and abroad have been actively exploring new types of waterless fracturing technology and conducting corresponding basic theoretical research. Cai *et al.* [4] explored the application of liquid nitrogen jetting and fracturing in the transformation

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of deep oil and gas reservoirs. Qin *et al.* [5] analyzed the evolution of pore structure in lignite under liquid nitrogen cyclic freeze-thaw conditions using nitrogen adsorption and mercury intrusion tests. While Liu *et al.* [6] conducted numerical research on microwave thermal recovery of shale gas based on fully coupled electromagnetic, heat transfer, and multi-phase flow models. Su *et al.* [7] experimentally studied the influence of liquid nitrogen on the permeability evolution and cracking characteristics of coal. Hou *et al.* [8] explores the influence of liquid nitrogen cooling state on the mechanical properties and fracture characteristics of coal. These studies indicate that waterless fracturing technology carrying thermal shock effects has a good cracking and permeability enhancement effect.

Therefore, based on this, conducting mechanical experiments on liquid nitrogen induced fracturing of shale, exploring the possibility of liquid nitrogen enhanced permeability of shale reservoirs, and understanding the impact of liquid nitrogen on shale structure and mechanical damage characteristics have become the focus of research. To this end, a three-point bending test was conducted to obtain the changes in shale fracture toughness before and after liquid nitrogen treatment, and the evolution characteristics of crack paths were analyzed. It is expected to provide theoretical basis and technical guidance for the liquid nitrogen production increase of shale gas.

Material preparation and methods

The shale used in the laboratory comes from the Chongqing area. According to X-ray diffraction analysis results, the main mineral components of all shale are kaolinite and illite, accounting for about 48%, quartz accounting for about 27%, feldspar accounting for about 14%, organic matter accounting for about 3%, and others accounting for about 8%. In addition, the shale density was measured to be 2.46 g/cm^3. In this study, international standard specimens were used, with a diameter of 50 mm, an artificial seam length of approximately 12.5 mm, and an artificial seam width of less than 1 mm, as shown in fig. 1. In addition, a schematic diagram of three-point bending loading is shown in the figure.

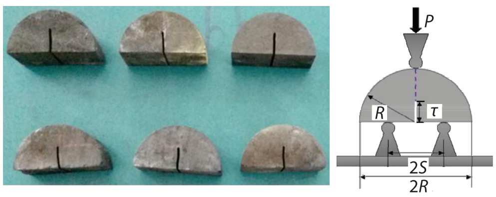

Figure 1. Shale specimens and their experimental loading schematic diagram

The specimens were divided into two groups, with each group containing three samples. These groups were the control group, the LN_2 group (frozen for half hour and then returned to room temperature). For the LN_2 group, three wave velocity measurements were taken before and after treatment at the same measuring point. The three-point bending test was carried out using displacement loading at a speed of 0.2 mm per minute, as illustrated in fig. 1. In addition, in order to observe the effect of LN_2 on the micro-structure of shale, a cylindrical sample with a diameter of 25 mm and a height of 50 mm was prepared and treated with LN_2 using the same method. The evolution of the micro-structure at the three positions before and after LN_2 treatment, as well as the upper, middle, and lower positions, was observed.

Wave velocity and fracture toughness

From fig. 2, it can be observed that the standard periodicity of the ultrasonic waveform of shale before and after LN_2 is lost, and the duration of a single cycle increases. As the cycle increases, the amplitude of the ultrasonic wave gradually decreases. This indicates that the absorption capacity of shale for ultrasonic energy is significantly enhanced after LN_2 treatment, resulting in waveform distortion when sound waves penetrate the shale structure. This suggests that the shale structure has changed after LN_2 treatment. According to the characteristics of ultrasound, this is inevitably due to an increase in the looseness of the structure, which results in structural damage.

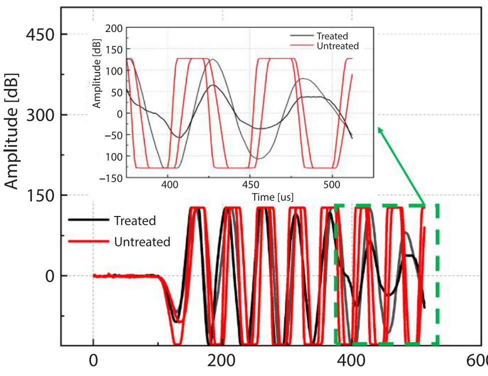

Figure 2. Changes in ultrasonic waveform of shale before and after LN_2 treatment

The decrease in wave velocity is the most direct reflection of the initiation and propagation of microcracks in rocks. As shown in fig. 3, compared with untreated shale, the maximum decrease in wave velocity after treatment is 7.8%, the minimum decrease is 3.2%, and the average decrease is about 6.2%. The main reason is that the large temperature difference of LN_2 leads to the shrinkage and tensile damage of shale matrix, especially for shale, which is a layered rock. The weak bonding surface between layers has very low tensile performance, and under the action of large temperature difference, it is easy to become the source of crack initiation and propagation.

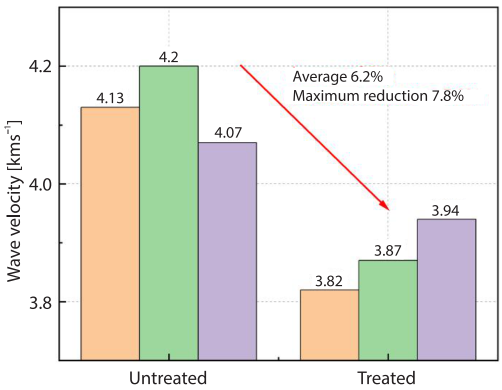

Figure 3. Wave velocity of shale before and after LN_2 treatment

The fracture toughness is one of the most important indicators in rock mechanics. It is commonly used to measure the ability of rocks to resist crack initiation and propagation under external forces, which is of great significance for analyzing and evaluating the engineering mechanical properties of rock reservoirs and the brittleness or toughness of rocks. The fracture toughness of rocks is a comprehensive indicator that reflects the ability of rock materials to resist crack propagation. It is not only related to the geological and mineral composition of rocks, but also influenced by the internal micro-structure of rocks [8]:

$$K_I = \frac{P}{2RB}\sqrt{\pi\tau}Y_I \tag{1}$$

where P is the applied load, R – the sample radius, B – the sample thickness, τ – the notch length, and Y_I – the geometry factor for mode I crack. The value of Y_I depends on parameters including the distance between the two supports, S, sample radius, R, and notch length, τ. According to the result in [8], the geometric factor for the mode I crack in this experiment is confirmed as 4.035.

Using eq. (1), the fracture toughness can be calculated as shown in fig. 4. It can be seen that the fracture toughness of the pre-treated shale is reduced, indicating that pre-treatment can reduce the difficulty of crack initiation. Specifically, compared with the untreated, the shale samples treated by LN_2 decreased by about 18.8% averagely. It can be seen that LN_2 treatment is beneficial for weakening the crack resistance of shale, which is very important in shale gas extraction engineering. The reduction of the difficulty of reservoir fracturing directly affects the effectiveness of reservoir transformation, accelerates the formation of shale fracture network, and also helps to reduce equipment requirements and save costs.

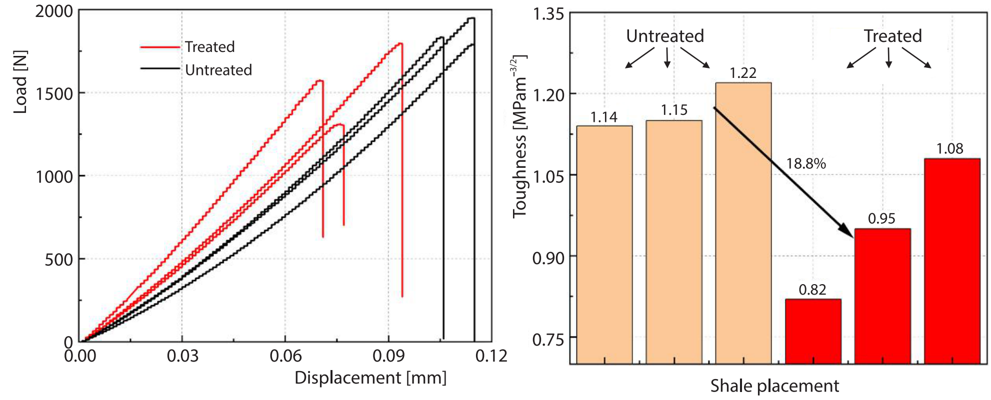

Figure 4. Changes in fracture toughness of shale under LN_2 treatments

Changes of fracture pathways and applied energy of shale treated with LN_2

The change in crack path can intuitively reflect the damage effect of LN_2 on shale. As shown in fig. 5, the crack path of untreated shale after loading failure is relatively straight, which is a typical tensile failure. The tortuosity of the crack path in the processed shale significantly increases, and the initial crack initiation direction deviates significantly.

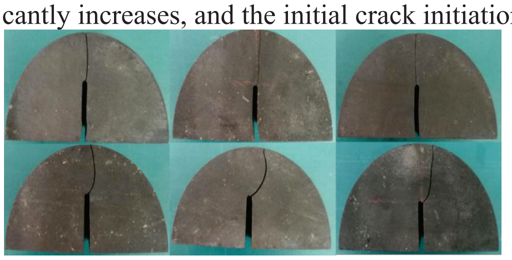

Figure 5. Fracture paths of shale before and after LN_2 treatment

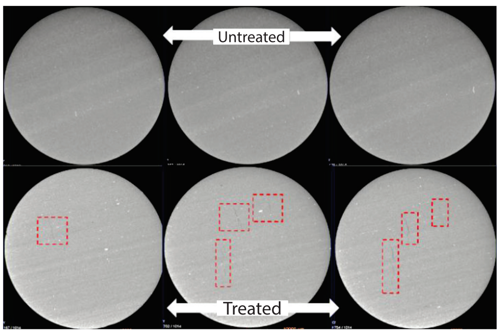

Figure 6. Shale CT scan images before and after LN_2 treatment

In addition, CT scan images before and after shale processing showed obvious microcracks inside the processed shale, and the crack direction was perpendicular to the bedding direction, indicating that the large temperature difference of LN_2 caused tensile deformation inside the shale matrix, ultimately leading to transgranular fracture inside the matrix, as shown in fig. 6. The existence of microcracks makes the distribution of internal damage in shale matrix more widespread, especially the presence of cutting tips, which can lead to stress concentration and exacerbate the randomness of micro damage distribution at cutting tips. These micro damages, in the process of participating in the formation of crack paths, change the initial direction of crack initiation. During the formation of crack paths, the direction of micro damage aggregation always expands and advances, but the aggregation of micro damages is random, ultimately leading to an increase in the curvature of the crack path, as shown in fig. 5.

Discussion

Fracture toughness is an indicator of a rock's ability to resist crack propagation, while fracture energy represents the energy absorbed during the rock's fracture process [7]. The total applied energy is equal to the work done on the fracture surface during the crack propagation process. The work done on a rock sample can be calculated by multiplying the applied load by the corresponding displacement. The calculation method for applied energy to rock samples is shown in fig. 7.

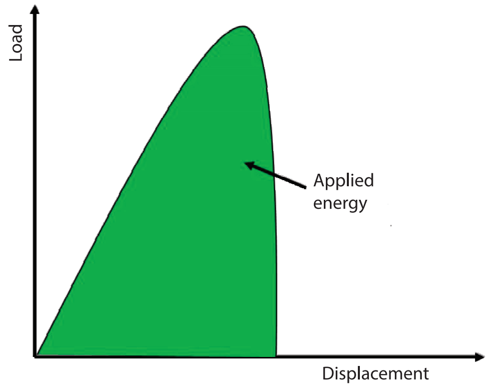

Figure 7. Schematic diagram of the applied energy calculation

The applied energy is the energy required to break the rock. Figure 8 shows the relationship between applied energy and LN_2 pretreatment. The applied energy is not only influenced by the internal micro-structure characteristics of shale, but also closely related to its fracture propagation mechanism, including tensile failure, shear failure, or a combination of both. In theory, the applied energy is mainly used to form new fracture surfaces, so larger fracture surfaces typically require more energy. However, as shown in fig. 8, after LN_2 treatment, the fracture path and fracture surface of shale increased, but the required application energy decreased by about 35%. This phenomenon indicates that the involvement of internal micro damage significantly reduces the energy required for matrix fracture during the formation of shale fracture surfaces, and CT scan results also support this conclusion. In addition, considering that shale contains a large number of nanopores, the large temperature difference of LN_2 may lead to stress concentration at the pore edge, resulting in crack initiation at the pore edge. The aggregation of these damages may appear externally in the form of microcracks, as further confirmed by CT scan results. Under the stimulation of LN_2, shale reservoirs exhibit significant fracture and damage effects, and can form relatively tortuous crack paths, forming complex crack networks that effectively enhance reservoir permeability and increase shale gas production.

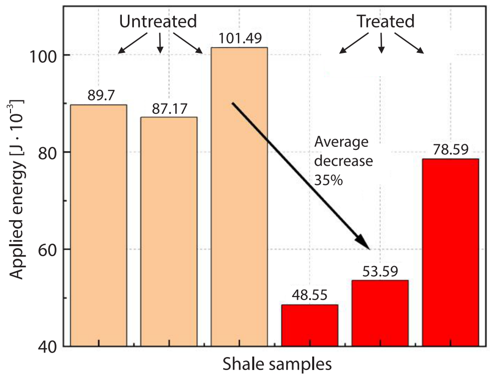

Figure 8. Applied energy of shale before and after LN_2 treatment

Conclusion

A series of shale I-model fracture tests were conducted under LN_2 treatment. It was found that LN_2 stimulation can reduce shale wave velocity and fracture toughness, the average reductions were 6.2% and 18.8%, respectively. In addition, it has been found that large temperature differences in LN_2 can induce transgranular fracture of shale matrix, increase the randomness of initial damage distribution, and thus lead to an increase in the tortuosity of shale crack paths and an increase in fracture area. The application analysis shows that the application can be reduced by about 35% after LN_2 treatment. From this, it can be seen that LN_2 stimulation is beneficial for the development of micro damage in shale matrix, making crack propagation easier and low energy consumption, which is very advantageous for enhancing the permeability of shale reservoirs.

Nomenclature

B – thickness, [m]
K_I – toughness, [MPam$^{2/3}$]
P – applied load, [N]
R – radius, [m]
Y_I – geometry factor, [–]

Greek symbol

τ – notch length, [m]

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