

## PRE-TREATMENT INFLUENCE OF LIQUID NITROGEN AND MICROWAVE ON THE MODE I FRACTURE CHARACTERISTICS OF GRANITE

by

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*Microwave and liquid nitrogen have shown great potential applications in the mining, geological, petroleum, and particularly metal mining fields. In order to further promote their application in rock tunneling engineering, this paper conducted a series of I-model fracture tests on hard rock (granite) to explore the impact of liquid nitrogen, microwave, and liquid nitrogen and microwave pretreatment on the fracture characteristics of hard rock. The results showed that compared with the control group, all pretreatment groups had varying degrees of reduction in wave speed and fracture toughness, especially the microwave and liquid nitrogen group.*

Key word: microwave, LN<sub>2</sub>, fracture toughness, roughness, hard rock

### Introduction

The rock-breaking technology that carries cold and hot impact energy has been successfully applied in various fields, including soil and rock tunnel excavation, enhanced permeability in unconventional oil and gas reservoirs, and mining operations [1, 2]. Liquid nitrogen and microwave, as cold and hot impact mediums, have attracted significant attention due to their excellent rock-breaking abilities. Liquid nitrogen, at a temperature of  $-196\text{ }^{\circ}\text{C}$  under normal pressure, has a gas expansion ratio of 1:696 [3]. The instantaneous contact between liquid nitrogen and rocks generates a thermal-pressure coupling effect and undergoes phase change, which weakens the mechanical properties of the rocks and promotes the generation and development of internal pores and fractures in the rock matrix, thereby enhancing permeability [4, 5]. Liquid nitrogen has achieved remarkable results in the practice of unconventional oil and gas development [6]. Compared to conventional heating, microwave radiation has the characteristic of selective heating. It can quickly raise the temperature of rocks, leading to non-co-ordinated deformations between rock mineral particles, promoting the generation and development of internal pores and fractures in rocks, effectively reducing the mechanical properties of the rocks, and improving permeability [7, 8]. Microwave has also been applied in the development of unconventional oil and gas fields such as shale oil and coalbed methane [9, 10].

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In this study, a series of type I hard rock fracture tests were conducted to investigate the influence of liquid nitrogen, microwave, and microwave and liquid nitrogen pre-treatment on the wave velocity, fracture toughness, and surface roughness of hard rocks. The research results will provide new technical solutions and scientific guidance for hard rock tunnel engineering excavation.

### Material preparation and methods

The granite used in the experiment was taken from Rizhao City, Shandong Province. According to the X-ray diffraction (XRD) analysis results, the main mineral components of the granite used are quartz (25%), plagioclase (39%), potassium feldspar (22%), and biotite (14%). The average density of the granite is  $2654 \text{ kg/m}^3$ . In the three-point bending tests of this study, semicircular specimens with a diameter of 76 mm and a thickness of 30 mm were used. Artificial fractures with a length of 14 mm and a width of less than 1 mm were introduced on the specimens, as shown in fig. 1. The machining precision of all specimens meets the ISRM standard.

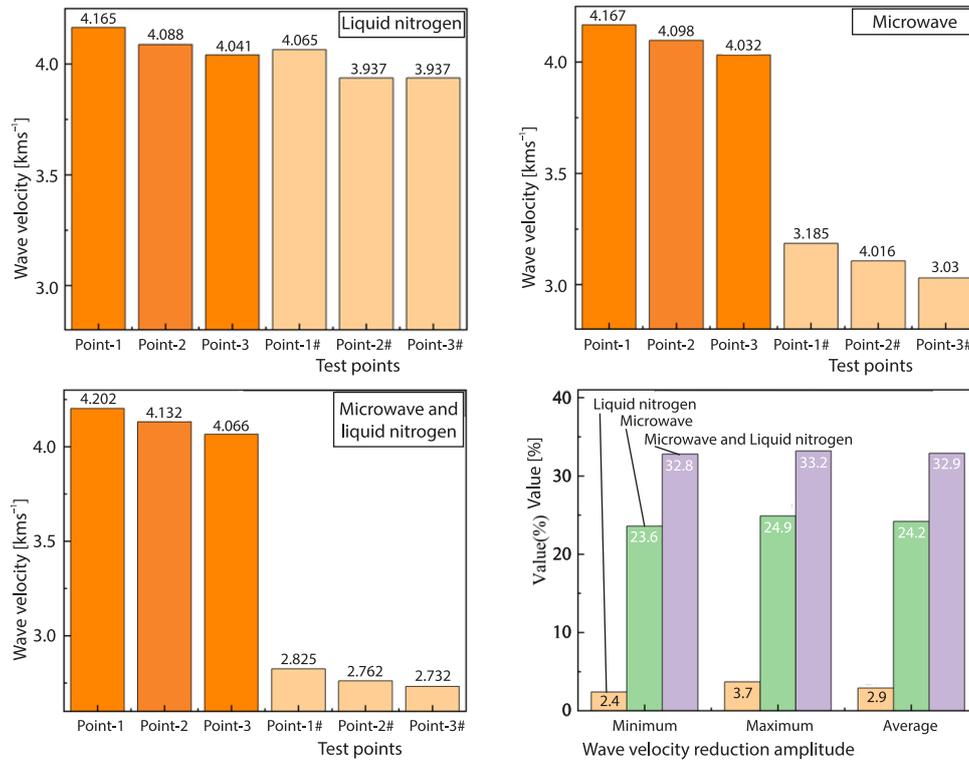


Figure 1. Granite specimens and their experimental loading schematic diagram

The specimens were divided into four groups, with each group containing two samples. These groups were the control group, the liquid nitrogen group (frozen for half hour and then returned to room temperature), the microwave group (heated for 10 minutes and then returned to room temperature), and the microwave and liquid nitrogen group (microwaved for 10 minutes, followed by liquid nitrogen treatment for half hour and then returned to room temperature). For each 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 0.3 mm per minute, as illustrated in fig. 1. After the experiment was completed, the fracture surface of the specimen was scanned using the VR-5000 3-D profiler, and the roughness of the crack was analyzed.

### Wave velocity and fracture toughness

From fig. 2, it can be observed that the wave velocities of granite decreased after pre-treatment with liquid nitrogen, microwave, and microwave and liquid nitrogen. Specifically, compared to untreated samples, the maximum reduction in wave velocity after liquid nitrogen pre-treatment was 3.7%, the minimum reduction was 2.4%, with an average decrease of 2.9%. After microwave pre-treatment, the maximum reduction in wave velocity was 24.9%, the minimum reduction was 23.6%, with an average decrease of 24.2%. For microwave and liquid nitrogen pre-treatment, the maximum reduction in wave velocity was 33.2%, the minimum reduction was 32.8%, with an average decrease of 32.9%.

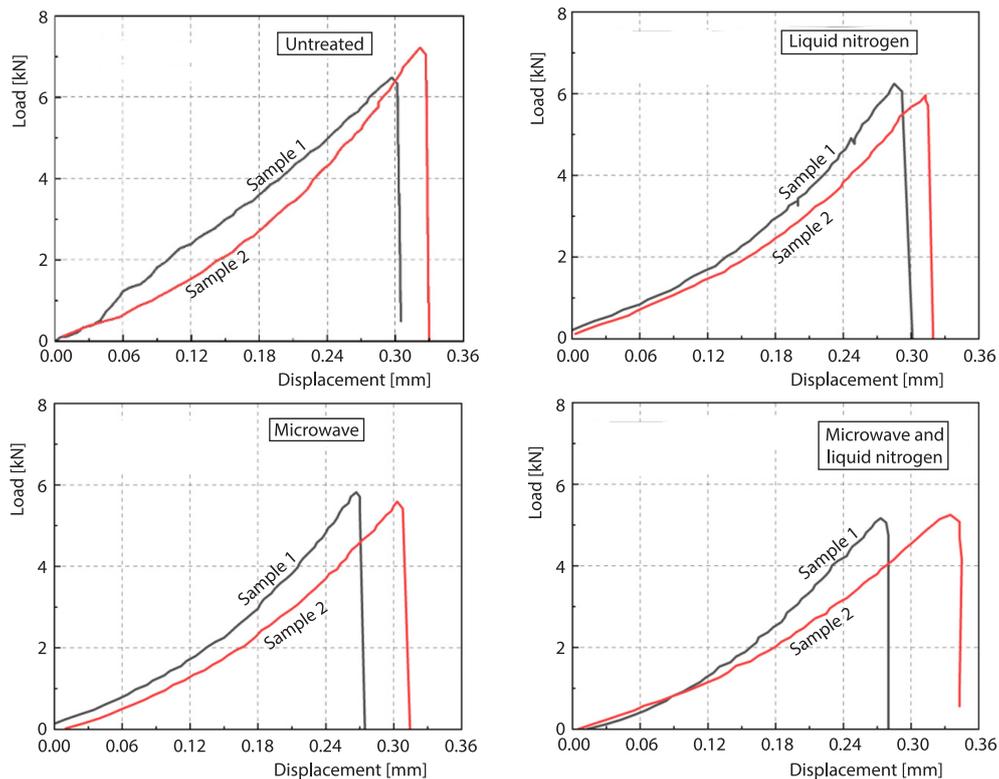


**Figure 2. Influence of different pre-treatments on the wave velocity of granite**

The reduction in wave velocity suggests that there is pore development, micro-crack initiation, crack propagation and other damage in the granite matrix. Through comparison, it was found that the damage to granite was minimized after liquid nitrogen treatment, followed by microwave treatment, and was greatest after microwave and liquid nitrogen treatment. Moreover, in terms of wave velocity reduction, the damage caused by their combined action was greater than the sum of the damage caused by them separately. The reason for this is that the high temperature generated by microwave heating and the low temperature of liquid nitrogen create a large temperature gradient. Under this gradient, there is a large difference in thermal properties between mineral particles inside the granite matrix, which inevitably results in large temperature stresses and strains. When the strain or stress exceeds the bearing limit of the matrix, it will lead to pore development, micro-crack initiation and propagation.

Comparing the load-displacement curves under different pretreatments, it can be observed that the compacting stage is significantly increased in all groups compared to the untreated group, especially in the microwave and liquid nitrogen group from fig. 3. This indirectly indicates the development of internal pores and cracks in the granite after pretreatment, leading to an increased compacting stage. In the elastic stage, the slope of the curves is arranged untreated group > liquid nitrogen group > microwave group > microwave and liquid nitrogen group, indicating that pretreatment weakens the stress feedback capability of the granite, especially in the microwave and liquid nitrogen pretreatment. No obvious yielding stage was found in the first three groups, while a slight yielding phenomenon appeared in the microwave and liquid nitrogen group. In the failure stage, the peak load of the pretreated groups showed vary-

ing degrees of decrease, and all exhibited typical brittle fracture, particularly in the microwave and liquid nitrogen group, where the peak load decreased most significantly. The maximum displacement at the peak also exhibited weak ductile fracture characteristics.



**Figure 3. Load-displacement curves of granite under different pre-treatments conditions**

Fracture toughness is one of the most important indexes of rock mechanics. It is usually used to measure the ability of rock to resist crack initiation and propagation under external forces and is of great significance for analyzing and evaluating the engineering mechanical properties and factorability of rock reservoirs. The calculation formula can be expressed [11]:

$$K_I = \frac{P}{2RB} \sqrt{\pi\tau} Y_I \quad (1)$$

where  $P$  is the applied load,  $R$  – the sample radius,  $B$  – the sample thickness,  $\tau$  – 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,  $\tau$ . According to the result in [11], 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 granite is reduced to varying degrees, indicating that pre-treatment can reduce the difficulty of crack initiation. Specifically, compared with the untreated group, the liquid nitrogen group decreased by about 11%, the microwave group decreased by 16.7%, and the microwave and liquid nitrogen group is decreased by 23.9%. Compared with the liquid nitrogen group, the microwave group is decreased by 6.5%, and the microwave and liquid nitrogen group decreased by 14.6%. Compared with the microwave

group, the microwave and liquid nitrogen group is decreased by 8.7%. It can be seen that the weakening effect of microwave coupling with liquid nitrogen on granite fracture toughness is the strongest, and the combined single treatment effect is significant. It is recommended to use both methods in a cyclic manner for pre-treatment of hard rock in relevant excavation projects, which will greatly improve excavation efficiency and reduce mechanical equipment loss.

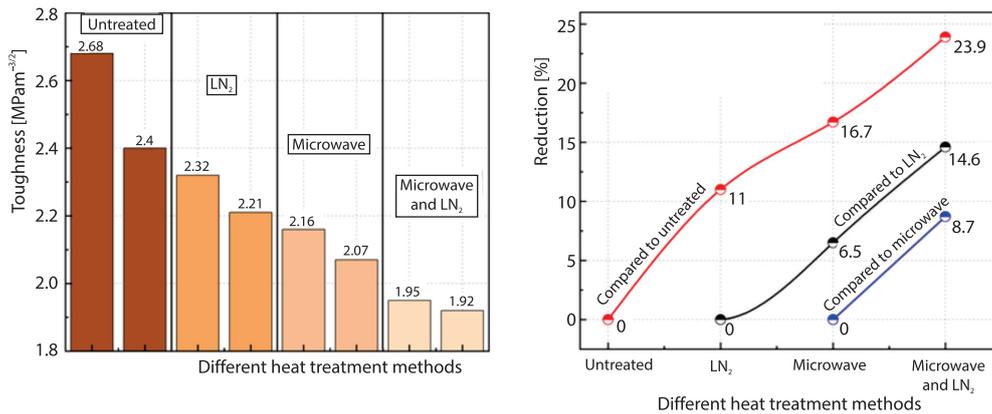


Figure 4. Changes in fracture toughness of granite under different pre-treatments conditions

### Characteristics of fracture surface roughness

The roughness of the cross-section has a significant impact on the efficiency of TBM excavation. Therefore, it is necessary to investigate the roughness transformation of the cross-section under different pre-treatment methods. In order to more comprehensively reflect the roughness of the cross-section, the diagonal of the approximate rectangular cross-section is taken to reflect its roughness characteristics. In this study, the VR-5000 automatic 3-D profilometer was used to obtain the digital information of the diagonal, as shown in fig. 5. From the curve shape, it can be seen that compared with the untreated group, the vertical distance between the peak and valley of the curve increases after pre-treatment, and the complexity of the curve shape is enhanced, especially in the microwave and liquid nitrogen group.

In order to further quantify and analyze the change in fracture surface roughness, two roughness evaluation indicators  $R_a$  and  $R_z$  were introduced. The  $R_a$  is the arithmetic mean of the absolute deviation of the contour within the sampling length,  $L$ . The  $R_z$  is the sum of the average height of the five largest contour peaks and the average depth of the five largest contour valleys within the sampling length. The numerical values of  $R_a$  and  $R_z$  were calculated and obtained as shown in fig. 6. It can be observed that the order of magnitude for both  $R_a$  and  $R_z$  is untreated group < liquid nitrogen group < microwave group < microwave and liquid nitrogen group. Specifically, compared to the untreated group, the increase in  $R_a$  and  $R_z$  for the liquid nitrogen group is 7.2% and 8.2%, respectively. For the microwave group, the increase is 26% and 30.2%, and for the microwave and liquid nitrogen group, the increase is 36.7% and 36.8%. The increase in roughness, which corresponds to a greater degree of surface irregularities, inevitably leads to increased stress concentration and stress concentration area when the tool contacts the rock during excavation, resulting in increased rock fragmentation. Based on the previous research sections, it can be concluded that the combined action of microwave and liquid nitrogen can effectively degrade hard rock, increase the roughness of the cross-section, and further enhance excavation efficiency.

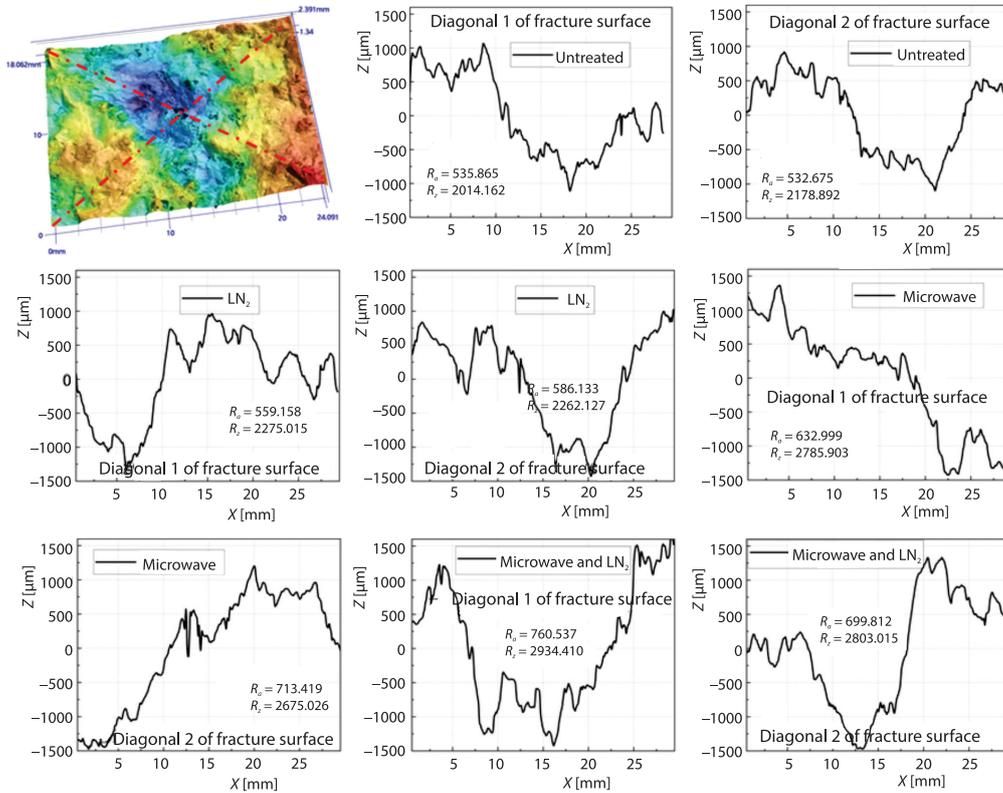


Figure 5. Diagonal Morphology of granite fracture surface under different pre-treatment conditions

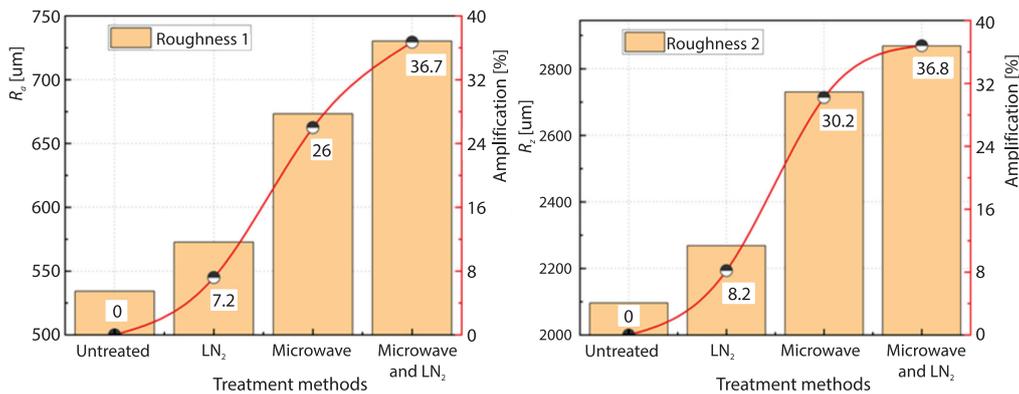


Figure 6. Diagonal roughness  $R_a$  and  $R_z$  of granite fracture surface under different pre-treatment conditions

### Conclusion

A series of granite I model fracture tests were conducted under different pre-treatment conditions in this work. It was found that liquid nitrogen and microwave pre-treatments can effectively reduce granite wave velocity and fracture toughness, and increase cross-sectional roughness. This study shows that the combined action of liquid nitrogen and microwave can ef-

fectively improve the construction efficiency in hard rock excavation projects, as well as reduce tool wear and maintenance costs.

### Nomenclature

$B$ – thickness, [m]	$R_a$ – roughness index, [ $\mu\text{m}$ ]
$I$ – geometry factor, [–]	$S$ – distance, [m]
$K_I$ – toughness, [ $\text{MPam}^{-2/3}$ ]	Greek symbol
$P$ – applied load, [N]	$\tau$ – notch length, [m]
$R$ – radius, [m]	

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