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Research Article

Influence of Freeze-Thaw Cycles on Engineering Properties of Tonalite: Examples from China

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The deterioration of the physical and mechanical properties of tonalites subjected to freeze-thaw cycling under three different temperature ranges was explored using several experimental techniques. Uniaxial compression and three-point bending tests were conducted on untreated and treated tonalite specimens. Clear decreases in uniaxial compressive strength (UCS), Young's modulus, and fracture toughness were observed in tonalite specimens with frost damage. Although Young's modulus and fracture toughness did not show clear decreases as the minimum temperature of the freeze-thaw cycle decreased from −30°C to −50°C, the UCS decreased almost linearly. The macromechanical characteristics of the tonalites can be explained by changes in mineral content and microstructure. The intensity of X-ray diffraction (XRD) peaks of minerals in tonalites that had not been freeze-thaw cycled were approximately 10 to 20 times higher than the peaks for the specimens subjected to freeze-thaw cycling, implying that the internal structure of tonalite is less compact after frost damage. The microstructures of the tonalite specimens were also examined using scanning electron microscopy (SEM). Increased amounts of fragmentation and breaking of structural planes were observed as the minimum temperature of the freeze-thaw cycle decreased.

1. Introduction

Weathering, for example from frost damage, heat exposure, or chemical processes [1–3], is common in nature. The deterioration of rocks due to weathering has recently attracted increasing concern from both academic researchers and engineers. In China, granitic rock is an important construction material in civil engineering, mining engineering, tunnel construction, and railway engineering. Granitic rock masses occupy approximately 9% of the land area of China, or up to 800000 square kilometres. There are many tunnels in northeastern and northwestern China, some of which are located in cold regions and experience different degrees of damage resulting from frost action [4]. Concerns regarding the effect of cooling and heating on rocks has also arisen in many engineering projects, such as the storage of liquefied natural gas at the extremely low temperature of −160°C [5].

An understanding of rock properties resulting from freeze-thaw cycling is valuable for engineers working on railway engineering, tunnelling, and underground construction in cold regions. When used as a construction material, tonalite is required to meet high-quality standards to ensure optimum behaviour under all conditions. Cold regions account for approximately 43% of the land area of China. Recently, weathering induced by the action of freeze-thaw cycles with different temperatures and cycle count has been extensively studied [3, 6, 7]. Compared with other weathering processes, freeze-thaw cycling can lead to a high degree of rock deterioration [8]. When porous brittle material such as rock falls below 0°C, the liquid in micropores can expand by 7% to 9%, concentrating tensile stresses and generating cracks in areas adjacent to the micropores [8–10]. When the rock mass returns to higher temperatures, the cracks become filled with liquid, which further exacerbates the deterioration of the rock mass [9, 11–13]. The effect of freeze-thaw cycling on the physical properties of rocks, including mass, density, P-wave velocity, porosity, and Young's Modulus, has previously been studied in [8, 14–17]. Extensive investigations into the mechanical properties of various rocks subjected to freeze-thaw cycling have examined uniaxial and triaxial compressive strength, tensile strength, and deformation characteristics [18–22]. However, the damage mechanism associated with freeze-thaw cycling in granitic rocks, i.e., ice crystallization cracking, is not fully understood, as it depends on a number of factors including temperature range, frequency of freeze-thaw cycles, applied stress, and water composition [23, 24].

The physicomechanical behaviours of granitic rocks under macroscales and microscales are different but related. Mineralogy, texture, structure, and weathering decide the mechanical properties of granitic rock. However, even when possessing the same mineral composition, the mechanical properties of rocks may vary [25]. Freeze-thaw cycles will affect the microstructural characteristics [26], grain boundaries [27], mineral shape, and spatial arrangement [28] of granite. The effects of weathering on rocks can be studied by examining mineral composition, phase transitions, and microstructures. X-ray CT imaging has been used to measure crack openings [29, 30], but to date, there has been limited research into changes in the microstructure and mineral composition of rocks subjected to freeze-thaw cycling. Changes in mineral composition and diffraction intensity of tonalite induced by freeze-thaw cycling can be identified using X-ray diffraction (XRD). This is an effective method to analyse and estimate the structural degradation of minerals at a microscale. The physical deterioration of minerals as a result of freeze-thaw cycling can be observed directly using scanning electron microscopy (SEM). In this study, SEM and XRD have been used to investigate rock properties on a mesoscale.

The main objective of this research is to investigate the deterioration of physical and mechanical properties of tonalite resulting from freeze-thaw cycling under different temperature ranges. To this end, uniaxial compression and three-point bending tests were conducted on tonalite specimens, and XRD and SEM were used to explore changes in mineral composition and microstructure resulting from freeze-thaw cycling. The findings of this study regarding the effects of mineral composition and microstructure on the mechanical properties of tonalite are summarized.

2. Geologic Background

The rock mass of Nan'an city is the product of late Mesozoic magmatism, and with an age of 135–136 Ma. Figure 1 shows the location of the outcrop where tonalite samples were taken. The geologic structure of the area surrounding Nan'an city is shown in Figure 2. The granitic rock in this region belongs to magmatic rock and has a fine-grained texture and massive structure. The primary minerals are quartz, potassium feldspar, plagioclase, and biotite. The quartz crystals have granular crystals. The majority of potassium feldspar has a hypidiomorphic tabular shape, and Carlsbad twinning can be observed under a microscope. Biotite exhibits a brown colour and eminent cleavage.

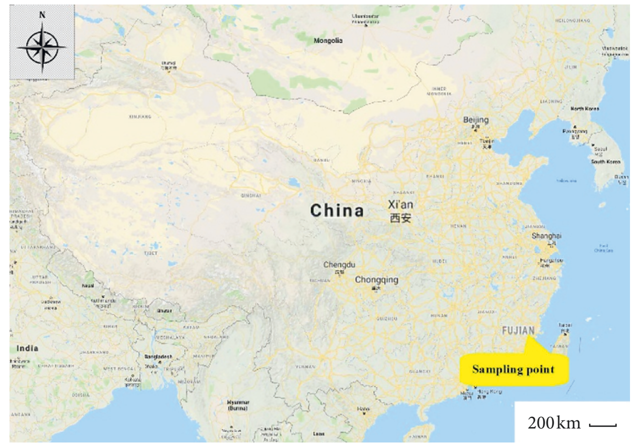


Figure 1: Location of outcrop from which tonalite samples were taken.

3. Experimental Testing

3.1. Sample Preparation. For uniaxial compressive strength (UCS) tests, the cylindrical specimens were cored perpendicular to the bedding plane from a depth of 2 m and cut to a height of 100 mm (Figure 3). The specimens were then processed to a diameter of 50 mm according to International Society for Rock Mechanics (ISRM) standards. Twenty specimens were prepared for UCS tests.

Single-edge-notched (SEN) specimens shown in Figure 4 were used for the three-point bending test, with a geometry shown in Figure 5. The length, height, and thickness of the SEN specimens were 250 mm, 50 mm, and 50 mm, respectively. In each of the specimens, a preprepared crack with a depth of 26.5 mm was introduced at a location midway between the clamps (Figure 5). Sixteen specimens were prepared for the three-point bending test.

3.2. Physical Properties. The physical properties were determined using standard methods. The recommended ISRM procedures for sample preparation and measurement were used as follows:

(1) Dry weight, dry density, and saturated density determined by saturation and calliper method

(2) Pore volume values and porosity obtained by water saturation

The results of these measurements are listed in Table 1.

3.3. Freeze-Thaw Tests. The selection of freeze-thaw cycle parameters does not have a global standard. Most of the research has employed freezing and thawing times of 4 hours each, which we follow here. The tonalite specimens were placed in a WGD-501 freeze-thaw device and subjected to freeze-thaw cycling. The specimens were divided into three groups: in all cases, the identical maximum temperature (20°C) was used, but minimum temperatures varied between groups (−30°C, −40°C and −50°C). The temperature settings used during freeze-thaw cycling are shown in Figure 6. There

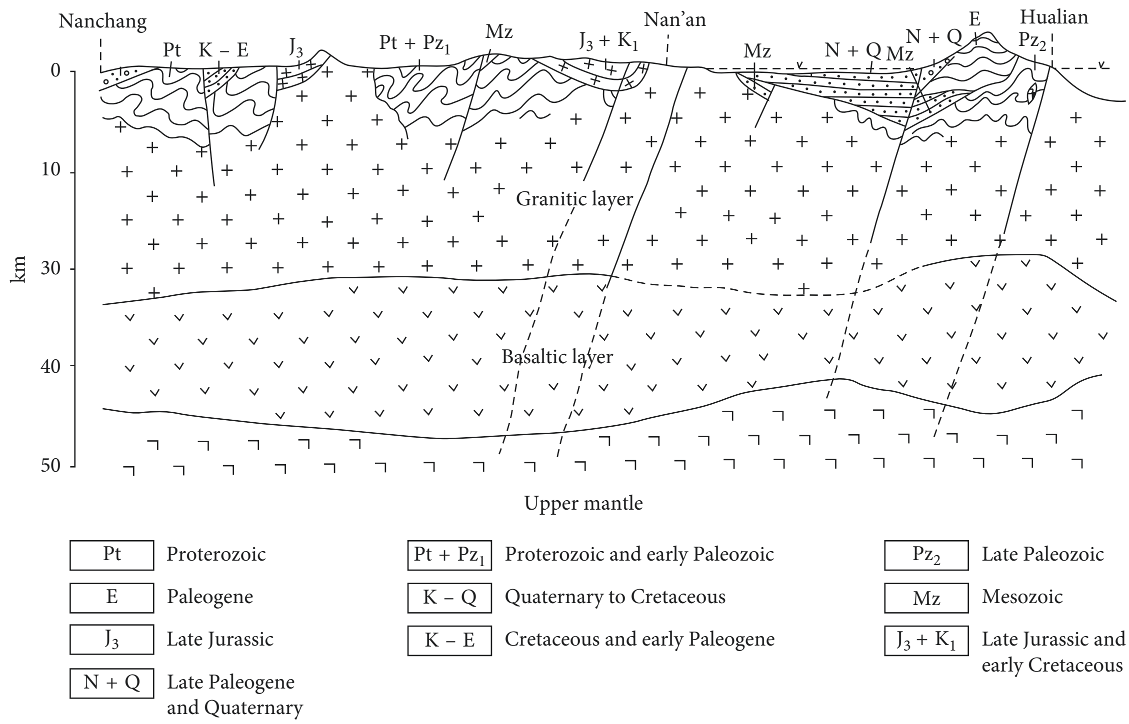


Figure 2: The geologic structure of the area around Nan'an city.

Figure 3: Tonalite specimen for uniaxial compression test.

were four stages to each freeze-thaw cycle: (1) the temperature was decreased from 20°C to the minimum temperature over 30 minutes; (2) the temperature was maintained at the minimum temperature for 4 hours; (3) the temperature was increased from the minimum temperature to 20°C over 30 minutes; and (4) the temperature was maintained at 20°C for 4 hours. Each freeze-thaw cycle therefore lasted 9 hours. This heating-cooling activity was repeated for 30 cycles. For all groups, the durations of stages (1) and (3) were 30 minutes, so the rate of temperature change varied depending on the minimum temperature of the cycle. The freeze-thaw cycle was set to 30 times.

3.4. XRD and SEM Test. The mineral content before and after freeze-thaw cycling was determined using an X-ray diffractometer (Bruker/D8 Advance). Five specimens in each temperature group were prepared for XRD analysis.

Figure 4: Tonalite specimen for three-point bending test.

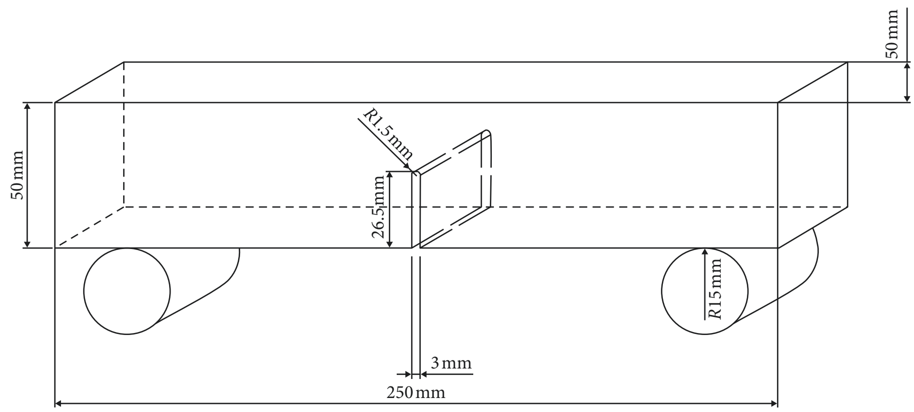


Figure 5: Geometry of three-point bending test specimens.

Table 1: Original properties of tonalite.

Dry weight (g)	Dry density (g/cm^3)	Saturated density (g/cm^3)	Saturation moisture content (%)	Porosity (%)
538.13	2.740	2.744	0.083	0.23

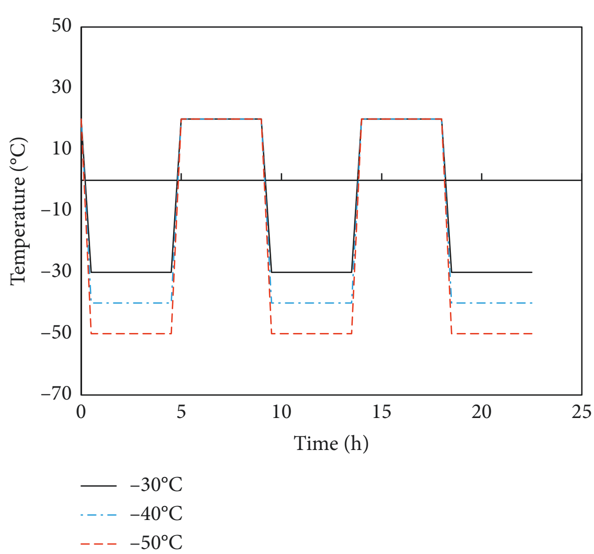


Figure 6: Temperature variation during freeze-thaw cycling.

Sample microstructures were observed using the Tescan Vega 3 scanning electron microscope, and 5 specimens in each temperature group were prepared for SEM analysis.

3.5. Uniaxial Compression Tests. Uniaxial compression tests were conducted using an electrohydraulic servo compression test machine manufactured by the Xi-an LETRY Testing

Table 2: Mineral composition (%) of the tonalite samples.

Plagioclase	Mica	Quartz	Potassium feldspar	Kaolinite	Amphiboles	Unknown
45–50	25–30	15–20	3–5	2	2	1

Machine Company, with a load capacity of 2000 kN. Five specimens in each temperature group were prepared for uniaxial compression testing. After freeze-thaw cycling, uniaxial compression tests were performed under load control at a loading rate of 0.5 kN/s until failure.

3.6. Three-Point Bending Tests. Three-point bending tests were conducted using a SANS electrohydraulic servo compression test machine with a load capacity of 2000 kN. Four specimens in each temperature group were analysed. These tests were performed under load control at a loading rate of 50 N/s until failure.

4. Results and Analysis

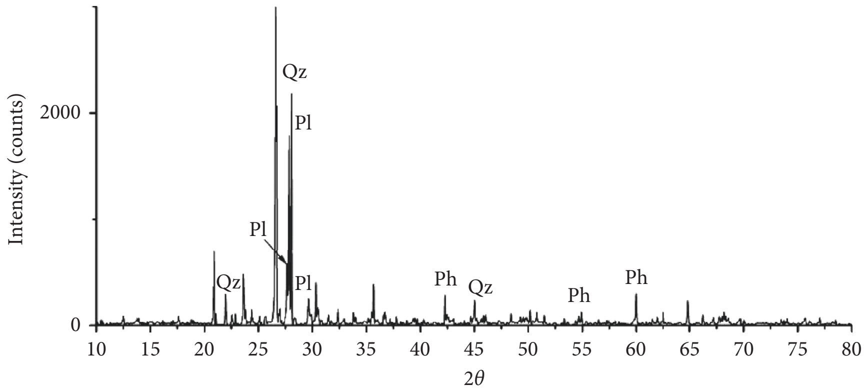
4.1. Tonalite Mineral Composition. The mineral composition of tonalite specimens without treatment was determined using XRD analysis (see Table 2). The results were analysed by the Microspectrum Technology Company and show that plagioclase, mica, and quartz are the three main mineral categories in the material studied.

Diffraction peak pattern results for the tonalite before and after freeze-thaw cycling are shown in Figure 7. It is shown that the main mineral composition (plagioclase, phlogopite, and quartz) does not change obviously before and after freeze-thaw cycles; however, peak intensities change. He [31] reported that the intensity of a peak is proportional to the crystallinity of the mineral and reflects the integrity of the mineral's internal structure. According to the data in Figure 6, the intensities prior to freeze-thaw cycling are approximately 10 to 20 times greater than those values recorded after freeze-thaw cycling, implying that the mineral components have less compact internal structure following freeze-thaw cycling.

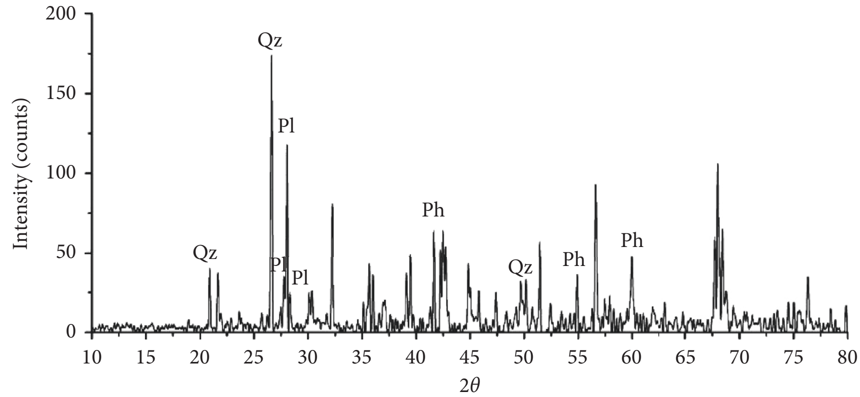
4.2. Stress-Strain Curves. Axial stress-strain curves obtained from tonalite specimens with and without exposure to freeze-thaw cycling are shown in Figure 8. The curves corresponding to the samples that have been subjected to freeze-thaw cycling are similar, whereas the curve corresponding to the natural tonalite specimen is quite different. The figure indicates that a significant decrease in the uniaxial compressive strength (UCS) occurs as a result of freeze-thaw cycling, and as the minimum temperature of the freeze-thaw cycle decreases, the stress-strain response moves further away from the reference curve. In addition, freeze-thaw cycling greatly reduces the strain at UCS, as the untreated tonalite specimens had much higher strain at failure compared to the samples that had been exposed to freeze-thaw cycling. However, the maximum strain of the freeze-thaw cycled specimens does not change significantly with the minimum temperature of the cycle.

4.3. Uniaxial Compressive Strength (UCS). The uniaxial compressive strength (UCS) of tonalite with and without exposure to freeze-thaw cycling is plotted as a function of minimum cycle temperature in Figure 9. Although there is variability in the UCS measurements for each temperature, the overall trend indicates that the UCS declines almost linearly with a decrease in the minimum temperature of the freeze-thaw cycle. Frost damage in rock is primarily caused by the 7% to 9% volumetric expansion of water as it changes to ice [22, 32]. Considering only this mechanism, the UCS of specimens subjected to freeze-thaw cycling should be the same irrespective of the minimum cycle temperature. Therefore, it can be speculated that there must be an alternative explanation for the observed deterioration in properties. Figure 10 shows the relationship between the diffraction intensity of minerals and the UCS of tonalite specimens. The diffraction intensity of quartz and plagioclase, which has similar trend as the UCS of tonalite specimens, decreases with decreasing freeze-thaw minimum temperature. Compared to the initial state, the intensity of quartz peaks decreases by 94.3%, 97.5%, and 97.7% after exposure to −30°C, −40°C, and −50°C freeze-thaw cycling, respectively. Compared to the initial state, the intensity of plagioclase peaks decreases by 94.6%, 98.3%, and 98.1% after exposure to −30°C, −40°C, and −50°C freeze-thaw cycling, respectively. However, the intensity of phlogopite peaks does not exhibit any clear change. These results indicate that the microstructure of minerals is closely connected to the mechanical properties of tonalite, especially that of quartz and plagioclase. Before and after freeze-thaw cycling, phlogopite may become the most relatively stable mineral.

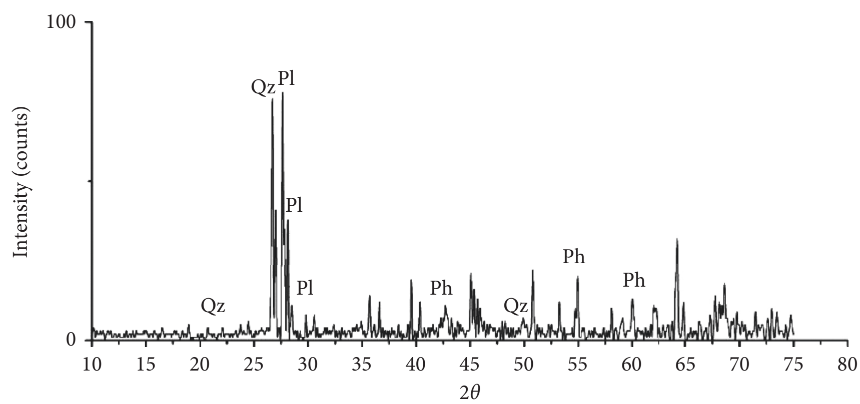
4.4. Young's Modulus. Young's modulus of a material is a measure of its stiffness and reflects the condition of its internal structure. In this study, Young's moduli were obtained from stress-strain relationships. Figure 11 shows the measured values of Young's modulus for the granite specimens with and without exposure to freeze-thaw cycling. The data show that there is a slight decrease in Young's modulus caused by the 30 freeze-thaw cycles, indicating a deterioration in the elastic properties of the granite. While the natural granite specimens had an average Young's modulus of approximately 21.92 GPa, those subjected to freeze-thaw cycling had an average Young's modulus of 18.98 GPa, corresponding to a reduction of 13.4%. However, as the minimum temperature of the freeze-thaw cycle decreased from −30°C to −50°C, Young's modulus remained constant. If it is assumed that the variation in Young's modulus is mainly related to porosity and microcracking, then it can be argued that the porosity and degree of microcracking are similar after freeze-thaw cycling with minimum temperatures between −30°C and −50°C.



(a)

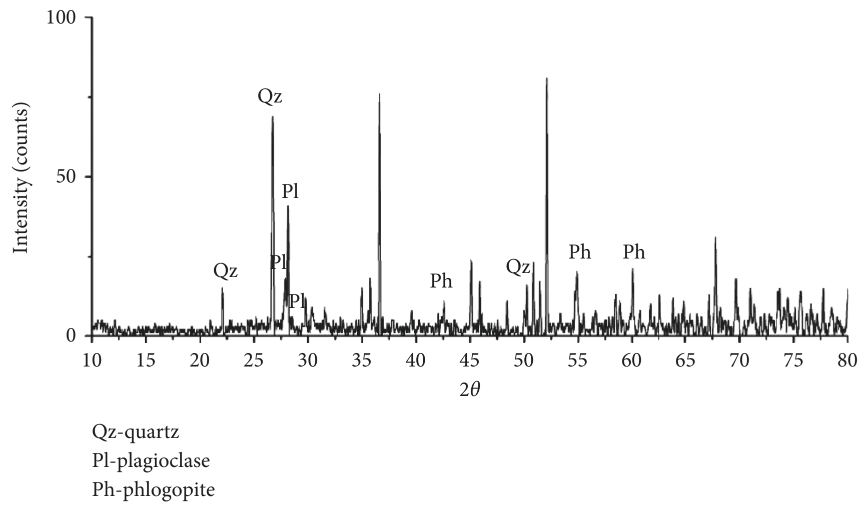


(b)



(c)

Figure 7: Continued.



(d)

Figure 7: XRD results for tonalite specimens: (a) under natural conditions; (b) after cycling between 20°C and −30°C; (c) after cycling between 20°C and −40°C; (d) after cycling between 20°C and −50°C.

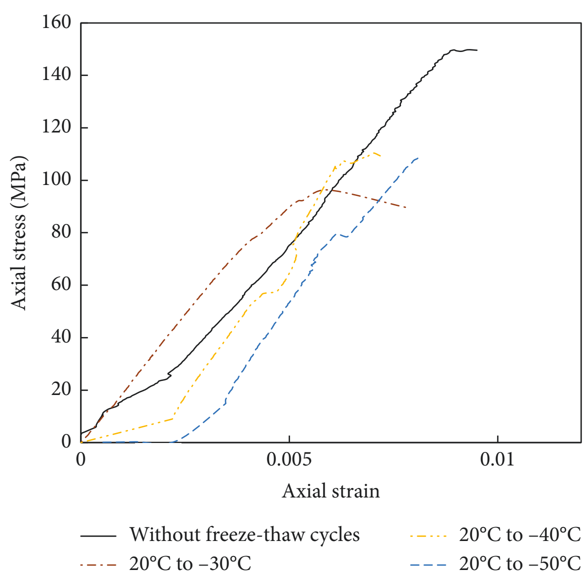


Figure 8: Stress-strain curves for tonalite specimens with and without exposure to freeze-thaw cycling.

4.5. Mode I Fracture Toughness, K_{IC}. Photos of three-point bending test specimens subjected to freeze-thaw cycles and mechanical testing are shown in Figure 12. No differences are observed on the surface of specimens before and after freeze-thaw cycling.

Three-point bending test was conducted to investigate the propagation of cracks under mode I loading, and the mode I fracture toughness, K_{IC}, was selected to characterize the failure of the tonalite specimens tested. Yin [33] developed a mathematical formula for determining the fracture toughness of tonalite specimens with a single-edged planar crack in three-point bending tests using boundary collocation as described below.

According to Williams's stress function, expression (1) can be used to calculate the mode I fracture toughness, K_{IC}:

$$4\frac{K_{IC}B\sqrt{W}}{PS} = -\sqrt{2}\left(\frac{4W}{S}\right)D_1, \quad (1)$$

where K_{IC} is the critical stress intensity factor; W, B, and S are the height, thickness, and length of the specimen, respectively; P is the external load applied to the middle of the specimen, and D_1 is a dimensionless parameter determined by the boundary conditions. In this study, the ratio of S to W is 5 and D_1 can be expressed in terms of a/W (where a is the depth of the single-edged planar crack). Therefore, equation (1) becomes

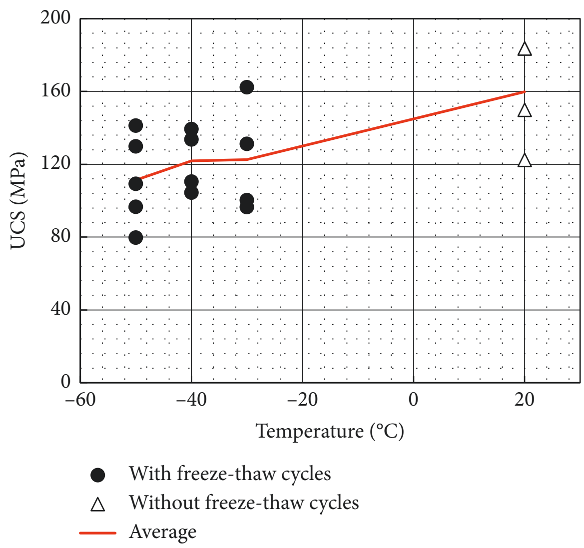


Figure 9: Uniaxial compressive strength (UCS) of tonalite with and without exposure to freeze-thaw cycling.

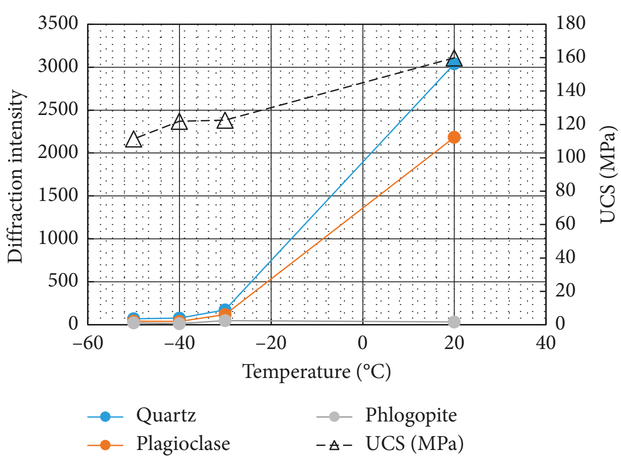


Figure 10: The relationship between the diffraction intensity of minerals and the UCS of tonalite specimens.

$$4\frac{K_I B\sqrt{W}}{PS} = F\left(\frac{a}{W}\right), \tag{2}$$

where $F(a/W)$ takes the form shown in the following equation (from the American Code SEM-E399):

$$F\left(\frac{a}{W}\right) = 11.6\left(\frac{a}{W}\right)^{1/2} - 18.4\left(\frac{a}{W}\right)^{3/2} + 87.2\left(\frac{a}{W}\right)^{5/2} - 150.4\left(\frac{a}{W}\right)^{7/2} + 154.8\left(\frac{a}{W}\right)^{9/2}. \tag{3}$$

The fracture toughness, K_{IC}, is therefore

$$K_{IC} = \frac{P_{max}}{BW^{1/2}} F\left(\frac{a}{W}\right), \tag{4}$$

where P_{max} is the peak load measured during testing. The mode I fracture toughness values of the tonalite specimens with and without exposure to freeze-thaw cycling are plotted in Figure 13.

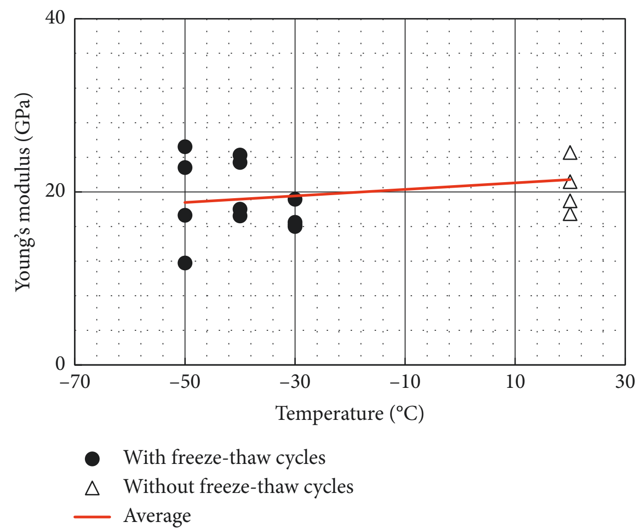


Figure 11: Young's modulus of granite with and without exposure to freeze-thaw cycling.

In general, the fracture toughness values of the tonalite specimens subjected to freeze-thaw cycling were lower than those of the natural tonalite specimens. The average fracture toughness values of the −30°C, −40°C, and −50°C minimum temperature freeze-thaw cycled specimens were 86.1%, 82.2%, and 81.8% of the average value for the natural tonalite specimens, respectively. Thus, as the minimum temperature decreases, the fracture toughness shows a slight decreasing trend.

4.6. Griffith Fracture Principal. According to Griffith's failure criterion [34], the critical fracture stress, σ_{cr}, in three-point bend testing can be described by

$$\sigma_{cr} = \sqrt{\frac{2E\gamma}{\pi c}}, \tag{5}$$

where c is half the length of the preprepared crack; E is Young's modulus; and γ is the surface energy density, which is related to the surface tension of the material. While [35] provides the surface energy densities for many minerals, that study excludes some granite minerals.

In the three-point bending tests, the crack length was fixed. Therefore, according to equation (5), if the mineral compositions of the tonalites were similar for all specimens, the value of σ_{cr} should only be affected by Young's modulus after freeze-thaw cycling. The results discussed previously support this hypothesis, as the variation in Young's modulus is similar to the variation in fracture toughness following freeze-thaw cycling. In other words, variation in the minimum temperature of the freeze-thaw cycle between −30°C and −50°C has no discernible effect on either Young's modulus or fracture toughness.

4.7. SEM Images. Scanning electron microscopy (SEM) was employed to investigate the effect of freeze-thaw cycling on the microstructure of tonalite. Figure 14(a) shows an SEM

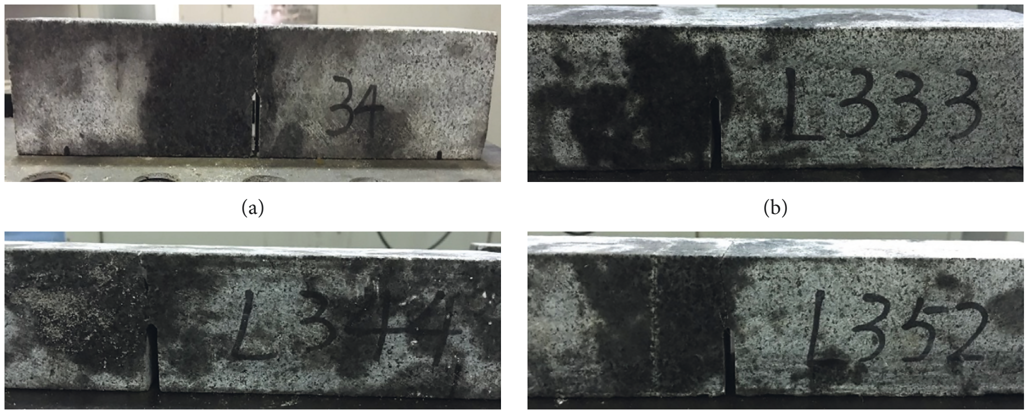


FIGURE 12: Three-point bending samples subjected to freeze-thaw cycling and mechanical testing: (a) 20°C; (b) −30°C; (c) −40°C; (d) −50°C.

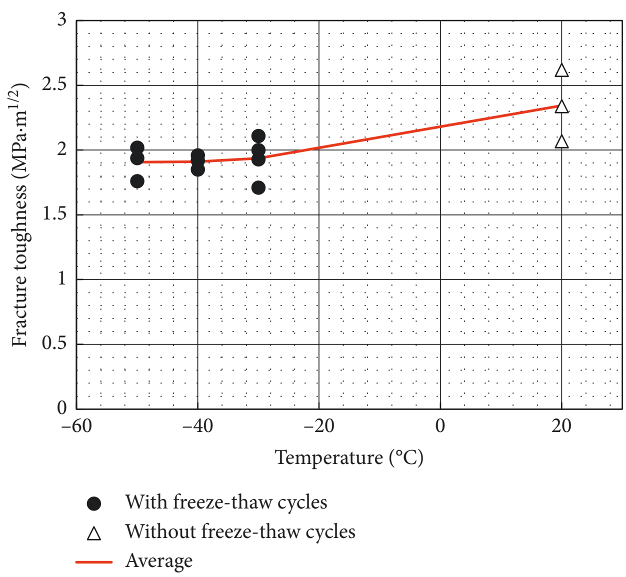


FIGURE 13: Fracture toughness of tonalite with and without exposure to freeze-thaw cycling.

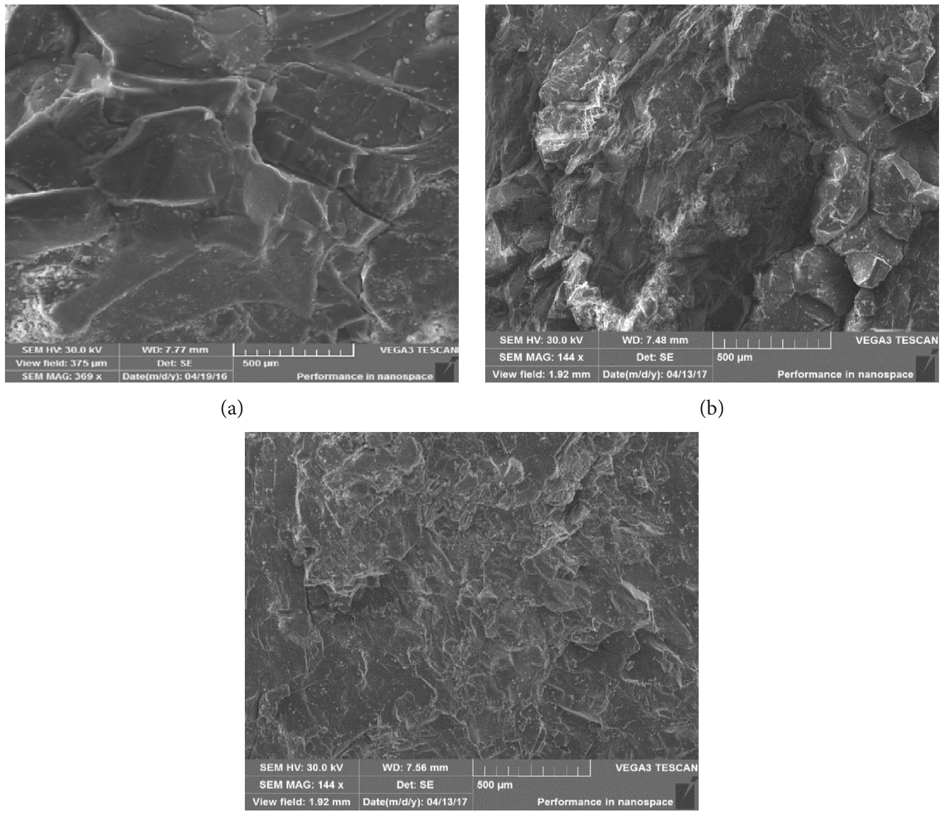
image of a tonalite specimen without freeze-thaw treatment and Figures 14(b) and 14(c) show SEM images of tonalite specimens subjected to freeze-thaw cycling with minimum temperatures of −30°C and −50°C, respectively. Comparison of microstructures suggests that the tonalite specimen subjected to the minimum temperature of −50°C is less compact and has more fragments and broken structural planes on the surface than the specimens subjected to the minimum temperature of −30°C with no treatment. The mineral structure of the specimen without treatment is more compact than those exposed to freeze-thaw treatment, and microcracks are much less abundant.

A higher magnification image of the microstructure of the tonalite specimen subjected to a minimum temperature of −40°C is shown in Figure 15. This image displays micropores on the surface, some of which are filled with fragments.

Figure 16 shows a lower magnification image of a phlogopite before and after freeze-thaw cycling with a minimum temperature of −50°C. After freeze-thaw cycling, cleavage in the phlogopite can be observed. The schistose structure of phlogopite is highly susceptible to shear loading, and an applied force along the surface of schistose will easily cause fracturing. However, as the surface tension and surface energy density of the mineral are not affected by freeze-thaw cycling, only the UCS of the tonalite is affected. There is no obvious degradation of the phlogopite observed after treatment.

5. Summary and Conclusions

Several techniques were used to examine the deterioration of tonalite resulting from freeze-thaw cycling. Physical and mechanical property data obtained from uniaxial compression



(a) (b)

(c)

Figure 14: SEM images of tonalite (a) without treatment and after freeze-thaw cycling with (b) a minimum temperature of −30°C and (c) a minimum temperature of −50°C.

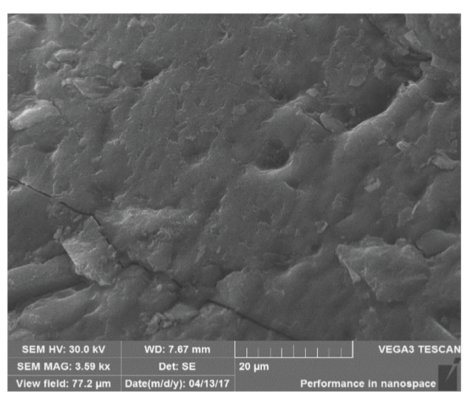
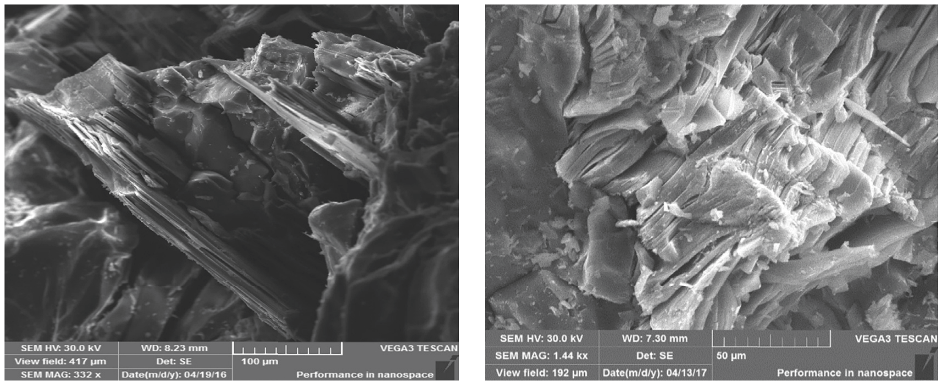


Figure 15: SEM image of tonalite after freeze-thaw cycling with a minimum temperature of −40°C.

testing, three-point bend testing, and elastic wave velocity measurements were analysed and compared with the results of mineral composition and microstructure investigations obtained from XRD analysis and microscopy. It was found that there is relationship between the degradation of macro-mechanical properties and the changes in mineralogy and microstructure of tonalite associated with freeze-thaw cycling. The following conclusions can be drawn from this study:

(1) Frost damage decreases the UCS, Young's modulus, and fracture toughness of tonalite specimens subjected to freeze-thaw cycling. The value of UCS decreases almost linearly with a decrease in the minimum temperature of the freeze-thaw cycle. However, Young's modulus and fracture toughness remain relatively stable for minimum cycle temperatures between −30°C and −50°C.



(a) (b)

Figure 16: SEM images of phlogopite: (a) without treatment and (b) after freeze-thaw cycling with a minimum temperature of −50°C.

(2) The minimum temperature of the freeze-thaw cycle affects the compactness of tonalite. Increasing amounts of fragmentation and breaking of structural planes can be observed with decreasing minimum temperatures. These microstructural surfaces and microcracks generated by freeze-thaw cycling affect the mechanical properties of tonalite, especially its bearing capacity.

(3) There is a close relationship between the diffraction intensity of minerals and the uniaxial compressive strength of tonalite specimens subjected to freeze-thaw cycles, especially for quartz and plagioclase. There is no observable relationship between phlogopite diffraction intensity and the UCS of tonalite specimens. Thus, phlogopite may possess the highest relative stability during freeze-thaw cycling.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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