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Determination of the mode I fracture toughness under different temperatures and pressures

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Abstract

Determining the mode I fracture toughness (K_{IC}) is essential for various rock engineering applications, including tunnel boring, rock drilling, hydraulic fracturing, and oil exploration. Although extensive research has been conducted to measure K_{IC} under ambient conditions using various methods, the effects of temperature and pressure, two key factors in engineering environments, remain insufficiently explored. This study employs the pseudo-compact tension (*p*CT) testing method to investigate K_{IC} across a wide range of temperatures and pressures. Experiments were conducted on Blanco Alba granite (Grt), Crema Palancar limestone (Lst), and Corvio sandstone (Sst). The experiments were performed after thermal annealing and shocking treatments (i.e. both tests performed at room T conditions) and under thermostatic conditions at prescribed below room temperature (i.e. tests performed at the target T), while the effects of confining pressure were performed using both jacketed and unjacketed specimens under pressures of up to 25 MPa. Results reveal that in Grt specimens, K_{IC} decreases after thermal shocking and annealing, and this is likely due to the pre-induction of microcrack during the thermal treatments. In contrast, K_{IC} increases in thermostatic tests for progressively lower temperatures. The influence of confining pressure varies depending on specimen type: In jacketed specimens, K_{IC} shows a linear increase while increasing it, whereas for unjacketed specimens K_{IC} decreases when confining pressure also increases. The reduction of K_{IC} for unjacketed specimens can be attributed to pore pressure unbalances near the crack tip, which is controlled by the permeability of rock. These findings provide valuable insights into the design and optimization of underground engineering projects operating under extreme temperature and pressure conditions.

Keywords

Mode I fracture toughness, Pseudo-compact tension (*p*CT) test, Temperature effects, Pressure effects

1 Introduction

The mode I fracture toughness (K_{IC}) is an important parameter to evaluate the stability of rock structures in various underground engineering applications. In the laboratory environment, the measurement of K_{IC} is typically performed under ambient conditions. However, there is a growing demand of new data and procedures to gather fracture toughness data at increasingly higher temperatures and pressures (e.g. geothermal energy production, ultra-deep mining, etc.), what in turns requires a deeper understanding of the processes affecting the behavior of K_{IC} under these challenging conditions. For instance, in high-level radioactive waste repositories, the heat released during radioactive decay (up to ~300 °C) raises the temperature of the surrounding rocks and may increase crack propagation velocities and microcrack density, with a potential impact over its mechanical performance (Zou et al. 2017). Similarly, in CO_2

sequestration projects, CO_2 may be injected at temperatures lower than the corresponding reservoir (e.g. Reppas et al. 2024), what may cool down the surrounding rocks, particularly during blowouts or non-continuous CO_2 injection (Vilarrasa and Rutqvist 2017). Likewise, geothermal systems involve injecting low-temperature fluids into hot dry rock masses to enhance the efficiency of heat exchange. The fast thermal changes induced may enhance the degradation of the rock matrix due to thermal shock (Shao et al. 2022). However, it is also important to bear in mind that, being the topic of interest, these fundamental thermomechanical aspects are not the only ones requiring comprehension since other non-straightforward phenomena induced by the experimental approach itself may affect the results and, by extension, the derived conclusions.

To date, extensive laboratory experiments have shown that temperature variations significantly affect fracture toughness and other fracture characteristics of rock. At moderate to high temperatures, rocks undergo microstructural alterations, including crystal expansion, microcrack nucleation and propagation, hot melting and phase transformations. That leads to complex and often unpredictable macroscopic effects (Zou et al. 2017; Fan et al. 2018; Guo et al. 2023; Hu et al. 2023; Qiu et al. 2024). Conversely, literature data at sub-zero temperatures indicates that the fracture toughness of dry rocks typically increases as the temperature decreases due to particle shrinkage, pore closure, and enhanced rock cohesion and internal friction angle. In addition, the water present in pores may freeze, what fills the pores and bonds strongly mineral grains, what further improves the resistance of rocks to fracture (Dwivedi et al. 2000; Zhang et al. 2023).

Pressure effects are also worth of consideration. The in-situ stresses operating over rock masses increase with burial depth. Published data shows that K_{IC} increases in jacketed dry specimens under higher confining pressures. This has been conventionally interpreted as resulting from the closure of microcracks (Yang et al. 2021) and the reduction in the size of the fracture process zone (i.e. the non-linearly deformed region near the crack tip; Fuentealba et al. 2024). However, rock masses are inherently porous media containing fluids in liquid or gas phases (Jing 2003). In addition to the influence of in-situ stresses, fluid pressure may also affect fracture behavior. Although, few researchers have performed confined K_{IC} tests without jacketing their specimens (Müller 1986; Kataoka et al. 2015; Yang et al. 2021; Balme et al. 2004; Muñoz-Ibáñez et al. 2023), the available experimental data on the role of confining fluids over the fracture toughness of rocks still remains limited.

This study aims to contribute to fill the previous gaps by illustrating above-ambient pressure and temperature testing of mode I fracture toughness of three rocks (granite, sandstone and limestone) using the pseudo-compact tension (*p*CT) approach. The thermal experiments are performed with three different approaches: Thermal annealing (slow heating/cooling), thermal shocking (fast heating/cooling) and thermostatic testing (T remains constant during the experiment). In the first two (which are the most used approaches across the literature) fracture toughness is determined at room T while in the third toughness is assessed at a given target temperature. In addition, the effect of sample jacketing (i.e. with and without penetration in the sample of the confining fluid) was assessed at room temperature from 0.1 MPa to 25 MPa.

2 Materials and methods

2.1 Materials and specimen preparation

Three different rocks have been used in this research. In the case of thermal processes, we have used the Blanco Alba granite (Grt), which is a coarse-grained, high-strength rock with a dry density of 2.6 g/cm³. Additional properties are summarized in Table 1. To investigate the effect of specimen jacketing on fracture toughness, in addition to the previous granite, two additional rocks were examined: Crema Palancar limestone (Lst) and Corvio sandstone (Sst). The properties of these materials are also detailed in Table 1.

Careful attention was paid to ensure homogeneity of the specimens used for the pseudo-compact tension (*p*CT) tests (Fig. 1). These were cut using a high-pressure water jet cutting system and their average dimensions are as follows: Diameter (D) = 50.0 mm; Thickness (B) = 25.0 mm; Notch length (a) = 18.0 mm; Distance from the base of the groove to the bottom of the specimen (b) = 45.0 mm. The a/b ratio is equal to 0.38 and the prefabricated notch width is 1 mm, what corresponds to the diameter of the water jet. While these dimensions served as guidelines, minor deviations due to system errors from the water jet cutting machine were corrected by carefully measuring each specimen with a digital caliper before testing. After preparation, specimens were dried in an oven at 60°C for at least 48 hours and then stored in a desiccator to maintain dryness. Fig. 1d shows a jacketed *p*CT specimen specifically designed for pressure tests, which will be described in detail in Sec. 2.4.

Table 1 Mechanical and physical characteristics of tested materials. (Grt=granite, Lst=limestone, Sst=sandstone)

Material	**Elastic modulus [GPa]**	**Compressive strength [MPa]**	**Tensile strength [MPa]**	**Permeability [m²]**
Blanco Alba Grt	52.6	109.9	11.2	7.32×10^{-19}
Crema Palancar Lst	60.0	98	8.4	1.87×10^{-17}
Corvio Sst	14.7	39.9	2.5	6.93×10^{-15}

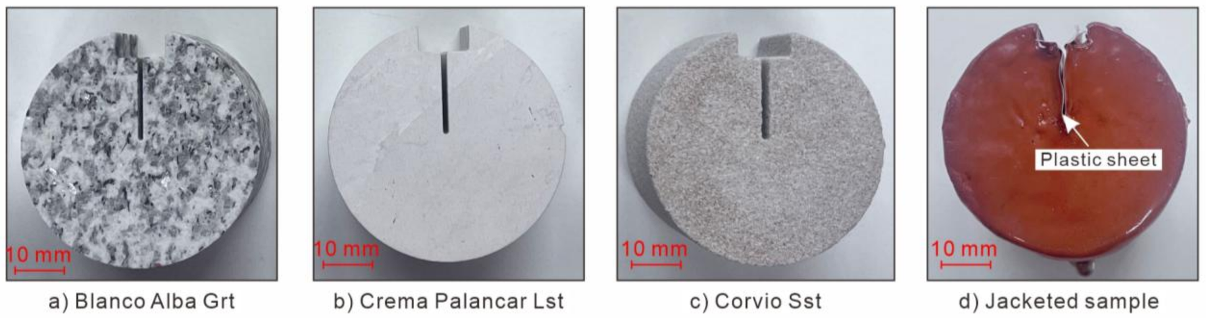

Fig. 1 Pictures of the specimens used in this research: a) Blanco Alba Grt specimen; b) Crema Palancar Lst specimen; c) Corvio Sst specimen; d) a jacketed sample of a granite specimen.

2.2 *p*CT tests under room conditions

The K_{IC} values determined under ambient conditions served as the baseline for each type of rock. The testing apparatus is the original device described in detail by Muñoz-Ibáñez et al. (2020) and a modified version also presented by Li et al. (2024). For high-pressure testing, a special pressure cell was also adapted. Test procedures followed the guidelines outlined by Muñoz-Ibáñez et al. (2020) and Li et al. (2024) and the load displacement rate was set to 0.1 mm/min, and all tests were conducted at ~22°C.

2.3 Thermal experiments

2.3.1 Thermal annealing

To study the effect of thermal treatment on granite, specimens were heated in a muffle furnace to the target temperature within 2 hours, maintained at that temperature for 24 hours, and then allowed to cool naturally to room temperature (25°C). The target temperatures included 200°C, 400°C, 550°C, 600°C, and 800°C. Once cooled, the specimens were tested using the original *p*CT device. This treatment is referred as thermal annealing.

2.3.2 Thermostatic testing

To perform the *p*CT tests at non-ambient, sub-zero temperatures, a constant low-temperature chamber was constructed using four isolation panels equipped with Peltier modules and a recirculation cooler. Specimens were maintained at the target temperature for 8 hours before testing. The achieved target temperatures were -4.5°C, -11°C, -15°C, -19.5°C, -22°C, and -22.1°C. This type of test is referred as thermostatic. For lower temperatures, the high-pressure *p*CT vessel described in Muñoz-Ibáñez et al. (2023) was thermally isolated to create a controlled environment. Dry ice ($CO_{2,dry}$) and liquid nitrogen (LN_2) were used to further reduce the temperature of the experimental chamber. With such approach, it was possible to attain up to -42.6°C and -44.7°C.

2.3.3 Thermal shocking

To evaluate the impact of sudden temperature changes over the K_{IC} of the granite rock, two different scenarios were examined. In the first case, a granite specimen was immersed in a mixture of dry ice plus acetone, resulting in a fast temperature drop from 22 °C to -70 °C. In the second scenario, a granite specimen was heated to 100 °C over a 2-hour period in a muffle furnace and kept at that temperature for 24 hours before being suddenly immersed in liquid nitrogen (-196 °C). That induced a significantly larger thermal shock in the rock.

2.4 Specimen jacketing effects

To conduct these tests, we used the high-pressure *p*CT cell described by Muñoz-Ibáñez et al. (2023), to which we introduced several modifications. One of them was the introduction a pressure-balancing system to reduce friction stresses on the loading rod caused by internal cell pressure. As shown in Fig. 1d, the jacketed *p*CT specimens were covered with waterproof rubbers to isolate them from the confining fluid. The notch surfaces were also covered with rubber, and the confining fluid was allowed to contact the notch, enabling the application of calculation equation derived under ambient conditions without modification.

To simulate in-situ conditions, unjacketed *p*CT specimens were tested directly under confining pressure. These specimens were exposed to fluid to better represent realistic field conditions. Distilled water was selected as the confining fluid.

2.5 Procedure and calculations

The load displacement rate for all tests was maintained at 0.1 mm/min. The applied load (P) was automatically recorded during each test, and the K_{IC} was calculated using Eq.1:

$$K_{IC} = Y' \frac{\mathrm{P_{max}}}{bB} \sqrt{\pi a} \tag{1}$$

Where $\mathrm{P_{max}}$ Value of the peak load
B Thickness of *p*CT specimen
b Distance from the base of the groove to the bottom of the specimen
a Notch length

The dimensionless stress intensity factor (Y') in Eq. 1 was computed using the expression proposed by Muñoz-Ibáñez et al. (2020) for a 50 mm diameter *p*CT specimen:

$$Y' = 12.651 - 47.054\left(\frac{a}{b}\right) + 157.72\left(\frac{a}{b}\right)^2 - 247.17\left(\frac{a}{b}\right)^3 + 296.33\left(\frac{a}{b}\right)^4 \tag{2}$$

3 Results and discussion

3.1 K_{IC} values under ambient environment

The Mode I fracture toughness (K_{IC}) values for Blanco Alba granite (Grt), Crema Palancar limestone (Lst), and Corvio sandstone (Sst) were determined under ambient conditions using three different *p*CT testing devices: the original device (Muñoz-Ibáñez et al. 2020), the modified device (Li et al. 2024), and the high-pressure *p*CT cell (pressure cell). The results obtained from these devices were consistent, demonstrating the good comparability of the experimental setups.

As shown in Fig. 2, Grt exhibits the highest average K_{IC} value of 1.25 MPa·m$^{1/2}$, indicating its larger resistance to crack propagation under ambient conditions. In contrast, Sst shows the lowest average K_{IC} value of 0.11 MPa·m$^{1/2}$, what reflects its relatively poor ability to resist fracture initiation and propagation. This is likely due to its lower strength and higher porosity. Lst showed an intermediate K_{IC} value of 1.03 MPa·m$^{1/2}$.

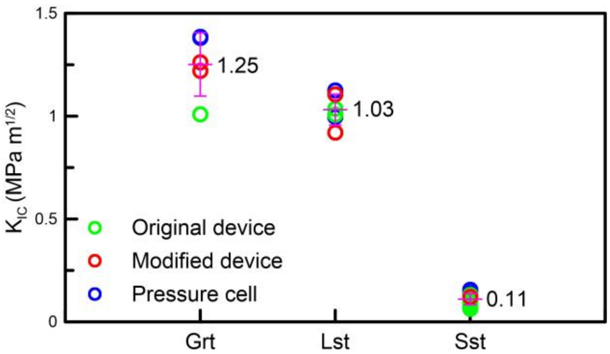

Fig. 2 Mode I fracture toughness (K_{IC}) results of rocks using different device under ambient environment.

3.2 K_{IC} values after different thermal conditions

The test results of K_{IC} obtained with the different experimental thermal approaches are presented in Fig. 3a. At 800°C, thermal cracking is generalized and de-cohesion of the grains (primarily due to thermal expansion and microcrack coalescence) led to specimen failure and the inability to obtain reliable K_{IC}

data at this temperature. This observation is consistent with previous findings, which report that increasing the temperature from 600°C to 800°C causes extensive crack propagate, leading to the structural breakdown of mineral networks (Fan et al. 2018).

In Fig. 3a we observe that the K_{IC} of the thermally annealed specimens progressively decreases as the treatment temperature increases from 200°C to 600°C. Compared to the ambient condition, K_{IC} decreases by 0.6%, 32.7%, 69.1%, and 78.3% at 200°C, 400°C, 550°C, and 600°C, respectively. This reduction is mainly attributed to the progressive development of thermally induced microcracks, as described in studies by Guo et al. (2023), who highlights the significant influence of temperature-induced microcrack density on the mechanical behavior of granite.

Fig. 3b shows that the specimen treated at 600°C shows obviously ductile failure behavior before reaching peak load compared to those treated at 550°C, what indicates a significant thermal softening effect. Similar non-linear deformation trends have been reported by Hu et al. (2023), who reported quasi-brittle failure of granite is observed up to 500°C, transitioning to more ductile failure at 600°C. In addition, Zhang et al. (2022) and Guo et al. (2023) also observed that the brittle-ductile transition of granite begins at 600°C. This phenomenon may be related to the α-β phase transition of quartz at 573°C (Mahanta et al. 2016).

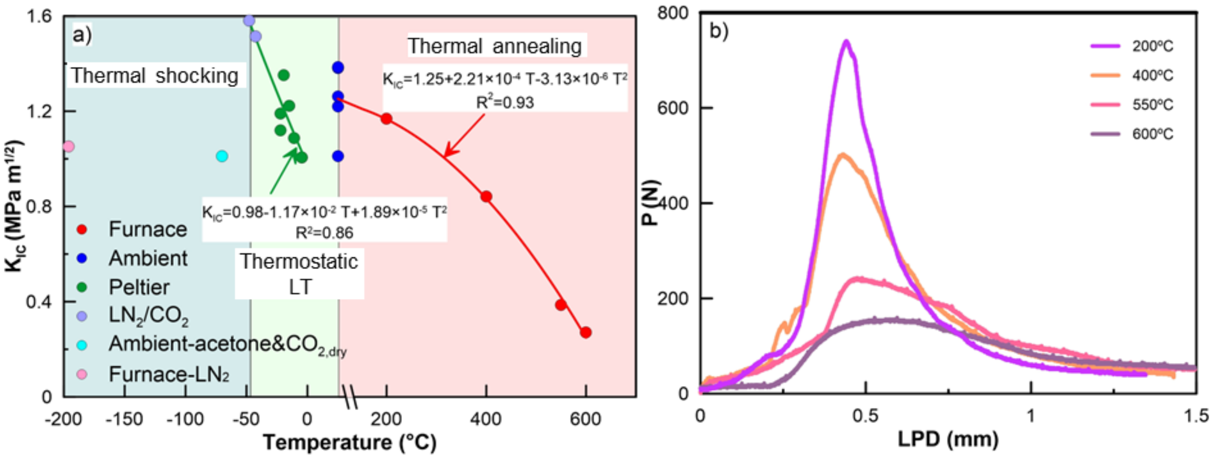

Fig. 3 a) Effects of temperature on mode I fracture toughness (K_{IC}); b) Loading curves of *p*CT specimens after thermal treatment.

In contrast, during the thermostatic low-temperature (LT) testing, K_{IC} shows an increasing trend as the temperature decreases from 0°C to -50°C. That points to a toughening effect on the Blanco Alba Grt. This can be primarily attributed to mineral grain contraction and the shrinkage of pre-existing cracks at lower temperatures, which enhances crack closure and strengthens the granite (Zhang et al. 2023). On the other hand, although the specimens were dried before testing, some residual water may have remained in small pores, potentially contributing to the observed increase in fracture toughness. Previous studies by Dwivedi et al. (2000), Mardoukhi et al. (2021) and Liu et al. (2024) have suggested that this toughening mechanism may be particularly effective at sub-zero temperatures, where the bridging effect of pore ice enhances cohesion along fracture surfaces.

The results under thermal shock conditions (Fig. 3a) indicate that rapid temperature changes reduce the strength of rock materials compared to room temperature conditions, consistent with findings by Shao et al. (2022) and Wang et al. (2023). This underscores the importance of considering sudden thermal gradients in practical applications, such as geothermal energy extraction and underground energy storage.

3.3 K_{IC} effects associated with smpecimen jaceketing

The K_{IC} values under different confining pressures (σ_{conf}) are illustrated in Fig. 4, revealing a linear relationship between K_{IC} and σ_{conf} is for the tested rocks. For Blanco Alba Grt (Fig. 4a) and Crema Palancar Lst (Fig. 4b), the jacketed material remained intact until the specimen failure, allowing for successful determination of K_{IC} under confining pressure. However, tests on jacketed Corvio Sst specimens were unsuccessful due to the high porosity and permeability of sandstone, which made it difficult to effectively apply a waterproofing layer.

As shown in Fig. 4, jacketed specimens exhibit a consistent increase in K_{IC} with increasing confining pressure, aligning with findings from previous studies. In contrast, unjacketed specimens show lower K_{IC} values compared to their jacketed counterparts. Linear fitting of the results shows that unjacketed specimens have lower coefficients of determination (R^2), indicating a greater variability in K_{IC}. Despite this variability, the trends in K_{IC} for different rocks under increasing pressure can still be analyzed.

For Grt and Sst specimens, K_{IC} decreased with increasing confining pressure, with reductions of ~25% and ~65%, respectively, at the confining pressure of 25 MPa. The decreasing trend of Grt is likely caused by its low permeability (7.32×10^{-19} m^2). According to Darcy's law, under a 25 MPa pressure head (maximum pressure applied in this study), water penetration depth into the Grt specimen within 20 minutes is only 11.8 mm, while the specimen thickness is 25 mm. This limited water penetration reduces the effect of pore pressure within the Grt specimen. However, direct contact with the prefabricated notch leads to a localized pore pressure increases near the crack tip, a phenomenon commonly observed in hydraulic fracturing (Bruno and Nakagawa 1991). This localized pore pressure rise near the crack tip may facilitate crack initiation and propagation, contributing to the observed reduction in K_{IC}.

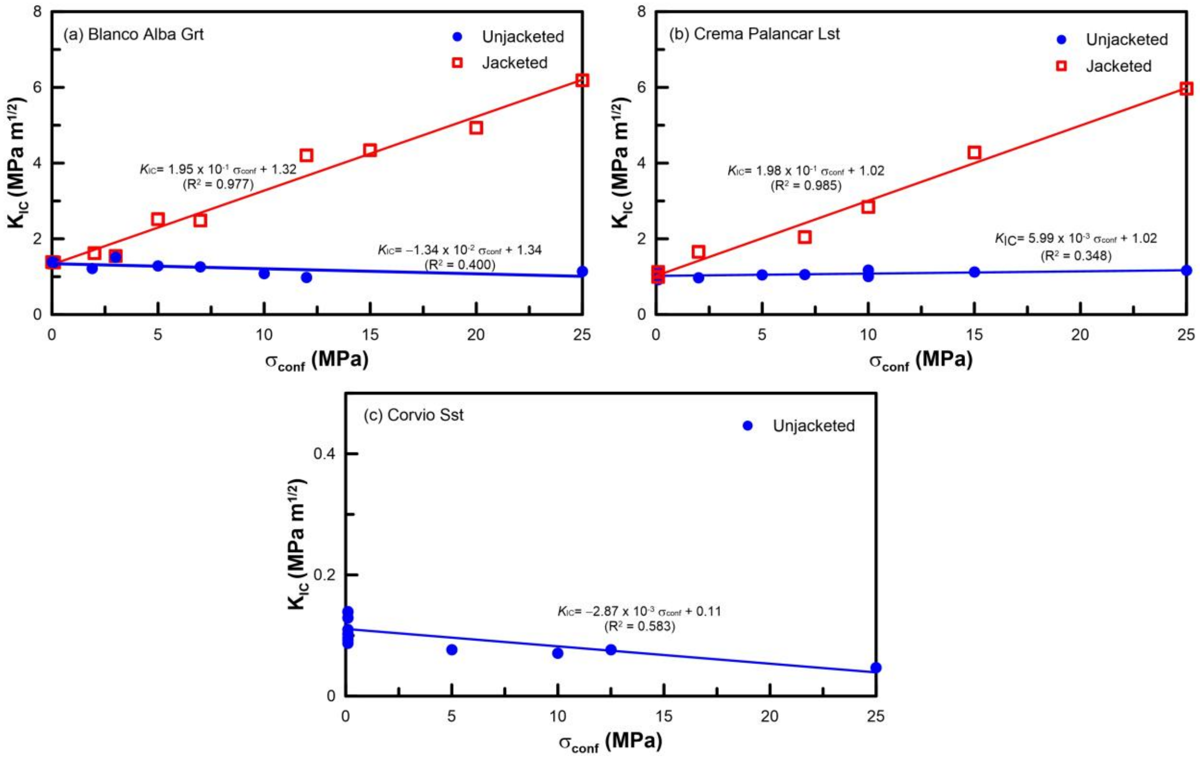

Fig. 4 Effects of confining pressure on mode I fracture toughness (K_{IC}) for different rock materials.

The permeability of Lst (1.87×10^{-19} m^2) allows for rapid pore pressure transmission, ensuring that pore pressure near the crack tip equilibrates with confining pressure. on time is shorter than the duration of the test. This prevents any additional influence of confining pressure on the crack tip, maintaining relatively stable K_{IC} values across different confining pressure. However, Sst specimens, which have significant higher permeability (6.93×10^{-15} m^2) than Lst, exhibit a decreasing trend in K_{IC} with increasing pressure (Fig. 4c). This reduction is likely due to the low strength of Sst, where the development of effective stress under pressurization further weakens the specimen. In addition, stress corrosion induced by water may contribute to the observed reduction in fracture toughness. Kataoka et al. (2015) found that exposure to water vapor pressure reduces K_{Ic}; however, since the exposure time in this study was only 20 minutes, significantly shorter than the 6 hours in their study, the influence of stress corrosion is expected to be minimal.

4 Conclusions

This study investigates the Mode I fracture toughness (K_{IC}) of three rock types under different temperature and pressure conditions using the pseudo-compact tension (*p*CT) testing method. The results provide valuable insights into rock fractured behavior under complex environmental conditions, with implications for underground engineering applications. The main conclusions are summarized as follows:

(1) Increasing treatment temperature of granite leads to significant reductions in K_{IC} of granite. The brittle-ductile transition for tested granite occurs around 600°C.

(2) Under real-time low-temperatures (sub-zero) conditions, K_{IC} increases with decreasing temperature, likely due to mineral contraction and residual pore water freezing, enhancing fracture resistance. However, thermal shock at low temperatures causes a reduction in K_{IC}, likely duo to rapid microcrack propagation from sudden temperature changes.

(3) Jacketed specimens show a linear increase in K_{IC} with confining pressure, whereas unjacketed specimens exhibited a decreasing K_{IC} values with confining pressure and higher variability compared to jacketed specimens. The reduction in K_{IC} for unjacketed specimens is attributed to unbalanced pore pressure near the crack tip, which is controlled by rock permeability.

Overall, this study provides an understanding of rock fracture behavior under temperature and pressure conditions as well other phenomena more connected with the experimental procedures. Future research will focus on the combined effects of temperature and pressure, extended temperature ranges, and long-term fluid exposure to address the complexities of rock behavior under in situ conditions.

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