DOI: https://doi.org/10.68732/9daef88b5cfcf46f

ISBN: 978-9989-2053-6-1

Mode I Fracture Toughness Tests of Limestone under High-Temperature Isothermal Condition

Y. Li, M. Herbón-Penabad, A. Muñoz-Ibáñez, J. Delgado-Martín
Centro de Innovación Tecnolóxica en Edificación e Enxeñería Civil (CITEEC)
Universidade da Coruña, A Coruña, Spain
Yan.li@udc.es
0000-0001-8986-3387

Abstract

Understanding the fracture behavior of rocks at elevated temperatures is essential for evaluating the stability of deep geological engineering systems, such as geothermal reservoirs, deep mining excavations, underground energy storage facilities, and nuclear waste repositories. Although many studies have investigated so farthe thermal effects on rock properties, most of them have been conducted on specimens tested only after cooling to room temperature, while fracture data under isothermal, high-temperature conditions remain limited. In this study, an improved pseudo-compact tension (*p*CT) apparatus is presented to determine the mode I fracture toughness (K_{Ic}) of limestone under controlled thermo-mechanical conditions. Specimens of the so-called Crema Palancar limestone has been tested under two thermal paths: isothermal heating (IT), in which specimens were heated and tested directly at the target temperature, and thermal annealing (TA), in which specimens were heated, naturally cooled to room temperature, and then tested. In both cases, the heating rate was 2°C/min, and the target temperatures ranged from room temperature to 600°C. The results show that the thermal path has a significant influence on the measured K_{Ic} of this limestone. Under IT conditions, K_{Ic} first increases and then decreases with increasing temperature, reaching a maximum at ~ 400°C. Under TA conditions, however, K_{Ic} decreases monotonically with increasing treatment temperature. At the same target temperature, the K_{Ic} values obtained under IT conditions are consistently higher than those measured under TA conditions. These contrasting trends indicate that different thermal stress paths govern the initiation, accumulation, and propagation of microcracks, thereby controlling the fracture resistance of limestone. The results highlight that post-treatment fracture tests do not represent the fracture behavior of rocks at elevated temperatures.

Keywords

Mode I fracture toughness, Pseudo-compact tension (*p*CT) test, Thermo-mechanical conditions, Crack initiation and propagation, High temperature conditions.

1 Introduction

With the expansion of deep mining, geothermal energy extraction, underground energy storage, and nuclear waste disposal, rock masses are increasingly exposed to high-temperature conditions (Guo et al., 2023). Temperature can substantially alter rock properties, including strength, elastic modulus, wave velocity, porosity, and crack development. These changes mainly arise from the heterogeneous thermal responses of mineral constituents, which induce incompatible deformation, thermal stress concentration, grain-boundary debonding, and microcrack initiation. In polycrystalline rocks such as granite, thermal expansion mismatch among quartz, feldspar, and mica is especially critical, making fracture behavior highly temperature-dependent. Because tensile crack initiation and propagation govern rock failure and permeability evolution, mode I fracture toughness (K_{Ic}) is a key parameter for assessing the stability and integrity of thermally affected rock masses.

Although the effects of high temperature on rock properties have been widely studied, most existing work has tested specimens after heating them to a target temperature and then cooling them to room temperature, a thermal path referred to here as thermal annealing (TA) (Guo et al., 2023). Some studies have also examined thermal shock (TS), where heated specimens are rapidly cooled to simulate severe thermal disturbance, such as cold-fluid injection during geothermal stimulation (Wong & Liu, 2025). By contrast, fracture behavior under isothermal heating (IT) remains less explored. In IT tests, specimens are heated to and maintained at the target temperature during fracture loading. Such tests are technically challenging because they require stable high-temperature loading, precise thermal control, and effective protection of the measurement system (Zhou et al., 2024a; Zhu et al., 2018).

TA tests measure the residual fracture toughness after thermal damage and cooling, whereas IT tests measure the fracture resistance of rock at elevated temperature, where thermal expansion, crack closure, and stress redistribution remain active. Thus, TA and IT reflect different thermo-mechanical histories. Although K_{Ic} under TA conditions generally decreases with temperature, IT behavior may be more complex due to the competition between thermal damage and temporary crack closure. Therefore, comparing TA and IT is essential to assess whether cooled specimens can represent the in-situ fracture behavior of hot rock masses.In this study, an improved pseudo-compact tension (*p*CT) apparatus was used to measure the K_{Ic} of limestone under high-temperature IT and TA conditions. Specimens were heated at 2 °C/min to temperatures up to 600°C and tested either at temperature or after natural cooling. The aim is to clarify the effect of thermal path on limestone fracture toughness and support more realistic evaluation of high-temperature rock engineering.

2 Methodologies

2.1 Rock materials and specimen preparations

The *Crema Palancar* limestone was used in this study, and its basic mechanical properties are reported in Li et al. (2026). A total of 28 *p*CT specimens were prepared, including 21 specimens for IT tests and 7 specimens for TA tests. For the IT tests, 3 specimens were tested at each temperature from room temperature (25 °C) to 600 °C, while for the TA tests, one specimen was tested at each temperature over the same range. The *p*CT specimens were cut using a high-pressure water jet system, with average dimensions of D = 50.0 mm, B = 25.0 mm, a = 18.0 mm, and b = 45.0 mm. Thus, the a/b ratio was 0.38. The specimen geometry and notation followed Muñoz-Ibáñez et al. (2020).

2.2 Experimental equipment

To determine the K_{Ic} of rocks under isothermal high-temperature conditions, the original *p*CT test configuration was modified, as shown in Fig. 1.The enhanced *p*CT device employs a stepper motor connected to a small backslash planetary gearhead to drive a precision ballscrew connected to the movable jaw at a constant velocity. That provides stable and controllable load/displacement to the tested specimen. To conduct the experiments at above-ambient temperature conditions, the system incorporates a 130 mm-diameter, 150 mm-length Herge 2500W, ceramic band-heater shielded with two thick refractory plates. The heater is regulated with a universal industrial temperature control unit (CJD-9000P) able to be programmed with temperature ramps and PID control based on a type-K thermocouple. The parts of the bench submitted to high temperatures (push rod and pulling jaws) have been manufactured with *Inconel*® 600 steel alloy (UNS N06600), which offers excellent mechanical performance and thermal resistance at high temperatures (yield strength ~90 – 120 MPa at ~600 ºC). As shown in Fig. 1(a), in order to reduce thermal transmission to sensitive parts of the device (e.g.

load cell, LVDT), the *Inconel*® push rod is sleeved with an external SS316 cooling jacket through which a cooling fluid is continuously recirculated with the aid of a 800 W industrial chiller (CW-5000). This cooling arrangement plays a critical role in ensuring both operational safety and measurement reliability during the high-temperature testing.

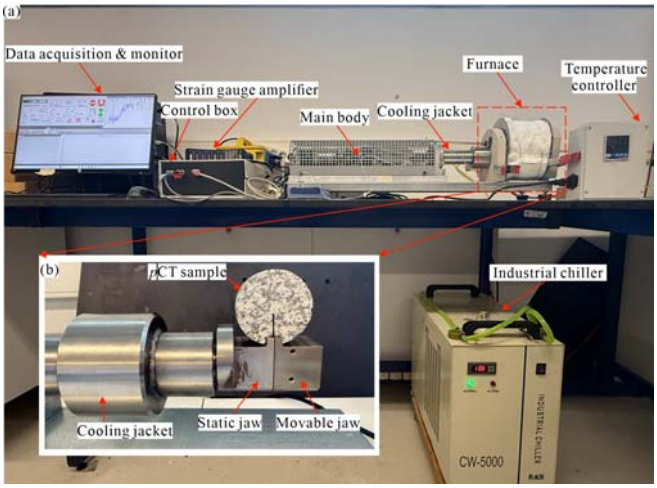


Fig. 1 Photos of the enhanced high-temperature *p*CT testing system: (a) overall view of the apparatus; (b) detailed view of a granite *p*CT specimen and pulling jaws.

Figure 1(b) shows a granite *p*CT specimen positioned inside the furnace. Compared with the original arrangement of the *p*CT test described by Muñoz-Ibáñez et al. (2020), in this enhanced device the sample is self-supported, lying on its weight on top of the pulling jaws. This allows the specimen to self-adjust its position in close groove/pulling jaws contact even when direct visual observation is not possible inside the furnace chamber.

2.3 Experimental procedures

For the IT group, specimens were tested directly in the high-temperature *p*CT apparatus. The furnace temperature was increased at a rate of 2 °C/min to the target temperatures. A separate finite element simulation, based on the specific heat capacity, density, and thermal conductivity of the limestone, showed that under this heating rate the internal and external temperatures of the *p*CT specimen increased synchronously and that the internal temperature field remained relatively uniform. In addition, tests performed at 100 °C, 200 °C, and 400 °C indicated that the holding time (0 – 4 h) at the target temperature did not significantly affect theresults. Even so, each specimen was held at the target temperature for 1 h before fracture testing to ensure thermal homogenization and to allow sufficient time for temperature-induced effects to develop.

For the TA group, specimens were thermally treated in a *Hobersal* muffle furnace with a maximum operating temperature of 1200 °C. The heating rate was the same as that used for the IT group (2 °C/min), but the holding time at the target temperature was extended to 12 h. After thermal treatment, the specimens were removed from the furnace and allowed to cool naturally to room temperature, after which the *p*CT test was carried out under ambient conditions.

For all tests, either IT or TA, the displacement loading rate was maintained at 0.1 mm/min. The applied load P was continuously recorded during the test, and theK_{Ic} was calculated using the expression proposed by Muñoz-Ibáñez et al. (2020) for a 50 mm diameter *p*CT specimen, as shown in Eq. (1) and Eq. (2):

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

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

Where P_{max} Peak value of the applied load during *p*CT test
B Thickness of *p*CT specimen
b Distance from the base of the groove to the bottom of the specimen
a Notch length
Y’ Dimensionless stress intensity factor

3 Results and discussion

Figure 2 presents photographs of the *p*CT specimens after testing at different temperatures for the IT (top) and TA (bottom) groups. In both groups, the apparent color of the specimens changes progressively with increasing temperature, evolving from white at low temperature (25 – 200°C) to grey and then dark grey at higher temperatures (400 – 600°C). A particularly notable feature is observed in the TA specimen treated at 600°C, whose outer surface is partially covered by a whitish layer, while the interior remains dark grey. This surface whitening is likely associated with reactions occurring during cooling and subsequent exposure to ambient moisture, as the whitish layer was not present immediately after the specimen was removed from the furnace. At high temperature, partial decomposition of calcite ($CaCO_3$) may produce lime (CaO), which can subsequently react with moisture in the air during cooling to form portlandite ($Ca(OH)_2$)) (Tomás et al., 2025; Vigroux et al., 2022). This process is accompanied by volume expansion and may promote local surface spalling, thereby exposing the darker interior of the specimen.In addition, the crack pattern observed in the TA specimen treated at 600°C is more complex than that of the corresponding IT specimen, with more pronounced crack branching. This observation suggests that the heating–cooling process generates more severe and heterogeneous damage than isothermal loading.

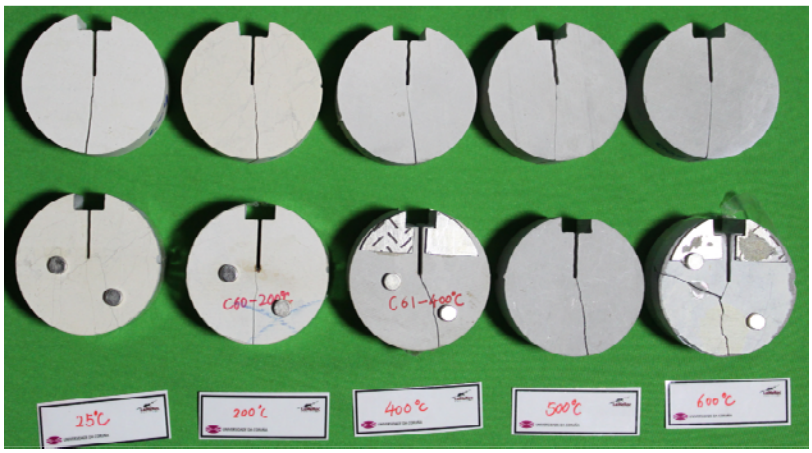


Fig. 2 Photographs of *p*CT specimens after testing under different temperatures for the IT group (top) and TA group (bottom).

The measured K_{Ic} values for limestone under IT and TA conditions are shown in Fig. 3. Although three IT specimens were tested at each temperature, some results are not reported because these tests were considered invalid: failure did not initiate from the tip of the pre-existing notch, and thus no reliable K_{Ic} value could be obtained. Overall, the K_{Ic} values obtained under IT conditions are consistently higher than those measured under TA conditions at the same target temperature. More importantly, the two thermal paths display distinctly different temperature-dependent responses. Under TA conditions, K_{Ic} decreases monotonically with increasing treatment temperature. The decrease before 400°C is relatively mild, whereas a more pronounced reduction occurs between 400 and 600°C.Under IT conditions, in contrast, K_{Ic} first increases and then decreases, reaching a maximum at an intermediate temperature of approximately 400°C. Notably, even at the highest temperature considered (600°C), the K_{Ic} value under IT conditions remains higher than the room-temperature reference value.

The more complex response of K_{Ic} observed under IT conditions can be interpreted as the result of a competition between temporary strengthening and progressive thermal damage while the specimen remains at elevated temperature during loading. Limestone, which is mainly composed of calcite may exhibit anisotropic thermal deformation. During heating, calcite may expand along one crystallographic direction while contracting along another, generating incompatible deformation between adjacent grains and inducing non-uniform thermal stresses within the rock (Rosenholtz & Smith, 1949; Tomás et al., 2025; Wong & Liu, 2025). At relatively low to moderate temperatures (25 –400°C), thermal expansion may partially close pre-existing pores and microcracks, thereby increasing resistance to tensile crack initiation and propagation. This crack-closure effect may explain the initial increase in K_{Ic} under IT conditions. With further increase in temperature (500 – 600°C), however, thermally induced damage becomes progressively more significant (Wang et al., 2025a; Wang et al., 2025b). The anisotropic thermal response of calcite enhances stress concentration around grain boundaries, pores, and cemented contacts, promoting the initiation and growth of new

microcracks. At sufficiently high temperature, partial decomposition of organic components and calcium carbonate may further increase pore development and weaken the internal structure of the rock. Consequently, after reaching a peak value, K_{Ic} decreases because thermal damage gradually outweighs the temporary crack-closure effect.

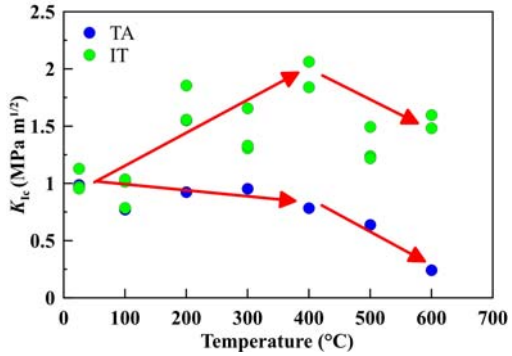


Fig. 3 Measured K_{Ic}values for TA specimens (blue dots) and IT specimens (green dots). The solid lines represent quadratic fitting curves, and the arrows indicate the general trend of each curve.

By contrast, the monotonic decrease in K_{Ic} under TA conditions is mainly associated with the residual thermal damage after cooling. During the heating–cooling process, the anisotropic deformation of calcite persists, and cooling leads to further incompatible shrinkage and stress redistribution. Consequently, microcracks formed during heating may reopen, propagate, or coalesce during cooling, while additional cracks may also develop due to differential contraction between adjacent mineral grains. The fracture toughness measured at room temperature after thermal treatment therefore mainly reflects the cumulative effect of irreversible thermal damage, resulting in a continuous reduction in K_{Ic} with increasing treatment temperature. In contrast, the TA specimens were held in the muffle furnace for 12 h, which is substantially longer than the holding duration used for the IT specimens. This prolonged thermal exposure may also be a contributing factor to the lower K_{Ic} values observed in the TA group, although further investigation is needed to confirm this effect.

Therefore, the difference between the IT and TA results is mainly attributed to their different thermal paths. Direct comparisons between IT and TA conditions remain limited because mechanical testing at elevated temperature is technically challenging. Nevertheless, previous studies support this interpretation. Hu et al. (2023) reported that the K_{Ic} of granite measured at elevated temperature is generally higher than that after thermal annealing, owing to the formation and retention of more irreversible microcracks during cooling. Similarly, Liu et al. (2020) found that sandstone exhibits higher elastic modulus at elevated temperature than after cooling, mainly because pre-existing cracks temporarily close during heating. Although these studies concern different rocks and mechanical parameters, they consistently show that thermal path strongly affects rock mechanical behavior by influencing crack closure, initiation, and accumulation. This agrees with the present limestone results, where K_{Ic} under IT conditions is higher than that under TA conditions at the same target temperature.

4 Conclusions

An enhanced pseudo-compact tension (*p*CT) apparatus has been employed in this study to investigate the mode I fracture toughness (K_{Ic}) of limestone under isothermal, high-temperature conditions and to compare the results with those obtained after thermal annealing treatment. The results show that the thermal stress path has a significant influence on the measured K_{Ic}: At the same target temperature, the K_{Ic} values obtained under TI conditions are consistently higher than those measured under TA conditions. Under TI conditions, K_{Ic} shows a non-monotonic variation with temperature, first increasing and then decreasing, which suggests that the fracture response is controlled by the competition between temporary crack closure and progressive thermal damage. Under TA conditions, however, K_{Ic} decreases monotonically with increasing treatment temperature. This difference is closely associated with the carbonate mineralogy of the limestone, which is dominated by calcite. Owing to its anisotropic thermal deformation, calcite generates non-uniform thermal stresses during both heating and cooling. The cooling stage, in particular, promotes stress redistribution, crack initiation, and crack propagation, thereby explaining the continuous reduction in fracture toughness observed in the TA specimens. At sufficiently high temperatures, possible decomposition of calcium carbonate may further weaken the rock structure. Overall, these results indicate that post-thermal treatment fracture

tests cannot directly represent the fracture behavior of limestone at elevated temperature. The thermal path must therefore be explicitly taken into account when assessing the fracture initiation and propagation of limestone in high-temperature engineering.

Acknowledges

The work has been supported by funds of the project PID2021-126419NB-I00 (FEDER) funded by MCIN/AEI/10.13039/501100011033 and "ERDF A way of making Europe". A.M.I. acknowledges support from the European Union's Horizon Europe Research and Innovation programme under the Marie Skłodowska-Curie grant agreement No. 101106038. Y.L. gratefully acknowledges the financial support from the China Scholar-ship Council (No. 202206560006).

References

Guo P, Bu M, Zhang P, Wang J, Luan Z,He M (2023). Mechanical properties and crack propagation behavior of granite after high temperature treatment based on a thermo-mechanical grain-based model. *Rock Mechanics and Rock Engineering*, *56*(9), 6411–6435. https://doi.org/10.1007/s00603-023-03408-x

Hu Y, Hu Y, Jin P, Zhao G, Zhao Z, Li C (2023). Real-time mode-I fracture toughness and fracture characteristics of granite from 20 °C to 600 °C. *Engineering Fracture Mechanics*, *277*(July 2022), 109001. https://doi.org/10.1016/j.engfracmech.2022.109001

Li Y, Herbón-Penabad M, Muñoz-Ibáñez A, Alejano LR, Alvarellos-Iglesias J, Canal-Vila J,Segura-Serra JM, Delgado-Martín J (2026). Effects of Confining Pressure on Mode I Fracture Toughness Considering Jacketed and Unjacketed Conditions. *Rock Mechanics and Rock Engineering*, *59*(1), 1393–1403. https://doi.org/10.1007/s00603-025-04878-x

Liu W, Zhang L, Luo N (2020). Elastic modulus evolution of rocks under heating-cooling cycles. *Scientific Reports*, *10*(1), 1–9. https://doi.org/10.1038/s41598-020-70920-3

Muñoz-Ibáñez A, Delgado-Martín J, Costas M, Rabuñal-Dopico J, Alvarellos-Iglesias J, Canal-Vila J (2020). Pure mode I fracture toughness determination in rocks using a pseudo-compact tension (*p*CT) test approach. *Rock Mechanics and Rock Engineering*, *53*, 3267–3285. https://doi.org/10.1007/s00603-020-02102-6

Rosenholtz JL, Smith DT (1949). Linear thermal expansion of calcite, var. Iceland spar, and Yule marble. *American Mineralogist: Journal of Earth and Planetary Materials*, *34*(11–12), 846–854.

Tomás R, Benavente D, Martínez-Ibáñez V, Garrido ME (2025). How do high temperatures affect rock properties? A comprehensive review of experimental thermal effects and underlying mechanisms. *Engineering Geology*, *357*(July), 108323. https://doi.org/10.1016/j.enggeo.2025.108323

Vigroux M, Sciarretta F, Eslami J, Beaucour AL, Bourgès A, Noumowé A (2022). High temperature effects on the properties of limestones: post-fire diagnostics and material's durability. *Materials and Structures*, *55*(10), 253. https://doi.org/10.1617/s11527-022-02086-5

Wang C, Zhang Q, He M, Guo S, Jia Y (2025a). Mechanism of fracture process zone development in sandstone and limestone under mode I loading wet-dry cycles and temperature. *Rock Mechanics and Rock Engineering*, *58*(7), 7307–7328. https://doi.org/10.1007/s00603-025-04519-3

Wang C, Meng Q, Lv S, Xiao L, Shen T, Luo L (2025b). Research on the fracture characteristics and thermal-damage mechanisms of coral reef limestone after high-temperature treatment. *Marine Georesources and Geotechnology*, *44*(4), 1–19. https://doi.org/10.1080/1064119X.2025.2550661

Wong LNY, Liu Z (2025). Thermal shock in rocks: A review of mechanisms, impacts, and applications in underground engineering. *Journal of Rock Mechanics and Geotechnical Engineering*, *17*(12), 8172–8197. https://doi.org/10.1016/j.jrmge.2025.01.036

Zhou Q, Zhu Z, Liu W, Lu H, Fan Z, Nie X, Li C, Wang J, Ren L(2024a). Hydraulic fracturing behaviors of shale under coupled stress and temperature conditions simulating different burial depths. *International Journal of Mining Science and Technology*, *34*(6). https://doi.org/10.1016/j.ijmst.2024.06.005

Zhou Q, Zhu Z, Zhang R, Fan Z, Nie X, Gao W, Li C, Wang J, Ren L (2024b). Rock fracture initiation under deep extreme in situ conditions: A review. *Journal of Rock Mechanics and Geotechnical Engineering*, *16*(12), 5297–5324. https://doi.org/10.1016/j.jrmge.2024.02.020

Zhu Z, Tian H, Jiang G, Cheng W (2018). Effects of high temperature on the mechanical properties of chinese marble. *Rock Mechanics and Rock Engineering*, *51*(6), 1937–1942. https://doi.org/10.1007/s00603-018-1426-0