# Influence of temperature on physico-mechanical properties of granite with reference to microcrack analysis

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# Abstract

Crucial engineering tasks such as the extraction of deep resources (geothermal energy extraction, deep mining of coal) or nuclear waste storage spark the pursuit in the examination of the physico-mechanical behaviour of rock materials under extremely high temperatures. Rock materials undergo significant changes in their physical, mechanical and chemical properties when exposed to extremely high temperatures for an extended duration. The rate and duration of high-temperature exposure, the state of stress and the mineral composition weigh how the rock materials behave. Although multiple researchers have focused on the changes in physico-mechanical behaviour of granites subjected to different thermal exposures, changes in mineralogy and the microstructures still require further investigation.

The primary objective of this study is to analyse changes observed in the physico-mechanical properties of granite before and after exposure to extremely high thermal conditions. To achieve the objective, the core specimens of Granites from Malanjkhand, Madhya Pradesh, India were systematically prepared and progressively subjected to treatment at 600 °C across several cycles. The physical properties, including effective porosity, dry density, and p-wave velocity, were quantified. Thereafter, mechanical tests, such as the Brazilian splitting test, are performed on the treated samples. Finally, microcrack analysis is conducted on the samples prior to and subsequent to the treatment, with the aid of an optical petrographic microscope and scanning electron microscope. The results acquired are thoroughly compared and analysed.

# Keywords

Thermal shock test, Brazilian tensile strength test, Rock fatigue, Physical properties.





# 1 Introduction

In the times when fossil fuel energy is being replaced with renewable resources to reduce the impact of environmental pollution, high-temperature rock property investigation of deep-seated rocks, such as granites, is crucial. Evaluating the physico-mechanical properties of granites in high-temperature conditions under thermal stress can be used for various engineering projects, for instance, deep rock drilling, establishment of nuclear storage facilities, hydraulic fracturing, geothermal energy exploration and extraction (Mardoukhi 2017; Zhang et al. 2021). Thermal shock can also be used to weaken rocks during drilling projects (Saksala and Ibrahimbegovic 2020). Additionally, during deep drilling or coring of rock, a strong temperature contrast can be observed between the high-temperature side-wall rocks and the cold drilling fluid (Xiao et al. 2021). Therefore, studying the behaviour of granites and its cracks influenced by thermal stress is important in assessing its durability and stability.

Thermal stress in rock builds up from the differential thermal expansion and contraction of its interior and exterior parts. This non-uniform temperature distribution leads to the alteration of physico-mechanical properties in various rock types, including granite (Yu et al. 2020). The test, namely thermal shock, induces these alterations caused by thermal stress in an accelerated manner (Wang et al. 2016).

The Brazilian splitting test is a commonly employed effective and feasible index test, which measures the indirect tensile strength of the rocks. The ease and practicality of preparing specimens have rendered it the most prevalent indirect method for assessing rock tensile strength (Coviello et al. 2005).

There are some studies which highlighted high temperature granites from various parts of the world (Chen et al. 2017; Dong et al. 2020; Fan et al. 2020; Heloisa et al. 2022). The majority of these studies have primarily focused on lower heating rates because high heating rates create microcracks rapidly. There is limited research on how a high heating rate affects the rocks and how microcracks behave. Additionally, research materials regarding physico-mechanical behaviour under the high-temperature influence of Malanjkhand granite, India, are scarce.

Therefore, in this study, several sets of granite specimens collected from Malanjkhand, India, were subjected to a high-temperature thermal shock test of 600 °C heating at a rate of 20 °C/min to understand its stability and durability. Following the thermal shock test, P-wave velocity and effective porosity were measured. Finally, the specimens underwent the Brazilian test to ascertain how the thermal change affected the tensile strength of the rock material.

# 2 Materials

# 2.1 Collection of samples

The core samples used in this investigation, which belong to the Malanjkhand granite group, were collected from Malanjkhand, Madhya Pradesh, India. The diameter of the cores was approximately 47 mm, which followed the standards of ASTM (2016).

# 2.2 Preparation of specimens

To abide by the objective of this study, the specimens are prepared in Brazilian discs and 10 cm cores in accordance with the standard ASTM (2016). Microscopic thin sections are also made for petrography and further SEM analysis.

# 2.3 Specimen details

The granite specimens were grey, coarse-grained and showed typical interlocking texture. It mainly contained quartz and feldspar (plagioclase and K-feldspar), along with moderate amounts of biotite and hornblende. The proportion of mafic minerals is uniformly distributed throughout the specimens. Deuteric alteration was prevalent in these specimens. This is primarily due to the specimen's close association with hydrothermal copper deposits. Most of the feldspar grains present in the rock samples were sericitized, as shown in Fig. 1. Some of the biotite and hornblende grains also underwent chloritization.



Fig. 1 (a), (c) PPL and (b), (d) XPL photomicrographs of the granite specimen. Qtz: Quartz, Fsp: Feldspar, Bt: Biotite, Hrn: Hornblende.

# 3 Methods

#### 3.1 Thermal shock cycles

The granite specimens were subjected to heat of 600 °C at a constant rate of 20 °C/min using a hightemperature furnace. After taking 30 minutes to reach the peak temperature, the specimens were held at that temperature for 2 hours. Subsequently, the heated granite samples were taken out from the furnace and immediately submerged in a container of 10 °C water. After cooling for 1 hour, the first cycle was concluded. The cycles were continued until the specimen failed due to thermal stress. In this investigation, the specimen gave away during the 6<sup>th</sup> cycle, i.e., the specimens failed inside the furnace due to thermal stress.

#### 3.2 Effective porosity and dry density

Porosity ( $\eta$ ) can be defined as an intrinsic property of the rock and a quantitative measurement of the voids present in the rock, such as pores, fissures and joints. There can be the presence of interconnection between the voids, or they can be separated from each other. In this study, the determination of effective porosity is considered. Effective porosity ( $\eta_e$ ) is the measure of the interconnectedness of the voids with reference to the permeability of water (Basu and Mishra 2014). The Brazilian disc specimens of the granite, which are above 50gm each, are taken into a container, and it is filled with water to submerge the samples completely. Then, they are shifted into a desiccator and kept in a vacuum for at least 24 hours, broadly following the standard mentioned in ISRM (1979). The effective porosity ( $\eta_e$ ) is determined with Eq. 1.

$$\eta_e = \left(\frac{M_{sat} - M_{dry}}{M_{sat} - M_{sus}}\right) * 100 \%$$
<sup>(1)</sup>

Where  $\eta_e$  effective porosity

- $M_{sat}$  Mass of saturated rock specimen with water
- $M_{dry}$  Mass of dry rock specimen
- $M_{sus}$  Suspended mass of the rock specimen immersed in the container of water

Dry density  $(\rho_{dry})$  of the specimen can be described as the dried mass of the rock specimen per unit volume (Basu and Mishra, 2014). See Eq. 2.

$$\rho_{dry} = \frac{M_{dry}}{M_{sat} - M_{sus}} \tag{2}$$

Where  $\rho_{dry}$  dry density

*M<sub>sat</sub>* Mass of saturated rock specimen with water

 $M_{dry}$  Mass of dry rock specimen

 $M_{sus}$  Suspended mass of the rock specimen immersed in the container of water

In this study,  $\eta_e$  and  $\rho_{dry}$  of the untreated specimens are recorded, followed by the treated samples after the thermal shock test. In this study, dry density is referred to as only density.

#### 3.3 P-wave velocity

The changes of wave velocities demonstrate the microstructural changes of the specimens, which includes mineralogy, shape, size, density, porosity, temperature, depth, direction and orientation of pores and grains (Basu and Aydin 2006). The index test used in this study determines the longitudinal velocity. The velocities determined depend on the elastic properties and the density of the specimens, where the microcracks or fissures, if present, produce an overriding effect. This effect helps to suggest the degree of fissuring within the rock core sample (Goodman 1989). An ultrasonic wave velocity test system is used. The transmitting transducer and the receiving transducer were held at the end faces of the core, and an ultrasonic wave was transmitted across the sample. The longitudinal wave velocity, also known as P-wave velocity ( $V_p$ ) is then noted from the ultrasonic digital datalogger. In this investigation, firstly, the  $V_p$  is noted for the untreated granite 10 cm core specimens and subsequently,  $V_p$  of the treated specimens is also recorded.

#### 3.4 Brazilian test

The Brazilian granite discs are loaded in between two flat platens, wherein compression is applied, which results in uniform tensile stress distribution in the specimen perpendicular to the loading direction. During this experiment, the displacement rate of the loading platens is kept constant at 0.5 mm/min. Brazilian tensile strength ( $\sigma_t$ ) is measured with Eq. 3.

$$\sigma_t = 2P/\pi LD \tag{3}$$

Where  $\sigma_t$  Brazilian tensile strength

P Peak load

*L* Length of the rock specimen

*D* Diameter of the rock specimen

Both untreated and treated specimens are mechanically tested, and data are recorded and further analysed.

# 4 Results and discussion

A high temperature of 600 °C influences any kind of rock to a greater extent, but to what extent? Physical changes are seen in the granite samples as shown in Fig. 2. After cycle 1, the formation of a redder tinge on the specimens was observed. Additionally, the biotite flakes showed a prominent brownish tinge, and as the cycle increased, the red/brown colouration became more prominent. This can be attributed to the oxidation of the iron compounds present in the rock specimens (Li et al. 2017).



Fig. 2 Comparison of the granite specimens untreated (cycle 0) and thermally treated to 600 °C (cycle 1, 3 and 5) side-byside. Yellow arrows show tiny cracks.

It is also seen in Fig. 2 that with increasing cycles, the development of more tiny cracks takes place. Cycle 1 hand specimens show rare minor cracks, whereas after cycle 5, the number of cracks increased drastically.

The average P-wave velocity  $V_p$  drastically decreased 68.51 % in the first cycle from 6065.09 m/s to 1914.64 m/s. The rate slowed down with the corresponding increase in the number of cycles, with the  $V_p$  being 702.99 m/s after cycle 5 (Fig. 3a).

Fig. 3b shows  $V_p$  decreases with the increase in  $\eta_e$  (Fig. 3b).



Fig. 3 (a) P-wave velocity with respect to number of cycles, (b) P-wave velocity with respect to effective porosity.

The effective porosity increased from 0.04 % (cycle 0) to 5.6% (cycle 5), which shows an increase of approximately 1400 %, as presented in Fig. 4a and 4b. It is also observed that the density of the granite decreased from 2.75 g/cc to 2.6 g/cc, which shows a decrease of 5.5 % (Fig. 4c and 4d). When comparing the Brazilian tensile strength, it can be observed that the strength decreased in a similar trend to  $V_p$ , i.e., after cycle 1 the strength drop is maximum (55.45 %), and then it decreases gradually to 81.3% after 5 cycles (Fig. 5). It is also seen that  $\sigma_t$  decreases with the increase in porosity. Additionally, Fig. 5c also shows a positive correlation between tensile strength and  $V_p$ .



Fig. 4 (a) Effective porosity, (b) change in effective porosity, (c) density and (d) change in density with respect to number of cycles



Fig. 5 (a) Brazilian tensile strength with respect to number of cycles, (b) effective porosity and (c) P-wave velocity.

The formation of microcracks increases the porosity disperses the P-waves traversing through it thus reducing  $V_p$  as well as reducing the tensile strength. Therefore, it can be fairly said that a series of microcracks is developed with the introduction of thermal stress in the granite specimen and with the increase in the number of cycles, the microcracks enhance, and hence, the damage increases until thermal fatigue. To analyse the depth, microcracks analysis is performed with the help of scanning electron microscopy (backscattered electron images) as shown in Fig. 6.



Fig. 6 (a)-(d) Scanning electron microscope images, (e) Avg. and Max. crack width with respect to number of cycles.

It is observed that from cycle 0 to cycle 1, hairline cracks are introduced along the grain boundaries, forming intergranular cracks. The average crack width of 2.16  $\mu$ m is increased to 14.48  $\mu$ m in cycle 3, the intergranular cracks widen, and the formation of intragranular cracks is seen (Fig. 6c). After cycle 5, the average crack width widens to 67.52  $\mu$ m, the network of intergranular cracks widened dramatically with a formation of many intragranular and transgranular cracks. However, only intergranular cracks have been quantified for comparison here.

# 5 Conclusions

This study was conducted to understand the stability and durability of Malanjkhand granite, which is in close association with copper deposits, under the influence of a high temperature of 600 °C, which is significant for quartz-containing rocks as  $\alpha$ -quartz is converted to  $\beta$ -quartz at 573 °C, with a high heating rate. From this study, it can be said that the development of microcracks and widening of intergranular pore spaces takes under extremely high temperatures.

Effective porosity increases by about 1400 %, whereas density decreases by 5.5 %. P-wave velocity decreases by 88.37%, and finally, Brazilian tensile strength also decreases by 81.3% after cycle 5.

When looking at multiple cycles of high-temperature treatment, intergranular cracks form at an earlier stage, followed by intragranular cracks, which leads to the formation of transgranular cracks (a combination of both intergranular and intragranular cracks) at later cycles. Therefore, it can be said that interconnection of the pores and cracks are enhanced by high temperatures.

# Acknowledgement

The servo-controlled compression test machine utilised in this study was funded by the Science and Engineering Research Board, File No.:CRG/2019/000899. The authors express gratitude to Hindustan Copper Ltd. (Malanjkhand Copper Project) for supplying core samples. The aid and support of IIT Kharagpur is sincerely appreciated.

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