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Study of Thermal Behaviour of Micro-Cracks in Granulite

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ABSTRACT

Presence of micro-cracks significantly affects physico-mechanical properties of rocks. Behaviour of micro-cracks changes when rock is subjected to high temperatures. Effect of micro-cracks' on behaviour of granulite (also known as charnockite in India) rock samples was studied at temperatures, 30°C (room temperature), 65°C, 100°C, 125°C, 160°C and 200°C by carrying out permeability and ultrasonic tests. In addition, study of visual micro-cracks' was also carried out at aforementioned temperatures using Scanning Electron Microscope (SEM). The heat treated granulite rock specimens were cooled to room temperature before carrying out the studies presented here. The study revealed that development of new micro-cracks and increase in length of pre-existing micro-cracks started considerably from 100°C upwards.

Keywords: Micro-cracks; Granulite; P-wave; Permeability; SEM

1. INTRODUCTION

Micro-cracks in rocks are the rock defects which influence its physico-mechanical properties (Friedman and Johnson, 1978; Baur and Johson, 1979; Siegfried and Simmons, 1978; Alm et al., 1985; Etinne and Houpert 1989; Darot and Reuschle, 2000). In addition to mineralogy, structure, stress and time, the mechanical properties of rocks also depend upon the temperature (Darot and Reuschle, 2000; Dwivedi et al., 2008). The activities where rocks are subjected to high temperature like recovery of geothermal energy and underground disposal of high level nuclear waste, knowledge of thermal behaviour of micro-cracks is imperative (David et al., 1999). In conventional deposition of high level nuclear waste in the deep underground tunnels in rock, the nuclear waste stored on surface for sufficiently long time is subsequently planned to be buried underground. The rock temperature in such conventional storage therefore may not rise beyond 250°C (USDE 1980; Bergman, 1980). In this view, the present study deals with micro-cracks' behaviour of granulite (popularly known as charnokite in India) rock at high temperatures in the range of 30 - 200°C is presented.

Permeability, a transport property is directly correlated to porosity. It depends strongly on pore space microstructure, i.e., connectivity, pore/crack geometry, pore/crack density, tortuosity, etc. (Ravalec et al., 1996) and the pores get influenced by high temperature. Thus study of permeability also indicates change in connecting pores/fractures within the rock specimens.

For the study, the granulite rock specimens were treated at various temperature levels up to 200ºC and cooled down to room temperature to obtain the heat-treated specimens. For each test, five rock specimens were tested at one temperature. These heat-treated specimens were then subjected to various tests viz., permeability, ultrasonic P-wave velocity and Scanning Electron Microscope (SEM). During cooling of specimens at room temperature, widening of new and pre-existing micro-cracks takes place. Thus, the width of micro-crack would be little larger on cooling the specimens as compared to the case of maintained constant high temperature. Hence, the results of thermal properties of heat treated (heated and cooled) specimens would be in a little conservative side, if utilized for design of underground high level nuclear waste repositories, where the rock mass continues to be at high temperature as there is no cooling.

The variation in temperature affects micro-cracks in granulite rocks because of its mineralogical components heterogeneity or phase transition of some components. The different minerals have different coefficients of thermal expansion leading to a creation of intergranular compressive and tensile forces. Depending upon the temperature intensity, thermal cracking can occur either between adjacent crystalline grains (intergranular cracks) or within grains (intragranular cracks) (Chaki et al., 2008). In the present study, micro-cracking is observed on heating and subsequent cooling of different rock mineral grains. The cracking phenomenon occurs due to different thermal expansions of minerals in poly mineral rocks like granulite and also in mono-mineral rocks which exhibit anisotropic expansions due to random orientation of minerals (Somerton, 1992). Thus, mineralogical composition is a main factor on which rock sensitivity to temperature variation depends.

Thermal treatment exhibits increase of volume of micro-cracks in the rock specimens and especially, cracks have an essential influence on the elastic properties of rock and hence they flow on velocity of seismic waves propagating in the fractured rock mass (Yoshimitsu et al., 2009; Kleczek and Idziak, 2008). Decrease in P-wave velocity with increase in temperature has been inferred here as increase in the volume of micro-cracks in the specimen.

2. PETROGRAPHIC ANALYSIS AND PHYSICAL PROPERTIES OF GRANULITE

2.1 Geology and Mineralogical Composition

Granulite, popularly known as charnokite in India, is a medium to coarse grained metamorphic rock consisting of quartz, feldspar (soda-calcic feldspar-plagioclase), hypersthene, biotite and other minerals like garnet. It is mostly greyish black in colour. Two microscopic views of granulite are shown in Fig. 1. Quartz and feldspar are comparatively smaller in size (approximately 0.15-0.25 mm) and occurred in clusters surrounded by hypersthenes. Hypersthene is coarse grained (grain size upto 2 mm) and are subhedral to anhedral. The granulite rock has maximum percentage of quartz and soda-calcic feldspar followed by hypersthenes and other minerals like biotite and oxides. Detailed mineralogical composition of granulite is given in Table 1.

Table 1 - Mineralogical composition and grain size of granulite

Minerals	Hypersthene	Quartz and Plagioclase	Biotite and Garnet	Oxides (Opaque)
MC (%)	20-30	60-70	<10	<1
GS (mm)	≈2	0.15-0.25	0.25-0.50	0.25-0.50

Notation: MC = Mineralogical composition; GS = grain size

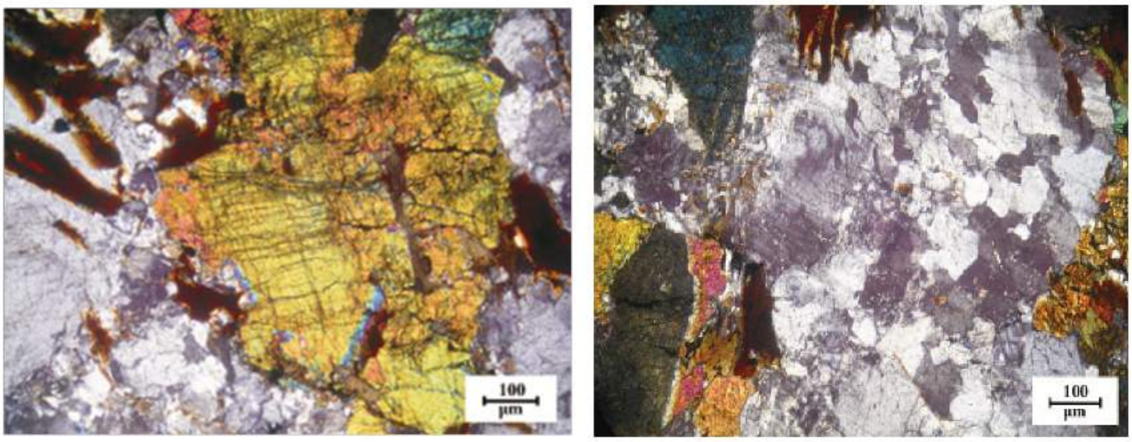


Fig. 1 - Microscopic views of granulite

The micro-cracks in some of the minerals such as hypersthene (pyroxene), garnet and biotite contain brownish opaque infillings whereas the micro-cracks in quartz and feldspars were found to have infillings of secondary minerals (Rao et al., 2006).

2.2 Physical Properties

The granulite has following physical properties at room temperature.

- Bulk density : 2.6×10^3 kg/m^3
- Permeability : 4.58×10^{-19} m^2
- Porosity : 0.8%

3. SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

Four cubic specimens of 30 mm size were used for this study. For clarity of pre-existing micro-cracks, one surface of each specimen was polished. Subsequently, golden polish was applied to the polished surface of the specimen in order to make it a conductor of electricity. The polished surface was studied by SEM. The surface area of the specimen was magnified by about 1000 times for observation of micro-cracks.

At first, SEM images of various micro-cracks on the polished surface of each specimen were studied at room temperature (RT), i.e. 30°C. All the specimens were then heated up to 65°C and maintained this temperature for six hours and cooled. After such heat treatment, the surface of each specimen was scanned through SEM to observe for the new micro-cracks and the temperature effect on pre-existing micro-cracks. The same procedure was adopted to study the micro-cracks at 100°C, 125°C, 160°C and 200°C. In this study, the same portion of rock specimens' surface was studied at every temperature level to observe the clear effect of temperature on pre-existing micro-cracks.

It was observed that new micro-cracks started developing from 100°C upwards. Maximum number of new micro-cracks developed at 200°C. In addition, the thermal treatment excites all the initial micro-cracks and their length and width increased with rise in temperature.

3.1 Micro-Crack Measurement

Measured length of micro-cracks was adjusted according to Galton's laws. Generally, application of these laws is relevant for all rocks (Warren, 1982). Crack length mode for the every heat treated specimen was determined. Value of crack length mode increases by 12.4% for the specimen treated at 200°C as compared to the non-treated specimen (Fig. 2). Crack length mode is the length of micro-crack which was most commonly or frequently observed during SEM analysis.

Width of pre-existing micro-cracks was also observed to be increased with temperature but the increment was not so significant in the tested temperature range i.e. between 30°C and 200°C, therefore not measured.

Cracking density is determined by the ratio of cumulated crack length to unit observed area. Value of crack density (C_D) was observed to follow increasing trend with temperature like crack length mode (Fig. 3). C_D-values were determined to be 1.72mm^{-1} and 2.41mm^{-1} at room temperature and 200°C respectively (Fig. 3).

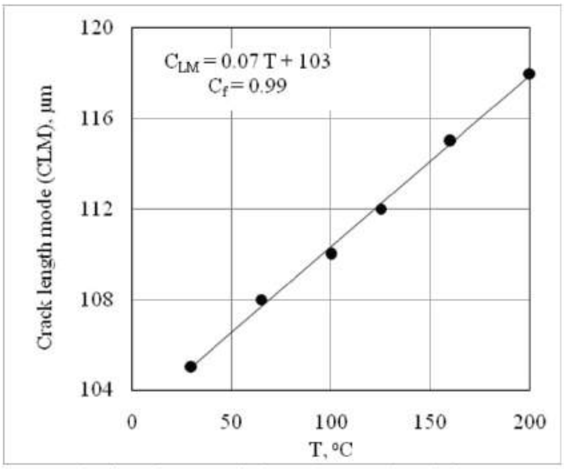


Fig. 2 - Variation in crack length mode with temperature

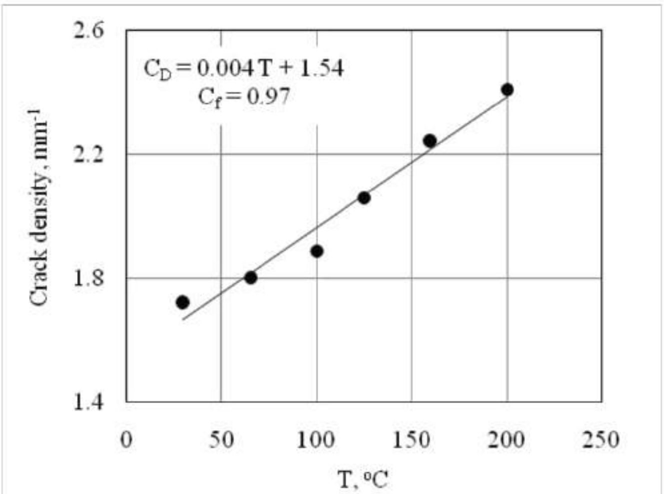


Fig.3 - Variation in crack density with temperature

4. P-WAVE VELOCITY

The velocity of longitudinal waves (P-waves) was measured at room conditions using the ultrasonic pulse transmission technique. P-wave velocity depends on the wavelength and size of the cracks. If they are of the same order of magnitude, the waves are scattered and it results macroscopically in P-wave attenuation. On the other hand, if the cracks are much smaller than the wavelength, then they modify the effective elastic properties of the rock and the P-wave velocity decreases.

4.1 Experimental Setup

The experimental setup includes a waveform generator, two piezoelectric transducers mounted on specimen holders, a small loading frame and a digital oscilloscope. The signal sent to the transmitter is a single sinusoidal period with 700kHz dominant frequency which matches the resonance frequency of the transducers. Velocity is calculated from the travel time of the P-waves through the rock sample (Bungey et al., 2006). A constant pressure was symmetrically applied (with a constant mass) to ensure a tight contact between the sample and the transducer. In addition, the quality of the recorded signals was improved applying a gel onto the contact surfaces of the specimen holders.

4.2 Specimen Preparation and Testing Procedure

Granulite rock cores of 54 mm diameter (NX size) and length 135 mm were used for the study. P-wave velocity measurements were carried out in three orthogonal directions. Each specimen was provided eight pairs of data points for radial measurements (four pairs in *OX* direction and other four pairs in *OY*-directions) and four pairs of data points for the axial measurements (in *OZ* or borehole direction) (Fig.4). The testing was carried out for six temperature viz. 30°C (room temperature), 65°C, 100°C, 125°C, 160°C and 200°C. After heat-treatment at a temperature level, the rock specimen was cooled down to room temperature before starting the ultrasonic study. During the ultrasonic testing the travel time was recorded for all pairs of points. Then the same rock specimen was heated at higher temperature level and again travel time for ultrasonic wave was recorded and so on. So, there is repeated heating and cooling of same specimen for five times. Five rock specimens were tested. The study therefore will also show the repeated heating and cooling effect.

For heat treatment of rock specimens, one environmental chamber was designed and fabricated indigenously. It is capable of maintaining maximum temperature of 300±2°C. Heating rate can be regulated between 0.5 and 3.0°C / min (Dwivedi et al., 2002). The specimens were heated at slow rate (1.5°C / min) to avoid thermal shock. To ensure achievement of desired temperature inside the rock core, a dummy specimen was used of the same rock. A thermo couple was inserted into this dummy specimen to record the actual temperature at the centre of the core. After achievement of the desired level of temperature inside the core, the specimen to be tested was left at such temperature for 6 hours. The environmental chamber was then switched off and the rock specimens were left inside the chamber to attain the room temperature before starting the test.

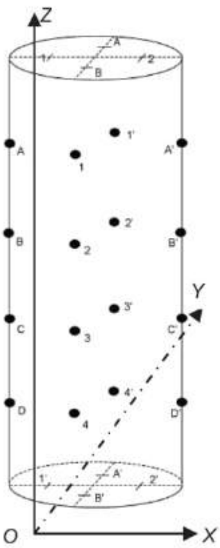


Fig. 4 - Twelve pairs of data points marked on the specimen.

4.3 Results and Discussion

Figures 5 and 6 show the values of average P-wave velocities in granulite at various temperatures in the radial and axial directions. In general, it was observed that P-wave velocity decreased with increase in temperature upto 200°C for all the tested rock specimens. For example, V_p-value was observed to be 5334m/s and 3748m/s at 30°C (room temperature) and at 200°C respectively along radial direction of the specimen (Fig. 5). As such a decrease in V_p-values of 30% in the radial direction and 23.4% in the axial direction were recorded as compared to the respective values at room temperature (Fig. 6). V_P-value decreases at the rate of 10m/s per °C in radial directions (Fig. 5) and at 9m/s per °C in axial or borehole direction of the specimens (Fig. 6).

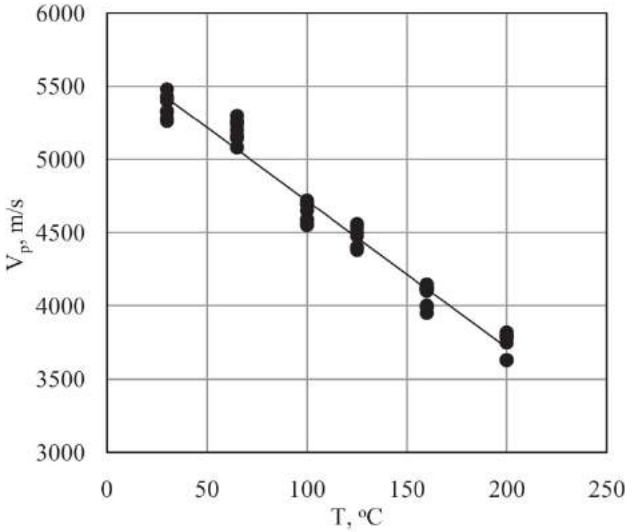


Fig.5 - P-wave velocity in the radial directions

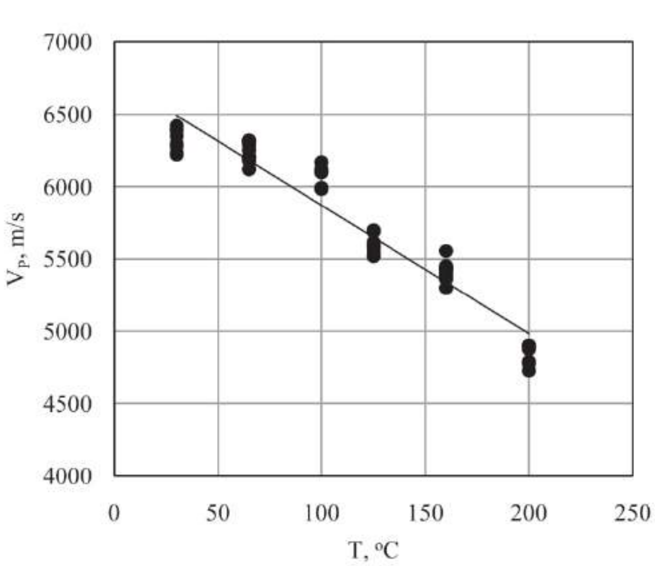


Fig.6 - P-wave velocity in the axial (OZ) or borehole direction

V_p-values at room temperature are higher along the axial direction as compared to the radial directions. It indicates that the orientation of maximum pre-existing micro-cracks is along the axial direction. Slope of plot in Fig. 5 is greater than that of the plot in the Fig. 6. It shows that the rate of decrease in V_p-values is faster in radial directions as compared to the axial direction. It indicates that the orientation of maximum newly developed micro-cracks and extended and widened pre-existing micro-cracks were in the axial direction of the specimens. In all the three orthogonal directions, P-wave velocity decreased with increase in temperature. Decrease in P-wave velocity with temperature is due to development of new micro-cracks and extension & widening of pre-existing micro-cracks took place especially from 100°C onwards.

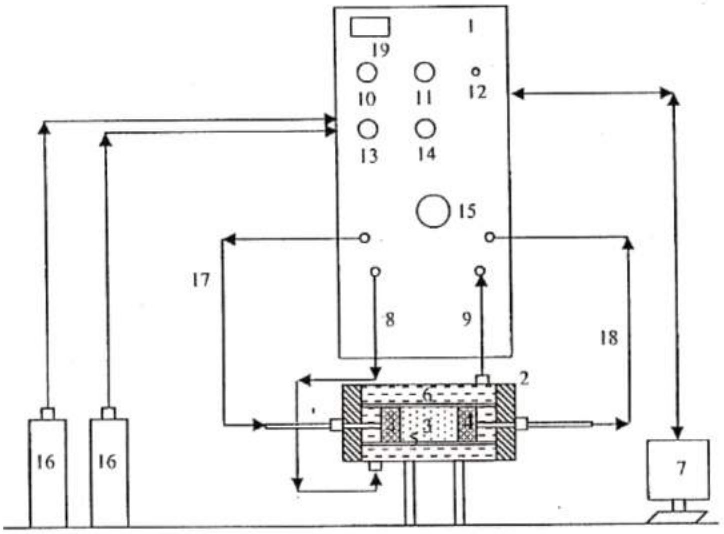
5. PERMEABILITY

5.1 Equipment

Permeameter (TEMCO, USA) was used for determination of permeability. It uses the constant gas (N_2) flow rate technique and provides an accurate and non-destructive measurement of permeability of rock core specimens. A computer data acquisition system attached to the equipment automatically computes the permeability and stores the data. Main parts of this equipment are core holder, back-pressure regulator and confining pressure pump (Fig. 7). The computer software automatically calculates permeability of the core specimen using the Darcy's equation. This equipment is designed to measure permeability in the range of 10^{-20} m^2 to 10^{-11} m^2.

5.2 Specimens for the Permeability Study

Granulite core specimens of diameter 36mm were used for permeability test. The length of each specimen was kept as 150mm. The tests were conducted on heat treated specimens at 30°C (room temperature), 65°C, 100°C, 125°C, 160°C and 200°C. To avoid thermal shock, the specimens were heated at a slow rate of 1.5°C per minute. A total of five specimens were tested at one temperature level.



1.Control panel; 2.Confining pressure system; 3.Rock specimen; 4.Distribution plug; 5.Sleeve; 6.Confining fluid; 7.Data acquisition system; 8.Confining bottom; 9.Flow from confining bottom; 10.Gas regulator inlet; 11.Back pressure regulator (BPR); 12.Fine controller; 13.Confining pressure regulator; 14.Fine controller (confining pressure); 15.DP display; 16.N2 cylinder; 17.Gas flowing to core; 18.Gas flow out from core; 19.Inlet pressure display

Fig.7 - Line diagram permeability testing equipment

After testing the permeability at room temperature, the same specimens were then heated to 65°C for 6 hours and tested after cooling down to room temperature. Again, these were heated to 100°C and tested at room temperature and so on. Thus, the same rock specimens were tested at five temperature levels and each specimen was subjected to five thermal cycles. These thermal cycles gave rise to increased volume of micro-cracks. The specimens were tested for permeability at a confining pressure of 4MPa and an axial upstream pressure of 3MPa.

5.3 Results and Discussion

P-Wave and SEM study of the heat treated specimen revealed that development of new micro-cracks and extension of pre-existing micro-cracks increased with temperature. The degree of permeability depends on the size and shape of the pore space and the extent of their interconnections (Geraud, 1994). Increasing trend of gas permeability (K_g) for the heat treated specimens at high temperatures also indicate that volume of interconnected pores increased with increase in temperature (Fig. 8).

Permeability increased with the increase in temperature up to 200°C for rock specimens (Fig.8). Newly developed micro-cracks and widened pre-existing micro-cracks with temperature as revealed by SEM analysis provide more apertures for gas-flow and hence increased permeability. This may be attributed to the repeated heating and cooling of specimens. Therefore, permeability of granulite increased with temperature. A significant increase of about 50% in permeability values takes place from 100°C to 200°C (Fig. 8). This is due to volumetric increase of micro-crack volume in this temperature range. Page and Heard (1981) used porosity measurements of Climax granite (CIG) up to a temperature of

500°C to estimate permeability and concluded that permeability increases with increase in temperature (Page and Heard, 1981).

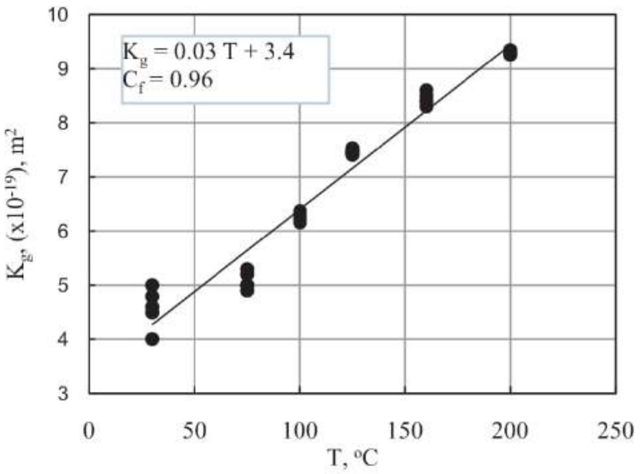


Fig. 8 - Changes in permeability (K_g) of granulite rock at high temperatures

Granulite is a poly-mineral rock mainly containing quartz, plagioclase and hypersthenes having coefficient of linear thermal expansion (α) values 13.7 x 10^{-6} / °C, 4.7 – 6 x 10^{-6} / °C, 12.1 x 10^{-6} / °C respectively causing high differential thermal expansion leading to more widening of pre-existing micro-cracks (Rzhevsky and Novik, 1971; Mukhopadhyay et al., 2007). In addition to this, high difference in grain sizes of the minerals constituting the rock also accrues the differential thermal expansion effect (Table 1).

6. CONCLUSIONS

Scan electron microscope (SEM), ultrasonic P-wave and permeability studies indicate that there is no practical effect of temperature upto 65°C, whereas considerable decrease in V_P-values and increase in permeability with temperature from 100°C upwards indicate that volume of micro-cracks increased considerably with increase in temperature from 100°C to 200°C. SEM tests also revealed that there was no significant variation in micro-cracks upto the temperature of 65°C. It started significantly changing from 100°C upwards. Development of new micro-cracks started considerably at 100°C and increased with temperature upto 200°C. Crack density was observed to be 1.72mm^{-1} and 2.41mm^{-1} at room temperature and 200°C respectively (Fig. 3). Widening of pre-existing micro-cracks was also observed to be increased with temperature but the increment was not significant. Increase in length of pre-existing micro-cracks was observed to be considerable from 100°C upwards. Crack length mode was determined to be 105μm and 118μm at room temperature and 200°C respectively (Fig. 2). Further, higher V_P-values in axial direction of the specimens, indicate that majority of the micro-cracks were aligned along the major axis of the rock-cores.

Above conclusions indicate that considerable widening of micro-cracks in granulite took place in the temperature range of 100°C to 200°C. The credit for this effect may be given to its large sized grains. Structural parameter, like granularity can amplify differential expansions, porosity and cracks, both leaving free space for expansion (Cooper and Simmons, 1977; Rzhevsky and Novik 1971). Large grains are most sensitive to temperature increase (Mukhopadhyay et al., 2007). The granulite is also a metamorphosed granular rock having maximum mineral grains size of 2mm and hence resulted in rise to differential thermal expansion on heating.

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References

Alm, O., Jaktlund, L. L. and Kou. S. (1985). The influence of micro-crack density on the elastic and fractrue mechanical properties of Stripa granite, Physics Earth Planet Interiors, 40, pp. 161-169.

Baur, S. J. and Johson, B. (1979). Effects of slow uniform heating on the westerly and charcoal granites, Proceedings of 20th Symp. on Rock Mech., Austin, Texas and also in: ASCE, New York, pp. 7-18

Bergman, M. S. (1980). Nuclear waste disposal,Subsurf Space, 2, pp. 791-1005.

Bungey, J. H., Millard, S. G. and Grantham, M. G. (2006). Ultrasonic pulse velocity methods, Testing of concrete in rock structures (Pub: Taylor & Francis Group), pp. 51-81.

Chaki, S., Takarli, M. and Agbodjan, W. P. (2008). Influence of thermal damage on physical properties of a granite rock: Porosity, permeability and ultrasonic wave evolutions, Construction and Building Materials, 22, pp. 1456-1461.

Cooper, H. W. and Simmons, G. (1977). The effect of cracks on the thermal expansion of rocks, Earth Planet Sci. Lett., 36 pp. 404 -12.

Darot, M. and Reuschle, T. (2000). Acoustic wave velocity and permeability evaluation during pressure cycles on a thermally cracked granite, Int. J. Rock Mech. Min. Sci., 37, pp. 1019-1026.

David, C., Menendez, B. and Darot, M. (1999). Influence of stress induced and thermal cracking on physical properties and microstructure of La Peyratte granite, Int. J. Rock Mech. Min. Sci., 36(4), pp. 433-448.

Dwivedi, R. D., Goel, R. K., Prasad, V. V. R. and Sinha, A. K. (2008). Thermo-mechanical behaviour of Indian and other granites, Int. J. Rock Mech. Min. Sci., 45, pp. 303-315.

Dwivedi, R. D., Goel, R. K. and Prasad, V. V. R. (2002). Thermo-mechanical behaviour of Indian granite, Proceedings of ISRM Indian Symp. on Advancing Rock Mechanics Frontiers to Meet the Challenges of 21st Century, New Delhi, India, pp. II 39-II 44.

Etinne, H. F. and Houpert, R. (1989). Thermally induced micro-cracking in granites: characterization and analysis, Int. J. Rock Mech. Min. Sci. Geomech. Abstr., 26, pp. 125-134.

Friedman, M. and Johnson, B. (1978). Thermal cracks in unconfined Sioux quartzite, Proceedings of 19th Symp. on Rock Mechanics, Austin, Texas and also in: ASCE, New York, pp. 35-40.

Geraud, Y. (1994). Variations of connected porosity and inferred permeability in a thermal cracked granite. Geophys. Res. Lett., 21(11), pp.979-982.

Kleczek, I. S. and Idziak, A. F. (2008). Anisotropy of elastic properties of rock mass induced by cracks, Acta. Geodyn. Geomater., 5(2-150), pp.153–159.

Mukhopadhyay, A. K., Neekhra, S. and Zollinger, D. G. (2007). Preliminary characterization of aggregate coefficient of thermal expansion and gradation for paving concrete, Texas Department of Transportation and Federal Highway Administration Report, 0-1700-5.

Page, L. and Heard, H. C. (1981). Elastic moduli, thermal expansion, and inferred permeability of climax quartz monzonite and Sudbury Gabbro to 500°C and 55 MPa, Lawrence Livermore national Laboratory Report, UCRL-85736.

Rao, M. V. M. S., Prasanna, Lakshmi K. J. and Sarma, L. P., Chary, K. B. (2006). Elastic properties of granulite facies rocks of Mahabalipuram, Tamil Nadu, India, J. Earth Syst. Sci., 115 (6), pp. 673-683.

Ravalec, M., Darot, M., Reuschlé, T. and Guéguen, Y. (1996). Transport properties and microstructural characteristics of a thermally cracked mylonite, Pure and Applied Geophysics, 146(2), pp. 207-227.

Rzhevsky, Y. and Novik, G. (1971). The Physics of Rocks, Mir publishers, Moscow, 320 p.

Siegfried, R., Simmons, G. (1978). Characterization of oriented cracks with differential strain analysis, Geophys. Res., 83, pp. 1269-1278.

Somerton, W. H. (1992). Thermal Properties and Temperature Behaviour of Rock/Fluid Systems, Elsevier (UK), 256 p.

U. S. Department of Energy (1980). Statement of the position of the United States Department of Energy in the matter of rulemaking on the storage and disposal of nuclear wastes, Report DOE/NE-0007, April.

Warren, N. (1982). Statistical characterization of complex crack and petrographic texture: application to predicting bulk physical properties, CR 23rd Symp. on Rock Mech., Berkeley, USA, pp. 132-139.

Yoshimitsu, N., Kawakata, H. and Takahashi, N. (2009). Broadband P-waves transmitting through fracturing westerly granite before and after the peak stress under a triaxial compressive condition, Earth Planets Space, 61, pp. e21–e24.