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Determination of Thermal Properties of Rock Samples Using Modified Thermal Block Method

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Abstract

Thermal properties are usually the key parameters governing the heat transfer through any material. Hence in modeling the thermal behavior of materials, accurate determination of thermal properties is quite imperative. This paper deals with the determination of thermal conductivity, thermal diffusivity and volumetric heat capacity of rocks by block method with thermal contact resistance consideration and the validation of the results with that of KD2 device. Thermal properties of granite, limestone and gneiss rocks were determined in the laboratory with the use of thermal interface material (TIM) Arctic Silver to find out the effect of contact resistance. KD2 probe was also used with and without TIM to compare thermal conductivities results. Thermal conductivity of granite, limestone and gneiss increased from 2.96 to 3.96 W/mK, 2.02 to 2.68 W/mK, and 1.64 to 2.20 W/mK respectively while Thermal diffusivity increased from 0.41 x 10^{-4} to 0.67 x 10^{-4} m^2/s, 1.09 x 10^{-4} to 7.38 x 10^{-4} m^2/s, and 0.65 x 10^{-4} to 1.44 x 10^{-4} m^2/s respectively. Thermal conductivities with and without TIM were tested statistically using (FPLSD) and it was found that using TIM to correct contact resistance was significant at ($P > 0.05$).

Keywords: Rocks, Thermal Properties, Block Method, Thermal Interface Material, Thermal Contact Resistance.

Introduction

The study of heat transfer has continued to find significance in understanding thermal behavior, especially in relation to determining underground heat flux. Thermal property determination is also important in studying water balance and mass exchange processes occurring across porous media surfaces. Thermal contact resistance (TCR) is the resistance to heat transfer at an interface due to poor physical contact between adjoining objects of differing shapes or roughness and is a function of the amount of air in contact with the measuring probe. Thermal contact resistance has been the greatest concern with regards to accuracy of thermal properties measurements (Hadas, 1974, 1977; Sauer *et al.*, 2003, 2005, 2008). The correlation between thermal properties and moisture content of soils and rocks obtained during laboratory experiments have also been used to determine these properties in the field (Akinyemi and Mendes, 2007a).

Samples used in this study were collected from south western Nigeria comprising rocks of the Precambrian basement. This region of the country is very much affected by geological exploration activities due to well-logging and bore-hole construction. Nigeria lies between latitudes 50 and 140N and longitudes 30 and 140E and crystalline basement rocks of Precambrian age underlie about 50 % of the country (Muotoh *et al.*, 1988). These are unconformably overlain by sedimentary rocks of Cretaceous to recent age (Fig.1). Investigators have measured thermal properties of rock materials using various methods (Blackwell, 1945; ASTM D5334-00, 2000; ASTM D5930-01, 2001; Fasunwon *et al.*, 2008), but little attempts have been made so far on the use of block method to determine thermal properties of rocks. Contact method had been used in the past exclusively on granular materials but little is known on its use on rock materials (Bruijn *et al.*, 1963; van Haneghem *et al.*, 1983; van Wijk, 1964, 1967; Akinyemi and Sauer, 2007b; Akinyemi *et al.*, 2011; Stigter, 1968). Moreover, scientists have raised accuracy concerns arising from thermal contact resistance between the block and the sample surface thereby necessitating further research on the method (Schneider, 1969). The objectives of this work, therefore, were to apply the block technique on rock samples of known thermal properties using thermal interface materials to address the contact resistance errors, and to validate the results using KD2 thermal analyzer.

Method and Instrumentation

Block device was used to make measurements of thermal conductivity of granite, limestone and gneiss while KD2 thermal analyzer was used to make instantaneous measurements of thermal conductivities for validation.

Block Device Setup:

Thermal Block device was made using Perspex (10 x 10 x 4 cm) with λ_p = 0.18568 W/mK, $C_{p\,=}$ 1.728 x 10^{-4} J/m^{3}K. Copper - constantan thermocouple line the flat surface and also upward at depths (2, 4, 8, 16 and 32 mm) from contact surface inside the block (Fig. 2), at which the initial temperature at the instant t = 0, which must be uniform, is measured. The device obtains measurements of thermal properties at the surface which no other method that use line source probes does including KD2 thermal properties analyzer. The block and the KD2 data should agree, however many times in soil, the surface is either much wetter or drier than below so that the block method is really the only way to get the true surface layer thermal properties. The block with an insulation cover (2.54 cm - thick Styrofoam) is placed in a thermostat and after a quarter of hour the temperature at the surface of the block and within it was recorded for a short time to measure the initial temperature of the block and ensure a uniform temperature. After removing the insulation plate covering the lower surface, the block is quickly placed on the sample surface, the time of contact being taken at t = 0, while the contact temperature was recorded through thermocouple data logger for about 5 minutes.

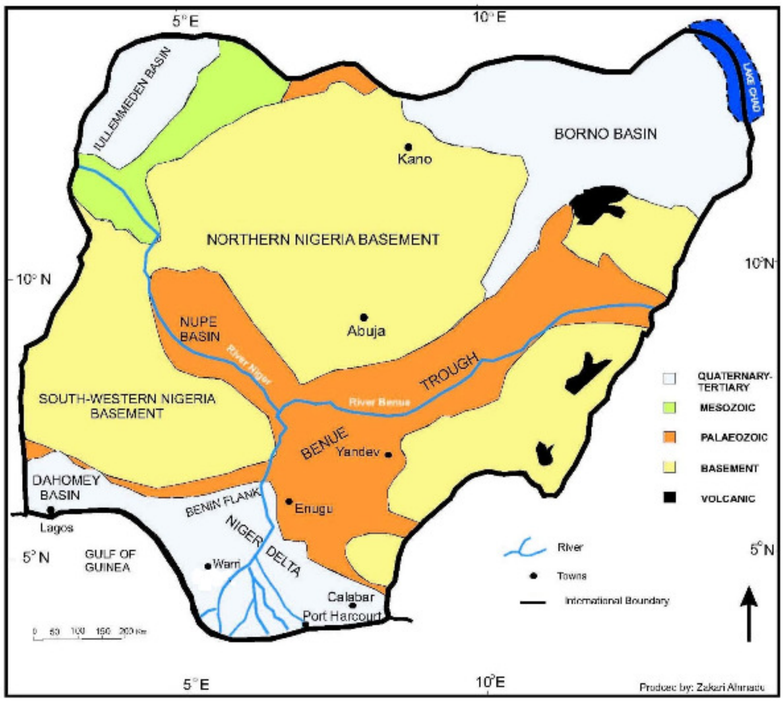


Fig. 1: Geological map of Nigeria.

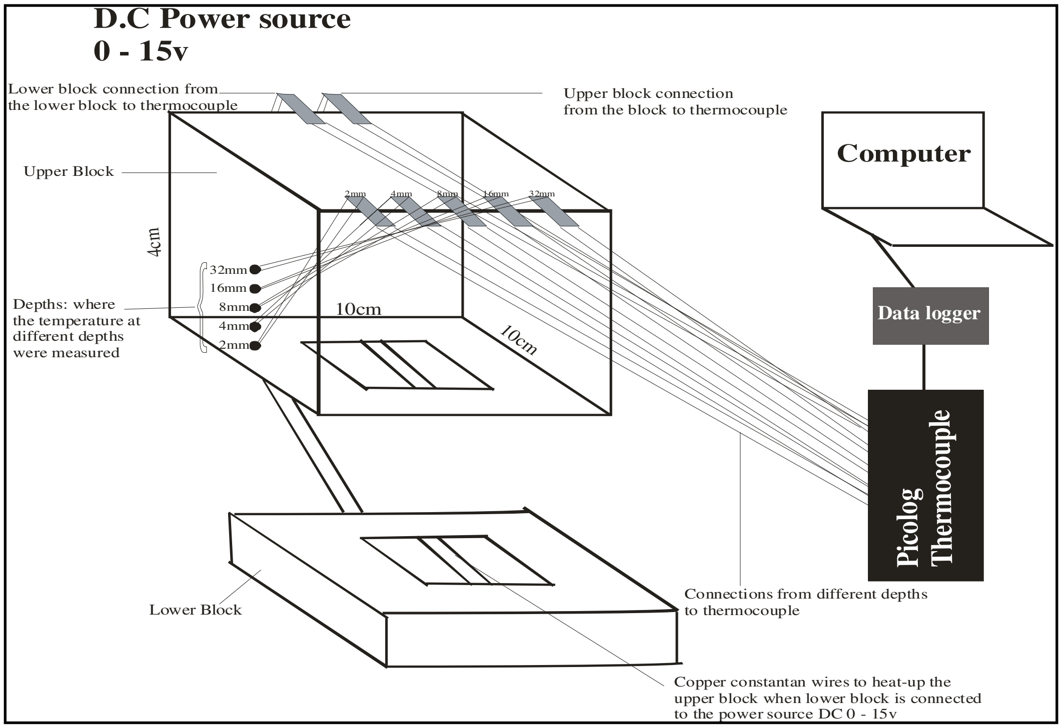


Fig. 2: Contact/ Block apparatus setup.

Fig. 3: KD2 probe in a drilled hole inside a sample slab.

The temperature near the center of the contact plane is calculated from the theory of two bodies suddenly brought into contact along the plane z = 0 at the instant t = 0. The temperature changes according to the equation (Carslaw and Jaeger, 1959):

$$\frac{\delta\theta_i(z,t)}{\delta t} = \alpha_i \frac{\delta^2\theta(z,t)}{\delta z^2} \qquad (1)$$

Where i = 1 for block, and i = 2 for sample, thermal diffusivity α (m^2/sec) = λ/ C = $\lambda/\rho c$, where λ (W/mK) is the thermal conductivity, C (J/m^3 K) is the heat capacity per unit mass, ρ (kg/m^3) is the density, and c (J/ kgK) is the specific heat. Using the Laplace Transform of θ_1 (z, t), the temperature of the block's contact plane is given as (Stigter, 1968):

$$T_1(0,t) = \frac{T_{1in}\sqrt{\lambda_1 C_1} + T_{2in}\sqrt{\lambda_2 C_2}}{\sqrt{\lambda_1 C_1} + \sqrt{\lambda_2 C_2}} + \frac{2}{\pi}\frac{\lambda_1 E_1 + \lambda_2 E_2}{\sqrt{\lambda_1 C_1} + \sqrt{\lambda_2 C_2}}\sqrt{t} \qquad (2)$$

where $T1_{in}$ is initial surface temperature of the block.

From equation 2, a plot of $T_1(0,t)$ vs. $\sqrt{t}$ yields a straight line graph which intersect $T_1(0,0)$ at t = 0. Using temperature readings from Block (1) and that from the porous medium (2), a set of two equations is generated which can be solved to determine the surface temperature of the porous medium at t = 0. The porous medium temperature T_2 (z, 0) beneath the block was measured at the depths of 2, 4, 8, 16 and 32 mm through thermocouple data logger temperature sensor connected at the depths.

Arctic Silver® used in this work as the thermal interface material is a high-density compound of silver, aluminum oxide, zinc oxide, and boron nitride in a polysynthetic oil base with thermal resistance rated as less than 0.0045°C-in²/W.

KD2 Thermal Properties Analyzer:

Instantaneous measurements of thermal conductivities were made using a 0.9 mm diameter KD2 Digital thermal probe (Decagon Devices Inc., Pullman, WA, USA). The thermal probe was considered as an infinitely long heat source in an isotropic medium. During measurements, the 60 mm long probe was put into the samples and the result was read out from the KD2. This was used to make measurement for the limestone and gneiss samples with and without Thermal Interface Material. The method used is generally called the transient line heat source or transient heated needle method. If heat at a constant rate *(q)* is applied to an infinitely long and infinitely small “line” source, the temperature response of the source over time can be described by the equation

$$\Delta T = \frac{q}{4\pi\lambda} E_i \left(\frac{-r^2}{4\alpha t} \right) \tag{3}$$

where λ is the thermal conductivity of the medium in which the line is buried, α is the thermal diffusivity of the medium, r is the distance from the line at which temperature is measured, and E_i is the exponential integral.

Sample Description

Granite is made of coarse mineral grains and consists of the quartz and feldspar, with or without a wide variety of minor minerals. The quartz and feldspar generally give granite a light colour ranging from pinkish to white, though light background colour, is punctuated by the darker accessory minerals.

Limestone is a sedimentary rock composed primarily of calcium carbonate in the form of the mineral calcite. It can be deposited as a sedimentary rock formed in shallow calm marine water from the shell and skeletons of dead marine life, and chemically from the direct precipitation of calcium carbonate from marine or fresh water.

Gneiss is a common and widely exposed rock type formed by high grade regional metamorphic processes from pre-existing formations that were originally either igneous or sedimentary rocks. Gneiss rocks are usually medium to coarse-foliated and largely recrystallised but do not carry large quantities of micas, chlorite or other platy minerals. Component analysis of the granite, limestone and gneiss rocks using ASTM D5334-00 is shown in Table- 1.

Sample Preparation:

Three sample slabs of approximately 15cm x 15cm x 10 cm was cut to size for surface contact with block apparatus and bored to slightly less than 1mm diameter on one side to allow for close insertion of the KD2 probe. Slab was placed one after the other inside a chamber where the temperature measurement was taken for block method calculation. A Picolog Data Logger (USB TC - 08 Thermocouple (USA)) was connected through temperature sensors connected to the block while 0.05cm-thick TIM was carefully applied to the contact surfaces of the block device and the sample. Measurements were made on granite, limestone and gneiss rocks to determine their thermal conductivities using both block method and KD2 analyzer (Fig. 3).

Table- 1: Sample Description

Samples	Colour	Grain size	Fabric	Mineral contents
Granite	Light grey	Block size 15cm x15cm 10cm	Isotropic	Quartz (30%), microcline (35%), plagioclase (30%) Others (5%)
Limestone	Light grey	Block size 15cmx15cm 10cm	Isotropic	Calcite (95%), quartz (3%) Others (2%)
Gneiss	Dark grey	Coarse	Foliated	Quartz (60%) plagioclase (30 %) opaque ore and sphene (5%) Others (5%)

Thermal Properties Determination

The theoretical calculations were presented below for the three rock samples. Graphical figures of temperature vs. time for Granite with TIM are only shown.

Granite with Thermal Interface Material:

From Fig. 4

$$31.77\,(1+\alpha) = 31.95 + \alpha T_2 \qquad (4)$$

$$31.42\,(1+\alpha) = 31.30 + \alpha T_2 \qquad (5)$$

Then, $\alpha = 0.8571$, $\sqrt{\lambda_2 C_2} = 1.1606 \times 10^{-2}$ while $T_{2\ in}$, the sample initial temperature is 31.55°. As the temperature gradient in the upper soil layer is known, λ_2 (thermal conductivity), C_2 (volumetric heat capacity and α_2 (thermal diffusivity) can be calculated separately:

$\sqrt{a_2} = \dfrac{\sqrt{\lambda_2}}{\sqrt{C_2}}$ = gradient x $\dfrac{\pi}{2}$ x $\dfrac{1}{E_2}$ x $\left(1+\dfrac{1}{\alpha}\right)$ where E is the temperature gradient in the upper soil layer.

$\dfrac{\delta\theta}{\delta z} = 31.26 - 0.124z$, $E_2 = 0.124$, $\sqrt{a_2} = 0.8161$

$\lambda_2 = \sqrt{\lambda_2 C_2}$ x $\sqrt{a_2}$ = 0.9471 cal. /cm sec. K = 3.96 W/m K

$C_2 = (\sqrt{\lambda_2 C_2} / \sqrt{a_2}) = 1.422 \times 10^{-2}$ cal/cm^{3}K = 5.95×10^{4} J/m^{3}K

$\alpha_2 = \lambda_2 / C_2 = 0.67 \times 10^{-4}$ m^2/s

Granite (without TIM):

From the plot of Temperatures against time for granite without TIM (Not shown), the following calculations were made:

$T_2 = 35.01^{\circ}$

$T_2\,(0, t) = 35.17 - 0.0289\sqrt{t}$

$T_2\,(z, t) = 28.14 - 0.089z$

$\alpha = 0.8125$

$\sqrt{\lambda_2 C_2} = 1.10025 \times 10^{-2}$

$\sqrt{a_2} = 0.64201$

$\lambda_2 = 0.70621 \times 10^{-2}$ cal. /cm sec. K = 2.956 W/mK

$C_2 = (\sqrt{\lambda_2 C_2} / \sqrt{a_2}) = 1.713 \times 10^{-2}$ cal/cm^{3}K $= 7.17 \times 10^4$ J/m^{3}K

$\alpha_2 = \lambda_2 / C_2 = 0.4122 \times 10^{-4}$ m^2/s

Limestone (without TIM):

$T_2 = 35.96^{\circ}$

$T_2\,(0, t) = 36.35 - 0.022\sqrt{t}$

$T_2\,(z, t) = 33.23 - 0.0726z$

$\alpha = 0.3452$

$\sqrt{\lambda_2 C_2} = 0.4675 \times 10^{-2}$

$\sqrt{a_2} = 1.0466$

$\lambda_2 = 0.4873 \times 10^{-2}$ cal. /cm sec. K = 2.04 W/m K

$C_2 = (\sqrt{\lambda_2 C_2} / \sqrt{a_2}) = 0.4466 \times 10^{-2}$ cal/cm^{3}K $= 1.869 \times 10^4$ J/m^{3}K

$\alpha_2 = \lambda_2 / C_2 = 1.09 \times 10^{-4}$ m^2/s

Limestone (with TIM):

$T_2 = 34.23^{\circ}$

$T_2\,(0, t) = 34.77 - 0.0204\sqrt{t}$

$T_2\,(z, t) = 31.66 - 0.0448z$

$\alpha = 0.1742$

$\sqrt{\lambda_2 C_2} = 0.2359 \times 10^{-2}$

$\sqrt{a_2} = 0.3275$

$\lambda_2 = 0.6417 \times 10^{-2}$ cal. /cm sec. K= 2.68 W/m K

$C_2 = (\sqrt{\lambda_2 C_2} / \sqrt{a_2}) = 0.08671 \times 10^{-2}$ cal/cm^{3}K = 0.363 $\times 10^4$ J/m^{3}K

$\alpha_2 = \lambda_2 / C_2 = 7.38 \times 10^{-4}$ m^2/s

Gneiss (without TIM):

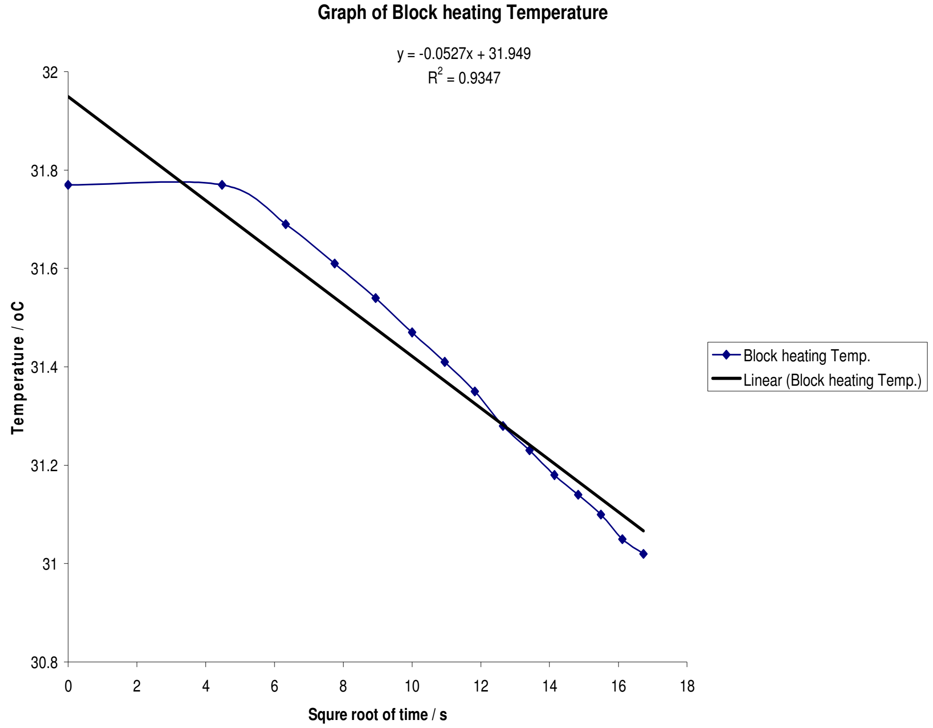
$T_2 = 36.87^{\circ}$

$T_2\,(0, t) = 37.16 - 0.023\sqrt{t}$

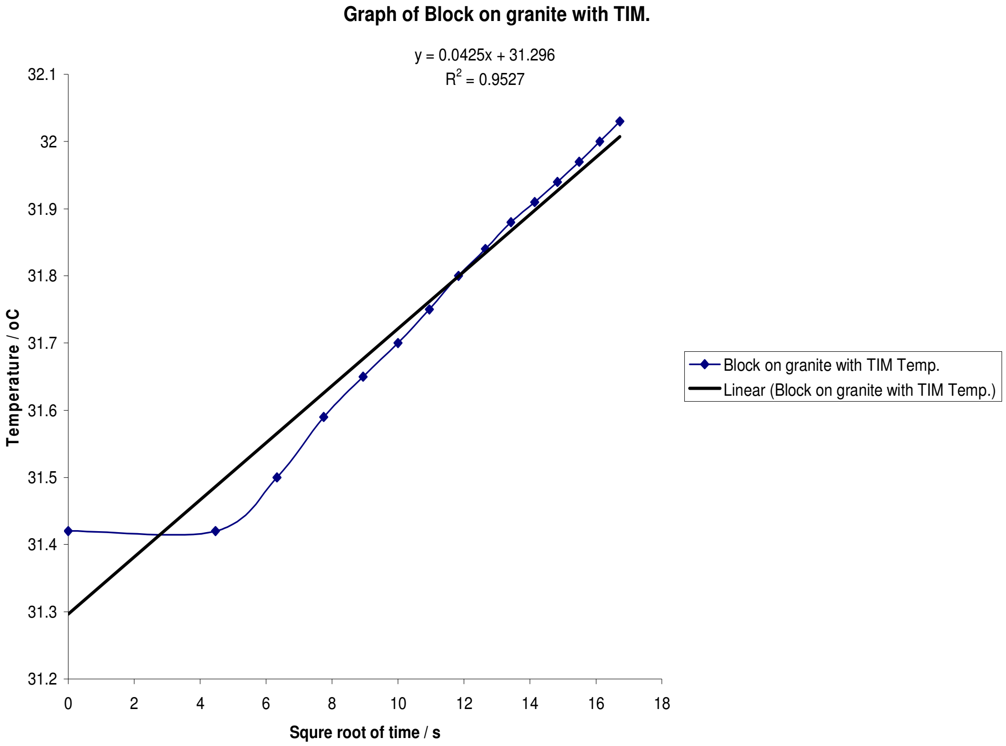
$T_2\,(z, t) = 34.08 - 0.0956z$
$\alpha = 0.3582$
$\sqrt{\lambda_2 C_2} = 0.485 \times 10^{-2}$
$\sqrt{a_2} = 0.8075$
$\lambda_2 = 0.3916 \times 10^{-2}$ cal. /cm sec. K= 1.64W/m K
$C_2 = (\sqrt{\lambda_2 C_2} / \sqrt{a_2}) = 0.60062 \times 10^{-2}$ cal/cm^{3}K = 2.514×10^4 J/m^{3}K
$\alpha_2 = \lambda_2 / C_2 = 0.6519 \times 10^{-4}$ m^2/s

Gneiss (with TIM):

T_2 36.25°
$T_2\,(0, t) = 36.47 - 0.0205\sqrt{t}$
$T_2\,(z, t) = 33.56 - 0.061z$
$\alpha = 0.3285$
$\sqrt{\lambda_2 C_2} = 0.4448 \times 10^{-2}$
$\sqrt{a_2} = 1.2030$
$\lambda_2 = 0.535 \times 10^{-2}$ cal. /cm sec. K= 2.24W/m K
$C_2 = (\sqrt{\lambda_2 C_2} / \sqrt{a_2}) = 0.3697 \times 10^{-2}$ cal/cm^{3}K = 1.547×10^4 J/m^{3}K
$\alpha_2 = \lambda_2\, C_2 = 1.44 \times 10^{-4}$ m^2/s



(a) Granite Surface contact Temperature



(b) Perspex Surface Temperature

Fig. 4 (a & b): Granite Contact temperature for the two blocks with TIM.

Discussions

Thermal conductivity of granite, limestone rocks sample and that of the gneiss rock sample increased with the application of TIM, and compared well with standard values (Kappelmayer and Haner, 1974). Thermal conductivity from Block method and that from KD2 device also compared favorably well. Thermal diffusivity from Block method also increased with the application of TIM. Results of thermal conductivities after measurements and calculations are presented in Tables-2.

Statistical analysis for KD2 Method:

Statistical analysis was carried out between the two methods as shown in tables 3 and 4 for the KD2 with TIM and without TIM through the analysis of variance (ANOVA) using Fisher's Protected Least Significant Difference. Thermal properties determined for granite, limestone and gneiss rocks are presented in Table- 3. $f_{cal} = 0.6769$ as P-value greater than 0.05 for KD2. Analysis indicates significant difference of thermal conductivities without TIM and with TIM.

Table- 2: Evaluation table of thermal properties of the samples

Samples	Block Exp. without TIM λ_{BO}	Block Exp. with TIM λ_{BW}	Difference λ_{BW}- λ_{BO}	% of difference	KD2 without TIM λ_{KO}	KD2 with TIM λ_{KW}	Difference λ_{KW}- λ_{KO}	% of difference	Standard range values W/mK (Kappelmayer and Heanel, 1974)
Granite Thermal conductivity (λ)	2.96	3.96	1.00	25.0	2.93	3.96	1.03	26.0	2.0–7.0(W/mK)
Thermal diffusivity (α)	0.41 x 10^{-4}	0.67 x 10^{-4}							(m^2/s)
Limestone Thermal conductivity (λ)	2.02	2.69	0.67	24.6	1.90	2.59	0.69	26.6	2.0–7.0(W/mK)
Thermal diffusivity (α)	1.09 x 10^{-4}	7.38 x 10^{-4}							(m^2/s)
Gneiss Thermal conductivity (λ)	1.64	2.20	0.56	30.0	1.54	2.18	0.64	29.4	2.0–7.0(W/mK)
Thermal diffusivity (α)	0.65 x 10^{-4}	1.44 x 10^{-4}							(m^2/s)

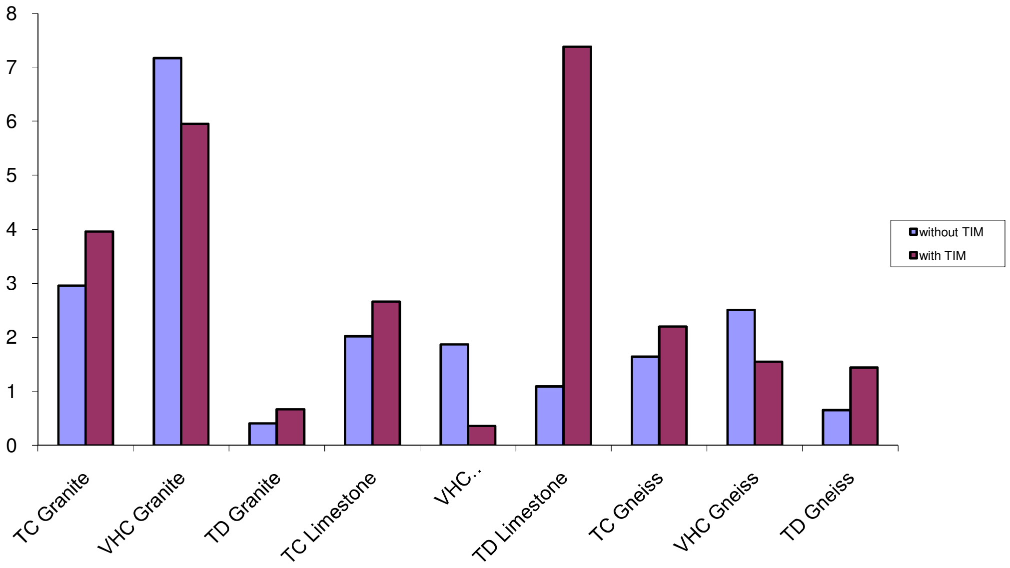


Fig. 5: Thermal properties of the samples without and with TIM.

Table- 3: Statistical Test of Significance for KD2 Method

SV	SS	Df	MS	f_{cal}
With/without TIM	0.1492	1	0.1492	0.6769
	0.4408	2	0.2204	
Total	0.6900	3		

Table- 4: Statistical Test of Significance for Block Method

SV	SS	Df	MS	f_{cal}
With/without TIM	0.16455	1	0.16455	0.6571
	0.50075	2	0.25040	
Total	0.66530	3		

Test of Significance for Block Method:

f_{cal} = 0.6571 as P-value is greater than 0.05 for block method Results which indicate significant difference of thermal conductivities without TIM and with TIM (Table- 4). This implies that using TIM to reduce the contact resistance error in the block method is effective.

Experiments on Granite, Limestone and Gneiss:

Thermal conductivity of granite increased from 2.96 to 3.96 W/mK with 26% difference for the block method with TIM, and from 2.93 to 3.96 W/mK with 24% difference for the KD2. Thermal diffusivity increased from 0.41 x 10^{-4} to 0.67 x 10^{-4} m^2/s. For limestone λ increased from 2.02 to 2.69 W/mK with 25% difference for the block method with TIM and from 1.90 to 2.59 W/mK with 27% difference for the KD2 measurement. Thermal diffusivity increased from 1.09 x 10^{-4} to 7.38 x 10^{-4} m^2/s. For the gneiss sample, λ increased from 1.64 to 2.20 W/mK with 30% difference for the block method and 1.54 to 2.18 W/mK with 29 % difference for the KD2. Thermal diffusivity increased from 0.65 x 10^{-4} to 1.44 x 10^{-4} m^2/s (Table- 2) where BO and BW represent block measurement without TIM and with TIM, and KO and KW represent KD2 measurement without TIM and with TIM respectively. Standard values of thermal conductivity for granite, limestone and gneiss range between 2.0 and 7.0W/mK, 1.0 and 5.0 W/mK and from 1.5 and 5.0 W/mK respectively (Kappelmayer and Haenel, 1974).

Conclusions

Thermal Block technique was applied on granite, gneiss and limestone with a view to measuring thermal conductivity, thermal diffusivity and volumetric heat capacity. Accuracy concern arising from contact errors was addressed using thermal interface materials. Measurements from KD2 thermal analyzer was used to validate the results from block measurements and results compared well. Thermal Interface materials improved values of Thermal Conductivity and thermal diffusivity of rock samples. Thermal conductivity of granite, limestone and gneiss increased from 2.96 to 3.96 W/mk, 2.02 W/mK to 2.68 W/mK, and 1.64 W/mK to 2.20 W/mK respectively. Thermal diffusivity of granite, limestone and gneiss also increased from 0.41×10^{-4} to 0.67×10^{-4} m^2/s, 1.09×10^{-4} to 7.38×10^{-4} m^2/s, and 0.65×10^{-4} to 1.44×10^{-4} m^2/s respectively. Thermal conductivity and thermal diffusivity without and with TIM showed significant difference at $P > 0.05$ and confirmed the effectiveness of thermal interface material in reducing contact resistance errors.

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