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ASPECTS OF THERMAL CONDUCTIVITY RELATIVE TO HEAT FLOW

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The various techniques and methodologies of thermal conductivity measurement have been conventionally based on the determination of the rate of directional heat flow through a material having a unit temperature differential between its opposing faces. The constancy of this rate depends on the material density, its thermal resistance and the heat flow path itself. The last of these variables contributes most significantly to the true value of steady-state axial and radial heat dissipation depending on the magnitude of transient thermal diffusivity along these directions.

The purpose of this paper is to exemplify the above features by defined parameters of heat flow measurement by existing methodologies. No new method is proposed here. Importantly, the relationship between the rate of heat transfer, total heat transferred and thermal conductivity at a given temperature under steady-state conditions for a fixed heat flow path will be illustrated.

Keywords: *DuPont vespel, granite, heat flow path, quartz, silica, thermal conductivity, water*

Introduction

The measurement of thermal conductivity involves a set of parameters that are common to different techniques and methodologies. Aside from variations due to the nature and type of samples, all methodologies require determination of the actual amount of heat transferred through the sample along and perpendicular to the heat flow path in a given thermal environment. The calculated value is expressed in the same unit as that provided for a standard of the same material. Conductivity, as opposed to conductance, provides dimensional attributes to the calculated value. Thus, thermal conductivity is related to a material property that denotes a rate process of heat transfer. Since diffusion is the primary mode of heat propagation in a solid body, thermal conductivity is a function of diffusivity, density and heat capacity. Whereas through-thickness thermal conductivity for fixed-dimension solids is primarily measured under steady-state conditions, accompanying transient diffusivity in the radial direction is taken into account by using the ratio of sample thickness to the total sample area as the heat flow path [1]. The relationship is expressed as:

$$\lambda = qL/A(t_1 - t_2) \tag{1}$$

where λ=thermal conductivity/W m^{-1} K^{-1}; q=time rate of heat flow/W; L=thickness of sample in the heat flow direction/m; A=area of sample/m^2; t_1=temperature of hot surface/K; t_2=temperature of cold surface/K. With the parameters thus established, simple methodologies can be developed additionally to measure heat flow rate, heat transfer value and thermal conductivity for materials not bounded by fixed dimensions i.e. for oil, grease and powders. The heat flow path is obtained from the X–Y axes of the fixed sample holder in these cases. Thermal conductivity, thermal diffusivity, heat flow rate, heat transfer value and thermal stability are the very important parameters for the characterization of materials ranging from gaseous through liquid to solid. Therefore, it is not surprising that many authors studied the thermal properties including thermal conductivity, thermal diffusivity, heat flow rate and heat transfer value to characterize various materials [2–52].

Experimental

For the purpose of this paper, measurement of heat flow using a standard DSC cell and also a modified DSC cell were used to exemplify the use of heat flow value for the determination of thermal conductivity. As a first step, the suitability of using the heat output value of a standard TA Instrument DSC cell for calculating thermal conductivity was checked by a run on water (65.0 mg) in a closed sample pan. An empty closed pan of the same dimensions and materials as the sample pan was used as a reference. The obtained value agrees well with the value obtained by measure-

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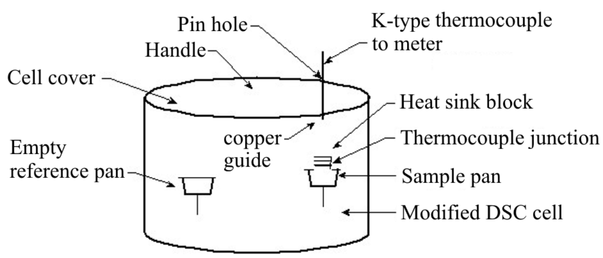
ment of temperature gradient across a water sample and a heated platinum wire in a glass cell [53].

The DSC cell modified by Chiu and Fair [54] was adapted for determination of the ratio of thermal gradients in sample and reference by adding an empty reference pan. This modified DSC cell contains an external thermocouple fixtured through the cell cover for the thermocouple junction to contact the sample pan from above. The body of the thermocouple is embedded in a hollow copper rod and a terminal heat sink block for measuring the temperature of the cold side of the sample. A sketch is provided below to show the details of the modified cell.



Sketch of modified DSC cell

Thermal matching of the sample and reference pans was checked by running a zero baseline prior to sample run. The sample was run by equilibrating the DSC cell at the measurement temperature. The temperature of the hot side of the sample is indicated by the thermocouple output of the DSC cell. The methodology calls for a second run under identical conditions of a standard material of known thermal conductivity value. The calculation of thermal conductivity of the sample (K_s) is simplified by using the respective ratios:

$$K_s=K_r(X_s/X_r)(L_s/L_r)(D_r/D_s)^2(\Delta T_r/\Delta T_s) \tag{2}$$

where K_r=thermal conductivity of standard; X_s=DSC output (thermal flux) of sample; X_r=DSC output of standard; L_s=thickness of sample; L_r=thickness of standard; D_r=cross sectional area of standard; D_s=cross sectional area of sample; ΔT_r=thermal gradient in the standard; ΔT_s=thermal gradient in the sample. Another experimental technique for the utilization of steady-state equilibrium heat flow value at the analysis temperature for thermal conductivity measurement, using the modified DSC cell was utilized. The methodology involves the following steps: i) initial equilibration and iso-tracking; ii) ramping to within 1°C of the analysis temperature, and iso-tracking to follow the sample temperature; iii) 1°C shift to analysis temperature and equilibration for total heat flow measurement; iv) temperature difference between the top and bottom of the sample was measured at equilibrium by the embedded thermocouple (type K) and v) calculation was made by the ASTM equation (Eq. 1) given earlier.

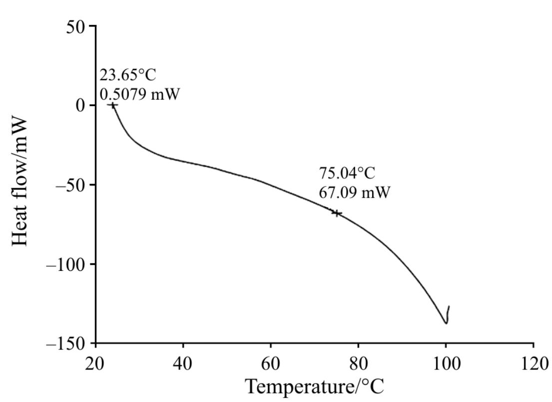


Fig. 1 Thermal conductivity of water

Results and discussion

Figure 1 shows the calculation of thermal conductivity value of laboratory tap water from the DSC net heat flow measurement at 75°C. The calculated value of $6.675\cdot10^{-3}$ W cm^{-1} °C^{-1} agrees well with the value of $6.663\cdot10^{-3}$ W cm^{-1} °C^{-1} reported by Jamieson and Tudhope [53] for unspecified regular water.

The principle of their method was based on the measurement of the temperature/resistance relationship of a heated thermal conductivity cell filament when the cell was filled with water. This method forms the basis of ASTM D-2717. The reported value has an accuracy of ±10%, which also allows for the variability of water from different sources. The DSC value is expressed in the same units as in the reference cited for direct numerical comparison. The calculation steps are as follows:

- Net Heat Flow @ 75ºC=66.5821 mW,
- $(66.582 \cdot 0.01433) / 1000 = 9.5432\cdot10^{-4}$ Kcal min^{-1},
- $[9.5432\cdot10^{-4}\cdot1000]/(60\cdot4.186)=6.65797\cdot10^{-2}$ W,
- $6.65797\cdot10^{-2}/(75\cdot0.133)=6.675\cdot10^{-3}$ W cm^{-1} ºC^{-1}.

This result establishes the suitability of using the DSC heat flow output for calculating the thermal conductivity value.

The use of Eq. (2) is illustrated in Figs 2 and 3. Figure 2 shows the measured heat flow values at equilibration temperatures for a quartz sample used as a standard and Fig. 3 shows the heat flow values at the corresponding temperatures for an unidentified proprietary polymer sample. The calculation parameters are shown in Table 1. The calculated thermal conductivity values at 25, 100 and 150°C were 0.157 W m^{-1} K^{-1}, 0.619 W m^{-1} K^{-1} and 0.725 W m^{-1} K^{-1}, respectively. The value of thermal conductivity at 100°C provided by the supplier was 0.612 W m^{-1} K^{-1} as opposed to the ob-

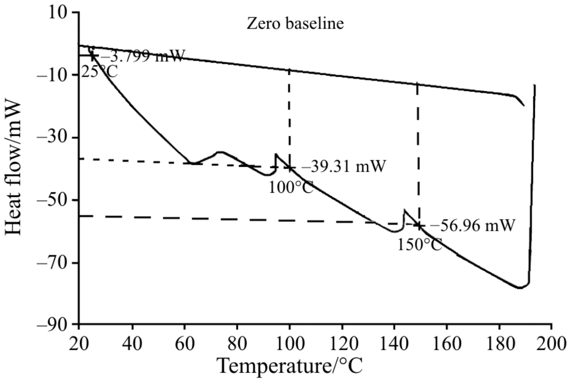


Fig. 2 Heat flow values for quartz

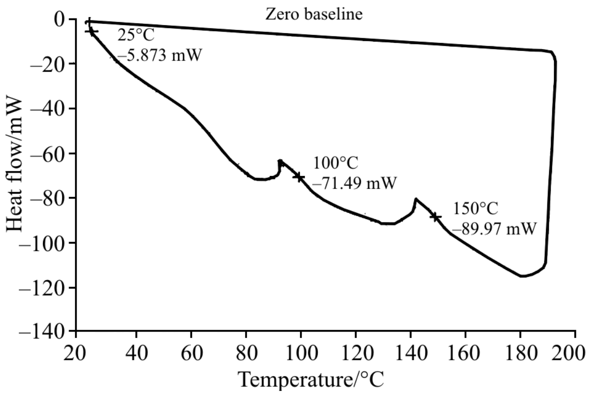


Fig. 3 Heat flow values of a polymer sample at different temperatures

Table 1 The calculation parameters

Temp/ ºC	K_r/ W m^{-1} K^{-1}	L/cm	D/cm	D^2/cm^2	$(\Delta T_r/\Delta T_s)$
25	1.763	0.254	0.681	0.4638	0.300
50	1.913	0.254	0.681	0.4638	1.007
150	1.980	0.254	0.681	0.4638	1.180

L_s and L_r as well as D_r and D_s have the same value, represented here by L and D, respectively

tained value of 0.619 W m^{-1} K^{-1}. Units were recalculated for direct comparison of values.

The applicability of the steady-state equilibrium heat flow method by modified DSC using Eq. (2) was tested by a DuPont compression molded Vespel solid rod sample (DuPont shapes and parts product). Figure 4 illustrates that the value obtained at 313 K is the same as the value provided in the manufacturer's data sheet at this temperature, but did not contain informa-

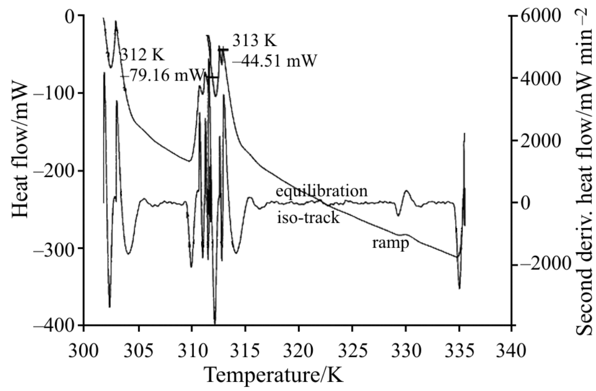


Fig. 4 Thermal conductivity of DuPont Vespel

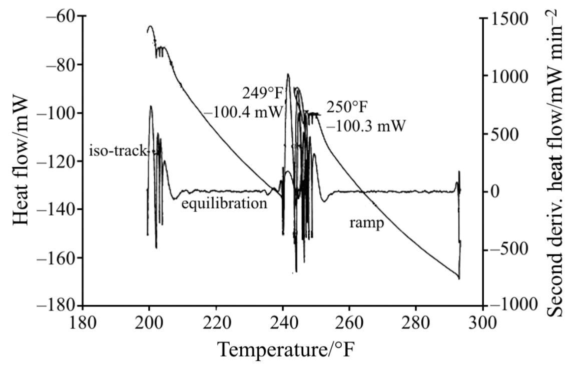


Fig. 5 Thermal conductivity of silica

tion on the analytical method used. The sample of Vespel used should not be confused with some grades of KAPTON film, which are also referred to as vespel because of their common polyimide resin base. These films have a much lower thermal conductivity value.

Finally, development of heat transfer data from the determination of thermal conductivity value is illustrated in Figs 5 and 6 for silica and granite, respectively. These measurements were made on the ºF scale, since data were needed for process engineering that was run on this temperature scale. In the interest of standard practice, the determined and calculated values are expressed in corresponding °C in Table 2. Customarily, heat flow and thermal conductivity values can be measured on any temperature scale and expressed in a converted temperature scale without loss of accuracy. These illustrations were chosen because these samples have high thermal conductivity, which leads to an easy calculation of heat transfer values. It is also because their thermal conductivity values can be verified from the literature.

Table 2 The determined and calculated values at 121°C

Sample	Area/cm^2	Thermal conductivity/ W m^{-1} K^{-1}	Equivalent heat transfer/ W	Heat transfer rate/ W K^{-1}	Total heat transferred/ W m^{-2}
silica	0.159	1.511	0.100	0.010	2.322
granite	0.159	3.421	0.181	0.021	2.943

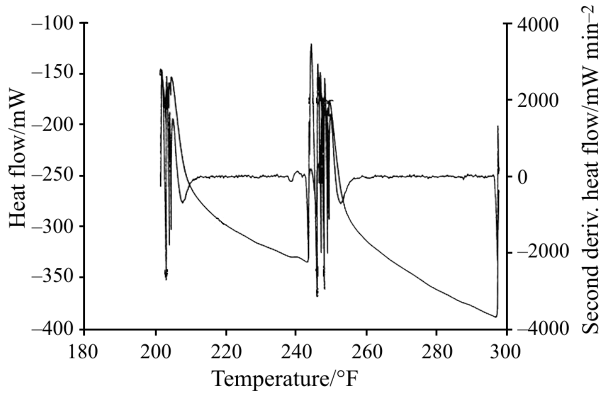


Fig. 6 Thermal conductivity of granite

Conclusions

Parametric measurement of heat flow for the determination of thermal conductivity has been exemplified in this work for samples not bounded by fixed dimensions, using existing methodologies. Development of thermal values such as temperature, specific heat transfer and heat transfer rate for such samples, required for engineering purposes have been demonstrated.

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