GEOLOGICAL SURVEY OF CANADA

OPEN FILE 5330

A review and assessment of current technologies and techniques for measuring the thermal properties of solids and porous materials

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2006

Executive Summary

The following report was prepared under a cooperative research initiative between the Geological Survey of Canada (GSC) and the Institute of Applied Energy (IAE), Japan conducted in 2002. The work was part of a larger initiative led in Japan by the New Energy Development Organization (NEDO). The broader agreement involved other Canadian partners, including the Stacie Institute of Molecular Science (SIMS) of the National Research Council of Canada (NRCC), and the University of British Columbia (UBC). Japanese partners included the Institute of Applied Energy (IAE), the Geological Survey of Japan (GSJ), Hokkaido National Industrial Research Institute (HNIRI), and the National Institute for Resources and Environment (NIRE). The specific research documented in this report was implemented through a Collaborative Research Agreement between the Institute of Applied Energy (IAE) and the Terrain Sciences Division of the GSC (FY 2001-2002).

The primary objective of the research was to identify and evaluate the utility and limitations of available technologies and techniques for measuring the thermal properties of solids and porous materials. A summary of current and landmark publications in the fields of physics, geophysics, geothermics, soil science, and material sciences form the basis of the assessment. Additional relevant information was obtained through a product survey of available commercial equipment and instrumentation, including specifications and operational data from manufacturers of specialized thermal property measurement systems. Current technologies and techniques were assessed for their suitability for simultaneous measurement of thermal conductivity and diffusivity of solid and porous materials with particular emphasis on:

1. The theoretical basis of the measurement technique;
2. The influences of transient mass fluxes (air/water transport and phase change);
3. Operational ranges of temperature and pressure;
4. Scale factors.

Finally, the report makes recommendations regarding optimal design configurations for simultaneous measurement of the thermal conductivity and thermal diffusivity of geologic material containing water and/or ice and/or air and/or gas hydrate. However, because few reliable data exist regarding the measured thermal properties of gas hydrate and gas hydrate bearing sediments, NRCan makes no claims as to the actual performance of any of the reviewed measurement systems for gas hydrate applications. The report does, however, incorporate the best available information regarding the current state-of-the-art technologies and techniques for thermal properties measurement in frozen porous materials, for which the complexity of heat and mass fluxes during measurement are thought to be similar to those encountered in the presence of gas hydrate.

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1.0   INTRODUCTION

Heat and mass transfer problems have been studied in various fields of science and engineering. They are of interest both in macro-scale as in geology, geothermics, earth science etc. and in micro-scale as in metallurgy, electronics, biology etc. Heat transfer through solids, porous materials and particulates take place primarily through conduction, diffusion and coupled process (pore fluid convection and phase change). Any heat conduction study requires a knowledge of thermal properties namely, thermal conductivity, diffusivity, and specific heat or volumetric heat capacity. If published values for these properties are not available or are not applicable, accurate measurements must be made which simulate the conditions in which the materials occur naturally or are employed in manufacturing and/or engineering applications.

Thermal conductivity is defined as the rate of heat flux conducted through a material under a unit thermal gradient. Heat capacity is defined as the amount of heat absorbed/released by a unit volume of material as it warms/cools by 1°C. Heat capacity (also known as volumetric heat capacity) is equal to specific heat divided by density of the material. The thermal diffusivity is defined as the ratio of heat conducted to heat absorbed.   Therefore: diffusivity = conductivity/heat capacity.   In the case of composite materials and porous solids with gas and liquid filled pores, the measured heat conducting properties can be regarded only as bulk parameters, which may vary significantly within the medium.

Heat conducting properties of porous materials (such as frozen and unfrozen soils and rocks) may be strongly dependent on the degree of pore saturation, ambient temperature, and pressure. Therefore it is important to measure thermal properties over the range of conditions. Researchers have developed a number of different techniques for measuring thermal properties of various materials such as metals, ceramics, offshore sediments, soils, and rocks. They range from simple and approximate methods suitable for field use to elaborate and accurate measurements conducted under controlled laboratory conditions. These techniques can be classified into two broad categories depending on the type of thermal fields induced in the test medium by the measurement techniques:

1. Steady state techniques;
2. Transient techniques.

Typically, steady-state techniques involve the application of a constant heat flux to the sample, and the resultant thermal gradient is measured after a steady-state thermal field is established. The thermal parameters are calculated using simple formulas selected on the basis of the physical setup and the thermal boundary conditions employed. While the theory and calculation procedures are simple, the experimental setup may be quite involved to ensure that the thermal boundary conditions established during measurement are consistent with those assumed in the theory. Also relatively long time periods may be required to establish steady-state conditions

within the test medium. In the case of porous solids, convection of fluids (water or air) and phase change of water and ice within the pore volume may invalidate the measurements. They are generally not suited for *in situ* measurements.

In transient techniques, the thermal response of a specimen or test medium to a known heat input is monitored over a period of time. The heat input may be either continuous and of a constant rate, or in the form of a short duration pulse. The heat source can be a line source, a point source, a cylindrical source, or a surface element source. The evolution of temperature may be monitored either at the heater-medium interface, at some distance from the heater or at the opposite boundary of the specimen. Some transient methods involve instantaneously altering the boundary temperature and monitoring the temperature-time response at a predetermined location in the test specimen. For all of these transient methods, data reduction involves matching of the measured temperature-time response with the theoretical or numerical solution to the transient heat conduction under specific boundary conditions.

The potential sources of error in all of the above mentioned measurement techniques are twofold:

1. Errors due to approximations made in model simulation and derivation of theoretical solutions used for calculating thermal parameters;
2. Errors in measurement and boundary simulations.

In selecting a measurement technique, the sources of error and methods of minimizing them should be considered. Recent advancements in mathematical modeling, computation techniques and instrumentation have resulted in more accurate measurement of thermal parameters under ambient conditions.

2.0 THEORETICAL MODELS

The phenomenological law for thermal conduction is:

$$q = kA(\Delta\theta /L)t \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (1)$$

Where, q = the amount of heat that flows through a cross-sectional area A under a thermal gradient $\Delta\theta/L$
   k = the proportionality factor called the coefficient of thermal conductivity or thermal conductivity

In differential form, eq. 1 can be written as:

$$dq/dt = -k\, d\theta/dl \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (2)$$

Considering a small-volume element of dimensions dx, dy, dz of a homogeneous isotropic solid, and assuming that thermal conductivity k is independent of temperature, time and direction, and that no heat is generated within the element, one obtains by the law of conservation of energy, the general equation for heat transmission in a body:

$$\partial^2\theta/\partial x^2 + \partial^2\theta/\partial y^2 + \partial^2\theta/\partial z^2 = c_{p}/k \ (\partial\theta/\partial t) \quad\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \ (3)$$

Where, k = the thermal conductivity

$\quad\quad c_p$ = the volumetric heat capacity of the solid

In cylindrical coordinates, the above heat conduction equation becomes:

$$\{\partial^2\theta/\partial r^2 + 1/r(\partial\theta/\partial r) + 1/r^2(\partial^2\theta/\partial r^2) + \partial^2\theta/\partial z^2\} = c_{p}/k \ (\partial\theta/\partial t) \quad\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \ (4)$$

For one-dimensional heat flow, eq.3 reduces to:

$$\partial^2\theta/\partial x^2 = 1/\alpha \ (\partial\theta/\partial t) \quad\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \ (3a)$$

Where, $\alpha = k/c_p$ = thermal diffusivity.

For radial heat flow from an axially placed heat source, eq.4 reduces to:

$$\partial^2\theta/\partial r^2 + 1/r(\partial\theta/\partial r) = 1/\alpha(\partial\theta/\partial t) \quad\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \ (4a)$$

Experimental methods available for the determination of thermal conductivity fall under two broad categories, namely:

(a) Steady-state methods for which eq.1 and eq.2 apply;
(b) Transient or non-steady methods for which eq.3 or eq.4 apply.

3.0   STEADY-STATE METHODS

Steady-state methods consist of measuring the thermal gradient established under a constant rate of heat input into a specimen once a steady-state condition has been established. Only the thermal conductivity (not thermal diffusivity) can be determined by steady-state methods.

Equation 1 is used to calculate thermal conductivity for one-dimensional heat flow. Guarded-hot-plate and divided-bar techniques fall under this category. In the guarded-hot-plate apparatus, flat heaters contain peripheral guard heaters to minimize the transverse heat flow. The thermal gradients developed across the sample under a constant heat flux are measured when steady-state

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conditions are reached. A detailed description of test procedure and the specifications for guarded hot plates are given in ASTM Standard C-177-71.

In the case of the divided-bar technique, a disc-shaped specimen is placed between two cylindrical metal bars of same diameter as that of the specimen. One-dimensional heat flow is established by heating the remote end of one bar while cooling the remote end of the other bar. This is accomplished by circulating thermostatically controlled water through coils embedded in the metal bars. The sides of the test specimen and metal blocks are insulated to prevent heat loss. When steady-state conditions are reached, the thermal gradients across the metal bars and the test sample are measured by thermocouples.

In the parallel-bar technique a thin flat heater is sandwiched between two identical samples. The sides of sample and heater are heavily insulated to ensure one-dimensional heat conduction through the test specimens. After a steady-state condition is reached, the heat flux and thermal gradients in both the samples are measured.

All of the above methods can be used for measuring thermal conductivity at a range of temperatures. High temperature measurements are commonly made for insulating materials, while sub-zero temperature measurements are made for frozen soils, rocks, and insulating materials.

4.0 TRANSIENT-STATE METHODS

Solutions to the transient heat conduction equations (3) and (4) for different boundary conditions have been used in designing experimental methods for determining thermal parameters α and k. The commonly used transient methods employ either a known heat input boundary or a constant temperature boundary. The heat input can be either of a constant rate or a short duration pulse. The line source method is extensively used for measuring thermal properties of soils, rocks, and other building materials such as concrete. In this method, a constant and continuous rate of heat input is applied to a line source (generally a linear probe heater) implanted in the test medium and the measured temperature-time data are used to derive the required thermal parameters. Alternative methods are also available in which a heat pulse is applied to a line source and the measured temperature-time response is analyzed to calculate thermal parameters.

A number of transient techniques are available for measuring thermal properties of solid and porous materials. These can be grouped into three broad categories:
   1. Line heat source based techniques;
   2. Surface heat source based techniques;
   3. Calorimeter based techniques.

Which are further sub-grouped as:

1. Radial heat conduction from line source:

   1.1 Single needle or probe using a constant line source
   1.2 Dual-needle or probe using a constant line source
   1.3 Dual-needle or probe using a heat pulse
   1.4 Pulsed line source heater on the cylindrical surface of sample

2. One-dimensional heat conduction from surface heat source:

   2.1 Constant rate of heat applied to sample surface
   2.2 Heat pulse applied to the sample surface
   2.3 Heat flash applied to sample surface

3. Calorimeter based techniques:

   3.1 Instantaneous rising or lowering of boundary temperature
   3.2 Adiabatic calorimeter
   3.3 Differential scanning calorimeter

The line source based techniques are suitable for the simultaneous measurement of thermal conductivity and diffusivity. The techniques based on surface heat source and calorimeter are generally suitable for determining only thermal diffusivity or heat capacity.

4.1   SINGLE PROBE TECHNIQUE

The single thermal probe (or needle probe) is the most commonly used method for determining the thermal conductivity of soils and particulate materials. It consists of an electric wire heater and a temperature sensor encased in a metal tubing or hypodermic needle. The probe is inserted into the test sample ensuring a good contact between the probe and the medium. A constant power is applied to the electric heater in the probe while the temperature at its mid-point is monitored by a thermocouple or a thermistor.

The data analysis is based on the heat flow solution for a constant line source in an infinite medium (eq.4). For this case, Carslaw and Jaeger (1959) derived an expression for the temperature rise T at a known distance r from heat source as a function of elapsed time t:

$$T\ (t)\ =\ -q/4\pi kEi(-r^2/4\alpha t) \dotfill (5)$$

Where, q = constant rate of heat input per unit length of the line source
        k = thermal conductivity

α = thermal diffusivity of the medium

Ei = exponential integral

Eq.5 can be expanded in the form:

$$T(t) = -q/4\pi k \ [ln(4\alpha t/r^2) - \gamma + r^2/4\alpha t - 1/4(r^2/4\alpha t)^2 + 1/9(r^2/4\alpha t)^3 - \dots \ ] \ \dots\dots\dots\dots\dots\dots (6)$$

Where, γ = Euler's constant (0.5772)

For small values of $(r^2/4\alpha t)$, i.e. for small distance r and long time t, the above equation can be approximated to:

$$T(t) = -q/4\pi k \ [ln(4\alpha t/r^2) - \gamma] \ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (7)$$

Which can be rearranged into the following form:

$$T(t) = -q/4\pi k \ [lnt + ln \ (4\alpha/r^2) - \gamma] \ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (8)$$

From eq.8 and Fig. 1, it can be inferred that if temperature rise is plotted as a function of $ln(t)$, the thermal conductivity k can be calculated from its slope β at long times by:

$$k = q\beta/4\pi$$

Also, the thermal diffusivity α can be calculated from the intercept value I obtained by extrapolating back the straight line portion of $T(t)$ vs. $ln(t)$ plot to $ln(t)$ axis as shown in Fig. 1:

$$\alpha = r^2/4 \ exp \ [\gamma - I] \ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (9)$$

For experimental results to agree with the above theoretical solutions the experimental procedures should comply with the following assumptions:

1. No thermal contact resistance between the heat source and test medium;
2. The probe is an instantaneous and constant heat source;
3. Test data are free from boundary effects;
4. The probe is infinitely long and heat flow through the test medium is radial;
5. Conduction is the only mechanism of heat transfer.

In practice, the thermal probe will have finite dimensions, some heat capacity, and is likely to have some contact resistance. The impact of these factors on the test results and the methods of accounting for them are described below.

Figure 1. Plot of the temperature change vs. ln(time) for a cylindrical probe with contact conductance.

Leads from heater & temp.

Insulation

Thermal probe with line source & temp. sensor

Test sample

Temperature sensor

Insulation

Temperature Change

Intercept I = $\left[\, \ln(4\alpha/r^2) - \gamma + 2k/rH \,\right]$

$T(t) = (q \,/\, 4\pi k) \left[\, \ln(t) + \ln(4\alpha/r^2) - \gamma + 2k/rH \,\right]$

ln(t)

Thermal conductivity: $k = q\,\beta\,/4\pi$
Thermal diffusivity: $\alpha = r^2 / 4 \exp[\gamma - 2k/rH - I]$

q = Rate of heat input per unit length
β = Slope of straight line portion of temperature-time response curve
r = Radius of probe
H = Contact conductance
γ = Euler's constant
I = Intercept on time axis

4.1.1 Contact Resistance/Conductance

Blackwell (1954) developed an approximate solution for the case of constant contact resistance between the cylindrical heat source and infinite medium, which is similar to eq.8:

$$T(t) = -q/4\pi k \ [ln(t) + ln(4\alpha/r^2) - \gamma + 2k/rH] \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (10)$$

Where, H = contact conductance (inverse of resistance) at the heater-media interface, which is assumed to be constant for the full duration of the test

Hartley and Black (1976) suggested estimating the acceptable minimum time beyond which the temperature vs. time data can be used to achieve an acceptable degree of accuracy in the calculation of thermal conductivity. In practice, it has been found that the contact conductance and heat capacity affects only the initial transient and it does not appear in the calculation of thermal conductivity k. Large diameter probes (up to 25 mm by 2 m) have been successfully used for offshore thermal surveys (Radhakrishna and Steinmanis, 1981)

The contact conductance affects the thermal diffusivity α, which can be calculated as:

$$\alpha = r^2/4 \ exp \ [\gamma - 2k/rH - I] \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (11)$$

To determine the thermal diffusivity from a thermal probe of cylindrical shape, it should be possible either to accurately estimate the contact conductance H such that $2k/rH$ becomes very small (approaches zero) or it should be large enough to neglect the term $2k/rH$ in the above equation.

Carslaw and Jaeger (1959) estimated the contact conductance H for different gap widths of an air filled space between the probe and the test medium:

H (cgs units)	Gap width (cm)
0.5	0.0001
0.05	0.001
0.005	0.01
0.0005	0.1

The relative error in the calculated α due to the omission of contact conductance is:

$$\Delta\alpha/\alpha = 2k/rH$$

Table 1 presents the percentage error in the calculated value of α due to neglecting the contact resistance for different probe diameters and contact conductance values assuming a typical value of $k = 0.016$ W/°C-cm for rock (Carslaw and Jaeger, 1959). The data suggest that the accuracy of α is very poor for probes thinner than 2 mm, even in cases where the contact between the probe and the test medium is good.

Table 1. Percentage of modeling error in thermal diffusivity (α) by neglecting contact conductance

Radius of probe b (cm)	Contact conductance H (cgs units)			
	0.5	0.05	0.005	0.0005
0.02	80%	800%	>1000%	>>1000%
0.2	8%	80%	800%	>1000%
2	0.8%	8%	80%	800%
10	0.2%	2%	20%	200%

Crowe *et al.* (1962) computed the percentage of error in the calculated thermal diffusivity value α for different levels of error in the estimation of intercept *I,* slope of *T* vs. *ln(t)* curve, and the contact conductance *H*. This was done for different gap widths and different probe diameters (Table 2).

Table 2. Maximum percentage of error in thermal diffusivity corresponding to relative errors in intercept, in slope, and in contact conductance (Crowe et *al.*, 1962).

	Contact conductance H (cgs units)			
Probe radius b (cm)	0.5	0.05	0.005	0.0005
Case 1:E_I=15%, E_A=5%, E_H=50%				
0.02 – Wire or small needle probe	30%	300%	>1000%	>>1000%
0.2 – large needle probe	20%	40%	400%	>1000%
2 – small borehole probe	70%	80%	120%	400%
10 – large borehole probe	120%	130%	140%	200%
Case 2: E_I=5%, E_A=1%, E_H=50%				
0.02	10%	40%	>1000%	>>1000%
0.2	10%	30%	450%	>1000%
2	10%	20%	50%	400%
10	20%	20%	30%	100%

Note: E_I= relative error in intercept *I*, E_A = relative error in slope *A* and E_H= relative error in contact conductance *H*.

It is apparent from Table 2 that with the highest contact conductance (or lowest resistance) that can be realized in practice, the error in the thermal diffusivity will be 100% or more in the case of wire or small diameter needles. However, it may be possible to lower the error to 50% or less if the probe radius is greater than 0.2 cm. From tests on soils with a probe of 1 cm in diameter the relative error in α was about 30-40% (Boggs *et al.*, 1980, Radhakrishna *et al.*, 1981). Mason and Kurtz (1952) suggested an empirical method for correcting the temperature-time response curve from a cylindrical probe to account for the effects of probe heat capacity and contact resistance. They demonstrated that by plotting the values of (dt/dT) against time t, the time lag t_0 could be estimated as the intercept of the straight-line portion extrapolated back to t-axis. The temperature-time response curve is corrected for this time-lag effect before calculating the conductivity and diffusivity from its slope and intercept. However, this technique is susceptible to estimation errors.

It should be noted that none of the above methods (including eq.10) apply if the contact conductance changes during the test, as may be the case with small diameter probe in coarse-grained particulate materials.

4.1.2   Heat Capacity of the Probe

The heat capacity of the cylindrical probe will also affect the measured thermal response of a test specimen, especially at short test durations. This introduces an error in the calculated thermal diffusivity. However, the thermal conductivity determination is unaffected by the heat capacity of the probe, provided the slope of temperature vs. *ln(t)* is obtained after initial transients have dissipated.

In addition to contact conductance at the heater-medium interface, several other factors influence the temperature-time response of a cylindrical probe. These are:

1. The heat capacity of probe;
2. The location of the heater in the probe;
3. The location of temperature sensor in relation to the heater;
4. The length to diameter ratio of the probe.

The heat capacity (or thermal mass) of the probe delays propagation of the thermal front through the test medium and thus introduces an error in intercept I and hence in the thermal diffusivity (α). Some researchers have included the heat capacity term along with contact conductance and corrected the intercept I by empirical procedures (Mason and Kurtz, 1952). Boggs *et al.*, (1980) used the more rigorous non-linear least-squares fit of the entire temperature-time response curve to Blackwell's (1954) model of a cylindrical thermal probe to calculate thermal diffusivity. This analysis takes into account both the heat capacity of probe and contact conductance. For the

known value of the probe's heat capacity, the thermal diffusivity (α) and contact conductance (H) are obtained as fit parameters from this regression analysis. Good results were obtained for tests on soils under carefully controlled conditions (Boggs *et al.*, 1980).

The heat capacity of the probe can be determined by heating it under a low constant power while wrapped in a high-density thermal insulation. The heat capacity of the probe was calculated from the straight-line portion of the measured temperature vs. linear time response (Boggs *et al.*, 1980) as:

$$C = q(\Delta T/\Delta t) \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(12)$$

Where, C = probe heat capacity
q = constant heat input per unit time
$\Delta T/\Delta t$ = constant rate of temperature rise of probe

4.1.3   Location of Heater and Temperature Sensor

To minimize the initial time lag and associated errors in the measured temperature–time response, the heater should be placed as close as possible to the contact surface between the probe and the test medium. The inside space of the probe should be filled with a material with high thermal conductivity, low heat capacity, and good electrical insulation. Also, if the temperature sensor is not in direct contact with the probe wall, the value of r used in thermal diffusivity calculations will be different than the probe radius. The 'effective radius' of the probe may be determined by conducting a thermal test in glycerol and using the "TDFIT" computer program to fit the measured temperature-time response (Boggs *et al.,* 1980). Glycerol was selected because of its high viscosity and very good contact conductance.

4.1.4   Length to Diameter Ratio of Probe

To ensure that the conduction of heat from the probe into the test medium is truly radial for the test duration, the probe must have a sufficient length to diameter ratio. Blackwell (1954) provides the methodology for choosing an appropriate ratio based on his theoretical analysis. A probe length to diameter ratio of 20 or more was found to be satisfactory for thermal conductivity measurement in soils (Boggs *et al.*, 1980). If the probe is equipped with multiple temperature sensors along its length to make multiple measurements, the distance from the top- or bottom-most sensor to the nearest heater tip must be at least 10 times the probe diameter.

4.1.5 Sample Boundary Effects

Equations 8 and 10 are based on the assumption of a line or a cylindrical heat source in an infinite medium. When the measurements are made on a cylindrical specimen of finite diameter, the boundary conditions will begin to affect the temperature after a certain time. According to deVeries and Peck (1958) this limiting time can be estimated from:

$$exp\ (R^2/4\alpha\ t_b\ ) << \Phi \quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\quad(13)$$

Where, R = sample radius

$\quad\quad\quad \alpha$ = thermal diffusivity

$\quad\quad\quad t_b$ = time when the thermal front impinges on the boundary

$\quad\quad\quad \Phi$ = dimensionless factor (which is <<1), say = 0.02 (Wechsler, 1966)

After performing tests on 5 cm diameter samples of soil under extended probe heating durations, Steinmanis (1981) concluded that time periods longer than t_b are required for boundary effects to influence the temperature-time response at the probe-medium contact. Therefore, eq.13 can be used as a guide to limit the test duration for the given size of test specimen. If the available test samples are smaller in diameter than the minimum required, they can be still tested by placing them in a media having similar thermal conductivity to that of the test material. Non-homogeneity in a sample can effect the measurement, especially if it is in the direction of the conduction path (radially in the case of line source). Philip and Kluitenburg (1999) have investigated these effects in detail.

In addition to the above sources of error due to deviations in the measurement system from the theory of line source, there can be errors in the measurement of several parameters that enter into the calculation of thermal conductivity and diffusivity by the single probe method. These are primarily in the power (heat) input and temperature measurement. There has been considerable improvement in this area with the use of programmable power supplies with stable output and the use of heater wire having a low thermal coefficient. A feed-back loop can be built into the power control to compensate for changes in the electrical resistance of the probe heater during the test. Because the slope and intercept of the temperature-time curve (rather than the absolute temperature) are required for thermal property calculations, the resolution and precision in temperature sensing is more important than absolute accuracy. In cases for which a thermal probe is used as a thermometer prior to heating, improved absolute accuracy is desirable. Traditionally thermocouples have been used in most thermal probe designs. However, the development of thermistors with higher resolution, stability, and low line losses for long leads has given better performance than thermocouples when used as temperature sensors in thermal probes.

4.2   DIFFERENTIAL LINE SOURCE TECHNIQUE

In principle, this technique is similar to the single probe technique except that the temperature sensor is placed at a short radial distance r from the heater. It consists of measuring the temperature-time response due to a line source of constant rate q at a short radial distance r from the heater. The heat flow equation for this case is the same as for the single probe and is based on the line source theory:

$$T(t) = -q/4\pi k \; [Ei(-r^2/4\alpha t)] \dotfill (5)$$

Differentiating both sides with respect to time t,

$$dT/dt = (q/4\pi k) \; exp(-r^2/4\alpha t) \dotfill (14)$$

By plotting dT/dt vs. t, the values of $(dT/dt)_{max}$ and t_{max}, the time at which (dT/dt) reaches its maximum can be obtained:

$$t = t_{max} = r^2/4\alpha , \; dT/dt = (dT/dt)_{max}$$

Substituting above in eq.14 and solving for thermal conductivity of medium k:

$$k = 0.3679q/ \; 4\pi(dT/dt)_{max}$$

and $\alpha = r^2/4t_{max}$

Merrill (1969) found it was more accurate to read the time $t_{1/2}$ at which (dT/dt) reaches one-half of its maximum, which is equal to:

$$t_{1/2} = 0.37337 \; t_{max} = 0.37337 \; r^2/4\alpha$$

Thus by plotting (dT/dt) vs. t (Fig. 2) and locating $t_{1/2}$ at which $dT/dt = \frac{1}{2}(dT/dt)_{max}$, the thermal diffusivity of the test medium can be calculated as:

$$\alpha = 0.37337r^2/4t_{1/2}$$

This technique was found to be suitable for testing small amounts of particulate material such as lunar dust (West and Fountain, 1975). This method was also investigated for its suitability for field measurement of soil thermal properties by electric utilities but was considered impractical and prone to error with respect to the determination of r (probe separation) (Boggs *et al.,* 1980).

The sources of error in the above technique are:

1. Error in measurement of distance r between the heat source and the sensor;
2. Contact resistances between heater and medium and between sensor and medium;
3. Heat capacity of the probe.

The maximum error in the calculated thermal diffusivity can be expressed as:

$$\delta\alpha/\alpha = (\delta q/q) + (\delta t_{1/2}/t_{1/2}) + 2(\delta r/r)$$

The errors in heat input q can be minimized by careful selection of the power supply and controls. Errors in $t_{1/2}$ are primarily systematic in nature and they can be minimized by careful selection of instrumentation. The primary source of error in $t_{1/2}$ is in the estimation of $(dT/dt)_{max}$ from the response curves. The use of calibrated sensors and an automated data acquisition system can reduce this error. Both contact resistance and heat capacity of probe introduce a delay in t_{max} and $t_{1/2}$ and thus tend to underestimate the thermal diffusivity value α. West and Fountain (1975) estimated a maximum error in their measurement of thermal diffusivity on terrestrial basalt powder using this technique to be about 20%.

Drury (1987, 1988) used a single heating and cooling cycle for measuring thermal conductivity and diffusivity of rock cores and deep-sea sediments. He used a 6 cm long thin heater together with a thermocouple located 13 mm from the heater. Good measurements of thermal parameters from the temperature response were obtained during the heating portion of the heat cycle, while the measured data from the cooling curve did not match well with the model predictions. Therefore, using the simplified line source model for obtaining thermal parameters from the cooling curve was not recommended.

Figure 2. Thermal diffusivity determination by differential line source method.

$$dT/dt = q/4\pi kt \{\exp(- r^2/4\alpha t)\}$$

$(dT/dt)_{max}$

$t_{1/2}$

dT/dt in units of q/4πk (α/ r^2)

Thermal diffusivity
$\alpha = 0.37337r^2/4t_{1/2}$

Time (t) in units of ($r^2/4\alpha$)

Plot of (dT/dt) as a Function of Time
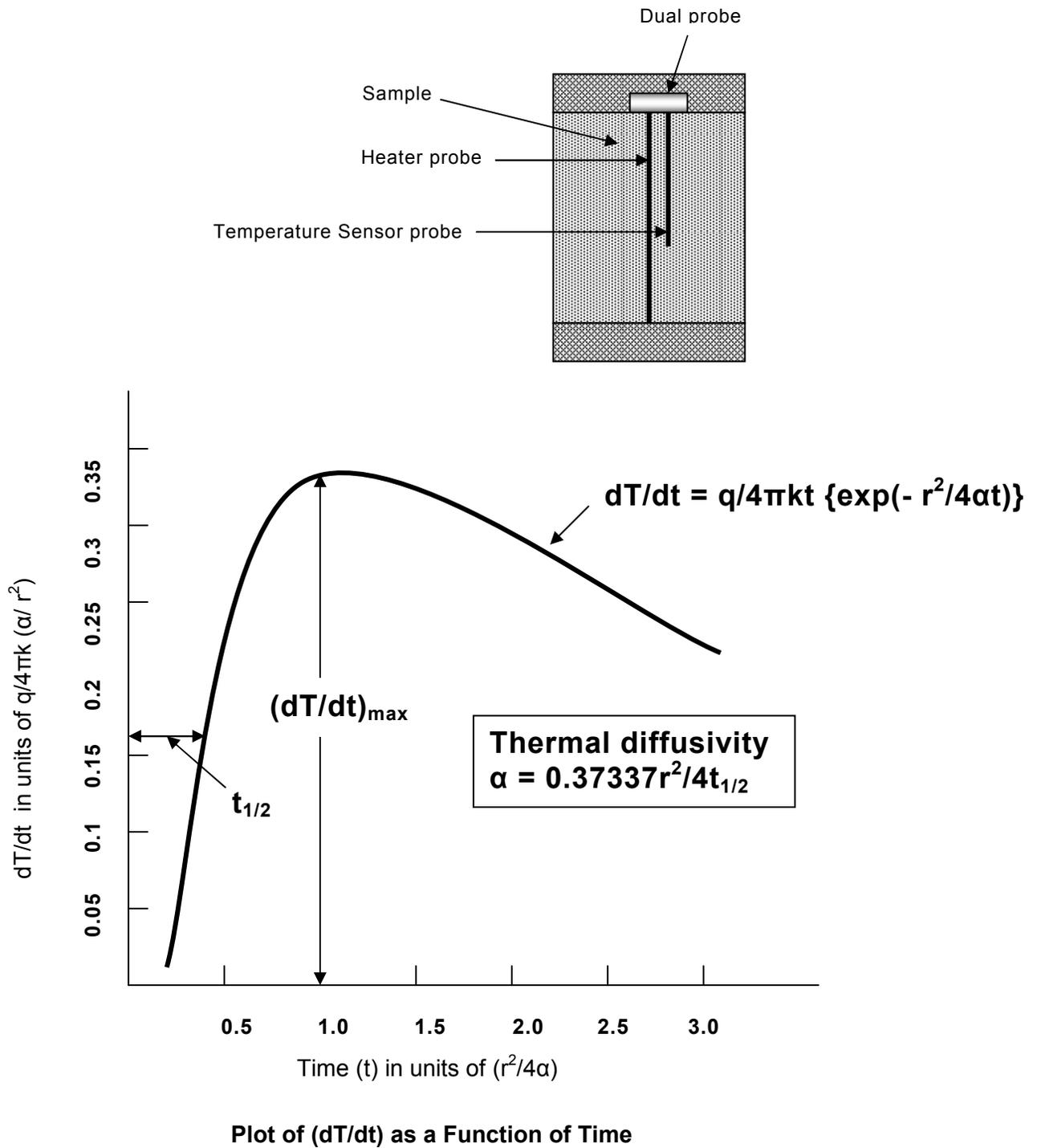
4.3   DUAL PROBE HEAT PULSE (DPHP) TECHNIQUE

A number of "heat-pulse" techniques have been employed for simultaneous measurement of thermal diffusivity and heat capacity by analyzing the transient heat flow through a test medium in response to a heat pulse of relatively short duration. The advantage of these methods over the constant line source method is that the temperature gradients established in the specimen will be comparatively small, therefore problems due to convection and migration of pore fluids are greatly reduced.

The heat-pulse technique, which employs a line source to produce a heat pulse, has been used extensively for simultaneous measurement of thermal conductivity and diffusivity of soils. This method is also called dual-probe heat-pulse (DPHP) technique. It is based on the theory of radial heat conduction of a short-duration heat pulse away from an infinite line source in an infinite medium. The temperature change at a radial distance r from the heat pulse source of strength q and duration t_0 is given as (deVries, 1952; Kluitenburg *et al.*, 1993; Bristow *et al.*, 1994)**:**

$$T(t) = -q/4\pi k \; [Ei(-r^2/4\alpha t)] \quad for \; t < t_0 \; \dotfill (5)$$

and

$$T(r, t) = q/4\pi\alpha\rho_c \; [Ei\{- r^2/4\alpha \; (t - t_0)\}- Ei\{-r^2/4\alpha t] \quad for \; t > t_0 \; \dotfill (15)$$

Where, t = time elapsed from the instant heat pulse q is applied.

Dury (1988) suggested an approximation to eq.15 and simplified it as:

$$T(r,t) = q/4\pi k \; ln(1+ t_o/t) \dotfill (16)$$

Where, t = time measured from the end of the heat pulse q which is applied for duration of t_o.

Thermal conductivity is calculated from the slope of T vs. $ln(1+ t_o/t)$ plot. Thermal diffusivity cannot be obtained from this approximate eq.16.

By differentiating eq.15 with respect time t and equating it to zero, Bristow *et al.* (1994) obtained the following solutions for α and ρ_c**:**

$$\alpha = (r^2/4)\{1/(t_m\text{-}t_0) - 1/ t_m\} / \{t_m /(t_m\text{-}t_0)\} \dotfill (17)$$

$$\rho_c = (q/4\pi T_m) \; [Ei\{- r^2/4\alpha \; (t - t_0)\}- Ei(-r^2/4\alpha t)] \dotfill (18)$$

The above analysis procedure is known as the 'single point method', because only the maximum temperature T_m and the time t_m at which it occurs are used to determine the thermal parameters (Fig. 3). It is interesting to note that the thermal diffusivity α is independent of the magnitude of temperature peak. However, the thermal conductivity is dependent on peak temperature T_m. The values of T_m recorded for an instantaneous versus a pulse heat source were found to be similar, but t_m for a pulse heat source was larger than that of an instantaneous source. Care is needed in extracting t_m from the response curve because of the noise in the data. Also, the temperature peak T_m may be difficult to identify with increasing separation r between heater and sensor. While the single-point method is easy to apply, the results are sensitive to the choice of t_m and T_m, which can be difficult to identify if the data are sparse and contain noise.

To overcome these problems Bristow *et al.* (1995), suggested solving eq.15 for several sets of values for α and ρ_c using a nonlinear least-squares curve fitting of the measured temperature-time response. This nonlinear model fit was found to match better with broad, flat peaks and sparse, noisy data (Bristow *et al.*, 1995). Once the values of α and ρ_c have been determined, the value for thermal conductivity can be easily calculated by $k=\alpha\rho_c$.

The key sources of error in the heat-pulse method are in the determination of r and t_m. The probe design, duration of heat pulse and frequency of temperature measurement should be carefully selected to achieve the desired level of accuracy in the thermal parameters obtained. Bristow *et al.* (1994) measured the thermal properties of soils using two thin needle-like probes (0.8 mm diameter by 28 mm long) at a distance of 6 mm apart. One needle contained a heat source (with or without a thermocouple) and the other contained a thermocouple at its center. A measured current was applied to the heater for about 8 s to produce a heat pulse that could be detected by the thermocouple. The results, based on four different types of soil, showed that a small increase in probe spacing r resulted in large decrease in T_m and a large increase in t_m. This effect was more pronounced with heat pulses of lower strength. The peaks in temperature-time response became less well-defined with increased probe spacing. Smaller probe spacing yielded better-defined signals, and hence T_m and t_m values, but increased the relative error in r and hence the error in thermal diffusivity. Therefore, the probe spacing must be optimized so that the response curve is well defined, the relative error is minimized, and at the same time the measurement volume is representative of the material being tested.

**Figure 3. Temperature response at the sensor due to a heat pulse from a
line source in the dual probe.**

$$\alpha = (r^2/4)\{1/(t_m - t_0) - 1/t_m\} / \{t_m / (t_m - t_0)\}$$
$$k = (q/4\pi T_m) [Ei\{- r^2/4\alpha (t - t_0)\} - Ei(-r^2/4\alpha t)]$$
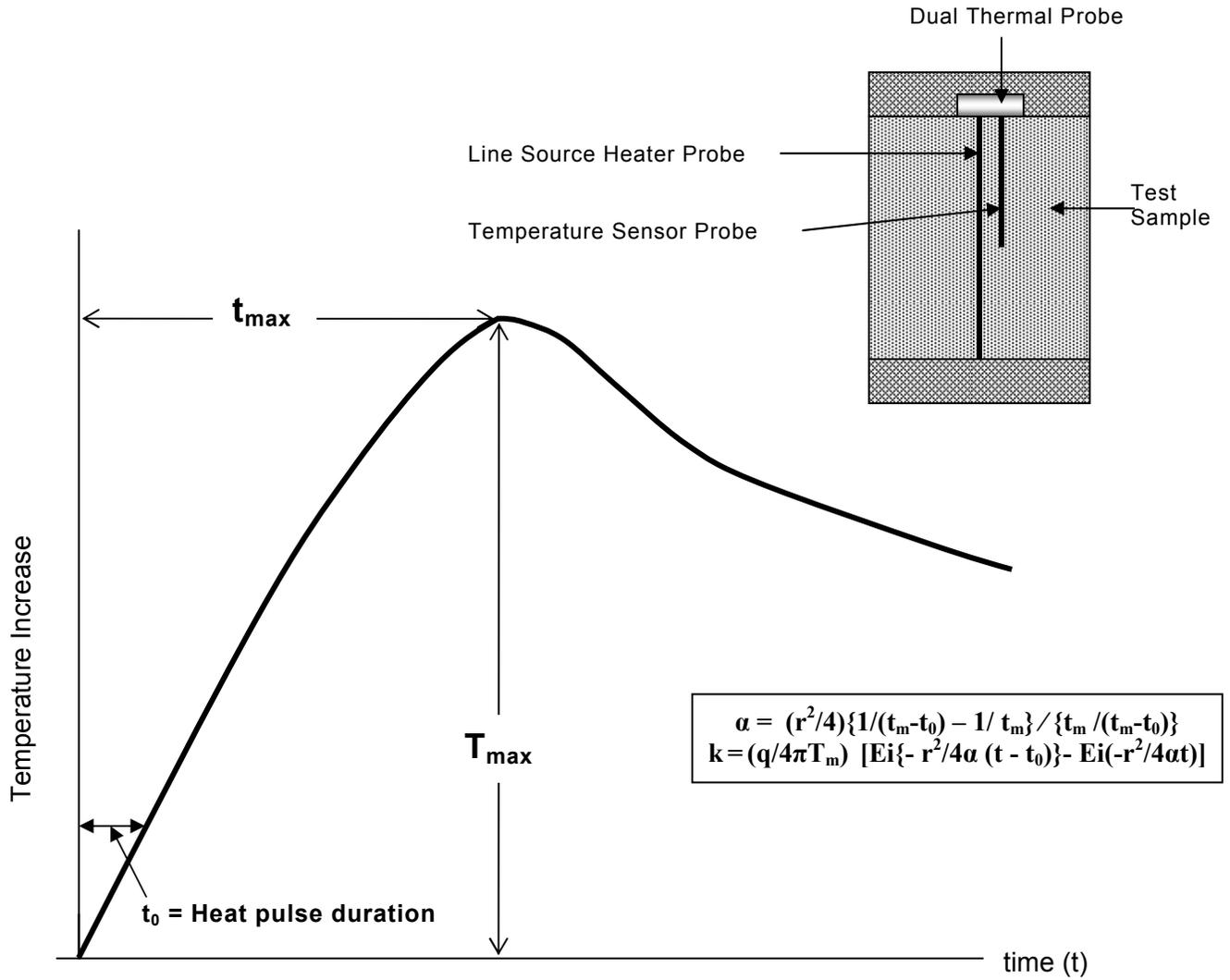
Two types of error occur in the dual-probe heat-pulse technique: (1) model errors and (2) measurement errors. Modeling errors arise due to approximations made in deriving equations (17) and (18), and due to non-compliance of the probe with the line source theory. Bilskie *et al.* (1998) conducted tests on aqueous colloidal suspensions of α-alumina and glycerol as well as on soil samples, and found that the modeling error in the measured thermal properties by pulse technique was about 3% in diffusivity and about 6% in conductivity values. They determined the thermal properties by using the nonlinear least-squares curve fitting of the measured temperature data.

The theoretical model and the solution for temperature-time response given in eq.16 are for a short duration heat-pulse from an infinitely thin and long line source. In practice the heater is a cylinder of finite length and diameter. To account for the finite length and diameter of the heater, a more rigorous solution for the radial heat conduction of a short-duration heat pulse from a cylindrical heat source was developed by Kluitenburg *et al.* (1993). They calculated the relative errors induced in the thermal diffusivity and heat capacity calculations due to finite heater length *2b* and heater diameter *a* for various heat-pulse durations t_0. Their analysis showed that both the finite length and diameter of the probe introduced model errors in thermal diffusivity calculations, while only finite length appear to give model errors in heat capacity calculations. They have produced curves showing the relative error in thermal diffusivity and heat capacity as function of a parameter ($r/2\sqrt{\alpha}$) for a range of values (*b/r*) and (*a/r*) relevant to their experimental study. The trend in these curves can be used for selecting the (*b/r*) ratio for the material being tested. It would appear that for measurements in soils a heater probe length of at least six times the probe separation would allow for negligible modeling errors.

Measurement errors arise mostly in determining the actual probe separation *r*, and in obtaining t_m and T_m from the temperature-time response curves. The error coefficient for *r* has a constant value of 2.0. Kluitenburg *et al.* (1993) presented the results of their analysis in a graphical form for estimating the error coefficients for t_m. Based on their error analysis they concluded that it is be possible to optimize the DPHP instrumentation so that the sensitivity to error in particular inputs is minimized.

4.4   HEAT PULSE FROM A POINT SOURCE

The probe used in this method consists of a central point source heater surrounded by several temperature sensors. The heating element is a small bead embedded in the tip of the central heater probe, while thermistors are housed in the tips of surrounding sensor probes. Both the heater probe and sensor probes can be rigidly mounted on an insulator block in a predetermined configuration. Larson (1988) used a "Geoflo" probe (a ground water flow meter with a center probe and multiple sensors around it) to serve as a pulsed point source for measuring thermal properties of soils (eolian silt). The center probe had a bead heater at its tip and the peripheral

probe had a thermistor at its tip. Both the heater and sensor probes were the same length. The thermistors used in the sensor probes were accurate to ±0.2°C.   The thermistor output was digitized at 1 s intervals. The pulse strength was 18.0 W and the pulse duration was 25 s. Temperature peaks ranged from 2.65°C to 4.70°C at elapsed times of 100 to 170 s.

The theoretical solution for the temperature-time response for pulsed point source has a similar from to that of a line source**:**

$$T(r,t) = q/4\pi k \left[erfc\{r/2\alpha^{1/2}\ t^{1/2}\} - erfc\{r/2\alpha^{1/2}(t - t_0)^{1/2}\} \right] \quad for\ t > t_0 \dots\dots\dots\dots\dots\dots\dots(19)$$

Where, *erfc* = error function
all other terms are same in eq.15.

Larson (1988) used the curve fitting method by generating a family of curves of *[erfc{r/2α$^{1/2}$ t$^{1/2}$} - erfc{r/2α$^{1/2}$(t - t$_0$)$^{1/2}$}]* vs. time *t* for a range of *α* values, and finding a match for the measured temperature-time response curve.   Subsequently, *k* was calculated from eq.19 using *α* and the peak values for the matched curves. The modeling errors due to axial heat loss through the heater probe have not yet been adequately investigated. The primary source of error is in the determination of the probe separation *r*.

 The advantages of the pulsed point source are that:

1. It requires only small amount of energy compared to constant heating of line source. This can be an advantage in battery powered system for field use;
2. Because of low energy input, the extent of pore fluid convection and phase change are minimized;
3. The errors due to heater dimensions in the case of line source dual-probe are eliminated;
4. The entire unit construction (heater and sensor assembly) can be more robust than a line source dual-probe.

The only disadvantage of the pulsed point source method is that the measured signals may be weak because of low energy input, especially with larger probe separation.

Figure 4. Determination of thermal conductivity and heat capacity from a pulsed line source along the side of a cylindrical specimen
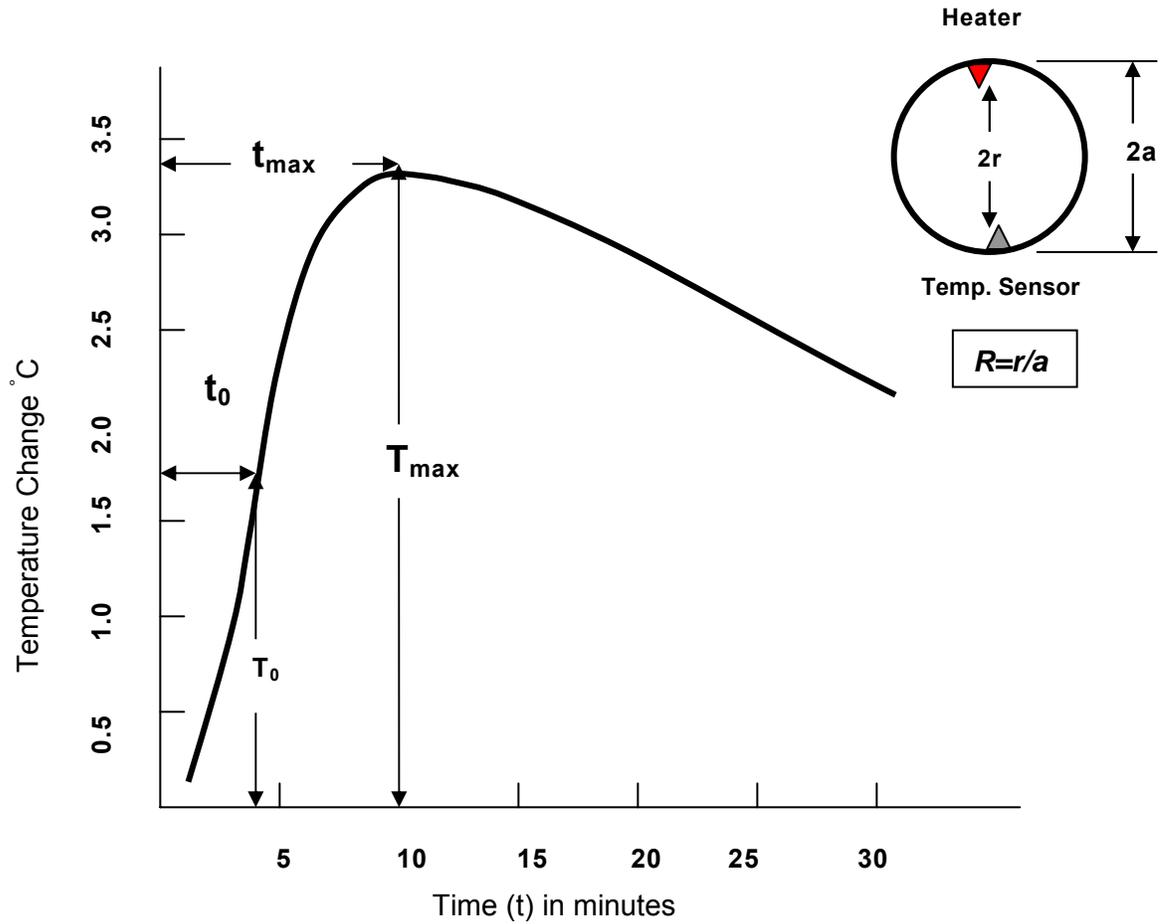
Thermal conductivity

$k = \{q/\pi(T_{max} - T_0)\}\{\ln(1+R) + \frac{1}{8} - \frac{1}{4} R^2\}$
heater turned off at $t = t_0$ large enough for the linear asymptote to have been atained

Heat Capacity

$C_p = (q /\pi a^2 M)$; where M= Slope of straight line portion of T vs t response curve ($t < t_0$)

4.5   PULSED LINE SOURCE ALONG THE SIDE OF A CYLINDRICAL SPECIMEN

Howard (1963) and Jaeger and Sass (1964) developed a simple and cost effective method for determining thermal parameters of core samples of rock by applying a heat pulse through a line-source heater thermally bonded to the cylindrical surface of the specimen. The temperature evolution at a point located diametrically opposite to the line-source heater was measured by a thermocouple (Fig. 4). The heater and the thermocouple were embedded in two shallow saw cuts in the sample.

4.6   SURFACE HEAT SOURCE TECHNIQUES

Surface heat source techniques produce one-dimensional heat conduction through the test specimen. The heat is applied either at a constant rate or as a short duration pulse to the flat surface of the specimen while the temperature evolution at the opposite parallel surface is monitored. The test specimens are prepared in the form of slabs or discs of uniform thickness with their heat application surface ground flat and sometimes polished.

4.6.1   Constant Surface Heat Source

This method consists of applying a constant heat flux to the plane surface of a specimen in the shape of a slab and measuring the temperature vs. time response at the opposite face which is completely insulated against any heat loss. The sides of the specimen are also insulated against heat loss. Thus it represents the case of 1-D transient heat conduction through a slab. The temperature T at the insulated face of a slab of thickness a at time t due to a constant heat flux q at the opposite face is shown to be (Carslaw and Jaeger, 1959):

$$T = q\alpha t/ak - qa/6k + transient\ terms \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots(21)$$

Where, t is large relative to $\alpha\pi^2/a^2$, the transient terms in the above expression can be neglected. In this case temperature vs. time behavior becomes linear, with slope m and intercept t_i on the time axis such that $m = q\alpha/ak$ and $t_i = a^2/6\alpha$ (Fig. 5). From this linear portion of the temperature-time response, the thermal diffusivity α and thermal conductivity k can be calculated by the slope m and intercept t_i respectively as follows:

$$\alpha = a^2/6t_i\ and\ \ k = qa/6mt_i$$

It is interesting to note that thermal diffusivity is independent of q the heat flux applied, but the thermal conductivity is a direct function of the heat flux q.

Figure 5. Determination of thermal conductivity and diffusivity from constant surface heat input to insulated thin specimen.
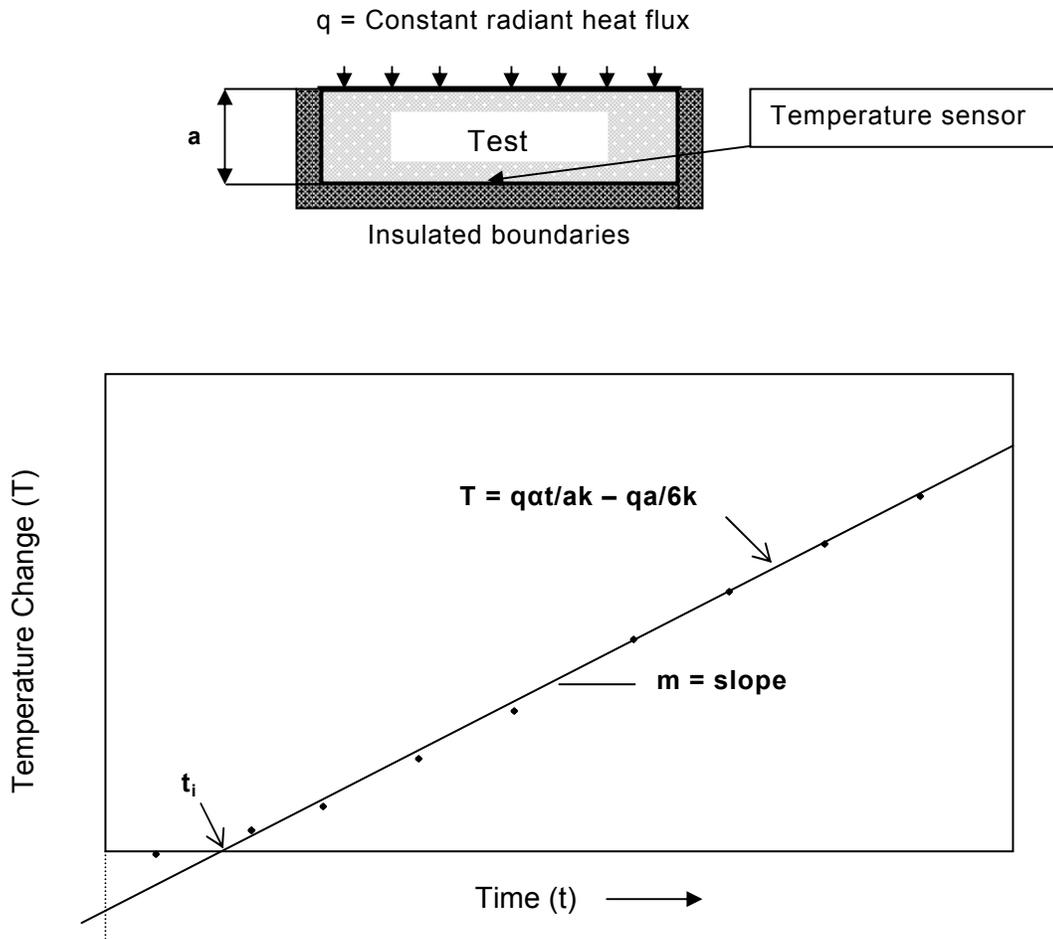
$$\text{Thermal conductivity } k = qa/6mt_i$$
$$\text{Thermal diffusivity } \alpha = a^2/6t_i$$

Middleton (1993) in his measurements of the thermal properties of rock specimens placed a constant current carrying coiled-wire element 1 cm above the top surface of the specimen. By creating an 'oven-effect', the constant heat flux boundary condition for the top surface of the test specimen was closely approximated, and contact resistance effects were eliminated. A digital thermometer with a 0.2 s time constant was used for monitoring the temperature at the base of the specimen. The temperature sensor was affixed to bottom face of the specimen. A thin leaf of aluminum foil was placed between the specimen and base insulation to improve the contact between the specimen and sensor, and the distribution of the base temperature.

The major sources of error in this method arise from the drying of surface moisture on the heated boundary and poor contact between the temperature sensor and the base of the specimen. The surfaces of test specimens should be air dried to minimize errors due to evaporation of free moisture. Middleton (1993) obtained good measurements of thermal diffusivity on rock samples from the sedimentary basin of Western Australia. The main advantage of this technique is that the analysis of results is very simple. The disadvantages of this technique are that the thermal conductivity value cannot be determined directly because the heat flux into the specimen is not measured. However, by using a heat flux meter on the top of specimen surface, this limitation may be overcome. The test setup is somewhat cumbersome and therefore is not suitable for field use.

4.6.2 Heat Pulse from a Surface Source

This technique consists of applying a heat pulse to the flat surface of a test specimen and estimating the heat conduction parameters from the temperature-time response recorded at a distance from the heat source. This technique was used for cubical specimens of rock by Seipold and Raab (2000), and for thin specimens of metals and ceramics by Bougrine *et al.* (2000). The heating element is usually of a foil type, which is glued to the surface of specimen with a thin coat of heat conducting varnish. The heater is covered with a radiation shield to prevent heat loss. The temperature sensor is fastened to the opposite face (Bougrine *et al.*, 2000). Alternatively, a setup similar to the parallel bar can be configured by sandwiching a plate heater between two identical specimens (Seipold and Raab, 2000). The temperature sensor is placed at a distance from the heater. The heat pulse is generated by passing constant current for a short duration and turning it off.

The thermal diffusivity, heat capacity, and thermal conductivity are obtained simultaneously by solving the heat conduction equation by means of a numerical technique and fitting it to the measured temperature-time response (Seipold and Raab, 2000). This technique is somewhat cumbersome both in experimental setup and in analysis procedures, therefore its application may be limited to laboratory-based measurements.

4.7   HEAT-FLASH TECHNIQUES

In the heat-flash method, a high-intensity short-duration radiant heat-pulse is applied to the surface of a test specimen by a non-contact heating source, which may be an electric heater, a laser flash, or a high-speed xenon discharge lamp. The temperature-time response at the rear surface of the specimen is recorded. Schilling (1999) used a non-contact electric heater as a radiant heat source in his setup for measuring thermal properties of crystalline rocks at elevated temperatures. The specimen sizes varied from 8 to13 mm in diameter and 3 to 18 mm in length. He attached two thermocouples to the ends of the test specimen: the first was placed between the heating element and the heated face of the specimen, while the other was placed in contact with the opposite face of the specimen.

Since the input heat flux is an irregular function of time in this case, there is no simple mathematical solution for the temperature response at the rear surface of the specimen. Instead, the temperature-time curve recorded by the sensor near the heat source is used to calculate the temperature-time response at the rear surface. Calculations are performed by finite difference method by using a large number of finite elements to model the one-dimensional transient heat flow through the specimen. The input values of thermal diffusivity are varied systematically to obtain a best-fit between the measured and calculated curves (least-squares algorithm). This evaluation procedure does not require absolute temperature or transferred heat values (Parker *et al.*, 1961). It is possible to include heat loss through radiation and conduction from specimen surfaces but the calculation procedures become tedious. Using this technique, Schilling (1999) obtained reliable measurements of thermal diffusivity on a range of materials with different mineral composition and crystalline structure.

In the laser flash method, a small disk–shaped sample is subjected to a very short burst of light energy using a laser or a xenon flash lamp with irradiation times of one millisecond or less (Parker *et al.*, 1960).   The temperature history of the rear face is monitored either by a thermocouple or infrared detector and recorded with a memory scope. Since the temperature rise is very small and of very short duration, this technique may be used for heat-sensitive and phase changing materials. Parker *et al.* (1960) used a commercially available quartz flash tube as the flash source for thermal diffusivity measurements of a number of metal specimens. Watanabe (1988) used a ruby laser as the flash source to determine the thermal diffusivity and heat capacity of thin specimens of synthetic sapphire.

By assuming that the heat input is an instantaneous pulse, the temperature rise at the rear surface of a cylindrical specimen with thermal diffusivity α is given by the expression (Parker *et al.*, 1961):

$$T(t) = Q / C_p L \left[1 + 2 \sum_{n=1}^{n=\infty} (-1)^n \exp\{-n^2 \pi^2 \alpha\, t / L^2\} \right] \quad\quad\quad (22)$$

Where, Q = radiant energy instantaneously and uniformly absorbed by the front surface

   L = specimen thickness

   α = diffusivity

   C_p = volumetric heat capacity

The shape of the temperature vs. time curve determines the thermal diffusivity and the maximum temperature at the rear surface determines the heat capacity.

From eq. 21 and by plotting the ratio (T/T_{max}) for the temperature rise at the back of specimen vs. dimensionless parameter $(\pi^2 \alpha t/L^2)$, the thermal diffusivity α and thermal conductivity k can be deduced as:

$$\alpha = 1.38L^2/\pi^2\, t_{1/2}$$

$$k = 1.38QL/\pi^2\, t_{1/2}\, T_{max}$$

Where, $t_{1/2}$ = time required for back surface temperature to reach half of the maximum

   temperature rise T_{max}

It is interesting to note that the amount of energy absorbed by the front surface of the specimen is required only for the determination of thermal conductivity but not for thermal diffusivity. The transferred heat flux to the front surface of the specimen is not easy to measure because it is an irregular function of time, as in the case of a radiating heat input. The success of the flash technique depends upon meeting the following boundary conditions:

1. Uniform irradiation of front surface of the sample in a short amount of time compared to the rise time of the back surface temperature;
2. Accurate measurement of the back surface temperature;
3. Minimum heat losses;
4. A temperature signal that is large and free of noise.

The irradiated sample surface is often coated with heat absorbing compound (carbon black, platinum paste, etc.) for uniform and improved heat absorption. The temperature-time signal can be monitored by an oscilloscope and captured by a camera. The half time $t_{1/2}$ can be scaled off from the trace in units of distance and converted to time by multiplying it by the sweep speed. A differential transistor preamplifier may be used to give a stable and linear gain under the conditions of operation. A detailed description of a simple setup used for measuring thermal diffusivity of metal samples is given by Parker *et al.* (1961).

The minimum thickness of the sample is determined by the requirement that the flash duration must be short compared to the time the temperature begins to rise at the back surface. A sample

that is too thin results in a low value for the thermal diffusivity obtained by the flash technique. On the other hand, if the sample is too thick, the sensitivity is reduced and the time for losses to occur is increased.

Another method of using the flash technique for determination of heat capacity is based on alternately measuring the responses of a (known) reference and the (unknown) test sample, and obtaining the results from their differential behaviour. The "test sample shot" should immediately follow the "reference shot" and the flash source must remain stable between these two shots. The temperature rise at the rear surface of specimen is detected with a fast sensing detector, while the ambient temperature is continuously monitored by a thermocouple adjacent to the sample holder.

4.8 INSTANTANEOUS CHANGE OF BOUNDARY TEMPERATURE

This method of measuring the apparent thermal diffusivity is based on an analytical solution for transient heat flow through a cylinder (eq. 4) under well-defined boundary conditions. Shannon and Wells (1947) presented a method for analyzing cylindrical samples of frozen and unfrozen soils. In their method, the external surface of the cylindrical sample is subjected to an instantaneous (step-wise) temperature change by immersing it in a constant temperature bath and monitoring the temperature at its center as a function of time. The measured response curve is then matched with the theoretical curve and the thermal diffusivity is calculated for the matched points in the curves (Fig. 6). Their analysis is based on the theoretical solution proposed by Carslaw (1945) for the heat flow in a cylinder of finite length subjected to an instantaneous boundary temperature change. The solution is expressed in the form of a curve of percent temperature change μ at the center of the sample vs. the dimensionless time factor T. By matching the measured temperature-time response with the theoretical curve at 50% temperature change, thermal diffusivity α is calculated as follows:

$$\alpha = D^2 T_{50} / t_{50}$$

Where, D = diameter of sample
   T_{50} = dimensionless factor at 50% temperature change
   t_{50} = time taken to reach for 50% temperature change

By using a thermal probe (line source) for measuring the temperature in the center of soil sample Steinmanis (1981) was able to perform thermal conductivity measurements before the same specimen was subjected to above test. Thus, both thermal conductivity and diffusivity values were obtained by combining the single probe technique for thermal conductivity and the *Shannon and Wells* (1947) method for thermal diffusivity.

Figure 6. Thermal diffusivity determination by instantaneous changing of boundary temperature.

Insulation & water proof cap

Metal container

Thermocouple

Test sample

Thermal probe

Insulation & water proof cap

Thermal diffusivity
$\alpha = D^2 T_{50} / t_{50}$
D = Sample diameter

Temperature at the center of sample ($^\circ$C)

Theoretical Model
Shannon & Wells (1947)

% Temperature

T_{50}

Time Factor, T

0.01

0.1

Experimental curve

Time (s)
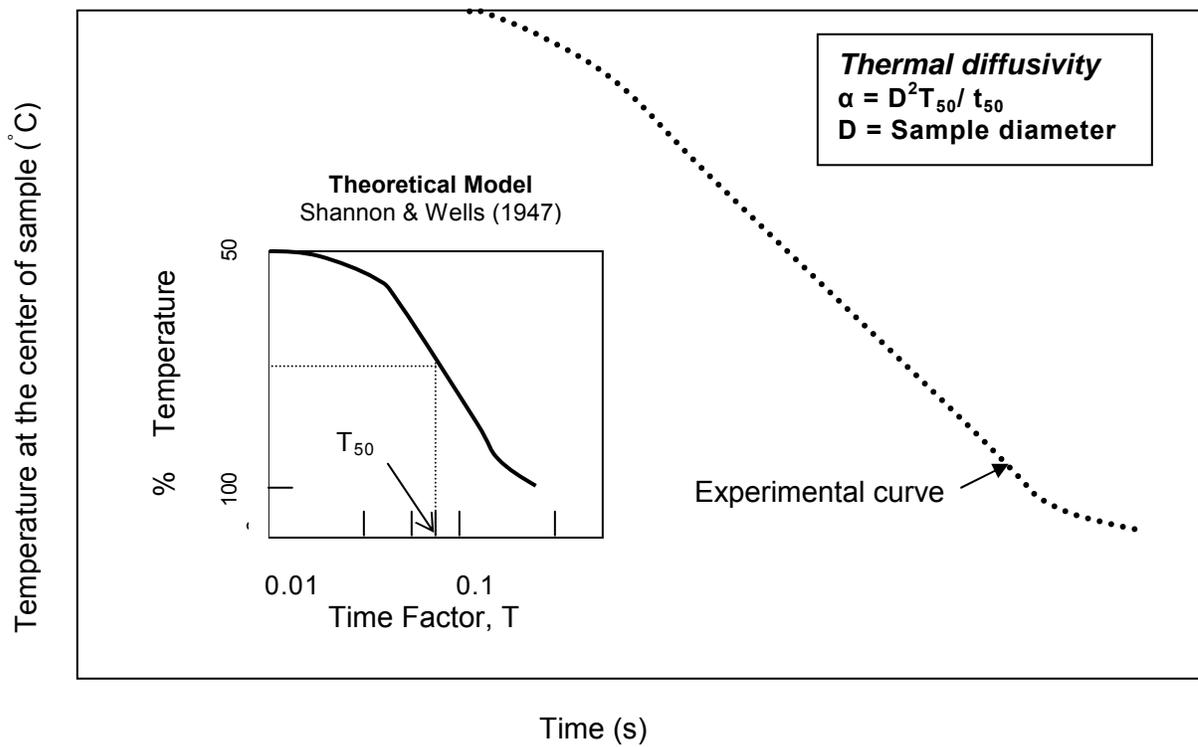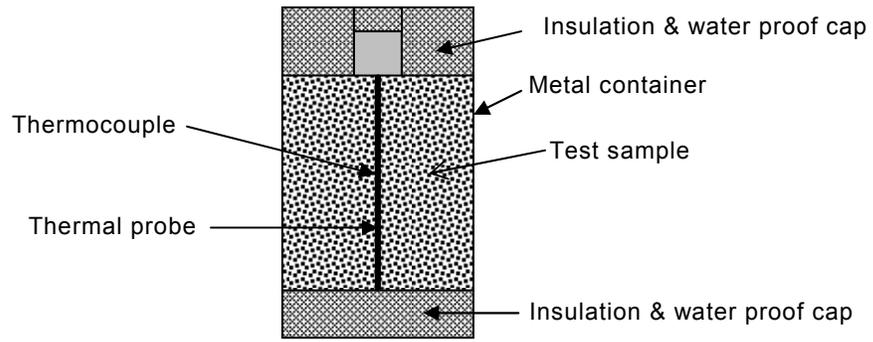
5.0   CALORIMETRIC METHODS

Calorimetric methods of determining the specific heat of solids and liquids are well known in the field of physics. In the case of porous materials and particulates, which may contain pore fluids, these methods have to be modified to include the contributions from pore volume and pore structure. Calorimetric methods can only determine the volumetric heat capacity and not the thermal conductivity or diffusivity.

5.1   ADIABATIC CALORIMETER

An adiabatic calorimeter consists of a thermally insulated container into which a known quantity of material is placed and subjected to uniform heating. A thermal guard is used to surround the insulated container to minimize heat losses (Johansson and Fervick, 1980). The equilibrium temperature for a measured heat input is recorded by a set of temperature sensors. By performing the heat balance calculations, the heat capacity of the test material is extracted. These tests can be repeated by increasing the heat input in steps and calculating apparent heat capacity as function of mean ambient temperature.

5.2   DIFFERENTIAL SCANNING CALORIMETER

A differential scanning calorimeter (DSC) is used commercially for determining the apparent heat capacity of a variety of solids, soil and rock samples, particulates, and liquids. The scanning calorimeter works by changing the temperature of a sample and a reference simultaneously at a constant rate. The temperatures of the test and reference specimens are measured separately while they are put through the same temperature course (heating or cooling). To maintain a constant rate of temperature change within the sample, a variable amount of heat must be supplied due to changing apparent heat capacity. To obtain an accurate measurement three scans are required:

1. An initial scan using an empty sample pan to obtain a base line signal B_T;
2. A second scan on the pan containing material of known heat capacity Cr_T resulting in the reference signal R_T;
3. A third scan on the pan containing the test material, resulting in the signal S_T.

At each temperature T, the apparent heat capacity Cp_T of the test material can be calculated from the following equation:

$$Cp_T = \{(S_T - B_T)/(R_T - B_T)\}(Vr/Vs)\ Cr_T \quad \text{...} (23)$$

Where, Vr = volume of the reference material
$\quad\quad\quad Vs$ = volume of the test material

In the modern DSC, the B_T and R_T signals are collected from high-speed data acquisition system and processed with computers. DSC is used commonly for frozen and unfrozen soils (van Loon *et al.*, 1993). DSC has also been effectively used to study the unfrozen water content in partially frozen soils (Oliphant and Tice, 1982).

6.0 ASSESSMENT OF AVAILABLE TECHONOLOGIES

In this section, the current technology and available techniques are assessed for measuring the heat conduction properties of relatively small samples of solids and porous materials containing water and/or ice and/or air. This review is based on considerations of:

1. Theoretical soundness, level of complexity in data analysis and accuracy;
2. Test device, instrumentation and ability to satisfy theoretical model;
3. Test duration, sample preparation, and size limitations;
4. Effects of mass transfer and phase change;
5. Adaptability for measurement at different ambient temperature and pressure conditions.

Table 3 summarizes the results of this assessment. The following is a brief discussion on the assessment of techniques:

Table 3. Summary of the review and assessment of techniques for measuring heat conduction properties of solids.

Technique	Heat Source	Parameters Measured	Theoretical Model	Modeling Errors	Measurement Errors	Total Errors	Sample Size, Shape	Sample Preparation	Test Duration	Adoptability for different temperature and pressure
Transient Single Probe	line source constant power	only conductivity not suitable for diffusivity	slope of T vs. ln(t)	contact resistance moisture migration boundary effects	power input temperature resolution	2 to 3% in conductivity	minimum 5cm for soils 8cm for rocks	insertion of probe into sample	5 to 10 min	adaptable for different temperatures
Transient Single Probe	cylindrical source constant power	conductivity and diffusivity	intercept of T vs. ln(t) line on t-axis or TDFIT	contact resistance and probe heat capacity	effective radius of probe	2 to 3% in conductivity up to 50% in diffusivity	minimum 5cm for soils 8cm for rocks	insertion of probe into sample	5 to 10 min	adaptable for different temperatures
Transient Dual Probe	line source constant power	conductivity and diffusivity	(dT/dt)max and t1/2 at 1/2(dT/dt)max	contact resistance and probe heat capacity	temperature resolution	2 to 3% in conductivity up to 20% in diffusivity	minimum 5cm for soils 8cm for rocks	insertion of probe into sample	5 to 10 min	adaptable for different temperatures
Transient Dual Pulsed Probe (DPHP)	heat pulse from a line source	conductivity and diffusivity	peak temperature Tmax and time tmax	contact resistance and probe heat capacity	power input, probe separation ' r ' and temperature	2 to 3% in conductivity up to 20% in diffusivity	minimum 5cm for soils 8cm for rocks	insertion of probes into sample	3 to 5 min	adaptable for different temperatures
Transient Dual Pulsed Probe	heat pulse from a point source	conductivity and diffusivity	temperature-time response curve matched with theoretical type curves	none identified	power input, probe separation ' r ' and temperature	2 to 3% in conductivity up to 20% in diffusivity	minimum 5cm for soils 8cm for rocks	insertion of probes into sample	4 to 5 min	adoptable for different temperatures
Transient heat conduction from pulsed line source	pulsed_line source attached to side of cylinder	conductivity and diffusivity	temperatures at end of heat pulse and at its maximum	heat loss at the source	input heat, effective radius and temperature resolution	5 to 10%	small cylindrical samples length to diameter ratio 5 or more	shallow saw cuts on the sample sides to embed heater and sensor	15 to 30 min depending on sample diameter and thermal properties	adoptable for different temperatures
Transient heat flow due to instantaneous change of boundary temperature	Sample transferred to warm or cold constant temperature bath	only diffusivity	matching temperature response with theoretical solution	heat transfer at sample boundary	moisture migration temperature resolution	less than 5%	cylindrical sample height to diameter ratio of 2	installing temperature sensor in the middle of sample and thermal sealing it	1 to 2 hours	adoptable for different temperatures
One-dimensional Transient heat flow	plate heater or radiant heater	both thermal conductivity and diffusivity	slope and intercept of linear temperature-time response	moisture evaporation at heat input boundary and contact resistance at temp. sensor	accuracy in the measurement of heat flux and resolution of temperature sensor	5 to 10%	disc shaped sample, size depends on heater size	cut to size and shape, surface leveling	few minutes	adoptable for different temperatures
One-dimensional Transient heat-pulse	heat pulse from a plate heater sandwiched between two identical samples	only thermal diffusivity	temperature-time response data matching with numerical simulation of 1-D heat-pulse propagation	heat loss at input boundary and contact resistance at sensor	sample thickness and temperature resolution	about 3% or less	pair of cylindrical or cubical samples of any size	cutting and leveling flat surfaces	2 to 5 min depending on thermal properties and sample thickness	adoptable for different temperatures and pressures

Technique	Heat Source	Parameters Measured	Theoretical Model	Modeling Errors	Measurement Errors	Total Errors	Sample Size, Shape	Sample Preparation	Test Duration	Adoptability for different temperature and pressure
Transient heat-flash	short burst of heat from xenon discharge or laser beam lamp on the flat face of sample	only thermal diffusivity	analysis of temperature-time response at the flash face and/ or opposite face of sample	none reported	accuracy and resolution of temperature sensing	not available	sample sizes depend on available equipment	smoothing surfaces and trimming to fit the holders	less than a minute	adoptable for different temperatures
Differential Scanning Calorimeter (DSC)	short burst of heat from xenon discharge or laser beam lamp on the flat face of sample	only thermal diffusivity	analysis of temperature-time response at the flash face and/ or opposite face of sample	none reported	accuracy of temperature sensor	not available	sample sizes depend on available equipment	smoothing surfaces and trimming to fit the holders	less than a minute	adoptable for different temperatures
One Dimensional Steady-State	guarded hot-plate supplying constant heat flux	only thermal conductivity	1-D steady-state heat conduction model	heat loss through insulated boundaries	heat input sample thickness	2 to 3%	depends on sizes of heater size	smoothing surfaces	several hours	adoptable for different temperatures
One-Dimensional Steady-State parallel bar	flat plate heater	only thermal conductivity	1-D steady-state heat conduction model	heat loss from sides	accuracy of temperature sensor and sample thickness	2 to 3%	depends on sizes of hot plates and guards	smoothing surfaces and trimming to fit the holders	several hours	adoptable for different temperatures
One Dimensional Steady-State divided bar	constant temperature fluid circulation	only thermal conductivity	1-D steady-state heat conduction model	none	accuracy of temperature sensing	3 to 3%	depends on size of divided bars	smoothing surfaces	several hours	adoptable for different temperatures and pressures

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6.1   THEORETICAL SOUNDNESS, DATA ANALYSIS, AND ACCURACY

Steady-state methods are based on simple 1-D or radial heat conduction theory. The computation procedures are simple and straightforward.   Therefore there are very few modeling errors provided the test setup conforms to the assumed boundary conditions. Measurement errors that occur are limited to the dimensions of the sample. Errors in measuring absolute temperatures do not affect the calculated thermal conductivity, which is a function of only the thermal gradients. Usually, an elaborate test setup is required to meet thermal boundary conditions and long test durations (directly proportional to thermal conductivity and sample thickness) are required to ensure that steady-state conditions are reached. These methods are suitable for measuring only thermal conductivity and not for measuring thermal diffusivity or heat capacity.

Thermal transient methods are based on matching the solution to transient heat conduction equation with the temperature-time response at the sensor location for different boundary conditions and different types of heat input. Different types of heat sources used are: point source, line source, cylindrical source, or plane source. The heat may be applied continuously at a constant rate or in the form of a pulse for a short duration. The heat can even be a short burst of radiant heat of variable flux, as in the case of flash methods. The computation methods vary from using simple and approximate analytical solutions to curve fitting methods, which employ numerical techniques and iterative procedures.

The line source theory for constant rate of heat input generally agrees with the modeled temperature-time response of a long cylindrical probe at long duration (after the initial transient period) from which the thermal conductivity value is easily calculated. The heat probe should have a length at least 20 times the probe diameter to satisfy the assumption of an infinite line source. The thermal diffusivity measurement however, is a bit more involved because of the effects of probe heat capacity and contact resistance. In the case of larger diameter probes, the radial position of the heat and temperature sensors become important. An 'effective diameter' can be determined by a test run in glycerol and using a curve fitting program (Boggs *et al.*, 1980). For the intercept method, modeling errors in calculating thermal diffusivity may be up to 100%. Improved curve fit methods, like TDFIT, can reduce modeling error to about 50% (Boggs *et al.,* 1980). Generally speaking, accurate and reliable measurements of thermal diffusivity are not possible with a laboratory-size single-probe of diameter less than 6 mm.

Dual-probe design is preferred to single-probe for thermal diffusivity measurement. The method of calculating thermal parameters from the dual-probe data appears to be less sensitive to initial transient conditions. The heater probe should be as thin as possible and have a heater length of at least 20 times the probe diameter to satisfy the assumption of infinite line source. The major source of error is in estimating the actual radial distance between the heater and temperature sensor (probe separation *r*). The thermal diffusivity values obtained from this technique can have an error of up to 20% (West and Fountain, 1975).

The principle behind the dual-probe pulsed heat-source (DPPH) technique is that the temperature measured at a short distance r from the line source reaches a peak T_{max} at time t_{max}. The thermal diffusivity and conductivity are calculated from the recorded peak temperature T_{max}, time t_{max} taken to reach the peak, the probe separation r, rate of heat input q, and the duration of pulse t_0. This calculation method is called the "single point method". Nonlinear least-squares curve fitting of the measured temperature-time data is recommended to obtain T_{max} and t_{max}. Modeling error in the measured thermal properties is less than 6% based on tests on aqueous colloidal suspensions of α-alumina and glycerol and on soil samples (Bliske *et al.*, 1998). More significant error (10 to 20%) can occur in calculated heat capacity due to error in the measurement of distance between the heater and the sensor r.

The theoretical basis and data reduction for the pulsed point source is similar to that of pulsed line source. For the known probe separation r and pulse duration t_0, the theoretical curves are generated for the expected range of thermal diffusivity values. The thermal diffusivity is calculated from the curve that best matches with the measured temperature-time response curve. The maximum error in the measured thermal diffusivity from the pulsed-point source is in the same range as that obtained from the pulsed-line probe. Again the principal source of error is in the measurement of distance between heater and sensor r.

Techniques which simulate 1-D transient heat conduction are ideally suited for measuring thermal properties of specimens prepared in the form of slabs. Heat is applied to one of the two flat parallel surfaces while the temperature is monitored at the other surface.

In one such technique, the top surface of slab is maintained as a constant heat flux boundary while the bottom surface is kept as an adiabatic boundary where the temperature is monitored as a function of time. At times long enough to ignore transient terms in the equation, the temperature at the adiabatic boundary is shown to rise linearly with time. The thermal diffusivity is calculated from the intercept on the time axis while the thermal conductivity is determined from the slope of the line, its intercept on time axis, and the constant heat flux. The primary sources of error in this technique are the heat losses at the boundaries and inadequate contact between the sensor and the sample base. The radiant heat from an electrical coil element with constant current input is found to produce near constant flux conditions. The thermal diffusivity is independent of the actual amount of heat flux applied, but the thermal conductivity is a direct function of heat flux, which is difficult to measure. Therefore this technique, in its present form, is not suitable for direct measurement of thermal conductivity. However, it may be possible to modify the test setup for measuring the input heat flux with newly developed heat flux sensors (e.g. NETZSCAN).

One-dimensional transient techniques using a radiant heat pulse of unknown distribution on the sample surface and monitoring the temperature-time response at the opposite face are used for cylindrical or cubical specimens of rocks. The analysis procedure is somewhat more involved

than those used for pulsed line and is accomplished by curve-fitting using numerical solutions in an iterative process. These techniques are becoming more common, however they are not suitable for estimating thermal conductivity.

Heat flash techniques involve applying a short burst of energy to the flat surface of a thin specimen and recording the temperature-time response at the other face. Techniques for calculating thermal diffusivity range from a simplified model which requires only the peak temperature (T_{max}) and the time $(t_{1/2})$ for the temperature to reach half its peak value (Parker *et al.*, 1961), to sophisticated numerical modeling and curve fitting methods (Watanabe, 1988). Accurate determination of thermal conductivity using the flash method is not possible since it requires an accurate estimate of the magnitude of the heat input pulse. These techniques, which were originally developed for metals, ceramics, and other composites, are becoming popular for use on rocks and other porous solids, but may not be suitable for routine measurements.

The Shannon and Wells (1947) method of determining thermal diffusivity by subjecting the external boundaries of a cylindrical sample to a sudden change in temperature, has a sound theoretical background and is a well proven technique for frozen and unfrozen soils as well as rocks. If a thermal needle is embedded in the center of the sample for temperature sensing, the thermal conductivity measurements can be made separately before the diffusivity measurement (Steinmanis, 1981).

The heat-pulse method suggested by Jaeger and Sass (1964) for rock cores is attractive because it is simple to perform. The method involves attaching a wire heater and a temperature sensor to the opposite sides of a cylindrical test specimen. For the case of no heat loss from its surface, the temperature rise vs. time becomes asymptotically linear over long periods of time. By applying a heat pulse long enough for the linear asymptote to be evident in the response curve (as suggested by Howard, (1963)), the thermal diffusivity can be calculated from the maximum temperature recorded T_{max} and the pulse duration t_0. The thermal conductivity can be calculated from the power of the heat pulse and two values of temperature: one at the end of heat pulse and the other at the maximum. The major source of error is the heat loss from the sample surface. The method of analysis to account for this heat loss is complex and requires an estimate of magnitude of the heat loss.

Differential Scanning Calorimetry involves comparing the thermal response of the test specimen with that of a reference material of same shape and size and subjected to same heat pulse. The analysis procedure is relatively simple. Sources of errors can be attributed to the preparation of the test sample and the reproduction of the heat pulse between reference and test measurements. These systematic errors can be eliminated by careful control of the test parameters. Since the thermal diffusivity is calculated as function of the ratio of the two temperature responses, systematic measurement errors are cancelled out. There has been considerable advancement in the development of test equipment, instrumentation, and analysis procedures, including the use of

laser flash techniques, infrared thermographs coupled with high-speed data acquisition, and data reduction systems. Note that only the thermal diffusivity or heat capacity can be obtained using these techniques.

6.2   TEST SETUP AND INSTRUMENTATION

Steady-state techniques require elaborate setups with proper insulation to prevent heat loss from specimen boundaries, together with plate heaters and guard heaters, or liquid circulated coils as heat sources. These methods are not suitable for soils and unconsolidated sediments because of the difficulty in preparing test specimens. The duration of testing can extend to several hours.

Thermal probes used in line source techniques are self-contained (heater and sensor) and are commercially available. The probes can be custom designed and fabricated for special applications. Thermal probes are extensively used in thermal testing of soils, soft sediments, particulate materials, and rock cores. Installation of probes in hard materials like rock and frozen soils require drilling of fine holes to insert the heater and sensor probes. High conductivity fillers must be used to set the heater probe in the specimen. It may be advantageous to eliminate the probe shell to minimize the thermal mass and contact resistance.

Dual probes of different probe separation may be required for different materials and different applications. Installation in soft sediments and soils can be accomplished by inserting dual-probes as a single unit. In hard materials such as rocks, two narrow, straight holes at proper spacing must be drilled for the heater and the sensor. Epoxy filling secures the heater and sensor within the material. The length of heater should be at least six times its distance from the sensor to conform to the assumption of infinitely long line source theory. For testing small diameter rock cores, it may be advantageous to drill a short length hole (less than the core radius) from the side for positioning the temperature sensor. The hole should be in-filled with a material of low thermal conductivity to minimize radial heat loss. For *in situ* measurements, accurate determination of r is likely difficult. Thus, over-coming challenges in probe installation and errors in estimating actual probe separation will be factors affecting the success of the dual-probe technique. Probe design and installation procedures should be carefully optimized for the material being tested so that the influence of these factors can be minimized.

State-of-the-art instrumentation for control of input power and data sampling are available for both the single and dual-probe techniques. Portable analyzers specially designed for the measurement of thermal properties (Thermal Property Analyzer or TPA) are available for single- and dual-probe techniques with constant or pulsed power input (Boggs *et al*., 1980). Improved accuracy and resolution in temperature measurement has been achieved by using thermistors instead of thermocouples. The programmable power supply used in these units can supply and regulate the electrical power input into the heater either in a continuous or a pulse mode.

Microprocessor based data acquisition systems can sample the power input and temperature evolution at specified time intervals at a resolution of up to 1 s.

The heat source for one-dimensional transient techniques can range from a simple coil or plate heater to expensive flash devices. In some cases the sample boundaries may have to be insulated against heat loss. Heat flash techniques require expensive flash sources such as laser or discharge tubes. The temperature sampling frequency must be at least once every second for good definition of the temperature peak. The temperature trace from the high-speed heat flash can be captured by a camera mounted on an oscilloscope, and the required instrumentation and data processors. These techniques have only recently been applied to testing of geologic materials.

6.3   SAMPLE SIZE AND TEST DURATION

The size and shape of the test specimen is governed by two factors:
1. The specimen should be large enough to be representative of the material being tested in terms of its grain size and texture;
2. The test method should accommodate samples small enough to be easily obtained without introducing sample boundary effects.

In general, steady-state methods require thin samples of relatively large diameter. The test specimen should be thick enough to include the largest size particle or conglomerate for measurements to be representative of the bulk properties. Thicker specimens require longer test duration to reach the steady-state, and therefore increase the potential for heat losses. The test setup and devices (as in the case of the guarded hot plate) are generally preset for one or two sample sizes. Testing of other sample sizes require major modifications to the test devices.

Transient methods (using a constant line source or point source) generally produce a small thermal perturbation in a localized zone around the heat source. Thus, smaller samples can be used for transient methods as compared to steady-state methods. The measurement durations in transient methods are kept short, with a maximum of 10 to 15 minutes, by minimizing contact resistance and heat capacity of probe. A further benefit of transient techniques is that any size or shape of samples, provided the minimum size requirement is met, can be used. Blackwell (1954) describes a method for determining the minimum sample size for the duration of heating involved in transient techniques. For temperature sensitive materials such as frozen solids, the transient methods can be optimized to avoid phase change effects. Transient techniques can also be used for testing materials at controlled ambient temperatures and pressures by making minor modifications to the test setup (van Loon *et al.*, 1993; Schilling, 1999).

One of the greatest advantages of the thermal probe technique is its suitability for both field and *in situ* testing (Radhakrishna *et al.*, 1981). If the material to be tested is naturally occurring and non-homogeneous (e.g. stratified deposits of soils and rocks) the transient methods offer the advantage of *in situ* testing of individual strata independently to obtain the parameters necessary for thermal modeling. Transient techniques, especially the line source methods, lend themselves to testing of anisotropic materials and assessment of the anisotropy in thermal parameters.

One-dimensional heat-pulse techniques, hot flash methods, and differential scanning calorimeters require only small, thin disc-like specimens, with thickness ranging from 5 to 15 mm. Since the amount of material tested is relatively small, they may not be suitable for coarse-grained or porous materials. Test durations are typically less than a few minutes. Newer flash sources and testing devises are being designed for measurements conducted on larger samples.

6.4   MASS TRANSFER AND PHASE CHANGE EFFECTS

In the case of porous materials, the thermal gradients set up by the application of heat during measurement of thermal properties can induce mass transfer in porous materials. This phenomenon has been well studied in saturated and partially saturated soils (Radhakrishna *et al.,* 1979; Hartley *et al.,* 1981). In the case of porous materials containing ice and/or water, there is potential for phase transition within the pores. Similarly in the case of materials containing gas (in free or dissolved form) within the pore space, there may be convective and/or diffusive flow in liquid and gas phases. The possibility of coupled mass transport processes occurring during thermal testing is a function of the induced thermal gradients and maximum temperatures, test duration, and the hydraulic properties of the test material (conductivity and diffusivity for the pore fluids). In the case of frozen soils, the ice content and ambient temperatures also become important.

The effects of test-induced mass transfer in test specimen are three fold:
1. The assumption that heat transfer is by pure conduction is violated;
2. The effects of convective heat transfer and latent heat can alter the measured thermal parameters;
3. The expected equilibrium conditions may not be reached in steady-state measurements.

Steady-state methods induce thermal gradients across the whole sample and require a long duration of heating to reach equilibrium conditions. They tend to be the most susceptible to mass transfer and phase change effects depending on the mean ambient temperature at which test is performed. Transient heat conduction methods require shorter duration of heating and induce only localized thermal gradients as compared to steady-state techniques. However, in the case of constant line source technique the temperature and thermal gradients adjacent to the heater may be high due to radial heat flow. Transient thermal probe measurements have been used to

characterize the thermal stability of soils by heating at different power levels over extended periods, and inducing moisture migration (Boggs *et al.*, 1980; Hartley *et al.*, 1981). In most cases, careful selection of power levels, test duration, and temperature sensor types can prevent the problem of mass transfer effects (Steinmanis, 1981). The choice of method would depend on the material being tested and the temperature range at which measurements are to be made.

7.0   SUMMARY

The following ranking of techniques for simultaneous measurement of thermal conductivity and diffusivity of relatively small geologic samples (less than 8 cm in diameter and 12 to 15 cm in length) is based on the foregoing review and assessment of available techniques for measuring the thermal properties of solids:

1.   The transient technique employing a single probe with a constant-heat source is by far the most commonly used method for measuring thermal properties of particulates, soils (frozen and unfrozen), marine sediments, and occasionally rocks.   The theoretical basis for thermal conductivity measurement is sound. The assumptions of the model can be easily fulfilled through careful design of the probe and by close adherence to the measurement procedures (Steinmanis, 1981).   Testing standards have been documented for the single probe method of thermal conductivity measurement of soils (ASTM Standard D 5334-92 and IEEE standard 442-1981). Thermal property analyzers specially designed for thermal probes typically exceed these standards. Determination of thermal diffusivity by the intercept method using a single probe is subject to a number of modeling errors, especially if the contact resistance cannot be estimated accurately.   Curve fitting methods such as TDFIT can improve the modeling accuracy. Steinmanis (1981) describes an alternative method for measuring both thermal conductivity and diffusivity with good accuracy using a single thermal probe. An initial thermal conductivity measurement is made using the thermal probe, followed by a diffusivity measurement on the same cylindrical sample by instantaneously changing its boundary temperature while using the probe as only a temperature sensor.   For rock cores and samples of cemented or frozen soils, pre-drilled holes for installing thermal probes should be in-filled with conducting material such as thermal epoxy or graphite paste.

2.   Dual thermal probes employing constant or pulsed heat sources offer a better method of simultaneously determining both thermal diffusivity and conductivity. The primary source of error is in determining the actual probe separation. It is not known if the dual-probes are available commercially as standard items, but can be obtained as a custom order. The dual-probes can be designed either as a single unit with two probes (one heater probe and another sensor probe) attached to a common head at a preset separation or as two probes installed separately at the required spacing. Single-unit dual-probes are more suited for particulate materials such as soil, into which the dual-probe can be pushed or around which the material can

be packed to a specified density. In this technique, the measured thermal diffusivity values are susceptible to error in the estimation of probe separation. Modeling errors in thermal diffusivity determination are relatively small compared to single probe systems. Total error in measured thermal diffusivity by the pulse technique can be in the order of 20%. Installing a dual-probe in rock cores and samples of cemented or frozen soils requires drilling of two fine holes close to each other. In the case of rock cores, it may be simpler to embed an electric wire heater and a sensor with heat conducting epoxy in two separate holes instead of using two probes. This will reduce the contact resistance and heat capacity of the heater probe.

3.   The pulsed point source has the advantage of requiring shorter length probes (one-half of the line source probe) to satisfy the minimum length to diameter ratio for the heater. The heater probe has a bead resistor in its tip with constant current flowing through it for the duration of the heat pulse. The sensor probe has a thermistor located in its tip. Larson (1988) used "Geoflo" (ground water flow meter with a center probe and multiple sensors around it) by modifying it for measuring thermal properties of soils. Calculation procedures are similar to those used for the pulsed line source. Estimation of probe separation remains the major source of error, but with the shorter length of probes the degree of error may be reduced. Also the degree of difficulty of installation is reduced compared to dual-line source probes. Experience with this type of technique is limited, but appears promising.

4.   For small diameter rock core samples, simultaneous measurements of thermal conductivity and thermal diffusivity can be obtained by applying a heat pulse from a line source attached to the side of a cylindrical sample, and monitoring temperature across the core diameter. The advantage of this transient technique is that no drilling is required for installation of the heater and sensor. A wire heater can be embedded into a thin shallow saw cut along the length of the core and a thermocouple or thermistor can be embedded into the side of the core diametrically opposed to the heater wire. The computation procedure is complex, but can be readily accomplished within a standard spreadsheet program. The primary source of error in this technique is the heat loss from the surface of core. Jaeger and Sass (1964) have outlined a method of factoring in the estimated heat loss in the calculation of thermal parameters from the measured response. The test specimens can be covered with insulating material or buried in a box of insulation beads. Jaeger and Sass (1964) obtained satisfactory results on 35 mm diameter cores of dolerite by using an eight minute heat pulse.

5.   One-dimensional transient techniques generally are only suitable for the determination of thermal diffusivity, not thermal conductivity. They involve the application of either a constant heat flux or a heat pulse to one of the two parallel flat surfaces of the specimen, and subsequent recording of the temperature-time evolution at the other face (which could be either an adiabatic, or conducting boundary). Generally a radiant heat is applied from a non-contact heat source such as electric coil heater, discharge lamp, or laser flash. It is difficult to accurately measure the

applied heat flux and hence the thermal conductivity. These techniques are not suitable for field use and experience with these techniques on geologic materials is limited.

6.   Differential Scanning Calorimetry is an emerging technology for testing geologic materials in frozen and unfrozen condition. Its main advantage is that there are no modeling errors, since it is based on comparing the thermal performance of test sample with a reference sample of same shape and size. Only thermal diffusivity can be measured by this technique.

8.0   DESIGN OPTIONS AND RECOMMENDATIONS

The most versatile configurations for simultaneous measurement of thermal conductivity and diffusivity of solids employ a line source with either constant rate of heat input or short duration heat pulse. There is considerable study and experience behind these techniques. For the determination of thermal diffusivity, the dual-probe configuration employing a heat pulse has less modeling error than the single probe, but extra care and precision is required for the installation of the probes and for the accurate determination of the probe separation. Calculation procedures are similar for line source or point source dual-probes. Dual probe configurations employing a point source may be advantageous because of their shorter length as compared to line source probes. Thermal probes of different sizes are available either as standard items or as custom orders. All of the instrumentation necessary for thermal probe based measurements is available as state-of-the art equipment. Thermal property analyzers designed especially for this purpose and suitable for both laboratory and field use are readily available commercially. For rock cores, a combination of the single probe method of thermal conductivity measurement and the Shannon and Wells (1947) method of thermal diffusivity measurement on the same sample may also be a suitable option.

The Geological Survey of Canada has considerable experience with measurement of the thermal properties of frozen earth materials (particularly thermal conductivity measurement), in which heat and mass flux characteristics are very complex, and for which the possibility of phase change of water during testing must also be considered. This complexity dictates that great care be taken during the course of such measurements, and also indicates that measurement results should be carefully evaluated in the context of the uncertainties identified above. Where thermal properties measurements are to be conducted on porous materials containing gas hydrates (as well as water and/or ice/and or air), one should appreciate that the geothermal factors influencing the measurement are even more complex. Thus there is a need for additional laboratory work to explore the sensitivity of different measurement techniques to the various physical and thermal factors influencing the measurement result.

Furthermore, where it is desirable to track the progress of gas hydrate formation/dissociation for industrial processes, available techniques for measuring the thermal properties of solids and/or

porous media are unlikely to be suitable. This is particularly so for measurement of the thermal properties of water/hydrate slurries which are flowed and/or continuously agitated. However, if the purpose of tracking changes in thermal properties is to quantify the progression of gas hydrate formation/dissociation associated with an industrial process, it is likely that a more reliable result can be obtained with non-thermal techniques. The 2000-2001 GSC Report to IAE indicated that measurement of the bulk dielectric properties of a porous medium containing gas hydrate can accurately track the progression of gas hydrate formation/dissociation within the medium. It is very likely that this technique can be successfully applied to tracking of gas hydrate growth in flowed or agitated slurries. The GSC recommends investigation of this possibility in future work.

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Product Information

1. Thermal Probes and Thermal Property Analyzer, Geotherm Inc.
2. Thermal Probe, Thermal Sensors and Heat Flux Sensors, Hukseflux
3. Specific Heat (Dual Probe) Sensors, Thermal Logic
4. Portable Thermal Property Sensor, Decagon Devices, Inc.
5. Quickline-30 thermal Property Analyzer, Antar Corporation
6. Flash line 300- Thermal Diffusivity System, Antar Corporation
7. Differential Scanning Calorimetry, Netzsch Group

APPENDIX

Temperature Sensors

A variety of temperature sensors are available for monitoring temperature in solids and liquids. The choice of sensor depends upon the application, the temperature range of interest, the required accuracy and the environment to which it is exposed.

Thermocouples**:**
A variety of these bi-metal wires are available to cover a full range of temperature measurements. The voltage output of this bi-metal junction is nearly proportional to the temperature, which can be read directly with a multi-meter by using a reference ice point. The most commonly used thermocouple type is type "T" (copper-constantan). It has very good resolution and accuracy for low range temperature measurement. For higher temperature range, type "J" (iron-constantan) or type "K" (chromel-allumel) are used. Thermocouples are fairly inexpensive and easy to use and can be read directly with a hand-held reader or can be hooked-up to automated data transmission and acquisition systems. The main advantages are its simplicity, versatility and low cost. Various types of insulation, jacket and wire gauge make them ideal for use in harsh environment. However, poor resolution and accuracy may not render them not well suited for applications where temperature measurements are applied to compute other parameters such as conductivity, diffusivity, etc. Another drawback is the effect of stray currents and corrosion that will affect their performance in terms of accuracy as well as resolution. For long lead lengths (in excess of a few hundred meters), the output signal has to be amplified to make up for the line losses.

Platinum Resistance Thermometers**:**
The principle of platinum resistance thermometers is that their electrical resistance changes as a function temperature. The measurement devices send in a small amount of current and measure the corresponding voltage. The devices are very precise and accurate, almost the industry standard. The main drawback is their size. They are too large to be incorporated easily into probes or other similar devices containing heater element.

Thin Film Detectors (TFD**):**
As the name implies, these devices look like thin films consisting of an electric circuitry. With an excitation voltage, the measured output is proportional to the temperature. Although they are fairly accurate and easy to use, the size and configuration make them somewhat cumbersome to incorporate into probes or other similar devices containing heater element.

Integrated Circuit Temperature Transducers (ICTT):

These are monolithic transducer chips, somewhat similar to the thin film detectors except they are relatively small in size (about 1.5 mm x 3.0 mm x 5.0 mm). When energized with a small voltage, the output current is 1 milli-amp per K over a wide range of temperature. This linear behavior makes them easy to use and to configure automation. The size is still not small enough to incorporate them in probes, etc.

Thermistors:

Thermistors are very small (0.3 mm to 2 mm diameter) beads made-up of various oxides that have negative coefficients of resistance change with temperature (exponential). They are available in various resistance values (2250 Ohms to a few hundred kOhms at 25°C) and for various temperature ranges (-80°C to 250°C). This makes them ideal for very high-resolution read-out of temperature. Over the past two decades, their quality, reliability, interchangeability and stability has improved to very high levels. They can be easily configured into measurement devices by using simple bridge networks. Their small size, high resolution and good accuracy make them the preferred sensors to be incorporated into thermal conductivity probes. They do have some self-heating effect if they are excited continuously and with a relatively high voltage.