

Why it's better to have options.

C-Therm Technologies Ltd.

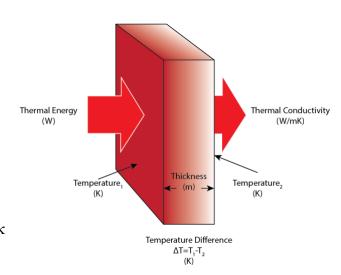


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What is Thermal Conductivity?

Thermal conductivity is described as the rate at which heat transfers through a material with a given temperature gradient. The thermal conductivity of a material is a measure of its ability to conduct heat. It is commonly denoted by k, λ , κ



Thermal Conductivity Properties: Putting Theory into Practice

The thermal conductivity of a material determines its suitability for engineering and specific applicationsⁱ.

While the thermal conductivities of pure materials are mostly known and can be accessed using standard reference sources such as NISTⁱⁱ and the CRC Handbookⁱⁱ, most technologically useful materials are not pure materials. Rather, they are alloys, compounds, polymers, composites, and mixtures that may exist in different physical forms such as solid, powder, paste, thin film, liquid or slurry.

Often, the thermophysical properties of materials in these different physical and chemical forms are not well-characterized. Consider the measured thermal conductivity of alumina ceramic materials: this property can have values that vary from an effective thermal conductivity of 0.12 W/mK for high-porosity alumina foamsⁱⁱⁱ to 30-40 W/mK for high-grade alumina ceramics^{iv}.

That is why in many engineering and research activities it is important to have an accessible analytical tool that provides rapid, accurate and precise determinations of relevant thermal conductivity of materials in the physical state and conditions of use for a given application. This is particularly important when dealing with materials that aren't well understood.

Some of the most common application areas where accurate thermal conductivity data is necessary include:

- Automotive
 Batteries
 Bio-Medical
- Building Materials
 Composites
 Electric Vehicles (EV)
- Explosives
 Geological
 Heat Transfer Fluids
- Insulation
 LED Lighting
 Metal Alloys
- Nanomaterials
 Oil & Gas
 Phase Change Materials
- Polymers
 Thermal Interface
 Metal Powders
 Materials

The Fundamentals of Thermal Conductivity Measurement Methods

Thermal conductivity can be measured using either steady-state or transient methods. Steady-state methods require the entire sample to be in a thermal steady-state during the measurement. Transient methods do not require a steady-state during the measurement and instead emit a momentary heat pulse. In both methods, the sample must be in thermal equilibrium with its surroundings before beginning the measurement procedure. This means that steady-state methods can require much longer thermal stabilization times for completion of a single test, particularly when testing materials with low thermal conductivity and relatively high specific heat capacity, such as polymers and most insulation materials.

While steady-state approaches provide highly precise and accurate determinations of the thermal conductivity of a sample, the combination of very long test times along with other drawbacks has largely resulted in a shift over the past decade, at least for routine measurements, towards transient techniques.

Table 1 shows selected steady-state and transient methods for the determination of the thermal properties of materials.

Steady-State Methods	Transient Methods	
Guarded Hot Plate	Laser Flash Diffusivity	
Heat Flow Meter	Transient Plane Source	
Guarded-Comparative-Longitudinal Heat Flow Meter	Modified Transient Plane Source	
Cut Bar Method	Transient Line Source	
	3ω Omega	

Table 1. Selected steady-state and transient methods for the determination of thermal properties.

Steady-State Methods

The *Guarded Hot Plate Method* is one of the primary steady-state methods for thermal conductivity measurements (ASTM STP 879; ASTM C177-13) for thermal insulators. While highly accurate and widely considered the default standard in building materials, certain requirements of this method severely limit its broad application as a routine analytical technique. First, the sample must be at thermal equilibrium during the measurement. Combined with the fact that the test often requires very thick samples (2cm+), this can produce exceedingly long testing times. Inhomogeneities in a sample can prolong the stabilization time further, particularly with very high porosity materials such as aerogels. Acceptable measurement accuracy requires the sample to have a large ratio of area to thickness that is flat and parallel within limits defined in ISO 8302. This requirement results in very exacting sample preparation.

Hard, higher thermal conductivity materials must be instrumented separately with temperature sensors to help minimize contact resistances between surfaces and to provide representative specimen surface temperatures, again resulting in sample preparation requirements that are tedious, time-consuming, and technically challenging. Another steady-state method is the *Radial Heat Flow Method*^{v vi}(ASTM C335 and ISO 8497) which, like the *Guarded Hot Plate Method*, suffers from tedious and time-consuming sample preparation issues and long testing times to achieve thermal equilibration.

Note: In steady-state methodology, the method needs to be chosen carefully, as most test methods are restricted in terms of thermal conductivity range. Guarded hot plate is typically restricted to materials with thermal conductivity of less than 1 W/mK, for example, while guarded-comparative-longitudinal heat flow meter is not appropriate to materials with a thermal conductivity of less than 10 W/mK. The reason for this is due to the primary sources of error for each type of measurement: When testing thermal conductors, the primary source of error is thermal contact resistance, so the apparatus and sample are designed in a way to minimize contact resistance effects, through the use of contact agents and long, thin samples with narrow cross-sections so that the contact resistance is insignificant compared to the resistance of the sample itself. When testing thermal insulators, the primary source of error is due to parasitic heat losses – e.g., through the wall of the instrument, or through air convection on the edge of the sample. For insulators, a large cross-section of sample is used to minimize heat losses at the edge and guard heaters are typically employed to reduce heat losses to the edges of the instrument. For this reason, steady state methods are restrictive in terms of the valid thermal conductivity range, and to fully equip a lab with steady-state instrumentation for thermal conductivity, three or four different instruments may be needed.

Transient Methods

Transient methods have become increasingly popular over the past 25 years, as their accuracy has significantly improved, and their flexibility provides a much more dynamic capability. For routine laboratory testing and field evaluations they have surpassed steady state methods. In transient methods, the heat source is applied in a periodic manner or as a pulse rather than continuously, as in steady-state methods. Since the measured thermal effects are transient, these methods do not require long thermal stabilization times, and this means dramatically shorter test times for transient vs. steady-state methods (minutes vs. hours). Additionally, the extremely short test times and small temperature increases allow less opportunity for the interference of other heat-transfer mechanisms, such as radiation or convection, which simplifies the experimental design requirements for avoiding parasitic heat losses (in most cases). Short test times also mean that there is less opportunity for the redistribution of any volatile components within a sample, producing a measurement that is more representative of the material in its unaltered state. The equipment required for transient measurements is, in most cases, relatively small (however, 30 methods for thin films are complicated). This means that it can easily be designed to migrate from dedicated R&D labs to manufacturing environments as a QC tool.

Innovations in Thermal Conductivity Testing – An Overview of Transient Methods

C-Therm has pioneered the advancement of transient methods, building a reputation for world class thermal conductivity instruments. The C-Therm Trident Thermal Conductivity Instrument represents a new paradigm in measurement of the heat transfer property by delivering three of the most powerful transient methods in one instrument. It's better to have options.

Trident offers the precision of MTPS (modified transient plane source method), the flexibility of TPS (transient plane source method), and the robustness of TLS (transient line source needle method), all in one modular package (Figure 1). These options support a wide range of applications without sacrificing ease of use.



Figure 1. C-Therm Trident Thermal Conductivity Instrument.





Figure 2. A Modified Transient Plane Source Thermal Conductivity sensor.

MTPS: Modified Transient Plane Source Method

The Modified Transient Plane Source (MTPS) Method (ASTM D7984-16) is an enhancement of the Transient Plane Source Method that requires only a single-sided interface with the sample during the measurement of thermal properties. It employs a one-sided interfacial heater/sensor (Figure 2) surrounded by an integrated heated guard ring. The patented heater/sensor applies a transient heat pulse to the sample, as shown in Figure 3 below. The heated guard ring provides a thermal barrier that surrounds the heater/sensor and eliminates lateral heat transfer at the sensor/sample interface. The heater/sensor/guard ring assembly resides on a base of low thermal effusivity to ensure that nearly all the heat transferred in a measurement occurs between the heater/sensor/guard ring and the sample.

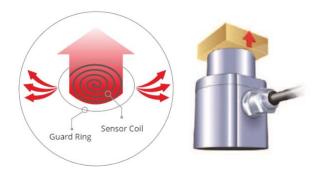


Figure 3. C-Therm MTPS Thermal Conductivity Sensor showing sensor assembly and specimen mounting.

In operation, the heater/sensor/guard ring assembly is placed in intimate contact with a sample, as illustrated in Figure 3 above. To ensure reproducibility in the contact between the sample and the heater/sensor and to minimize thermal contact resistance, a specific weight is typically placed on the sample. A momentary electrical current is simultaneously applied to both the spiral heater/sensor and the guard ring. The presence of an electrically heated guard

ring ensures that heat transfer between the heater/sensor spiral and the sample approximates one-dimensional heat flow. This specific incorporation of the guard ring in the design of the sensor is a significant innovation C-Therm developed for single-sided testing. No other vendor offers the patented guard ring technology.

The sensor temperature is factory calibrated using the relationship:

$$R(T) = R_0 + A \cdot T \tag{17}$$

and R(T) is the resistance of the sensor at a given temperature, R_0 is the resistance of the sensor at 0°C, T is the temperature and A is the slope of the graph of resistance vs. temperature. The slope is equal to:

$$A = R_0 \cdot TCR \tag{18}$$

where TCR is the temperature coefficient of resistance of the sensor. TCR is constant over the measured temperature range and the sensor is calibrated to determine the TCR within this range. Once the MTPS sensor has undergone TCR calibration, the instantaneous surface temperature of the sensor is then:

$$T = \frac{R(T) - R_0}{A} \tag{19}$$

The MTPS measures the sensor resistance directly:

$$R = \frac{V_0}{I} \tag{20}$$

or calculates it from the initial voltage, V₀ or power:

$$R = \frac{V_0^2}{P}$$
 (21)

When in contact with sample material, the short current pulse is applied to the heater/sensor to raise its temperature by 1 to 3°C. The rate at which the temperature at the heater/sample interface climbs during the current pulse is proportional to both the input power and the thermal losses due to conduction away from the interface by the sensor substrate and the sample.

Knowledge of the instantaneous temperature of the sensor allows the determination of the thermal characteristics of a material in contact with the sensor. The heat equation for the sensor in contact with the sample and with a constant supply of heat per second per volume, G', is given by:

$$\rho C_p \frac{\partial T}{\partial t} = \kappa \frac{\partial^2 T}{\partial x^2} + G' \tag{22}$$

Assuming that two semi-infinite media are in contact with the heat generated at the interface and that one medium is represented by the effusivity, e₁, of the sensor and that the other, e₂, is the tested sample material (Figure 4).

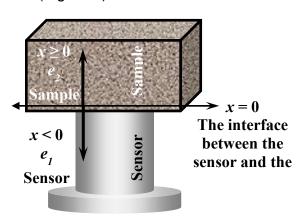


Figure 4.

Then, when temperature of both media at the interface is identical and in thermal equilibrium after contact has been established, the solutions to (31) for the sensor and sample are:

(Sensor)
$$\Delta T_1(x,t) = \frac{2G\sqrt{t}}{e_1 + e_2} ierfc \frac{|x|}{2\sqrt{a_1 \cdot t}} for x < 0, t > 0$$
 (23)

(Sample)
$$\Delta T_2(x,t) = \frac{2G\sqrt{t}}{e_1 + e_2} ierfc \frac{|x|}{2\sqrt{a_2 \cdot t}} for x \ge 0, t > 0$$
 (24)

where: ΔT = the change in sensor surface temperature (°C);

G =the power flux supplied to the sensor (W/m²);

t = time measured from the start of the process (sec);

 e_1 = equivalent effusivity of the sensor (= $\sqrt{\kappa_1 \cdot C_{p1} \cdot \rho_1}, \frac{W\sqrt{s}}{m^2 K}$);

e₂ = effusivity of the sample material (= $\sqrt{\kappa_2 \cdot C_{p2} \cdot \rho_2}$, $\frac{W\sqrt{s}}{m^2K}$);

 a_1 = equivalent diffusivity of the sensor, m^2/s ;

 a_2 = diffusivity of the sample material, m^2/s ;

 κ_1 = equivalent thermal conductivity of the sensor, *W/mK*;

 κ_2 = thermal conductivity of the sample material, W/mK;

 ρ_1 = equivalent density of the sensor, kg/m^3 ;

 ρ_2 = density of the sample material, kg/m^3 ;

 Cp_1 = equivalent heat capacity of the sensor, J/kgK;

 Cp_2 = heat capacity of the sample material, J/kgK;

ierfc = integral of the error function

If no thermal contact resistance exists at the interface between the sensor and the sample, then $T_1(x=0, t) = T_2(x=0, t)$ at all points having x=0 and Equations (23) and (24) become:

$$\Delta T(x=0,t) = \frac{2G\sqrt{t}}{e_1 + e_2} \cdot 0.5642 = \frac{1.1284G\sqrt{t}}{e_1 + e_2}$$
 (25)

From Equation (17), the change in resistance of the sensor at time, t, is:

$$\Delta R(t) = R(t) - R(t=0) = A \cdot \Delta T(x=0,t)$$
 (26)

And, from V=IR and Equation (26), the voltage change at time, t, is:

$$\Delta V(t) = I \cdot \Delta R(t) = I \cdot A \cdot \Delta T(x = 0, t)$$

$$= m\sqrt{t} = \frac{1.1284I \cdot A \cdot G\sqrt{t}}{e_1 + e_2}$$
 (27)

where
$$m=rac{1.1284I\cdot A\cdot G}{e_1+e_2}.$$

Figure 5 shows a plot of voltage change vs. the square root of time. Once the system has stabilized, the graph shows a linear response with slope *m*. The inverse of the slope, *m*, can be written:

$$\frac{1}{m} = \frac{e_1 + e_2}{1.1284l \cdot A \cdot G} = \frac{e_1}{1.1284l \cdot A \cdot G} + \frac{e_2}{1.1284l \cdot A \cdot G}$$
 (28)

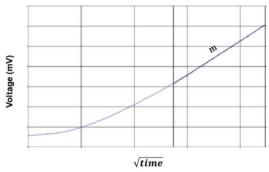


Figure 5. MTPS sensor voltage response vs. the square root of

If we consider a measurement in which the "sample" is a vacuum, then e_2 is the effusivity of vacuum, $e_2 = 0$, and Equation (28) reduces to:

$$\frac{1}{m}(vacuum) = \frac{e_1}{1.1284i \cdot A \cdot G}$$
 (29)

Values of the voltage/time slope, *m*, for a series of calibration materials with known effusivities can be determined experimentally using the MTPS sensor and a plot of 1/m for these materials is linear as shown in Figure 6.

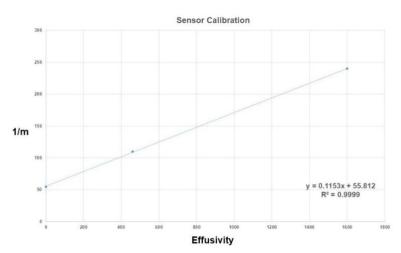


Figure 6. 1/m sensor effusivity calibration curve.

The slope of the effusivity calibration curve is *M* and the equation of the calibration line is:

$$\frac{1}{m} = M \times e_2 + C \tag{30}$$

where e_2 is the effusivity of the sample, C is the 1/m value of the sensor for vacuum, and the slope M is equal to:

$$M = \frac{1}{1.1284I \cdot A \cdot G} \tag{31}$$

Since:

$$C = intercept = \frac{1}{m}(vacuum) = \frac{e_1}{1.1284l \cdot A \cdot G}$$
 (32)

then the equivalent effusivity of the sensor, e_1 , is given by:

$$\frac{c}{M} = \left(\frac{e_1}{1.1284I \cdot A \cdot G}\right) \times \left(\frac{1.1284I \cdot A \cdot G}{1}\right) = e_1$$
 (33)

Thus, the effusivity calibration curve allows the determination of effusivity values for both the sensor and the sample. Knowledge of the slope of the voltage vs sqrt (time) response of the MTPS sensor provides 1/m values for unknown samples. So long as those 1/m value of the unknown material lies within the range of the 1/m calibration curve determined using calibration materials of known effusivity, the effusivity of the unknown sample, e_2 , can be determined from the graph in Figure 6 and Equation (30).

Two methods may be used to determine the thermal conductivity of a test sample. When the density, ρ , and specific heat, C_p , of the test sample are known, the thermal conductivity, κ , can be calculated using the effusivity, determined as described above, and the relationship:

$$\kappa = \frac{\varepsilon_2^2}{\rho \cdot C_n} \tag{34}$$

More commonly the density and specific heat of the sample are unknown, and the thermal conductivity is determined using a graphical method similar to that used for the determination of effusivity. The same 1/m data determined for the measurement of sample effusivity is used as input to a unique algorithm (the m^* algorithm vii) that employs an iterative process and the thermal conductivity of known materials to create a sensor calibration curve for thermal conductivity. The thermal conductivity calibration relationship has the form:

$$\frac{1}{m-m_*} = Slope \cdot k + Intercept \tag{35}$$

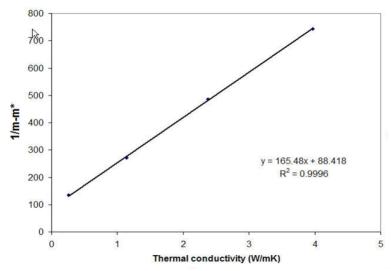


Figure 7. MTPS thermal effusivity and conductivity calibration plots.

$$\kappa = \left[\frac{\frac{1}{m-m^*} - Intercept}{Slope} \right]$$
 (36)

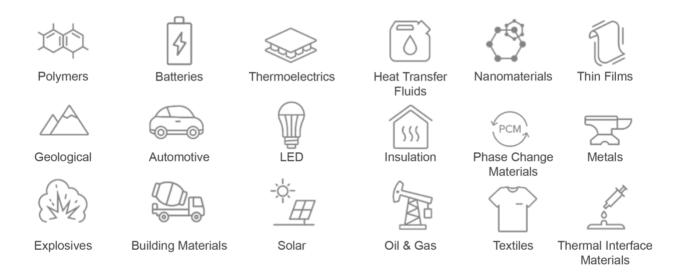
where: m is the value of the slope determined for the voltage vs. \sqrt{time} curve of the sample, shown in Figure 5, "Slope" is the slope and "Intercept" is the intercept of the $\frac{1}{(m-m*)}$ vs. thermal conductivity calibration curve shown in Figure 7, and m^* is a calibration factor that linearizes the measured 1/m with the known conductivities of the calibration materials as shown in Figure 7.

In the procedure for thermal conductivity calibration, values for m are determined for a series of calibration standards of known thermal conductivity that bracket the expected value of the test sample. The values for m and thermal conductivity of the calibration standards are then used as input to an iterative linear regression to solve Equation (36). m^* is treated as a variable fitting parameter during the regression with the value iteratively adjusted to provide the best least squares fit of the relationship to the data.

Maintaining high accuracy and precision across a broad range of thermal conductivity and effusivity using the MTPS technique requires that the sensor calibration is sub-divided into ranges that bracket the expected sample value, and the power and timing parameters must be optimized for a given group of materials. For example, longer test time and lower power settings are optimal for achieving a deeper depth of penetration when measuring insulation materials like foams. Conversely, shorter test time and higher power settings are optimal for higher-conductivity metals where the pulse travels very quickly through the material and a shorter test time provides for more flexibility in limiting the thickness requirements of the sample. Each group of materials has its calibration curve.

One thing to note is that the above discussion illustrates the theory of operation of the Modified Transient Plane Source method. However, on the user experience side, all MTPS sensors are factory calibrated. No user calibration is required, and the sensors are 'plug and play'. The MTPS sensor can measure thermal conductivity of materials in seconds, fast and easy to use. The user interface provides a tabular output of thermal conductivity, effusivity and temperature. The factory-provided calibration results in the easiest method for a user to obtain accurate and consistent thermal conductivity data.

MTPS Key Applications:



MTPS Application Highlight: Evaluating Thermal Performance of Advanced Materials

Thermal energy management is an important design criterion when designing new buildings and renovating existing structures. Recently, thermal energy storage within these building materials used in a structure has been gaining increasing interest and many new candidate materials are being evaluated for this purpose. One class of materials that have demonstrated promising characteristics in this application is phase change materials (PCMs). PCMs store latent heat energy not only as sensible heat in the thermal mass of the material but also in the latent heat associated with phase changes. This means that significantly less material is required to sequester thermal energy for heating and cooling the structure.

One recent study by Lee et al^{viii} provides a good representative example of the use of the MTPS method in quantifying the thermal energy storage characteristics of new materials. This study examined the thermal storage behavior of shape-stabilized PCMs (SSPCMs) of

exfoliated graphite nanoplatelets (xGnP) impregnated with fatty acid ester coconut oil and paraffin n-hexadecane. Fatty acid esters such as coconut oil are readily available, natural PCMs that are relatively inexpensive. Paraffins such as n-hexadecane are desirable in these applications owing to their large latent heat capacity and stability. However, both materials suffer from the fact that they have inherently low thermal conductivities and dimensional stability which makes them unsuitable for use as structural PCMs. These problems can be potentially resolved by incorporating the ester and paraffin materials into a stable material matrix that has acceptable thermal conductivity. xGnP provides such a substrate. xGnPs are porous, nanostructured substrate materials with high thermal conductivities that can absorb relatively large quantities of organic PCM substances. If the thermal conductivity of xGnP/coconut oil/n-hexadecane SSPCMs can be shown to retain high thermal conductivity, it provides a key qualification for their use as structural thermal storage materials.

Lee's study prepared samples of xGnPs infused with different amounts of coconut oil and n-hexadecane using a vacuum impregnation process that ensured good penetration of the PCM material into the layered, porous structure of the xGnP. xGnP test samples were loaded with 70/30, 50/50, and 30/70 mixtures of coconut oil/n-hexadecane. The samples were analyzed using electron microscopy (SEM), infrared spectroscopy (FTIR), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and MTPS thermal conductivity analysis. SEM analyses of the impregnated xGnP samples containing different loadings of PCM confirmed the saturation by the PCMs and that the impregnation process caused no significant change in the xGnP structure. FTIR analyses confirmed that the coconut oil and n-hexadecane were stable on absorption by the xGnP samples, with no indication of chemical changes apparent in the spectra. Individual peak intensities in the sample spectra were consistent with the different relative loadings of coconut oil and n-hexadecane. These analyses showed that the physical and chemical nature of the substrate and PCMs did not undergo significant change when the SSPCM was formed, a necessary qualification for use in structural thermal storage applications.

DSC analyses showed that the SSPCMs containing a 30/70 mixture of coconut oil/n-hexadecane exhibited a 30 percent increase in latent heat capacity over xGnPs with similar loadings of either of the PCMs alone. TGA analyses showed all mixtures of coconut oil/n-hexadecane in nGnP substrates to be thermally stable at the temperatures that might be encountered in structural thermal storage applications.

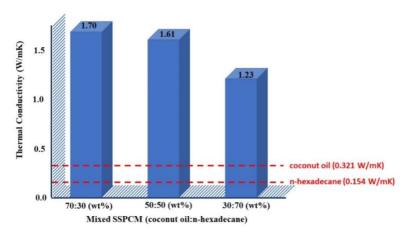


Figure 8. Thermal conductivities of mixed SSPCMs.

The thermal conductivity analysis used C-Therm's MTPS method (sensor) to determine thermal conductivities for all xGnP/coconut oil/n-hexadecane samples. The MTPS sensor was preferred for this study owing to the small sample requirements and the fact that it was necessary to determine the thermal conductivity of materials having physical properties that made them unsuitable for measurement using the TPS or TLS methods. The thermal conductivities of pure coconut oil and n-hexadecane were also determined using the MTPS method. Pure coconut oil had a thermal conductivity of 0.321 W/mK and pure n-hexadecane had a thermal conductivity of 0.154 W/mK. All tests were performed at 10°C and the results are shown in Figure 8. The thermal conductivity analyses using MTPS showed that impregnating xGnP with mixtures of coconut oil and n-hexadecane produced an SSPCM with thermal conductivity much superior to that of either coconut oil or n-hexadecane alone. Comparison of xGnP containing different mixture ratios of coconut oil to n-hexadecane showed that those with greater coconut oil concentrations had the highest thermal conductivity.

This study shows how the MTPS method, along with other physical, chemical and characterization tools, can provide data for the physical and thermal properties of advanced structural materials. This data is required as a design tool for the energy-efficient buildings that must be developed for a future that supports sustainable living.

TPS: Transient Plane Source Method



Figure 9. A Transient Plane Source sensor.

The Transient Plane Source Method (TPS), ISO 22007-2, ix uses a heated disc composed of a bifilar nickel spiral (contains two closely spaced spiral windings) encapsulated in a dielectric material such as Kapton® (Figure 10). During a measurement, the sensor is positioned between two identical samples of the material being measured as shown; a pulse of constant electrical power (up to minutes in duration) is applied to the spiral heater and the increase in temperature in the spiral is measured. The spiral in the sensor serves as both the source of heat and a dynamic temperature sensor during a measurement. The sensor and bridge circuitry is shown in Figure 10, including the DC power supply, V standard resistance, Re,s, and measurement points for the sensor voltage drop (V2) and the voltage drop across the standard resistor (V1).

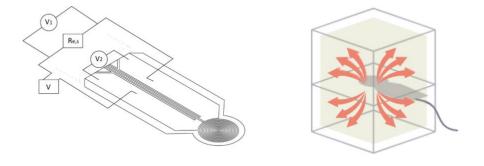


Figure 10. Left: The TPS probe configuration showing bridge circuit, contacts and bifilar spiral sensor; Right: Sensor positioning between sample pieces [19].

The sensor temperature response is factory calibrated using the temperature coefficient of resistance, TCR, of the nickel spiral. TCR relates to the change in metal resistance with a change in temperature. The use of this relationship, as employed in this application is described in ISO Standard 22007-2^x and TCR is defined in the British Standards Institute's BS EN 60751 Standard^{xi}. BS EN 60751 defines a quadratic or cubic model for the

temperature-resistance characteristics of a metal. For example, in the case of a platinum resistor, the temperature-resistance characteristic has the following form:

$$R(T) = R_0[1 + A \cdot T + BT^2]$$
 for $0 < T < 850$ °C (3)

$$R_T = R_0 [1 + AT + BT^2 + C(T - 100)T^3]$$
 for -200 < T < 0°C (4)

Where R(T) is the resistance of the metal at a given temperature, R0 is the resistance of metal at 0°C, T is the temperature, and A, B, and C are constants.

The current and time of the electrical pulse are calibrated to cause an expected temperature rise of one to several degrees in the sensor, based on the power delivered and the known TCR of the nickel spiral. As the temperature rises in the sensor, there is a corresponding increase in its resistance, and this produces a measurable change in the voltage drop over the sensor. To retain accuracy in the operational model of the TPS measurement, the duration of the measurement must be short for the thermal diffusivity of the sample. This allows the approximation that the sensor can be considered as being in contact with an infinite solid. Under this assumption, the sensor temperature is influenced only by the power input and the thermal transport properties of the solid in contact with the sensor, and not by any boundary effects.

The behavior of the TPS sensor during the transient pulse can be understood in terms of its time-dependent resistance, described by the equation (for small increases in temperature ΔT):

$$R(t) = R_0 \left[1 + a \overline{\Delta T(t)} \right] \tag{5}$$

where R(t) is the time-dependent resistance, R0 the initial resistance of the TPS element before the transient pulse, the TCR of the sensor element, and $\Delta T(t)$ is the mean value of the time-dependent temperature increase in the TPS sensor element. The temperature increase is dependent on the supplied power, the geometry of the sensor and the properties of the sample. It can also be expressed as $\Delta T(\tau)$ where the variable τ is a non-dimensional characteristic time parameter and is defined as:

$$\tau = \left(\frac{t}{\theta}\right)^{1/2}, \qquad \theta = \frac{r^2}{\alpha} \tag{6}$$

Where t is the time as measured from the start of the transient pulse, r the radius of the sensor spiral (largest ring) and α is the thermal diffusivity of the sample material.

In the case of a spiral sensor element (other geometries are possible), the time-dependent temperature increase of the sensor (Figure 11) is given by:

$$\overline{\Delta T(\tau)} = \frac{P}{\pi^{3/2} r_{\kappa}} D(\tau) \tag{7}$$

Where $\overline{\Delta T(\tau)}$ is the mean temperature rise of the sensor, P is the input power to the sensor, r is the sensor radius, κ is the thermal conductivity of the sample, and D(τ) is a shape function specific to the sensor geometry and depends on the thermal diffusivity of the sample α . The mean temperature the function can be modified to take into account the thermal resistance between the sample and the sensor:

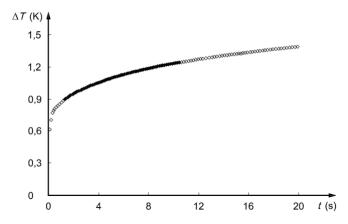


Figure 11. A typical temperature rise vs. time curve for a TPS measurement

$$\Delta T(\tau) = A + \frac{P}{\pi^{3/2} r \kappa} D \left[\left(\frac{\alpha (t - t_c)}{r^2} \right)^{1/2} \right]$$
 (8)

where A is the temperature drop on the total thermal resistance between the sensor and the sample, α is the thermal diffusivity and it is a time correction that takes into account thermal inertia of the sensor and various delays in the electronics of the apparatus. Equation (8) is true if and only if the thermal resistances on both sides of the sensor are equal, resulting in steady and equal heat flux to each side.

The TPS method can be applied in different measurement modes. These are Isotropic Bulk Mode, Anisotropic Bulk Mode, and Slab Mode. In all measurement modes, the setup must be symmetrical on both sides of the sensor.

TPS Isotropic and Anisotropic Bulk Measurement Mode: In the Isotropic Bulk Mode, the sensor is in contact with two identical isotropic samples such that their size in all directions is larger than the penetration depth of the heatwave over the entire measurement time. A bulk sample thus approximates an "infinite" sample size. The shape function for the isotropic bulk mode, $D(\tau)$, models the sensor as concentric ring sources and has the form:

$$D(\tau) = \frac{1}{n^2(n+1)^2} \times \int_0^{\tau} \frac{ds}{s^2} \sum_{l=1}^n l \sum_{k=1}^n k \, exp\left(-\frac{l^2 + k^2}{4n^2 s^2}\right) \, I_0\left(\frac{lk}{2n^2 s^2}\right)$$
(9)

where n is the number of concentric rings, I0 the modified Bessel function of the zeroth order and s is the integration variable, I and k are summation variables. In practice, Equations (5) and (6) are combined and the resulting equation is used, along with temperature measurements over a suitable time window. A data fitting procedure such as least squares regression is used that allows the determination of both the thermal diffusivity, α, and the thermal conductivity of the sample, κ .

The anisotropic bulk mode is limited to orthotropic samples, i.e. samples in which thermal transport properties are identical in all directions parallel to sensor surface but have different values vertical to the sensor surface. In this case, Equation (7) becomes

$$\overline{\Delta T(\tau)} = \frac{P}{\pi^{\frac{3}{2}} r \sqrt{\kappa_r \kappa_z}} D(\tau) \tag{10}$$

where κ_r is the thermal conductivity parallel to the sensor surface and κ_z is the thermal conductivity orthogonal to the sensor surface, and $D(\tau)$ is calculated with the thermal diffusivity in parallel to the sensor surface, α_r . Instead of Equation (6), Equation (11) is used for θ and τ .

$$\tau = \left(\frac{t}{\theta}\right)^{1/2}, \qquad \theta = \frac{r^2}{\alpha_r} \tag{11}$$

It is important to be aware of the assumptions inherent in the TPS method. It assumes that the sample provides an infinite medium for thermal transfer. In practical terms, this means that, ideally, a sample needs to be 2-3 times the sensor diameter in width and length and at least the sample diameter in thickness. The model treats the spiral sensor as concentric rings, an approximation that imposes some error in the measurement which increases as r decreases. The method assumes a complete symmetry on both sides of the sensor, meaning that the sensor insulation layers on both sides are identical and that any parasitic thermal contact resistance must be identical on both sides. While this condition may never be completely valid, applying a clamping force or weight on the sensor-sample system minimizes the resulting error. As well, the assumption that a sample's thermal properties don't change during measurement imposes a limit on how large of a temperature increase may be applied to the sample. These issues impose certain restrictions on the method:

- The method cannot be performed in an asymmetric sampling configuration since contact resistance affects the shape factor and the mathematical model has not yet been expanded to include contact resistance;
- Care must be taken in setup to ensure contact resistance is equal on both sides. This requires:
 - High pressure applied to both sides of the sample;

- Careful preparation of a smooth sample surface
 - Very small surface imperfections, if not symmetrical, can introduce very large errors into the measurement.
- Identical samples for multiple measurements
- Samples must be large enough to accommodate a valid test time. Practically, this
 means sample restrictions as follows:
 - Diameter: >5r, where r is the sensor radius
 - Thickness: >2r, where r is the sensor radius
- Test time must be chosen to be long enough that deviations from circular geometry do
 not affect the validity of the test result, but short enough that thermal diffusion effects
 are still significant in the shape factor
 - The test result is valid if the following inequality is satisfied:

$$1.1r < \sqrt{4\alpha t} < 2.0r$$

Where:

- r is sensor radius
- α is thermal diffusivity
- *t* is the maximum analysis time boundary

TPS Isotropic Slab Measurement Mode: The Isotropic Slab Mode is used when the approximation of an "infinite" sample size is no longer valid due to a restriction in the thickness of the sample. In this mode, thin sample slabs, or plates, can be used. The size of the samples must be adequately large ("infinite") in the directions parallel to the sensor surface, but relatively thin normal to the sensor surface. The external slab surfaces must be adequately insulated against heat loss, or alternatively, the measurement should be conducted in a vacuum. For high thermal diffusivity materials, this is the preferred method as it improves signal strength and decreases the sample size. As in the bulk mode, two identical samples are used.

In the Isotropic Slab Measurement Mode Equation (6) holds, but the shape function, $E(\tau)$ becomes:

$$E(\tau) = [n(n+1)]^{-2} \times \int_0^{\tau} s^{-2} \left[\sum_{l=1}^n l \sum_{k=1}^n k \, exp\left(\frac{-(l^2+k^2)}{4n^2s^2}\right) I_0\left(\frac{lk}{2n^2s^2}\right) \right] \left\{ 1 + 2\sum_{l=1}^{\infty} exp\left[\frac{l^2}{s^2}\left(\frac{h}{r}\right)^2\right] \right\} ds$$
 (12)

where n is the number of concentric rings, r is the sensor radius, h is the thickness of the two slabs, and s is the integration variable. One assumption made in deriving this function is that the material is isotropic – therefore this method cannot be used in measuring anisotropic materials.

Since thermal conductivities are obtained by fitting experimental data to the theoretical operating equations of the TPS method, the primary limitation of this method is that it is heavily dependent on the user's expertise and intuition for accurate results. This makes the technique prone to operator bias. The user must establish appropriate timing and power parameters and select sensors appropriate for specific types of measurements based on the expected thermal conductivity of the material. Lack of knowledge of the range of possible values for a sample's thermal conductivity requires multiple tests and can produce confusing results.

Limitations on test time, symmetry of test assembly, contact resistance, and sample diameter are identical for this method as for the bulk method. Thickness may be between 1mm and the lesser of the sensor radius or 10 mm.

TPS Key Applications:



Polymers



Geological



Metals



Building Materials

TPS Application Highlight: Electroceramic Materials

Repeatable electrical insulating characteristics are of obvious necessity in the ceramic insulating materials employed in electrical applications, but thermal properties are also critical. Manufacturers of electrically insulating components often use kaolin ceramics as the raw material and knowledge of the structural, mechanical and thermal properties of both the raw and fired ceramic is important. Recently, Krupa and Malinaric used the Transient Plane Source Method to measure the thermal conductivity of isostatic electroceramics at room temperaturexii. The authors followed ISO 22007-2 with the following modification: a constant,

A, was added to account for contact resistance between the sensor and sample. Provided the contact resistances on both sides of the sensor are identical, this does not affect the analysis.

The samples used in the study were made from weighed mixtures of the raw material that contained a precise ratio of clay, kaolin, feldspar, aluminum oxide, quartz and other materials that had been ball-milled to a fine powder. The dry mixture was isostatically compacted in a die-press and coupons were cut from the "green body" and precision-machined to a reproducible shape for firing. The samples were then fired at different temperatures using tightly controlled heating/cooling rates. Depending on the material mixture and firing conditions, different reactions, ranging from simple de-hydroxylation to chemical recombination were possible, producing fired samples with a variety of thermal, physical, chemical and electrical properties.

The authors used a 6.4 mm diameter sensor with 16 circular rings in a conventional TPS sampling configuration to evaluate the thermal conductivity of the fired sample coupons. They used a modified electrical bridge circuit that suppressed voltage source instability and removed the resistance of the sensor leads from the calculations. The authors employed the modified mean temperature function:

$$\overline{T(t)} = A + \frac{P}{\pi^{3/2} r \lambda} D \left[\left(\frac{\alpha (t - t_c)}{r^2} \right)^{\frac{1}{2}} \right]$$
 (13)

in which A corresponds to the temperature drop on the total thermal resistance between the sensor heater and the samples (i.e. including the sensor insulation layers) and time correction, etc, accommodates thermal inertia in the sensor and delays in the electronics.

Measurement uncertainty was evaluated using repeated measurements and by disassembling and reassembling the apparatus to evaluate any operator-dependent errors.

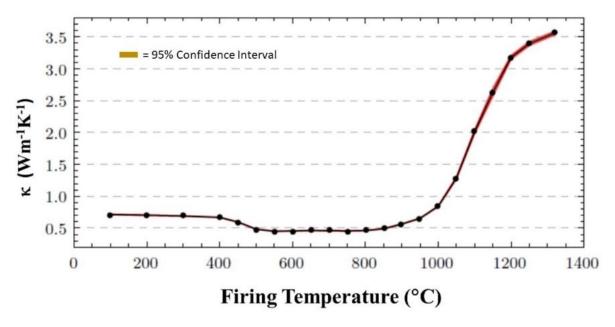


Figure 12. Thermal conductivity of the fired samples. (Source: Ref. [14])

Figure 12 shows the results of the thermal conductivity tests in this study. The shaded area surrounding the trace indicates the 95 per cent confidence interval. Measurement uncertainties were estimated as standard deviations resulting from repeated measurements.

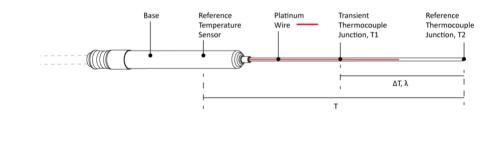
The TPS method used in this study provides a high degree of accuracy and precision in the measurement of the thermal conductivity of the fired electroceramic samples as is indicated by the narrow confidence interval in Figure 11. The confidence intervals further indicate that the firing procedure used in the study produced highly repeatable samples, at least in terms of their thermal conductivity. The thermal conductivity data for the electroceramic samples were found to be strongly dependent on the bulk density of the sample, which in turn depended on the firing conditions for the sample. This is consistent with simple physical considerations for thermal conductivity.

TLS: Transient Line Source Method



Figure 13. A Transient Line Source Probe.

The Transient Line Source Method (ASTM D5334, D5930, and IEEE Std 442-1981) uses a needle probe such as the one shown in Figure 13. During a measurement, the needle is inserted into a sample material such as a soft soil (ASTM D5334), plastic (D5930), or liquid that is at a uniform and constant initial temperature. A typical needle probe consists of a 150 mm long stainless-steel hypodermic tube as shown in Figure 13. Within the tube are the line heating source that provides a known amount of heat during the measurement and two thermoco2uple junctions positioned to measure the transient temperature rise (T₁) and reference temperatures (T₂) when the transient heat pulse is applied. In operation, a known amount of heat energy in the form of a heat pulse is applied to the line heating source (proportional to the known power supplied during the pulse) and this produces a heatwave that propagates radially away from the needle probe and into the sample. The thermocouple junction at T₁ measures the rate of increase of the temperature in the middle of the heating zone (Figure 14) and this is used to calculate the thermal conductivity of the sample.



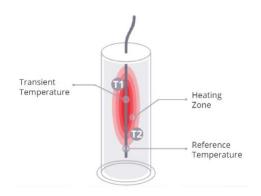


Figure 14. Typical needle probe components (Source: [15]).

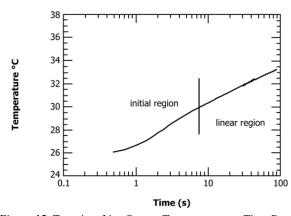


Figure 15. Transient Line Source Temperature vs. Time Response.

The temperature rise in the line source varies linearly with the logarithm of time as shown in Figure 15. The initial, non-linear portion of the curve in Figure 15, is due to heatwave propagation through the walls of the tube and to thermal contact resistance when it is present. The relationship between the rate of temperature rise and time in the linear region can be used to calculate the thermal conductivity of the sample using the relationship:

$$\kappa = \frac{cQ}{4\pi \cdot Slope} \tag{14}$$

where

$$Q = Heat input per length of wire (W/m) = I^{2} \frac{R}{L} = \frac{VI}{L}$$
 (15)

(I = Current, R = Wire Resistance, V = Voltage and L = Length of the heated wire)

and

$$Slope = \frac{T_2 - T_1}{\ln(^{t_2}/_{t_1})} K$$
 (16)

where T_2 and T_1 are temperatures recorded at times t_2 and t_1 , respectively, during the measurement. C is a calibration factor, $(C = \frac{\lambda_{material}}{\lambda_{measured}})$, determined by measurement of standard reference material. $\lambda_{material}$ is the known thermal conductivity of the reference material and $\lambda_{measured}$ is the thermal conductivity of the same material as measured by the method and apparatus being used.

As with TPS, it is important to maintain an awareness of the assumptions employed in TLS. The following assumptions are made in TLS measurements:

- 1. Thermal contact resistance is negligible;
 - a. This is only valid when the sample is symmetrical about the sensor.
- 2. The probe acts as a line source, with negligible heat capacity;
 - a. The sample volume must be much greater than the probe volume. For example,
 a 1mm diameter probe requires a 3cm sample diameter.
- 3. The probe does not significantly contribute to the heat-transfer of the system;
 - a. This is never valid; it is the reason that the system must be calibrated.
- 4. The sample is in thermal equilibrium before the start of measurement;
- 5. The probe (and heating wire inside) is exactly linear;
 - a. Deviations from linear geometry may severely affect the measurement accuracy.
- 6. Heat transfer occurs only through conduction;
 - a. TLS is inappropriate for measuring non-viscous fluids and other samples where convection or radiation may significantly contribute to the heat transfer.
- 7. The sample is isotropic;
 - a. TLS is not recommended for anisotropic samples.
- 8. Sample properties do not change during measurement;
 - a. e.g., samples such as moist soils can lose moisture during the measurement.

- 9. The sample is homogeneous;
- 10. The sample behaves as an infinite medium.

Fortunately, the T vs. t plot for TLS measurements indicates when certain of these assumptions are invalid in a sample measurement (6, 8, sometimes 9 and 10). For this reason, it is important in TLS measurements to carefully inspect the T vs. t plot.

TLS Key Applications:





TLS Application Highlight: Polymer Injection Molding

Injection molding (Figure 16) is the most commonly used manufacturing process for the fabrication of plastic parts. In an injection molding process, plastic is melted in the front end of the molding machine and forced into the mold using a screw. The plastic cools and solidifies into the final part in the mold section of the molding machine. The thermal conductivity of molten plastics is an important parameter in plastics processing since it affects temperature distribution and cooling behavior of the melt. Knowledge of the thermal conductivity of a polymer feedstock is important as it is used in developing reliable process simulations of the extrusion and injection of the plastic in the molding processes. Such simulations are used in



Figure 16. Plastic Injection Molding [18].

process control to achieve increased productivity and improved quality of the finished product.

Injection molding process development begins with the prediction of a polymer's melt behavior under the conditions expected in the injection mold machine. These predictions are accomplished using sophisticated rheological modelling that requires accurate knowledge of the thermophysical properties of the polymer. The absolute value and variability of the thermal conductivity of a polymer feedstock is a critical thermophysical parameter influencing process performance in injection molding. The thermal conductivity of the polymer as it passes through the transition from solid to melt and back dictates important process parameters such as the heating rate and cooling time needed to avoid undesirable flaws such as blistering, burn marks, warping or sink marks.

The injection molding industry has made the use of Transient Line Source (TLS) the industry standard for measuring the thermal conductivity of thermoplastics. Using the TLS sensor according to ASTM D5930 and a sample vessel such as the one shown figure 17, a powdered polymer can be melted in a bath or dry thermal chamber and its thermal conductivity measured as it passes through the phase transitions of melting and solidifying.

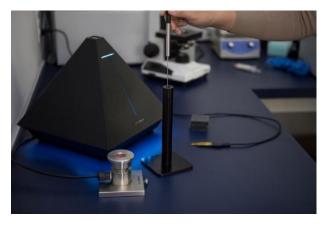


Figure 17. A TLS sensor and a sample vessel.

A typical polymer thermal conductivity measurement using the TLS is shown in Figure 18. The figure shows data for a sample of powdered polyamide 12 (Nylon-12), a thermoplastic material commonly used in injection molding. The sample had thermal conductivity measurements performed at 125°C, 150°C, and 200°C using a C-Therm Trident Thermal Conductivity Analyzer equipped with a Transient Line Source (TLS) sensor. As this application shows, C-Therm's TLS sensor provides researchers and manufacturing engineers in the polymer sector with a reliable, easy-to-use solution for measuring polymer melts.

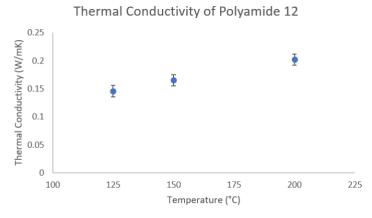


Figure 18. Thermal Conductivity Test Results of Polyamide 12.

Putting Trident to Work

Trident reflects C-Therm's years of experience working exclusively to provide labs, researchers and manufacturers, with the very best in thermal conductivity measurement.

	Modified Transient Plane Source MTPS GUARD RING TECHNOLOGY	Transient Plane Source FLEX FRANSIENT PLANSIENT	Transient Line Source NEEDLE TRANSIENT LINE SOURCE
Sample Preparation	None Sample simple rests on top of sensor. A few drops of contact agent may be required.	Moderate to Significant Samples (2) must be homogeneous with smoothed mating surfaces that sandwich the sensor.	None to Minimal Ranges from no preparation for simple soil probes to containment and heating for molten plastic samples.
Operator Training Requirements	Minimal	Significant	Minimal to Moderate
Non-Destructive	Yes	No	Sample Dependent
Method Development	Minimal	Significant	Minimal to Moderate
Material Phases	Solids, Liquids, Powders, Pastes	Solids, Liquids	Liquids, Powders, Pastes, Gels, Slurries, Melts
Testing Time	Seconds	Minutes	Minutes
Thermal Conductivity Range	0-500 W/mK	0-100W/mK	0.1-6 W/mK
Temperature Range	-50 - 200°C (500°C)	-100 - 700°C	-50 - 185°C

Table 2. A comparison of the characteristics of the MTPS, TPS, and TLS thermophysical analytical methods.

The simplest and quickest methods to implement and use are MTPS and TLS. These techniques require minimal to moderate sample preparation and operator training.

The MTPS method simply requires the operator to place the sample on the one-sided heater/sensor, add the supplied weight and "press go" on the analyzer. Analytical results for the sample are available within seconds of mounting it on the sensor. No sample preparation is needed, and an operator can be trained in minutes.

The use of TLS requires somewhat more operator training since, for example, powder and soil samples must be reproducibly packed into sample holders and the melting of samples for melt studies requires that heating and cooling characteristics are reproducible. Samples for TLS may also require some preparation to ensure reproducible and homogeneous samples. These requirements result in somewhat longer cycle times for TLS analyses as compared with MTPS.

The TPS method requires significantly more sample preparation and operator training than either the TLS or MTPS. The need for perfectly flat sample surfaces to "sandwich" the sensor adds to both operator training requirements and sample preparation time. TPS results can be highly dependent on the instrument operator.

The MTPS and TLS methods are the most broadly applicable in terms of sample types. Both methods can be easily configured to analyze liquids, powders and pastes. MTPS and TLS methods are complementary in that the MTPS probe is easily used with monolithic solids that are not readily probed by TLS, while TLS probes can conveniently determine dynamic thermophysical data for the melting behavior of plastic materials. The TPS method is generally more limited in the physical states that can be analyzed, restricted primarily to monolithic solids and liquid samples.

The choice of an analytical method is most often dictated by the expected value of the properties of interest of the sample under test and the physical conditions (i.e. temperature, pressure) for the material application.

In terms of thermal conductivity, the MTPS probe offers the widest analytical range, with a measurable thermal conductivity range of 0 to 500 W/mK under physical conditions that range in temperature from -50 to 500 °C and in pressure from vacuum to 135 atm (with appropriate testing accessories).

The TLS method is more limited in its thermal conductivity range with a maximum measurable value of 6 W/mK. Temperature limits for the TLS are also more restrictive with a maximum range of 200 °C.

The TPS method is limited to 100 W/mK in its thermal conductivity range when measuring uncharacterized samples. This range can be extended to 500 W/mK if the C_p and density of the sample is known. The TPS method is more broadly applicable for high-temperature analyses than either the MTPS or TLS method, having a maximum temperature range of 700 °C.

C-Therm software provides full data acquisition and analysis in one user interface, allowing full control of all three methods.

Trident: It's better to have options

Trident reflects C-Therm's years of experience working exclusively to provide labs, researchers and manufacturers, with the very best in thermal conductivity measurement. C-Therm products are globally distributed in over 65 countries around the world with full local support.

> Learn more at www.TridentThermalConductivity.com or contact info@ctherm.com

Glossary

density, ρ . The amount of mass per unit volume. In heat transfer problems, the density works with the specific heat to determine how much energy a body can store per unit increase in temperature. Its units are kg/m³.

heat flux, q. The rate of heat flowing past a reference datum. Its units are W/m².

specific heat, *c.* A material property that indicates the amount of energy a body stores for each degree increase in temperature, on a per unit mass basis. Its units are J/kg-K.

thermal conductivity, k. A material property that describes the rate at which heat flows within a body for a given temperature difference. Its units are W/m-k.

thermal diffusivity, α . A material property that describes the rate at which heat diffuses through a body. It is a function of the body's thermal conductivity and its specific heat. A high thermal conductivity will increase the body's thermal diffusivity, as heat will be able to conduct across the body quickly. Conversely, a high specific heat will lower the body's thermal diffusivity, since heat is preferentially stored as internal energy within the body instead of being conducted through it. Its units are m^2/s .

thermal effusivity, e. A material's thermal effusivity is a measure of its ability to exchange thermal energy with its surroundings. It is also referred to as "thermal inertia". Thermal effusivity is the square root of k, p (rho) and Cp, where p (rho) is density, Cp is the mass specific heat capacity at constant pressure, and k is the thermal conductivity. Thermal effusivity may be expressed equivalently in units of $Ws^{1/2}/m^2K$ or $J/s^{1/2}m^2K$.

thermal resistance, *R*. Thermal resistance is the temperature difference, at steady state, between two defined surfaces of a material or construction that induces a unit heat flow rate through a unit area, K·m²/W.

thermal conductance, *C.* Thermal conductance is the time rate of steady state heat flow through a unit area of a material or construction induced by a unit temperature difference between the body surfaces, in W/m²·K.

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