# TRANSIENT THERMAL CONDUCTIVITY MEASUREMENTS – COMPARISON OF DESTRUCTIVE AND NON-DESTRUCTIVE TECHNIQUES

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## Abstract

Transient thermal conductivity methods have benefited from the rapid nature of the testing, but at the same time these methods are destructive because a sample must be prepared. Laser flash diffusivity is an example of well established transient method of measuring thermal conductivity, which requires samples to be prepared to a diameter of 12.4 mm.

The newly introduced modified hot wire (MHW) and transient plane source (TPS) techniques are transient as well, but are nondestructive in nature. This is possible because both of these new techniques are reflectance methods that operate by applying and measuring heat at the same surface. Essentially any flat surface can be measured, with no maximum size constraints.

These transient techniques have been used to evaluate materials of differing size, structure and homogeneity. While results compare well for homogeneous materials from foams to ceramic; composite materials differ considerably. This is because the two methods penetrate the samples to different degrees, and the results are a reflection of the testing depth. As test times are varied in the reflectance techniques, information was gathered that has been related to the thermal conductivity as a function of position normal to the sample.
This paper will discuss the techniques and the comparison of results on homogeneous materials from two transient techniques.

## Keywords

thermal conductivity, transient technique, modified hot wire, non-destructive, material characterization, rapid, transient plane source, diffusivity, effusivity.

## Introduction

Thermal management is necessary for all areas of material processing and also in a growing number of end user applications. Products such as electronic assemblies, injection molding molds, automotive “under the hood” components and must transfer heat efficiently (Mathis 1999 a). Such products encounter cyclic loading and their ability to conduct heat must not deteriorate over the desired product life. To transfer heat rapidly, the materials used in these applications must have high thermal conductivity. Plastic can be a viable, light weight option for traditional metal products when it is compounded with conductive materials including glass, graphite or boron nitride.

A second family of products are designed with the opposite intention – to prevent heat transfer. Home insulation, fireman’s protective clothing, boiler liners and appliances represent a cross section of products that utilize materials designed for low thermal conductivity. New materials are developed for these markets in order to meet increasingly stringent energy or safety regulations. Foams and ceramics dominate these applications, with ceramics having the advantage for high temperature use.

Whether the goal is to expedite or retard heat transfer, the response and behavior of these products must be modeled and then measured. There are three modes of heat transfer: radiation, convection and conduction, and all require a driving force in the form of a temperature difference. Heat will flow from the hot surface to the cold surface. All modes of transfer occur in a given situation, but often one or two will have negligible contribution. The focus of this work will be the conduction mode.

Conduction occurs primarily through a solid material. Conduction is related to the materials thermal conductivity (k), the cross sectional area (A) for heat (Q) to flow through and the distance of travel (L) from the hot to cold side of the solid material. (Equation 1.)

$$Q = \frac{kA}{L}(T_{hot} - T_{cold}) \qquad \text{Equation 1}$$

When a material is being developed or produced for a thermal management application, it is the material properties that must be measured. Thermal conductivity, heat capacity and density are the primary properties of interest.

Thermal conductivity is a consideration at all levels when thermal engineers design for optimum heat dissipation, bonding and homogeneity. (Mathis 1999 b) Characterization of these new materials is crucial in the development cycle in order to optimize the production process. The same characterization is required on-line, to monitor the quality control during production.

There are two main categories of techniques available to measure thermal conductivity: transient and steady state. Both categories of measurement technique provide a temperature gradient and then monitor the response of the material to the gradient. Where the techniques differ include areas such as sample size, testing time, range and methodologies of measurement.

Steady state techniques, such as guarded hot plate described in ASTM C 518 -93 or the technique of ASTM D 5470 - 93, involve placing a solid sample of fixed dimension between two temperature controlled plates. One plate is heated while the other is cooled and temperatures of the plates are monitored until they are constant. The steady state temperatures, the thickness of the sample and the heat input to the hot plate are used to calculate the thermal conductivity from Equation 1. (See Figure 1)

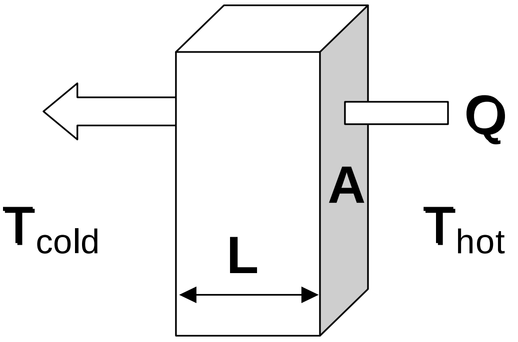

**Figure 1:** Experimental variables for steady state thermal conductivity tests

The difficulty with steady state techniques are that the measurement requires access to both sides of the sample, the sample must conform to specified dimensions and the test time is lengthy to allow steady state to be accomplished.

## Experimental Procedures

Transient techniques have gained popularity because of their speed. Measurements are conducted in seconds rather than minutes or hours. Two transient techniques have been established for a long enough period to have ASTM standards associated with them. They are described below.

***Hot wire*** (ASTM C 1113) is a transient technique that involves inserting an electrically heated wire into a material. This intrusive method is possible for foams and fluids and melted plastics. In some cases a hole can be drilled into the sample to allow insertion of the wire. The heat flows out radially from the wire into the sample and the temperature of the wire is measured. (See Figure 2) The plot of the wire temperature versus the logarithm of time is used to calculate thermal conductivity, provided that density and heat capacity are known. Testing can be conducted at a wide range of temperatures.

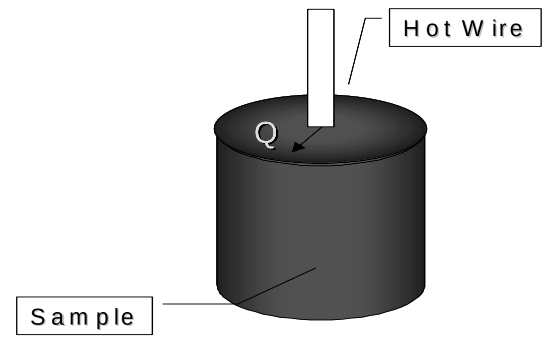

**Figure 2**: Experimental setup for Hot Wire experiments.

***Laser flash diffusivity*** (ASTM E 1491 - 92) is a transient method that involves applying a short pulse of heat to the front face of a specimen using a laser flash, and measuring the temperature change of the rear face with an infrared (IR) detector. (See Figure 3) The resulting temperature rise of the other face of the test specimen is monitored as a function of time and the half time to full temperature rise is used to determine the thermal diffusivity. (Equation 2a) This can be combined with density and heat capacity data to calculate thermal conductivity.

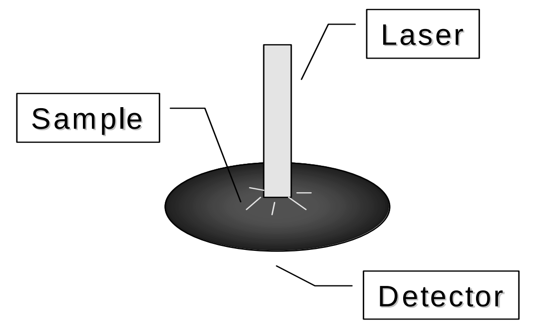

**Figure 3:** Experimental diagram for Laser Flash Diffusivity testing.

Both of these methods are considered destructive if the samples to be studied are solids. In the case of Hot Wire, the sample must either be melted or have a hole drilled into it. It is primarily a method for measuring liquids. Laser flash samples must be prepared to dimensions of 12.4 mm in diameter, thus destroying the solid product from which the sample was extracted. While the testing time was orders of magnitude faster than steady state methods, the sample preparation time was just as rigorous.

New techniques have been developed recently in order to address the needs of both research and quality control facilities that required the ability to test various sizes of samples non-destructively. ASTM standards are not available for these techniques at the date of this publication.

***Modified hot wire*** (MHW) is a transient heat reflectance technique similar to hot wire (Mathis 1998). The modification is that the heating element is supported on a backing, which provides a rectangular one-dimensional heat flow. (See Figure 4)

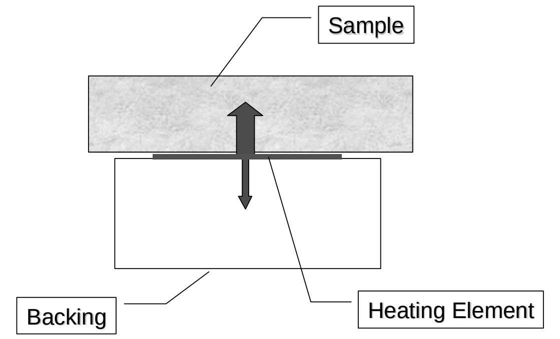

**Figure 4:** Experimental setup for Modified Hot Wire technique.

This eliminates the intrusive nature and allows for solids to be tested without being melted. The heating element relies on interfacial contact with a solid flat sample having minimum dimensions of 5 mm by 25 mm. The heating element is operated under constant current conditions and temperature of the heating element can be monitored during sample testing to measure temperature rise over time. The temperature rise of the element is inversely proportional to the ability of the sample to transfer the heat. From this relationship and calibration with characterised standard materials, the value of effusivity (sometimes referred to as thermal inertia) is measured. (See Equation 2b) From effusivity, thermal conductivity can be calculated by dividing by density and heat capacity.

If density and heat capacity are not available, alternative analysis methods are available to obtain thermal conductivity and/or thermal conductivity ratios from the MHW technique. (Mathis 1999 c)

$$Effusivity = \sqrt{k \rho c_p} \qquad \text{Equation 2a}$$

$$Diffusivity = \frac{k}{\rho c_p} \qquad \text{Equation 2b}$$

$Where:$

$k = thermal\ conductivity\ (W / m \cdot K)$

$\rho = density\ (kg / m^3)$

$c_p = heat\ capacity\ (J / kg \cdot K)$

***Transient plane source*** *(TPS)* is another transient heat reflectance technique similar to hot wire (Gustafsson et al ,1981-93). The heating element is encapsulated in a protective coating but is not supported on a backing. A three dimensional heat flow emanates from the circular heating element. (See Figure 5) The heating element relies on interfacial contact with two identical solid flat samples having minimum dimensions of 3 mm in diameter. As with MHW, the heating element is operated under constant current conditions and temperature of the heating element can be monitored during sample testing to measure temperature rise over time. The resultant data is curve fitted and iterated to determine thermal conductivity, diffusivity and volumetric heat capacity without the need for calibration. This technique can be used for solids, liquids and powders.

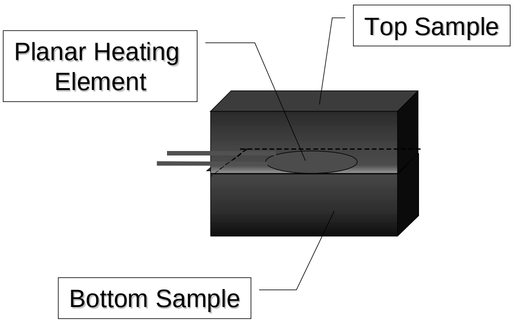

**Figure 5:** Experimental set up for Transient Plane Source testing.

Both hot wire and transient plane source techniques are interfacial; heat is applied and detected on the same side of the material. This facet of operation allowed for the system to be brought on-line for testing thermal properties during production. All other instrumentation for thermal conductivity measurement operates under principles that require that heat input at one side of a material to be detected on the opposite side. The drawback to interfacial/reflectance type testing is that the sample can not be completely penetrated during a test. If penetration does occur, the air on the other side of the sample would be factored into the test results produced by the instrument, thus providing a false representation of the thermal property value. To avoid this occurrence during testing, the techniques have methods and procedures to test films under 0.5 mm thick (Samuels 1999).

These instrument options are summarized in the chart below, along with some key benefits. Computer data acquisition, manipulation and display are standard.

**Table 1**: Comparison of Benefits and Features

| Benefits | Hot Wire | Laser Flash | MHW | *TPS* |
|---|---|---|---|---|
| No maximum sample size | | | ✓ | ✓ |
| Small sample size (< 250 mm$^2$) | | ✓ | ✓ | ✓ |
| Minimal sample preparation | ✓ | | ✓ | ✓ |
| Rapid testing (< 1 min) | | ✓ | ✓ | ✓ |
| Homogeneity testing method | | | ✓ | ✓ |
| Finished product testing possible | | | ✓ | ✓ |
| On-line QC ability | | | ✓ | ✓ |
| Thermal conductivity (k) directly | ✓ | | | ✓ |
| High k testing (> 10 W/m·K) | ✓ | ✓ | | ✓ |
| Low k testing ( < 0.05 W/m·K) | ✓ | | ✓ | ✓ |
| Range of testing temperatures | ✓ | ✓ | | ✓ |
| Liquid testing capability | ✓ | ✓ | ✓ | ✓ |
| ASTM compliant | ✓ | ✓ | | |
| No sample contact required | | ✓ | | |
| No calibration required | ✓ | | | ✓ |
| Complete unit ≤ $25,000 | | | ✓ | |

## Results

Testing was conducted on a series of homogeneous solid samples with different amounts of conductive alumina filler in a continuous matrix. The University of New Mexico supplied these samples and prepared them as 12.4 mm disks appropriate for laser flash diffusivity testing. After the samples were evaluated by laser flash, the same samples were evaluated by transient plane source technique.

The results of testing are given below as Figure 6. Both techniques illustrated that the thermal conductivity increased with level of filler. The sample with 30% fill gave a lower level of thermal conductivity than expected by both methods, showing that the two techniques are linearly related. This trend is more clearly indicated in Figure 7 that plots the results of thermal conductivity testing as a function of method and not fill level. A regression of the results plotted in Figure 7 give a regression coefficient of 0.99 when the intercept is set at zero. The slope of 0.8085 indicates that there is between the two techniques, with laser flash producing thermal conductivities consistently 20% higher than TPS.

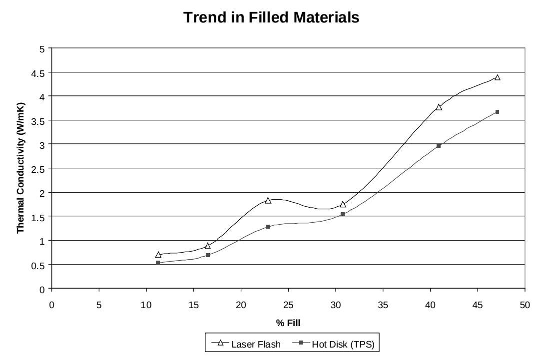

**Figure 6**: Thermal conductivity increased with percent fill of alumina.

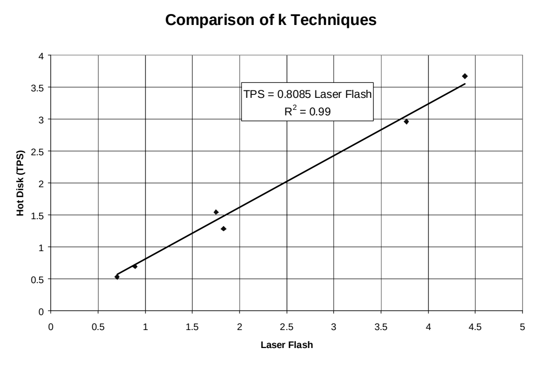

**Figure 7:** Results from the two techniques are show a linear relationship but with a 20% bias.

## Discussion

The bias between the two techniques may be related one or more of the following potential causes: sample surface, use of external data, outer boundary effects or sample non-homogeneity. All precautions were taken to avoid these errors, but they will be mentioned below to provide an understanding of what to take into account when testing using these techniques.

*Sample surface:* In order to prevent light scattering from the sample surface during laser flash testing, gold was sputtered onto the sample. This thin layer of gold is then incorporated into the testing and may cause a higher measured thermal conductivity –During the TPS measurement surface contact is required where it is not in laser flash measurements. If the surface contact were questionable, a low result would result from TPS measurements. Both of these are consistent with the trend in this work.

*External data*: A second reason may come from the fact that laser flash diffusivity relies on density and heat capacity measurements in order to calculate thermal conductivity from diffusivity. If either of these measurements had systematic errors that reproduced through the entire series, it could produce the type of bias that was found.

*Boundary effects:* A third reason for bias is that the samples may have experienced surface effects during TPS testing. If the heat wave penetrated to the far side or edges during testing, the thermal conductivity would have had a low bias as found in this study.

*Sample non-homogeneity*: If the samples being tested had a higher concentration of fillers in the center as compared to the edges, then laser flash results would have been higher because the method evaluated the entire thickness. TPS only evaluates a certain depth into the sample that is related to the test time and the sample properties.

## Conclusion

Transient techniques are a rapid method of evaluating thermal conductivity. Several options exist for instrument configurations that address sample specifications and testing environment requirements.

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