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ABSTRACT

This review is focused on the development of experimental methods for measuring thermal transport properties. Special emphasis is given to plane probes, which are used both as heat sources and as temperature recording devices. With such probes it is under certain conditions possible to determine both the thermal conductivity and the thermal diffusivity from a single transient recording. The conditions which must be fulfilled in an experiment are introduced by the use of the concept of a thermal probing depth, and from which the time window of observations can be defined. The ultimate goal of the dual determination of the thermal transport properties of a substrate is to reveal the anisotropy of the material from which the substrate should be a representative sample.

Keywords: thermal transport properties, transient method, thermal probing depth, anisotropy.

1. INTRODUCTION

Currently, a large number of experimental methods are available for measuring thermal transport properties such as thermal conductivity, thermal diffusivity, and specific heat per unit volume. Beside the traditional methods using non-varying temperature distributions like the Heat Flow Meter (International Standard: ISO 8301, 1991), the Guarded Hot Plate (ISO 8302, 1991), and Split Bar (Andersson, 1966), a series of experimental methods have, in recent decades, been developed which can be referred to as transient methods, namely the Laser Flash (International Standard: ISO 22007-4, 2008; Parker, Jenkins, Butler, & Abbot, 1961), Hot Wire (International Standard: ISO 8894-1, 2010), Monotonic Heating (ASTM E2584-07, n.d.; Maglic, Cezairlian, & Peletsky, 1989; Litovskiy, Issoupow, Horodetsky, & Kleiman, 2013), Hot Strip (Gustafsson, Karawacki, & Khan, 1979), Hot Disc (International Standard: ISO 22007-2, 2008; Gustafsson, 1991), 3ω Method (Cahill & Pohl, 1987), Temperature Wave (International Standard: ISO 22007-3, 2008), and Thermal Reflectance (Taketoshi, Baba, & Ono, 2001), etc. With the transient methods, a surface area of the sample material is heated and the temperature increase in one or several points or over a certain area of the sample is recorded. From this information one or several transport properties are then derived.

The transient methods can be classified in different ways. One would be to look at the number of transport properties that can be obtained from a transient recording, and the other to divide them into absolute methods and methods used as a means of interpolation. Looking at commercially available methods and their scientific basis (published papers or standardization documents) it is obvious that only a transport property (thermal conductivity, thermal diffusivity, or thermal effusivity) can be obtained from a transient recording with an Interpolating Method. However, the situation is different for the Absolute Methods in the sense that with some of these methods a transport property can be obtained, whereas from a few of them two thermal transport properties can be acquired from a single transient recording.

1.1 Interpolating method

When working with an Interpolating Method, it is possible to decide which transport property can be obtained from a transient recording. However, it is only possible to acquire a single property from a recording with such a method. In addition, prior calibration of the sensing element by the experimenter or by the supplier of the instrument is required. It must be remembered that as soon as the sensing element is placed in a slightly different environment (temperature, pressure, etc.) a new calibration of the sensing element is necessary. It is also important to keep in mind that it is easier to perform a good interpolation measurement if the calibration materials have properties close to the property of the material under study. Because of this, it is necessary to have a series of materials available against which calibrations can be performed.

1.2 Absolute method

From a transient recording with an Absolute Method, it is possible to calculate the transport properties if
the following information is available: (a) a recorded temperature increase, (b) the power supplied to the sample and (c) a length dimension typical of the method. This means that it is not necessary to calibrate an instrument based on these principles. One of the most celebrated methods for measuring thermal diffusivity, particularly of metals and other dense materials, is the Laser Flash method. By heating a surface with a short laser pulse of a small sample – typically with a diameter of 12 mm and a known thickness of a few millimeters – and recording the temperature increase on the other side of the single substrate, it is possible to calculate the thermal diffusivity. The reason for not directly calculating the thermal conductivity with the Laser Flash method is related to the fact that the amount of power transferred to the sample from the light source is unknown. Efforts are made to estimate the power by making comparisons with power transferred to similar materials with known optical properties, but this is not recommended or recognized by any international standardization body. The length dimension necessary for calculating the thermal diffusivity is the thickness of the sample.

Another versatile method for measuring thermal conductivity of liquids is the Hot Wire method. By suspending a thin wire vertically in a liquid and recording the temperature increase due to the electrical heating of the wire, it is possible to calculate the thermal conductivity from a single transient recording. The length dimension, required for calculating the thermal conductivity, is the length of the wire over which the temperature increase is measured. Claims are occasionally made that it is possible also to measure the thermal diffusivity with the Hot Wire method. Yet the problem is that this property has to be calculated from a correction term in the theory of the Hot Wire method containing the diameter of the wire as an important factor. Unfortunately, this diameter has to be kept small (~10 µm) to obtain a reasonable value of the electrical resistance of the wire.

There is a host of methods in which the temperature increase is measured at a distance from the heat source to obtain both thermal transport properties. In view of the large number of possibilities of designing transient methods there are established scientists who claim that every researcher in this field of thermal transport properties should develop his/her own method. The drawbacks of such a praxis, if adhered to, are of course limited by practical possibilities of independently comparing results, and difficulties in gaging whether results obtained in isolation are reasonable, unless Round Robin tests are organized or measurements are limited to materials, which have been standardized by reference laboratories.

There are, however, a limited number of methods, in which the heat source is unified with a temperature recording function. A typical example is the Hot Wire method. Specific advantages of these kinds of methods are: (a) the larger electrical heating current, which is used to determine the resistance of the sensing element, is actually providing a higher voltage variation and in that way boosting the sensitivity of the method and (b) there is no additional and disturbing sensing element introduced into the temperature field surrounding the heat source, which makes the experiment more ideal. There are essentially two experimental methods – the Hot Strip method (Gustafsson, Karawacki, & Chohan, 1986; Gustafsson et al., 1979) and the Hot Disc method (Gustafsson, 1991; Log & Gustafsson, 1995; Suleiman, Ul-Haq, Karawacki, Maqsood, & Gustafsson, 1993) – in which there is a unification of the heat source and the temperature recording, through which both the thermal conductivity and the thermal diffusivity and by this the specific heat per unit volume can be calculated.

The Hot Strip method is applied in the same way as the Hot Wire method with two essential differences: (a) when using a thin strip rather than a wire, it is easier to get good thermal contact between the heating element (the hot strip) and the sample, and for this reason it is possible to use the Hot Strip method also for measuring the thermal properties of solids; (b) the length and the width (typically in a ratio between 20 and 200) of the strip provides the basis for calculating both the thermal conductivity (strip length) and the thermal diffusivity (strip width) from a single transient recording. The Hot Disc method can be seen as a further development of the Hot Strip method with a view to easily measure the two transport properties of both solids and liquids from a single transient recording. In this case the sensing element consists of a bifilar spiral etched out of a thin metal sheet. The spiral with its electrical contacts is covered on both sides with a thin electrically insulating film. The possibility to obtain both the thermal transport properties from a single recording is dependent on the selection of the time window open for the transient recording.

This article will focus on the development of the Hot Strip and the Hot Disc as well as the Pulse Hot Strip methods and how these could and should properly be used. These methods have been developed to a stage, which allows experiments by which it is possible to:

(a) Obtain the true bulk properties of solid materials. This is an undertaking that has proved difficult and sometimes impossible to achieve with traditional steady state methods.

(b) Determine how deep into a studied material the properties are being measured by the introduction of the concept of thermal probing depth.

(c) Deduce both the thermal conductivity and the thermal diffusivity from a single transient
recording, and consequently to determine the anisotropy in the transport properties of the substrate material.

(d) Reduce the time scale of the transient recordings into the microsecond range and study the properties – including the anisotropy – of micrometer thin layers.

1.3 Optical method

As a historical note it can be mentioned that one of the first few papers on the measurement of thermal transport properties with plane sensors was published in 1967 (Gustafsson, 1967), and this demonstrated a way to study transparent liquids using water at room temperature as an example. The heating element was a rather wide metal foil, and the temperature increase in the liquid outside the foil was recorded with a wavefront-shearing interferometer close to the center of the heating element. Because the temperature was recorded at different distances from the heating element inside the liquid, it was possible to measure both the thermal conductivity and the thermal diffusivity. The width (12 and 60 mm) of the heating element (metal foil) furnished a very long optical path, which made the sensitivity of the arrangement such that the total temperature increase during an experiment could be limited to some 0.11K. This limitation in turn hindered the natural or thermal convection of disturbing the experiment until after ~20 seconds. Fringe patterns with a double image of the heating element were used for evaluating the experimental results. This method was subsequently employed for measurements of thermal transport properties in transparent molten salts, in particular molten alkali nitrates (Gustafsson, Halling, & Kjellander, 1968a, 1968b). The method could also be used to measure the properties of transparent solids.

2. HOT STRIP METHOD

Optical methods in combination with a wide heating element are probably the most ideal methods from a purely experimental point of view because the recording of the temperature can be made near the center of the heating element. A further asset with this setup is that the two end sections work as “guards” in relation to the heavy electrical leads, which supply power to the heating element. However the requirement of transparency is a severe limitation of the optical methods. This limitation is eliminated for the Hot Wire arrangement used specifically to measure the thermal conductivity of liquids. For comparatively small temperature increase, the electrical resistance of the wire can be expressed as:

\[ R = R_0 (1 + \alpha \Delta T) \]  

(1)

Here \( R \) is the resistance of the heating element, the temperature of which has been increased by \( \Delta T \) Kelvin. \( R_0 \) is the electrical resistance of the heating element (wire) before any heating has occurred and \( \alpha \) is the temperature coefficient of resistivity (TCR).

\[ \alpha = \frac{1}{R} \frac{dR}{dT} \]  

(2)

There is an obvious difficulty in applying the Hot Wire arrangement to studies of solids, and this was the prime motivation for developing the Hot Strip arrangement. Two reasons can be listed why Hot Strip measurements are to be preferred when studying solids:

1. There is a larger surface area between the heating element and the two sample pieces placed on both sides of the strip.
2. With a well-defined width of the strip both the thermal conductivity and the thermal diffusivity from a single transient recording may be determined, provided certain conditions are fulfilled (see in what follows).

Let us assume that we have a strip deposited on the surface of a homogeneous and isotropic substrate. If the strip has a length \( (2h) \), which is much longer than its width \( (2d) \), and the total output of power is \( P_0 \), then the average temperature increase of the strip is obtained as:

\[ \Delta T(\tau) = \frac{P_0}{2 \sqrt{\pi} \cdot h \cdot \lambda} \cdot f(\tau) \]  

(3)

Here

\[ f(\tau) = \tau^2 \cdot \text{erfc} \left( \frac{1}{\tau} \right) - \tau^2 \cdot (4\pi)^{1/2} \cdot \exp \left( -\frac{1}{\tau^2} \right) \cdot \left( \text{Ei} \left( -\frac{1}{\tau^2} \right) \right) \]  

(4)

with

\[ \tau = \left( \frac{t}{\theta} \right)^{1/2} \quad \text{and} \quad \theta = \frac{d^2}{\kappa} \]  

(5)

\( \lambda \) is the thermal conductivity and \( \kappa \) is the thermal diffusivity. If the hot strip is realized by a thin metal foil placed between two plane surfaces of two pieces of the sample material in question, the temperature increase becomes

\[ \Delta T(\tau) = \frac{P_0}{4 \sqrt{\pi} \cdot h \cdot \lambda} \cdot f(\tau) \]  

(6)

Whenever it is possible to perform an experiment and limit the \( \tau \)-value to <0.7, there is a very good approximation of the \( f(\tau) \) function (Gustafsson, Ahmed, Hamdani, & Maqsood, 1982) accordingly:

\[ f(\tau) = \tau - (4\pi)^{1/2} \cdot \tau^2 \]  

(7)

This approximation actually simplifies the evaluation of the transient recordings quite substantially. By making a
plot of the temperature increase as a function of square root of the time and then fitting a second-order polynomial to the experimental data points, it is possible to determine both the thermal conductivity and the thermal diffusivity from the coefficients of the second-order polynomial fit without having to initiate an iteration process.

2.1 Bulk properties

The temperature increase given in Equation (3) represents the temperature increase of the sample surface with a thin strip deposited on top and with the assumption that there is no thermal contact resistance between the strip and the surface. If we assume that a thin layer of material with low thermal conductivity is deposited on top of a sample surface under a Hot Strip, then a specific and well-defined thermal contact resistance can be obtained. An arrangement like this is necessary whenever the properties of an electrically conducting material (Gustavsson et al., 2006) are measured. To describe this situation consider the following equation:

\[
\Delta T(\tau) = \Delta T_i + \frac{P_0}{2 \cdot \sqrt{\pi \cdot h \cdot \lambda}} \cdot f(\tau) \quad (8)
\]

If the material separating the strip from the surface is thin – with a thickness of \( \delta \) and a thermal diffusivity of \( \kappa_i \) – it was shown in a communication at ITCC 24 (Gustavsson, Gustavsson, & Gustafsson, 1998) that for transients longer than say \( 4 \cdot \frac{\delta^2}{\kappa_i} \) the initial temperature increase \( \Delta T_i \) becomes a constant throughout the entire transient recording. The constancy was shown both experimentally and by numerical simulations. Considering the thin layer, the power \( (P_i) \) traversing the layer as well as its thickness \( (\delta) \) and its thermal conductivity \( (\lambda_i) \) are all constant during the transient, and hence:

\[
P_0 = 4 \cdot d \cdot h \cdot \lambda_i \cdot \frac{\Delta T_i}{\delta}
\]

or

\[
\lambda_i = P_0 \cdot \delta \cdot (4 \cdot d \cdot h \cdot \Delta T_i)^{-1} \quad (9)
\]

This way of determining the thermal conductivity of thin electrically insulating films – deposited on substrates with a substantially higher thermal conductivity – has subsequently been used successfully also by the 3\( \omega \) method. It should here be mentioned that possible thermal contact resistances between the Hot Strip and the thin film as well between the thin film and the underlying substrate might make the thermal conductivity value less reliable.

An important consequence of the behavior of a thin electrically insulating layer placed on top of a substrate and immediately under the Hot Strip, even to the effect that it is possible to determine its thermal conductivity, is that in this way it becomes possible to determine the temperature increase of the “first” solid surface of the substrate. Following on this fact and the possibility to experimentally eliminate any thermal contact resistances, as long as the latter can be modeled as thin insulating layers, the true bulk properties of a sample material can be obtained. Although perhaps not impossible when working with stationary methods, garnering such results involves a considerably more complex and time-consuming process.

2.2 Anisotropic properties

Because it is possible to determine both thermal transport coefficients from a transient recording, the Hot Strip method has turned out to be an excellent tool for determining the properties of anisotropic substrates. If we assume that the anisotropic material is of orthogonal symmetry, then the principal axes are perpendicular to each other and the hot strip is oriented along one of the axes – say the z-axis. The surface of the strip is then in the yz-plane and the x-axis is perpendicular to the strip surface. Following Carslaw and Jaeger, chapter 10 (Carslaw & Jaeger, 1971) yields:

\[
\Delta T(\tau_y) = \frac{P_0}{2 \cdot \sqrt{\pi \cdot h \cdot (\lambda_x \cdot \lambda_y)^2}} \cdot f(\tau_y) \quad (10)
\]

Here

\[
\tau_y = \left( \frac{t}{\theta} \right)^{\frac{1}{2}} \quad \text{and} \quad \theta = \frac{d^2}{\kappa_y}
\]

\( t \) is the time measured from the start of the current pulse, which initiates the transient recording, \( d \) is half the width of the strip and \( \kappa_y \) is the thermal diffusivity in the y-direction of the crystalline material. It has here been assumed that the material is homogeneous and anisotropic. In the most general case, strips are to be deposited in two or three different directions to obtain all the transport coefficients (Gustafsson, Karawacki, & Khan, 1981). Looking at a material with uniaxial structure and the properties along the x- and y-axes identical but different from those along the z-axis, a measurement with the strip along the z-axis would give the properties along the two axes with identical properties and consequently the specific heat per unit volume. With the strip oriented along the x-axis, one would get the properties along the y- and z-axis but one would not obtain the specific heat per unit volume. To get the full information of the properties of a uniaxial material, it is necessary to perform two transient recordings unless the specific heat is known from an independent measurement.

2.3 Thermal probing depth

In chapter 10 of Carslaw & Jaeger (1971), the authors discuss the concept of “mean square temperature
distribution of heat” in a transient experiment, indicating how far the heat has propagated into the substrate when it has been exposed to heat pulse of a certain duration. Based on this notion the concept of thermal probing depth ($\Delta p$) has been introduced (International Standard: ISO 22007-2, 2008), as follows:

$$\Delta p = \gamma \cdot (\kappa \cdot t)^{\frac{1}{2}} \quad (12)$$

This means that by selecting the length of a transient recording with a constant power pulse, how deep into the substrate the thermal properties are being probed is actually decided.

The question is then how the constant ($\gamma$) should be selected. It can be shown that this constant is related to the sensitivity of the experimental method. Imagine a plane heat source surrounded on both sides of equally thick slabs of the same material. Assume further that outside the substrate samples either a substance with zero thermal conductivity or a substrate with infinite thermal conductivity exists. By using the method of images it is possible to calculate to what extent the presence of the material outside the substrates will increase or decrease the temperature of the sensing heat source, if it is assumed that the thickness of the substrates is equal to the thermal probing depth ($\Delta p$). This means that for a given time the influence on the temperature increase from the properties of the material outside the substrate will be smaller for a large value of the constant ($\gamma$) and larger for a small value of the constant. With a typical over-all sensitivity of ~2% for the transient methods discussed here, an approximate value of the constant turns out to be ($\gamma = 2$) or:

$$\Delta p = 2 \cdot (\kappa \cdot t)^{\frac{1}{2}} \quad (13)$$

A further important consequence of the acceptance of the concept of thermal probing depth is, that it has become possible to rather precisely estimate to what depth the transport properties are being determined. With modern digital voltmeters, having sampling intervals in the milliseconds range, it is possible within a transient recording of a few seconds to collect several thousand data points. This opens up a possibility to actually probe a sample and see how constant its thermal properties are as a function of depth.

There are different ways to achieve such depth profiling. Suppose that data points in the time range from $t_a$ to $t_b$ with $t_a \ll t_b$ have been collected. The thermal conductivity using the data points from $t_a$ to $t_b$, with $a + c \leq k \leq b$ can be determined. The constant $c$ would define the first and shortest time window, from which the thermal conductivity would be calculated. In this way ever-increasing time windows will be arrived and the thermal conductivity could, for instance, be plotted as a function of the thermal probing depth using steadily increasing times numbered $\frac{a + c + k}{2}$.

A different way of analyzing the data from a profiling experiment would be to use time windows with a fixed number of data points. The calculations would then be made using the time windows from $t_a$ to $t_b$, with $a \leq k \leq (b - c)$. In this case one could decide to plot the thermal conductivity as a function of the thermal probing depth calculated using the times $t_b$.

A probing of the thermal properties as a function of depth is particularly recommended when looking for structural deviations in a specific material. During such experiments it is normally not possible to follow the conditions laid down for determining two transport coefficients from a single transient recording. Instead it is recommended to independently measure the specific heat per unit volume of the material under study and then use this value when following the structural variation in the sample. A variation in the structure is not likely to change the specific heat very much.

### 2.4 Thermal conductivity and diffusivity from a single transient recording

The most common arrangement in a transient experiment is to make a recording of the temperature increase versus time and from such a recording to derive a transport property. Typical examples of such experimental methods are: Laser Flash, Hot Wire, and Thermal Reflectance Methods. There are, however, some methods from which it is possible to derive both the thermal conductivity and the thermal diffusivity from a single transient recording (Gustafsson, 1991; Gustafsson et al., 1979). The theory behind this possibility involves the use of sensitivity coefficients $\beta_q$ defined accordingly:

$$\beta_q = q \frac{\partial [\Delta T(t)]}{\partial q} \quad (14)$$

Here $q$ is the thermal conductivity ($\lambda$), the thermal diffusivity ($\kappa$), or the volumetric specific heat capacity ($C$). $\Delta T(t)$ is the mean temperature increase of the probe. This means that different sensitivity coefficients are defined for thermal conductivity, the thermal diffusivity, and the specific heat per unit volume. It must here be remembered that the maximum sensitivity of one parameter does not necessarily coincide with that of another parameter, which means that the precision, with which the two parameters can be determined, is not necessarily the same. A typical pattern for the Hot Strip and the Hot Disc methods is that for both methods the thermal
conductivity can normally be determined with a precision which is somewhat higher than that of the thermal diffusivity. Some researchers claim that it is too difficult to get good precision when deriving two transport properties from a single transient. However, with modern power supplies and digital voltmeters in combination with a suitable electrical bridge circuit arrangement, it is possible to limit the experimental imprecision in both properties to a few percent.

Based on the theory of sensitivity coefficients it can be shown that the thermal probing depth of the experiment should be somewhere between half the strip width and the full strip width or values between 0.5 and 1.0. Longer-duration experiments do not necessarily improve the precision.

2.5 Electric circuit

A theoretical precondition for most transient measurements is that the electrical circuit is arranged in such a way that the output of power from the heat source is constant (Gustafsson, Karawacki, & Chohan, 1984). However, this is not strictly true if using the traditional circuit with a constant current \( I_0 \) through the heating/sensing element. In brief, the power increase at constant current \( \Delta P = \Delta R \cdot I_0^2 \) amounts to

\[
\Delta P = \Delta R \cdot I_0^2
\]  \hspace{1cm} (15)

From Equation (1) it is assumed that

\[
\Delta R = R_0 \cdot \alpha \cdot \Delta T
\]  \hspace{1cm} (16)

A typical and rather simple electrical circuit is displayed in Figure 1. It consists of a DC source \( V \), a constant resistance \( R_s \), and the heating/sensing element, the resistance of which is \( R = R_0 + \Delta R \). If we further assume that the voltage is constant, then:

\[
V = (R_s + R_0 + \Delta R) \cdot (I_0 - \Delta I)
\]  \hspace{1cm} (17)

and

\[
\frac{\Delta I}{I_0} = \frac{\Delta R}{R_s + R_0 + \Delta R}
\]  \hspace{1cm} (18)

The output of power in the heat source is:

\[
P_0 + \Delta P = (R_0 + \Delta R) \cdot (I_0 - \Delta I)^2
\]  \hspace{1cm} (19)

From these expressions it can be shown that

\[
\Delta P \equiv 0 \text{ provided } R_s = R_0 \text{ and } \Delta R \ll R_s + R_0
\]  \hspace{1cm} (20)

This means that it is preferable to use a simple electrical circuit with a constant driving voltage and a constant resistance in series with the heat source cum temperature sensor.

Because it is common to record the voltage increase across the heating/sensing element to measure the temperature increase, the following expression is obtained:

\[
U_0 + \Delta U = (R_0 + \Delta R) \cdot (I_0 - \Delta I)
\]  \hspace{1cm} (21)

Accepting the approximation by Equation (20) yields

\[
\frac{\Delta U}{U_0} = \alpha \cdot \Delta T \left[ \frac{R_s}{R_s + R_0} \right]
\]  \hspace{1cm} (22)

This is an expression commonly used for evaluating transient recordings.

3. HOT DISC METHOD

In most practical situations, samples are of a size between 10 and 100 mm, which makes it somewhat inconvenient to cut out strips of metal foils. These tend to have a very low electrical resistance because of the difficulty in working with foils with a thickness <10 µm. There are also specific constraints that must be considered when designing a hot strip heater/sensor. The length-to-width ratio should definitely be >20, and unless the sample is quite large it is difficult to reach reasonable thermal probing depths. It would also be desirable to have a handheld probe, which can be used repeatedly for many different samples. A probe, which meets these requirements, is the Hot Disc sensor (Gustafsson, 1991). This consists of a double spiral etched out of a thin metal film. Because the spiral design entails an inherently longer electrical path than that of a straight metal strip, it has been possible to increase the electrical resistance of the probe and at the same time to allow for larger thermal probing depth. The reason for the increased thermal probing depth is that it can be shown that the depth of probing is dependent on the total diameter of the spiral. The spiral and the electrical leads are kept together by thin electrically insulating layers on both sides of the heating and sensing metal spiral.

Two examples of Hot Disc sensors are shown in Figure 2. It has been demonstrated that such probes can be designed with diameters from 1 to 300 mm. This means that it is possible to measure the thermal transport properties of specimens with sizes ranging...
As indicated earlier, the temperature increase can be seen as consisting of two distinct parts: one part represents the temperature difference across the insulating layer of the probe and the other, the temperature increase of the specimen surface. This state of affairs can be expressed as:

$$\Delta T(t) = \Delta T_s(t) + \Delta T_i(t)$$ (23)

where $\Delta T_s(t)$ is the increase of the temperature over the insulating layers of the probe, and $\Delta T_i(t)$ is the increase of the temperature of the specimen surface. With the assumption that the bifilar sensor can be approximated by a number of concentric and equally spaced circular line sources, the solution of the thermal conductivity equation is given by:

$$\Delta T_s(t) = \frac{P_0}{\pi^2 \cdot r \cdot \lambda} \cdot D(t)$$ (24)

Here $P_0$ is the power output of the probe, $r$ is the radius of the outermost ring source, $\lambda$ is the thermal conductivity of the specimen material, $\tau$ is defined as earlier, but here the radius of the Hot Disc sensor replaces the half-width of the Hot Strip sensor. The dimensionless time function $D(t)$ is defined as:

$$D(\tau) = \sum_{m=1}^{\infty} \sum_{k=1}^{\infty} k \cdot \exp \left[ \frac{-l^2 + k^2}{4m^2\sigma^2} \right]$$

(25)

in which $m$ is the number of concentric ring sources, and $I_0$ is a modified Bessel function.

(Integration from $\tau = 0.02$ represents a small time correction to avoid the singularity at $D(0)$ — for $\tau$ values <0.02 a good approximation is $D(\tau) \approx \tau$. The temperature increase $\Delta T_i(t)$ becomes constant after a short-time interval provided the insulating layer is thin and the power output is constant. The time it takes to approach a constant value is determined by the relaxation time, which may be approximated as four times the thickness of the thin film squared and divided by the thermal diffusivity of the film material. The possibility to determine the thermal contact resistance experimentally (via an initially constant temperature difference) makes it possible — also in the case of the Hot Disc method — to determine the true bulk properties of the specimen material.

The calculation of thermal conductivity and diffusivity from Equations (24) and (25) starts with an iteration procedure using the diffusivity as the optimization variable. Through the iteration, a linear relationship between $\Delta T_i(t)$ and $D(\tau)$ is established — by a least-squares fitting procedure — and the diffusivity is obtained from the final step of the iteration calculation.

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**Figure 2.** Example of two Hot Disc sensors with different radii of the double spiral (2.0 mm left, and 6.4 mm right).

From a diameter of a few millimeters up to and beyond several hundred millimeters (the thickness of the substrates should be selected equal to the radius of the probe and the diameter of the substrates two times the spiral diameter (International Standard: ISO 22007-2a, 2008). The bifilar spiral is etched out of a ~10-µm thick metal foil and covered on both sides by thin (from 7 to 100 µm) electrically insulating film. The most common metals used as heating and sensing materials are nickel and molybdenum. These metals have a relatively high resistivity and also a good temperature coefficient of its electrical resistivity, which is important as the spiral is being used also for the temperature recording.

The selection of material for the thin insulating films is important particularly if the intention is to use the probe over a large temperature range. Polyimide, mica, aluminum nitride, and aluminum oxide have so far been used as insulating films, depending on the ultimate temperature of use. A recent development — initially for use at high temperatures — involves supporting the spiral and the leads with a thin layer only on one side and then attaching the uncovered spiral and the leads to a plane surface of the substrate with a thin layer of electrically insulating adhesive. After curing, the intention is to remove the insulating layer before performing the experiment. The removal can be arranged by prior heating or by chemical means. With an arrangement for keeping the thermal conductivity of the uncovered side much lower than that of the substrate, single-sided experiments can be performed.

Although there are different ways of solving the thermal conductivity equation to achieve a way to analyze the experimental data, the most commonly used solution so far is discussed here (Gustafsson, 1991; Yi, 2005a, 2005b).
Finally, $\lambda$ is determined from the slope of the straight line. The initial time window selected for the analysis may result in experimental points, which deviate from the straight line more than the general scattering. By removing such data points, a correct time window is obtained for the final analysis. A graph of residuals normally displays the deviating points very clearly. Such points normally appear at the beginning and at the end of the transient. Deviating points at the beginning of the transient regularly occur because of the thermal contact resistance between the sensing material of the probe and the sample surface, whereas those at the end of the transient arise because of the limited size of the sample. Based on the concept of sensitivity coefficients, it can be shown that the thermal probing depth as calculated from Equation (14) earlier should preferably be larger than the radius of the bifilar spiral but less than its diameter. This condition must be fulfilled, if the intention is to determine both the thermal conductivity and diffusivity from a single transient recording (Bohac, Gustavsson, Kubicar, & Gustafsson, 2000).

There are different arrangements for recording the temperature increase of the probe, which results in a resistance increase. The international standard ISO 22007-2 recommends the use of an electrical bridge circuit as depicted in Figure 3. During the transient the off-balance voltage is recorded as a function of time. In order to calculate the temperature increase from these voltage readings, the following relation applies:

$$\Delta T_s(\tau) = \left( R_s + R_l + R_0 \right) \cdot \Delta U(\tau) \cdot \left[ J_0 \cdot R_s - \Delta U(\tau) \right] \cdot (\alpha \cdot R_0)^{-1}$$  \hspace{1cm} (26)

Here the probe resistance is described by $R = R_s + \Delta R$, where $R_s$ is the initial resistance before the transient, and $\Delta R$ is the resistance increase during the transient heating. The inclusion of $R_s$ is for taking into account probe leads resistance, $R_s$ is the series resistance, and $\Delta U$ is the off-balance voltage created by the probe resistance increase $\Delta R$. The current $J_0$ is the initial current through the probe, and $\alpha$ is the TCR of the probe.

![Electrical bridge circuit for Hot Disc measurements as described by ISO 22007-2.](image)

Figure 3. Electrical bridge circuit for Hot Disc measurements as described by ISO 22007-2.

3.1 Slab substrates

A special application, which has been used for measuring the thermal transport properties of high conducting materials, is to arrange a setup with substrates in the form of thin slabs (Gustavsson, Karawacki, & Gustafsson, 1994). The sheet-formed specimens extend in two dimensions, but they must have a limited and well-defined thickness ranging from a few millimeters down to ~25 μm. Two such slabs of a material are clamped around a Hot Disc sensor and thermally insulated on the outer sides. The condition related to the thermal probing depth must be fulfilled in the plane of the probe but not in the through-thickness direction. This method has been used for measurements on materials with conductivities as high as 1500 W/m/K.

The thermal conductivity equation has then been solved using the mathematical “method of images” with the assumption that no heat loss occurs from the outer faces of the two specimen halves. The temperature increase can then be expressed as

$$\Delta T_s(\tau) = \frac{P_0}{\pi^2 \cdot r \cdot \lambda} \cdot E(\tau)$$  \hspace{1cm} (27)

Here

$$E(\tau) = \left[ m(m + 1) \right]^{-2} \int_{0.02}^{\tau} \sigma^{-2} \left[ \sum_{i=1}^{m} \sum_{k=1}^{m} k \cdot \exp \left\{ -\frac{(l^2 + k^2)}{4m^2\sigma^2} \right\} \cdot \left[ \frac{I_0 \left( \frac{lk}{2m^2\sigma^2} \right)}{1 + 2 \sum_{i=1}^{m} \exp \left\{ -\frac{i^2}{\sigma^2} \cdot \frac{h^2}{r^2} \right\} } \right] d\sigma \right.$$  \hspace{1cm} (28)

and $h$ is the thickness of each of the two slabs. For measurements with comparatively low conductivity it might be necessary to perform the experiments in vacuum, while still air and the use of three sharp points to support the sample pieces normally fulfills the condition, that the heat loss from the outer faces and the edges of the specimen is negligible.

3.2 Anisotropic substrates

It is also possible to study anisotropic materials with the Hot Disc method (Gustavsson, 2012; Gustavsson & Gustafsson, 2004; Keith, Hingst, Miller, King, & Hauser, 2006; Lundström, Karlsson, & Gustavsson, 2001; Miller et al., 2006). However, this method is limited to materials in which the thermal properties along two of the orthogonal and principal axes are the same, but are different from those along the third axis. This means that it is possible to study materials with a uniaxial structure and orthogonal axes such as layered structures, wood, fiber-reinforced materials, etc. It is important to remember that the surface of the probe must be oriented to coincide with the $xy$-plane in which the properties are the same but different from those in the $z$-plane. If the specific heat is available
from independent measurements, it is possible to obtain the thermal conductivity and the diffusivity in both directions from a single transient recording. The size of anisotropic specimens shall be chosen so that the requirements regarding the thermal probing depth are fulfilled along the principal axes.

If the properties along the x- and y-axes are the same, but different from those along the z-axis, the following expression for the temperature increase applies:

$$
\Delta T_x(t_x) = \frac{P_0}{\pi^2 \cdot r \cdot (\lambda_x \lambda_z)^{1/2}} \cdot D(t_x)
$$

(29)

where \( \lambda_x \) is the thermal conductivity along the x-axis, \( \lambda_z \) is the thermal conductivity along the z-axis,

$$
t_x = \left(\frac{t}{\theta}\right)^{1/2} \quad \text{and} \quad \theta = \frac{d^2}{\kappa_x}
$$

(30)

If the specific heat capacity per unit volume, \( C \), is known, then

$$
\lambda_x = C \cdot \kappa_x
$$

(31)

These equations demonstrate how to obtain the thermal transport properties along the two directions with the Hot Disc sensor.

### 4. PULSE HOT STRIP METHOD

Over the last couple of decades there has been an increased interest in measuring the thermal transport properties of micrometer-thin films with high-thermal conductivity, e.g., semiconductor-based material (Belkerk, Soussou, Carette, Djouradi, & Scudeller, 2012; Chien, Yao, Huang, & Chang, 2008; Li, Roger, Pottier, & Fournier, 1999; Taketoshi et al., 2001; Zhang et al., 2010). This is because of the necessity of ascertaining the thermal transport properties of structures used for electronic and optoelectronic components, like high-power transistors and diode lasers, in which considerable amounts of power must be dissipated to optimize their performance and not to over-heat neighboring components in integrated circuits. Anumber of transient methods, which have been developed or specially adapted for this kind of studies, include the Pulse Hot Strip method (Gustafsson, Chohan, Ahmed, & Maqsood, 1984), the Three-Omega method (Cahill & Pohl, 1987), and the Thermal-Reflectance method (Taketoshi et al., 2001; Zhang et al., 2010). However, it appears that so far only the Pulse Hot Strip method has the potential to conveniently deliver the anisotropic thermal properties of crystalline films. The other methods are effectively limited to studying isotropic films. The reason is that for a number of Transient Plane Source techniques, such as the Pulse Hot Strip method, which is a special adaptation of the Hot Strip method, both the thermal conductivity and thermal diffusivity can be retrieved from a single temperature versus pulse length recording. Moreover, Transient Plane Source methods have the advantage of clearly defining a thermal probing depth (as described earlier).

While working with the Hot Strip method on substrates with thermal diffusivities in the range 0.1–10 mm²/s and with transient recordings extended over a time ~10 seconds, the thermal probing depth, cf. Equation (14), will vary between 1 and 10 mm. However, if this probing depth could be reduced into the micrometer range, then square-shaped heating pulses in the microsecond range would be used. Fortunately, such square-shaped pulses are readily available from commercial pulse generators today. However, an important question to address in this context is how to record the short-voltage variations in the pulse-heated probe, which now is a micrometer-sized Hot Strip sensor evaporated on the surface of the sample. A way around the problem of making very fast recordings of the voltage variation has been developed by Rosenthal (1972) and researchers at Bell laboratories (English, Miller, Robinson, Dodd, & Chynoweth, 1978), who suggested the use of an AC-coupled electrical circuit (Ma, Gustavsson, Haglund, Gustavsson, & Gustafsson, 2014).

With the Pulse Hot Strip method the same simple electrical circuit as discussed above for the Hot Strip method (Figure 1) is employed, but with the addition of a blocking capacitor in series with the other components (Figure 4). With the aid of a low-pass filter the average voltage increase over the Hot Strip sensor as a function of the pulse length can be measured, keeping the duty cycle constant. The average voltage increases are in the micro- or millivolt ranges, but the recording can be extended over hundreds of seconds if necessary, and thus the average voltages can be measured quite precisely. Working in this way with pulses ranging from a few to perhaps a hundred microseconds it is possible to make a pulse-transient plot, from which both the thermal conductivity and diffusivity as well as the thermal anisotropy of surface layers down to an approximate thickness of \( \leq 10 \mu m \) may be obtained.

![Figure 4. Electrical circuit for Pulse Hot Strip measurements.](image-url)
Figure 5. A train of current pulses passing through the Hot Strip sensor. The zero net-charge condition of an AC-coupled network ensures that the gray-shaded regions have equal area.

The electrical current variation in the Hot Strip sensor for the AC-coupled circuit is shown in Figure 5. The presence of the blocking capacitor results in a total heating current that can be viewed as a series of current pulses in addition to a small constant “background” current being delivered to the probe. This constant “background” current establishes a small constant temperature difference between the probe and the constant temperature platform (i.e., heat sink) on top of which the wafer sample is placed. It is also clear that this temperature difference is dependent on the duty cycle of the pulse train. In experiments conducted so far a duty cycle of 5% has been used – with a view to minimize the temperature difference and still keep the sensitivity of the voltage readings at a reasonably high level. Under these conditions an analytical expression for the average temperature increase of the pulsed-heated Hot Strip sensor can be derived.

With the driving voltage from the pulse generator \( V \) and an internal resistance of the pulse generator \( R_s \), we have:

\[
V = (R_0 + R_s) \cdot (I_+ - I_-) \tag{32}
\]

and

\[
I_+ \cdot F = -I_- \cdot (1 - F) \tag{33}
\]

(i.e. zero net charge condition)

The negative current excursions – together with the same current during the positive pulse excursions – is creating an output of power \( R_0 \cdot (I_+)^2 \), which after a comparatively short time will result in a constant temperature difference \( \Delta T_b \) between the probe and the temperature controlled platform, on top of which the wafer sample has been placed. The constant temperature difference can be expressed as:

\[
\Delta T_b(t) = \frac{P_0 \cdot F^2}{2\sqrt{\pi} \cdot h \cdot (\lambda_x \cdot \lambda_y)^2} \cdot f_s(\tau_{2b}) \tag{34}
\]

where

\[
P_0 = \frac{V^2 \cdot R_0}{(R_0 + R_s)^2} \tag{35}
\]

is the total output of power in the probe, and

\[
f_s(\tau_{2b}) = \int_0^{\tau_{2b}} df(x) \cdot \left(1 + 2 \cdot \sum_{n=1}^{\infty} (-1)^n \cdot \exp \left(-\frac{n^2 \cdot v}{x^2}\right) \right) \cdot \left(1 - \frac{1}{x^2}\right) \tag{36}
\]

is a dimensionless time function with \( v = \frac{l^2 \cdot \kappa_y}{d^2 \cdot \kappa_x} \), and

where \( l \) is the thickness of the slab sample. As soon as the constant temperature difference has been established, the temperature increases, which will contribute to the voltage increases over the probe, can be expressed as follows:

\[
\Delta T(t) = \Delta T_{ho}(t) - \Delta T_b = -\frac{P_0 \cdot F^2}{2\sqrt{\pi} \cdot h \cdot (\lambda_x \cdot \lambda_y)^2} \cdot [f(\tau) - F^2 \cdot \beta] \tag{37}
\]

It is here assumed that the temperature variation, above the “background” temperature during the part of the period, when the pulse generator is idle, is obscured by the fact that the total “reverse” current is used to maintain the “background” temperature. The corresponding average temperature increase over a pulse “on-state” can then be expressed as:

\[
\Delta T_{MV}(FP) = T_x \cdot \frac{1}{FP} \cdot \int_0^{FP} [f(\tau) - F^2 \cdot \beta] \tag{38}
\]

where

\[
T_x = \frac{P_0}{2\sqrt{\pi} \cdot h \cdot (\lambda_x \cdot \lambda_y)^2} \tag{39}
\]

Equation (38) can be expressed as:

\[
\Delta T_{MV}(FP) = T_x \cdot \frac{2}{\tau_y} \cdot \int_0^{\tau_y} dx \cdot x \cdot f(x - \beta \cdot F^2) \tag{40}
\]

using the transformation

\[
\tau_y = \left(\frac{FP}{\theta_y}\right)^2 \quad \text{and} \quad \theta_y = \frac{d^2}{\kappa_y} \tag{41}
\]

Equation (40) can be transformed to:

\[
\Delta T_{MV}(\tau_y) = T_x \cdot H(\tau_y) - \beta \cdot T_x \cdot F^2 \tag{42}
\]
with

\[
H(\tau_2) = \frac{2}{\tau_y} \int_0^{\tau_y} dx \cdot f(x)
\]

(43)

This is the equation from which the thermal diffusivity in the direction perpendicular to the extension of the Hot Strip sensor – yet in the plane of the same – can be obtained using an iteration procedure. When the iteration is completed, the geometrical average of the two thermal conductivities in the two orthogonal directions perpendicular to the extension of the Hot Strip sensor is obtained from the slope of the straight line, which is the ultimate aim of the iteration. Figure 6 illustrates how to orient the strips to cover the possible anisotropies which might be present in the film sample, and Figure 7 shows an example of recorded average temperature increases versus “dimensionless time function”, \(H(\tau_2)\), after a successfully completed iteration.

5. CLOSING REMARKS

Over the years covered by this review – and indeed up to the present day – the effort by many experimental scientists working in the field of transient methods is to establish either one-dimensional (Laser Flash, Temperature Wave, Thermal Reflectance) or cylindrical (Hot Wire, 3Ω Method) heat flows. For the methods specifically discussed earlier (Hot Strip, Hot Disc, and Pulse Hot Strip), the effort is to avoid such extreme situations. These can be created for the Hot Strip by working with either very short (plane source range) or very long (cylindrical source range) experimental times, or for the Hot Disc by working either in the plane source or the point source range. The disadvantage of choosing not to work with these extreme situations has been that the solutions of the thermal conductivity equation are not covered by elementary mathematical functions. However, the advantage with the more general approach is that it is possible to retrieve both the thermal conductivity and the thermal diffusivity from a single transient recording. This dual information also makes it possible to identify the anisotropy of certain structures, which to date cannot be obtained by any other means. The symmetrical but complex temperature distribution around a Hot Disc sensor during a transient recording extending over a specific time with a thermal probing depth approximately equal to the radius of the probe is demonstrated in reference (Wang, Dinwiddie, Gustavsson, & Gustafsson, 2006).

Figure 6. Hot Strip sensor layout and orientation with respect to the crystallographic axes of the sample wafer.

Figure 7. Best straight line fit of calculated \(H(\tau_2)\) to measured \(\Delta T_{MV}\) on a fused quartz wafer using an 8-µm wide Hot Strip sensor and a maximum pulse length of 20 µs. The inset shows the deviation of \(\Delta T_{MV}\) from the straight line, resulting in a standard deviation of 2.5 mK. This yielded a thermal conductivity of 1.27 W/mK, a thermal diffusivity of 0.83 mm²/s, and a thermal probing depth of only 8.3 µm.

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