

MATERIALS SCIENCE SERIES

Thermal Properties Measurement of Materials

Yves Jannot and Alain Degiovanni



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Preface

Why write a book on thermal metrology and more specifically on the measurement of the thermal properties of materials?

The answer to this question is twofold:

- to know and improve materials;
- to have an input for calculating the modeling of complex systems.

Specialists in materials engineering are capable of manufacturing new materials with specific properties on demand, making it imperative to be able to characterize them as precisely as possible, and thus to guide new research.

The second challenge is to have an input for the calculation codes under good conditions. Indeed, the latter, by becoming more and more efficient, demand more and more precise parameter values; at each improvement of the codes, measurements need to be improved.

We could add a more societal reason to these two technical reasons: the control of energy, which goes through the use of efficient insulating materials in particular. The latter is of the utmost importance in highly energy-intensive sectors, in particular:

- industries that are very energy-intensive, such as glass furnaces, cement factories or the steel industry;
- buildings that need to develop more efficient thermal insulation;
- transport with reduced structures.

This book is therefore accessible to all, heat transfer specialists and non-specialists, who need to measure the thermal properties of a material. The objective is to allow them to choose the measurement method best suited to the material to be

characterized and to transmit to them all the information allowing the implementation of the measurement method with maximum precision.

The first chapter presents the different modes of heat transfer and the thermal properties of materials. Heat transfer equations are solved for a number of cases which will appear later in the presented characterization methods.

The second chapter describes the tools and methods of thermal metrology, beginning with the often underestimated problem of temperature measurement. Theoretical tools such as the study of reduced sensitivities and methods of parameter estimation are then presented.

The following three chapters describe the main thermal characterization methods classified into three main families, each method being presented at two levels:

- The first part describes the theoretical aspects of the method with a modeling of the heat transfer in the measuring device. This theoretical study most often makes it possible to determine the limits of the method's application in terms of values of the thermal properties and dimensions of the materials to be characterized on the one hand and of measurement duration in the case of a transient method on the other hand.

- The second part describes the experimental device, the measurement procedure and the mode of exploitation of the results in detail. This second part, being practical in nature, is intended for heat transfer non-specialists and can be approached independently from the first part. The application conditions set by the theoretical study will be systematically recalled in a simple manner.

Then, we present a synthesis chapter summarizing the fields of application of each of the methods presented and proposing the choice of the most adapted methods for different materials and dimensions. Finally, the strong analogy between heat transfer equations and equations governing other types of transfer is shown: for the case of mass transfer and flow of a fluid in a porous medium, for example. This allows a transposition of the thermal characterization methods to the estimation of other characteristic quantities and an application to the measurement of the permeability of a porous medium will be presented.

Happy measuring!

Yves Jannot
Alain Degiovanni
November 2017

Nomenclature

a	Thermal diffusivity	$\text{m}^2 \text{s}^{-1}$
Bi	Biot number	
c	Specific heat capacity	$\text{J kg}^{-1} \text{K}^{-1}$
D	Diameter	m
e	Thickness	m
E	Thermal effusivity	$\text{J m}^{-2} \text{K}^{-1} \text{s}^{-1/2}$
f	Frequency	Hz
$F(t)$	Transfer function in real space	
Fo	Fourier number	
g	Acceleration of gravity	m s^{-2}
k	Permeability	m^2
Gr	Grashof number	
h	Convective heat transfer coefficient	$\text{W m}^{-2} \text{K}^{-1}$
h_r	Radiative heat transfer coefficient	$\text{W m}^{-2} \text{K}^{-1}$

$H(p)$	Transfer function in Laplace space	
ΔH	Latent heat of phase change	J kg^{-1}
I	Intensity	A
I_0, I_1	Modified Bessel functions of the 1 st kind	
K_0, K_1	Modified Bessel functions of the 2 nd kind	
L, ℓ	Length	m
m	Mass	kg
$\dot{m}$	Mass flow	kg s^{-1}
Nu	Nusselt number	
p	Laplace variable	s^{-1}
P	Pressure	Pa
P_e	Perimeter	m
Pr	Prandtl number	
Q	Heat quantity	J
q	Surface energy density	J m^{-2}
$\dot{q}$	Power density	W m^{-3}
r, R	Radius	m
R_c	Contact resistance	K W^{-1}
Re	Reynolds number	
R_{el}	Electrical resistance	Ω
S	Surface area	m^2

t	Time	t
T	Temperature	K
u	Speed	m s^{-1}
U	Tension	V
V	Volume	m^3
x, y, z	Space variables	m
Z	Impedance	K W^{-1}

Greek letters

α	Radiation absorption coefficient	
β	Coefficient of volume expansion	
δ	Thickness	m
ε	Emissivity, porosity	
ϕ	Heat flux	W m^{-2}
Φ	Heat flow rate Laplace transform	
φ	Heat flow rate	W
λ	Thermal conductivity, wavelength	$\text{W m}^{-1} \text{K}^{-1}$, m
μ	Dynamic viscosity	Pa s
ν	Kinematic viscosity	$\text{m}^2 \text{s}^{-1}$
η	Yield or efficiency	
Ω	Solid angle	sr
ρ	Density, radiation reflection coefficient	kg m^{-3}

σ	Stefan–Boltzmann constant	$\text{W m}^{-2} \text{K}^{-4}$
τ	Time constant	s
θ	Laplace temperature transform	
ω	Pulsation	rd

Modeling of Heat Transfer

This chapter presents a reminder of courses on heat transfer limited to what is necessary to understand and master the methods of measuring the thermal properties of materials which will be described in the rest of this book.

1.1. The different modes of heat transfer

1.1.1. *Introduction and definitions*

We will first define the main quantities involved in solving a heat transfer problem.

1.1.1.1. *Temperature field*

Energy transfers are determined from the evolution of the temperature in space and time: $T = f(x, y, z, t)$. The instantaneous value of the temperature at any point of space is a scalar quantity called a temperature field. We will distinguish two cases:

- time-independent temperature field: the regime is called steady state or stationary;
- evolution of the temperature field over time: the regime is called variable, unsteady or transient.

1.1.1.2. *Temperature gradient*

If all the points of space which have the same temperature are combined, an isothermal surface is obtained. The temperature variation per unit length is maximal

in the direction normal to the isothermal surface. This variation is characterized by the temperature gradient:

$$\overrightarrow{grad}(T) = \vec{n} \frac{\partial T}{\partial n} \quad [1.1]$$

where: $\vec{n}$ is the normal unit vector;

$\frac{\partial T}{\partial n}$ is the derivative of the temperature along the normal direction.

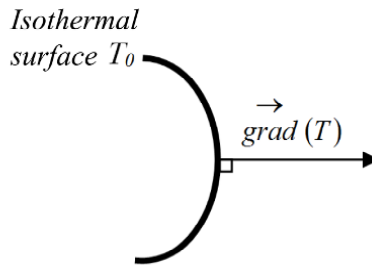


Figure 1.1. Isothermal surface and thermal gradient

1.1.1.3. Heat flux

Heat flows under the influence of a temperature gradient from high to low temperatures. The quantity of heat transmitted per unit time and per unit area of the isothermal surface is called the heat flux ϕ (W m^{-2}):

$$\phi = \frac{1}{S} \frac{dQ}{dt} \quad [1.2]$$

where S is the surface area (m^2).

φ (W) is called the heat flow rate and is the quantity of heat transmitted to the surface S per unit time:

$$\varphi = \frac{dQ}{dt} \quad [1.3]$$

1.1.1.4. Energy balance

The determination of the temperature field involves the writing of one or more energy balances. First, a system (S) must be defined by its limits in space and the

different heat flow rates that influence the state of the system must be established and they can be:

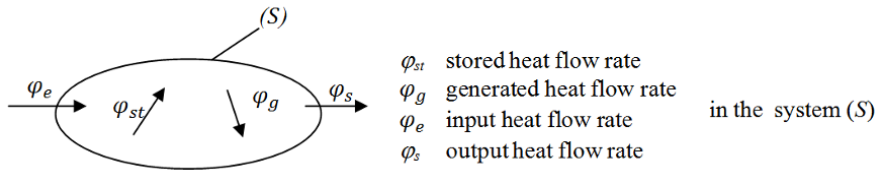


Figure 1.2. System and energy balance

The first principle of thermodynamics is then applied to establish the energy balance of the system (S):

$$\varphi_e + \varphi_g = \varphi_s + \varphi_{st} \quad [1.4]$$

After having replaced each of the terms by its expression as a function of the temperature, we obtain a differential equation whose resolution, taking into account the boundary conditions of the system, makes it possible to establish the temperature field. We will first give the possible expressions of the heat flow rates that can enter or exit a system by conduction, convection or radiation before giving an expression of the flux stored by sensible heat.

1.1.2. Conduction

Conduction is the transfer of heat within an opaque medium, without displacement of matter, under the influence of a temperature difference. The transfer of heat via conduction within a body takes place according to two distinct mechanisms: transmission via atomic or molecular vibrations and transmission via free electrons.

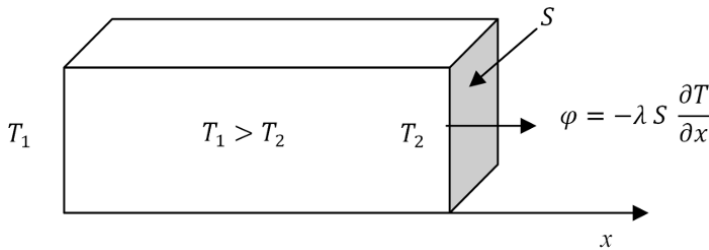


Figure 1.3. Conductive heat transfer scheme

The theory of conduction is based on the Fourier hypothesis: the heat flux ϕ is proportional to the temperature gradient:

$$\vec{\phi} = -\lambda \overrightarrow{\text{grad}}(T) \quad [1.5]$$

The heat flow rate transmitted by conduction in the direction Ox can therefore be written in algebraic form:

$$\varphi = -\lambda S \frac{\partial T}{\partial x} \quad [1.6]$$

where: ϕ is the conductive heat flux (W m^{-2});

φ is the conductive heat flow rate (W);

λ is the thermal conductivity of the medium ($\text{W m}^{-1} \text{K}^{-1}$);

x is the space variable in the heat flow's direction (m);

S is the surface area of the passage of the heat flux (m^2).

The values of the thermal conductivity λ of some of the most common materials are given in Table 1.1. A more complete table is given in Appendices A.1 and A.2.

Material	λ ($\text{W m}^{-1} \text{K}^{-1}$)	Material	λ ($\text{W m}^{-1} \text{K}^{-1}$)
Silver	419	Plaster	0.48
Copper	386	Asbestos	0.16
Aluminum	204	Wood (hard, soft wood)	0.12–0.23
Mild steel	45	Cork	0.044–0.049
Stainless steel	15	Stone wool	0.038–0.041
Ice	1.9	Glass wool	0.035–0.051
Concrete	1.4	Expanded polystyrene	0.036–0.047
Clay brick	1.1	Polyurethane (foam)	0.030–0.045
Glass	1.0	Extruded polystyrene	0.028
Water	0.60	Air	0.026

Table 1.1. Thermal conductivity of certain materials at room temperature

1.1.3. Convection

Here, we will only consider the heat transfer between a solid and a fluid, the energy being transmitted by the fluid's displacement. A good representation of this transfer mechanism is given by Newton's law:

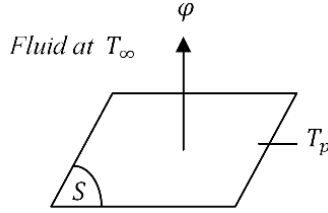


Figure 1.4. Convective heat transfer scheme

$$\varphi = h_c S (T_p - T_\infty) \quad [1.7]$$

where: φ is the heat flow rate transmitted by convection (W);

h_c is the convective heat transfer coefficient ($\text{W m}^{-2} \text{K}^{-1}$);

T_p is the solid's surface temperature (K);

T_∞ is the temperature of fluid away from solid surface (K);

S is the area of solid/fluid contact surface (m^2).

The value of the convective heat transfer coefficient h_c is a function of the fluid's nature, temperature, velocity or the temperature difference and the geometrical characteristics of the solid/fluid contact surface. The correlations in the most common cases of natural convection are given in Appendix 3, i.e. when the fluid's movement is due to temperature differences (no pump or fan).

Thermal characterization aims to measure the conductive and diffusing properties of a material. Convection most often occurs as a mode of "parasitic" transfer on the boundaries of the system by influencing the internal temperature field. We therefore have to take this into account. The correlations presented in Appendix 3 show that the coefficient of heat transfer by natural convection depends on the temperature difference between the surface and the surrounding fluid. Most often this difference is not perfectly uniform on surfaces and varies over time. It is therefore not possible to calculate it precisely and it will most often have to be estimated.

In natural convection, its value is generally between 2 and 5 W m⁻² K⁻¹. The radiation heat transfer coefficient that will be defined below is of the same order of magnitude. It will therefore be noted that placing a device under vacuum makes it possible to reduce losses by decreasing convective transfers but not canceling them, because radiation transfer is not affected by pressure.

1.1.4. Radiation

Radiation is a transfer of energy by electromagnetic waves (it does not need material support and even exists in a vacuum). We will only focus here on the transfer between two surfaces. In conduction problems, we take into account the radiation between a solid (whose surface is assumed to be gray and diffusing) and the surrounding environment (of large dimensions). In this case, we have the equation:

$$\varphi = \sigma \varepsilon_p S (T_p^4 - T_\infty^4) \quad [1.8]$$

where: φ is the radiation heat flow rate (W);

σ is Stefan's constant (5,67.10⁻⁸ W m⁻² K⁻⁴);

ε_p is the surface emission factor;

T_p is the surface temperature (K);

T_∞ is the temperature of the medium surrounding the surface (K);

S is the area of surface (m²).

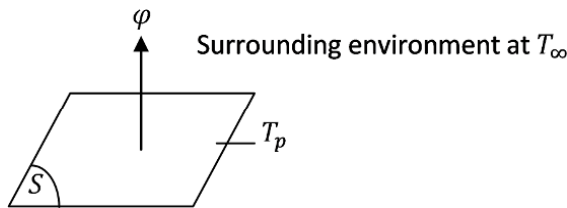


Figure 1.5. Radiation heat transfer scheme

NOTE.— In equations [1.6] and [1.7], temperatures can be expressed either in °C or K because they appear only in the form of differences. On the contrary, in equation [1.8], the temperature must be expressed in K.

1.1.4.1. Linearization of the radiation flux

In the case where the fluid in contact with the surface is a gas and where the convection is natural, the radiation heat transfer with the walls (at the average temperature T_r) surrounding the surface can become of the same order of magnitude as the convective heat transfer with the gas (at temperature T_f) at the contact with the surface and can no longer be neglected. The heat flow rate transferred by radiation is written according to equation [1.8]:

$$\varphi_r = \sigma \varepsilon S (T_p^4 - T_\infty^4)$$

It can take the form: $\varphi_r = h_r S (T_p - T_\infty)$

where h_r is called the radiation transfer coefficient:

$$h_r = \sigma \varepsilon (T_p^2 + T_\infty^2) (T_p + T_\infty) \quad [1.9]$$

The radiation transfer coefficient h_r varies very slightly for limited variations of the temperatures T_p and T_∞ and can be regarded as constant for a first simplified calculation. For example, with $\varepsilon = 0.9$, $T_p = 60^\circ\text{C}$ and $T_\infty = 20^\circ\text{C}$, the exact value is $h_r = 6.28 \text{ W m}^{-2} \text{ K}^{-1}$. If T_p becomes equal to 50°C (instead of 60°C), the value of h_r becomes equal to $5.98 \text{ W m}^{-2} \text{ K}^{-1}$, we get a variation of only 5%. When T_p is close to T_∞ , we can consider: $h_r \approx 4\sigma\varepsilon T_\infty^3$. It is also noted that the calculated values are of the order of magnitude of a natural convection coefficient in air.

It is to be remembered that when the convection exchange of a surface with its environment takes place by natural convection, we write the global heat flow rate (convection + radiation) exchanged by the surface in the form of:

$$\varphi = h S (T_p - T_\infty)$$

where $h = h_c + h_r$

1.1.4.2. Case of a high-temperature source

The radiation transfer also occurs in the exchange of a wall with temperature T_p with a high-temperature heat source T_s , for example, the Sun ($\approx$ black body at 5760 K). In this case, equation [1.8] becomes:

$$\varphi = K S (T_p^4 - T_s^4) \quad [1.10]$$

where K is a constant taking into account the surface and source emissivities as well as the geometric shape factor between the surface and the source.

Then equation [1.10] becomes: $\varphi = -K S T_s^4 = -\varphi_0$ and the radiation source is modeled by a constant heat flow rate φ_0 imposed on the wall (this is, for example, the case of a thermal sensor exposed to the Sun).

1.1.5. Heat storage

The storage of energy in sensitive form in a body corresponds to an increase of its enthalpy in the course of time from which (at constant pressure and in the absence of change of state):

$$\varphi_{st} = \rho V c \frac{\partial T}{\partial t} \quad [1.11]$$

where: φ_{st} is the stored heat flow rate (W);
 ρ is density (kg m^{-3});
 V is volume (m^3);
 c is specific heat ($\text{J kg}^{-1} \text{K}^{-1}$);
 T is temperature (K);
 t is time (s).

The product $\rho V c$ (J K^{-1}) is called the thermal capacitance of the body.

1.2. Modeling heat transfer by conduction

1.2.1. The heat equation

In its mono-dimensional form, this equation describes the one-directional transfer of heat through a flat wall (see Figure 1.6).

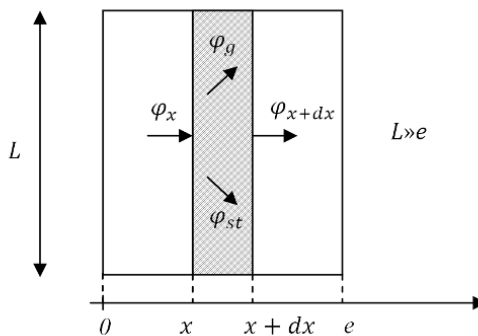


Figure 1.6. Thermal balance of an elementary system

Consider a system of thickness dx in the direction x and of section of area S normal to direction Ox . The energy balance of this system is written as:

$$\varphi_x + \varphi_g = \varphi_{x+dx} + \varphi_{st}$$

where: $\varphi_x = -\left(\lambda S \frac{\partial T}{\partial x}\right)_x$;

$$\varphi_{x+dx} = -\left(\lambda S \frac{\partial T}{\partial x}\right)_{x+dx} ;$$

$$\varphi_g = \dot{q}Sdx;$$

$$\varphi_{st} = \rho c S dx \frac{\partial T}{\partial t}.$$

By plotting in the energy balance and dividing by dx , we obtain:

$$\frac{(\lambda S \frac{\partial T}{\partial x})_{x+dx} - (\lambda S \frac{\partial T}{\partial x})_x}{dx} + \dot{q}S = \rho c S \frac{\partial T}{\partial t}$$

or: $\frac{\partial}{\partial x} \left(\lambda S \frac{\partial T}{\partial x} \right) + \dot{q}S = \rho c S \frac{\partial T}{\partial t}$

and in the three-dimensional case, we obtain the heat equation in the most general case:

$$\frac{\partial}{\partial x} \left(\lambda_x \frac{\partial T}{\partial x} \right) + \frac{\partial}{\partial y} \left(\lambda_y \frac{\partial T}{\partial y} \right) + \frac{\partial}{\partial z} \left(\lambda_z \frac{\partial T}{\partial z} \right) + \dot{q} = \rho c \frac{\partial T}{\partial t} \quad [1.12]$$

This equation can be simplified in a number of cases:

- a) if the medium is isotropic: $\lambda_x = \lambda_y = \lambda_z = \lambda$;
- b) if there is no generation of energy inside the system: $\dot{q} = 0$;
- c) if the medium is homogeneous, λ is only a function of T .

The hypotheses a) + b) + c) make it possible to write:

$$\lambda \left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right) + \frac{d\lambda}{dT} \left[\left(\frac{\partial T}{\partial x} \right)^2 + \left(\frac{\partial T}{\partial y} \right)^2 + \left(\frac{\partial T}{\partial z} \right)^2 \right] = \rho c \frac{\partial T}{\partial t} \quad [1.13]$$

d) If λ is constant (moderate temperature deviation), we obtain the Poisson equation:

$$\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} = \frac{1}{a} \frac{\partial T}{\partial t} \quad [1.14]$$

The ratio $a = \frac{\lambda}{\rho c}$ is called thermal diffusivity ($\text{m}^2 \text{s}^{-1}$), it characterizes the propagation velocity of a heat flux through a material. Values can be found in Appendix 1.

e) In steady state, we obtain Laplace's equation:

$$\nabla^2 T = \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} = 0 \quad [1.15]$$

Moreover, hypotheses a), c) and d) make it possible to write:

– heat equation in cylindrical coordinates (r, θ, z):

$$\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} + \frac{1}{r^2} \frac{\partial^2 T}{\partial \theta^2} + \frac{\partial^2 T}{\partial z^2} + \frac{\dot{q}}{\lambda} = \frac{1}{a} \frac{\partial T}{\partial t} \quad [1.16]$$

In the case of a cylindrical symmetry problem where the temperature depends only on r and t , equation [1.16] can be written in simplified form:

$$\frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial T}{\partial r} \right) + \frac{\dot{q}}{\lambda} = \frac{1}{a} \frac{\partial T}{\partial t}.$$

– heat equation in spherical coordinates (r, θ, φ):

$$\frac{1}{r} \frac{\partial^2}{\partial r^2} (rT) + \frac{1}{r^2 \sin(\theta)} \frac{\partial}{\partial \theta} \left[\sin(\theta) \frac{\partial T}{\partial \theta} \right] + \frac{1}{r^2 \sin^2(\theta)} \frac{\partial^2 T}{\partial \varphi^2} + \frac{\dot{q}}{\lambda} = \frac{1}{a} \frac{\partial T}{\partial t} \quad [1.17]$$

1.2.2. Steady-state conduction

1.2.2.1. Simple wall

We will place ourselves in the situation where the heat transfer is one directional and where there is no energy generation or storage.

We consider a wall of thickness e , thermal conductivity λ and large transverse dimensions whose extreme faces are at temperatures T_1 and T_2 (see Figure 1.7).

By carrying out a thermal balance on the system (S) constituted by the wall slice comprised between the abscissae x and $x + dx$, we obtain:

$$\varphi_x = \varphi_{x+dx} \Rightarrow \left(\lambda S \frac{dT}{dx} \right)_x = \left(\lambda S \frac{dT}{dx} \right)_{x+dx}$$

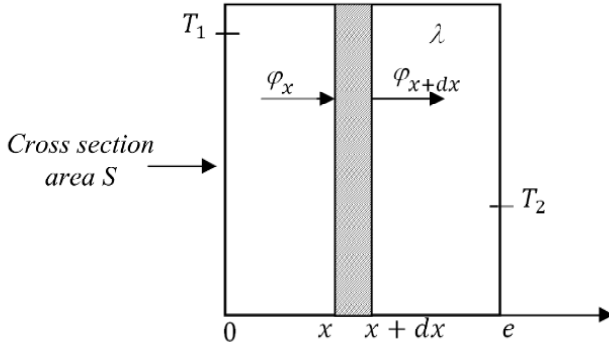


Figure 1.7. Basic thermal balance on a simple wall

where: $\frac{dT}{dx} = A$ and $T(x) = Ax + B$

with boundary conditions: $T(x = 0) = T_1$ et $T(x = e) = T_2$

$$\text{Thus: } T(x) = T_1 - \frac{x}{e} (T_1 - T_2) \quad [1.18]$$

The temperature profile is therefore linear. The heat flux passing through the wall is deduced by equation: $\phi = -\lambda \frac{\partial T}{\partial x}$

$$\text{Thus: } \phi = \frac{\lambda}{e} (T_1 - T_2) \quad [1.19]$$

Equation [1.19] can also be written as: $\phi = \frac{T_1 - T_2}{\frac{e}{\lambda S}}$, this equation is analogous to Ohm's law in electricity which defines the intensity of the current as the ratio of the electrical potential difference on the electrical resistance. The temperature T thus appears as a thermal potential and the term $\frac{e}{\lambda S}$ appears as the thermal resistance of a plane wall of thickness e , thermal conductivity λ and lateral surface S . We thus get the equivalent network represented in Figure 1.8.

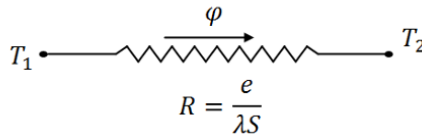


Figure 1.8. Equivalent electrical network of a single wall

NOTE.— The flux is constant, it is a general result for any tube of flux in steady state (system with conservative flux).

1.2.2.2. Multilayer wall

This is the case for real walls (described in Figure 1.9) made up of several layers of different materials and where only the temperatures T_{f1} and T_{f2} of the fluids that are in contact with the two faces of the lateral surface wall S are known.

In steady state, the heat flow rate is preserved when the wall is crossed and is written as:

$$\varphi = h_1 S (T_{f1} - T_1) = \frac{\lambda_A S (T_1 - T_2)}{e_A} = \frac{\lambda_B S (T_2 - T_3)}{e_B} = \frac{\lambda_C S (T_3 - T_4)}{e_C} = h_2 S (T_4 - T_{f2})$$

$$\text{where: } \varphi = \frac{T_{f1} - T_{f2}}{\frac{1}{h_1 S} + \frac{e_A}{\lambda_A S} + \frac{e_B}{\lambda_B S} + \frac{e_C}{\lambda_C S} + \frac{1}{h_2 S}} \quad [1.20]$$

It was considered that the contacts between the layers of different natures were perfect and that there was no temperature discontinuity at the interfaces. In reality, given the roughness of the surfaces, a micro-layer of air exists between the surface hollows contributing to the creation of a thermal resistance (the air is an insulator) called thermal contact resistance. The previous formula is then written as:

$$\varphi = \frac{T_{f1} - T_{f2}}{\frac{1}{h_1 S} + \frac{e_A}{\lambda_A S} + R_{AB} + \frac{e_B}{\lambda_B S} + R_{BC} + \frac{e_C}{\lambda_C S} + \frac{1}{h_2 S}} \quad [1.21]$$

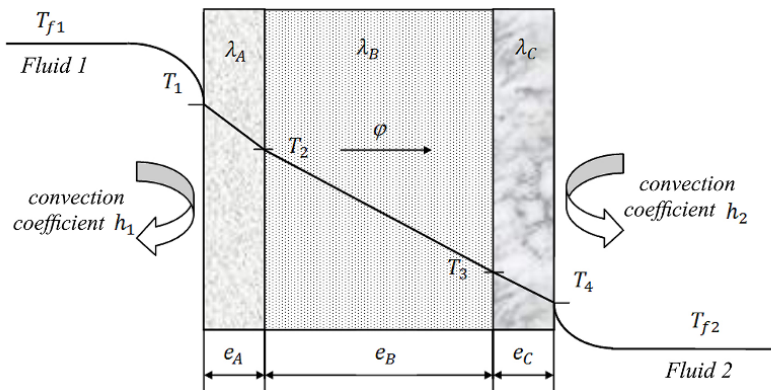


Figure 1.9. Schematic representation of heat flow and temperatures in a multilayer wall

The equivalent electrical diagram is shown in Figure 1.10.

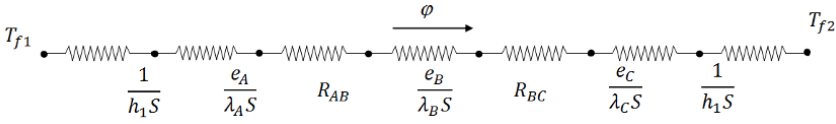


Figure 1.10. *Equivalent electrical network of a multilayer wall*

NOTES.—

- Thermal resistance can only be defined on a flux tube.
- The thermal contact resistance between two layers is neglected if one layer is an insulating material or if the layers are joined by welding.

1.2.2.3. Composite wall

This is the case most commonly encountered in reality where the walls are not homogeneous. Let us consider, by way of example, a wall of width L consisting of hollow agglomerates (Figure 1.11).

Considering the symmetries, the calculation of the wall's thermal resistance can be reduced to that of a unit cell defined by the diagram in Figure 2.6. This unit cell is a flux tube (isotherm at $x = 0$ and $x = e_1 + e_2 + e_3$, and adiabatic at $y = 0$ and $y = \ell_1 + \ell_2 + \ell_3$) and can therefore be represented by a resistance R .

The exact calculation of this resistance is complex because each medium does not constitute a flux tube. If there is no need for high accuracy, several approximate calculations are possible by making assumptions on isothermal and adiabatic surfaces.

For example, assuming the surfaces at $x = e_1$ and $x = e_1 + e_2$ to be isothermal and the surfaces at $y = \ell_3$ and $y = \ell_2 + \ell_3$ adiabatic and by using the laws of association of the resistors in series and in parallel, we obtain:

$$R = R_1 + R_2 + \frac{1}{\frac{1}{R_3} + \frac{1}{R_4} + \frac{1}{R_5}} + R_6 + R_7$$

where:

$$R_1 = \frac{1}{h_1 \ell L}; R_2 = \frac{e_1}{\lambda_1 \ell L}; R_3 = \frac{e_2}{\lambda_2 \ell_1 L}; R_4 = \frac{e_2}{\lambda_1 \ell_2 L}; R_5 = \frac{e_2}{\lambda_1 \ell_3 L}; R_6 = \frac{e_3}{\lambda_1 \ell L}; R_7 = \frac{1}{h_2 \ell L}$$

which can be represented by the equivalent electrical network shown in Figure 1.12.

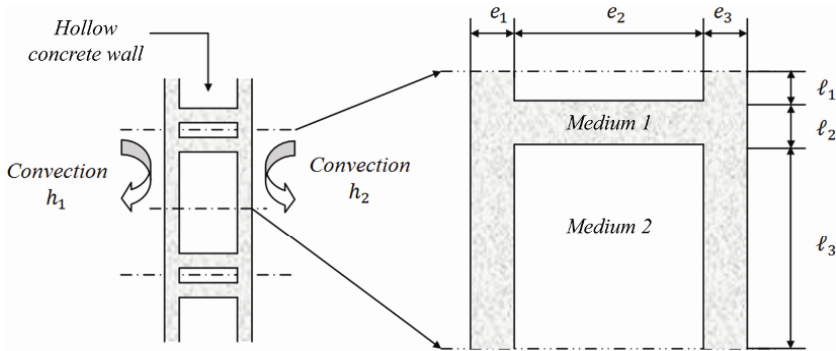


Figure 1.11. Diagram of a composite wall

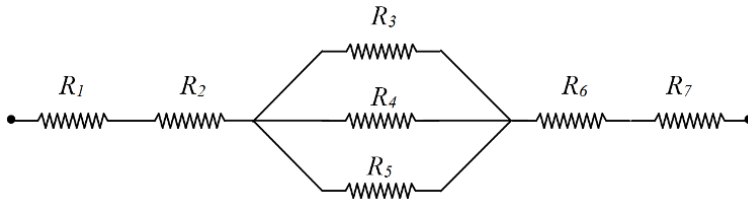


Figure 1.12. Electrical equivalent network of a composite wall

It will be noted that this solution is only approximate since the real transfer is 2D because of the differences between the thermal conductivities λ_1 and λ_2 .

1.2.2.4. Long hollow cylinder (tube)

We consider a hollow cylinder with a thermal conductivity λ , internal radius r_1 , external radius r_2 , length L , and with internal and external face temperatures being T_1 and T_2 , respectively (see Figure 1.13). It is assumed that the longitudinal temperature gradient is negligible compared to the radial gradient.

Let us carry out the thermal balance of the system constituted by the part of the cylinder comprised between radii r and $r + dr$: $\varphi_r = \varphi_{r+dr}$

where: $\varphi_r = -2\pi r L \lambda \left(\frac{dT}{dr} \right)_r$ and: $\varphi_{r+dr} = -2\pi(r + dr) L \lambda \left(\frac{dT}{dr} \right)_{r+dr}$

Thus: $-2\pi r L \lambda \left(\frac{dT}{dr} \right)_r = -2\pi(r + dr) L \lambda \left(\frac{dT}{dr} \right)_{r+dr}$ where: $r \frac{dT}{dr} = C$

with boundary conditions: $T(r_1) = T_1$ and $T(r_2) = T_2$

$$\text{where: } \frac{T(r) - T_1}{T_2 - T_1} = \frac{\ln\left(\frac{r}{r_1}\right)}{\ln\left(\frac{r_2}{r_1}\right)} \quad [1.22]$$

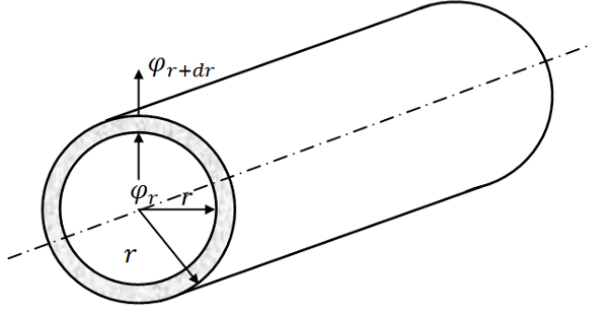


Figure 1.13. Diagram of transfers in a hollow cylinder

and by applying equation $\varphi = -2\pi r L \lambda \left(\frac{dT}{dr} \right)$, we obtain:

$$\varphi = \frac{2\pi \lambda L (T_1 - T_2)}{\ln\left(\frac{r_2}{r_1}\right)} \quad [1.23]$$

This equation can also be written as: $\varphi = \frac{T_1 - T_2}{R_{12}}$ with $R_{12} = \frac{\ln\left(\frac{r_2}{r_1}\right)}{2\pi \lambda L}$ and can be represented by the equivalent electrical network of Figure 1.14.

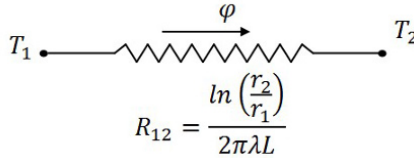


Figure 1.14. Equivalent electrical network of a hollow cylinder

1.2.2.5. Multilayer hollow cylinder

This is the practical case of a tube covered with one or more layers of different materials and where only temperatures T_{f1} and T_{f2} of the fluids in contact with the inner and outer faces of the cylinder are known; h_1 and h_2 are the global (convection + radiation) heat transfer coefficients between the fluids and the internal and external faces (see Figure 1.15).

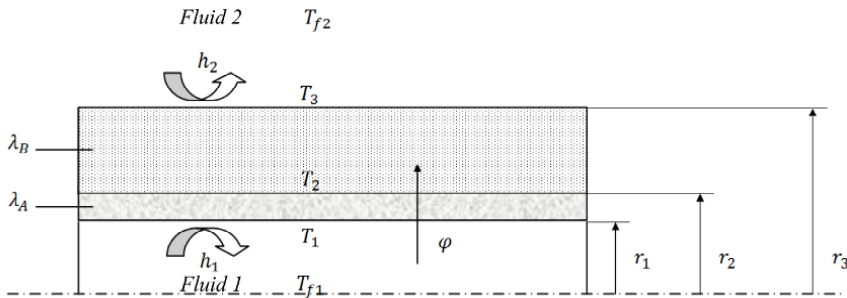


Figure 1.15. Diagram of heat transfers in a multilayer hollow cylinder

In steady-state conditions, the heat flow rate ϕ is conserved when passing through the different layers and is written as:

$$\phi = h_1 2\pi r_1 L (T_{f1} - T_1) = \frac{2\pi \lambda_A L (T_1 - T_2)}{\ln\left(\frac{r_2}{r_1}\right)} = \frac{2\pi \lambda_B L (T_2 - T_3)}{\ln\left(\frac{r_3}{r_2}\right)} = h_2 2\pi r_3 L (T_3 - T_{f2})$$

$$\text{Hence: } \phi = \frac{T_{f1} - T_{f2}}{\frac{1}{h_1 2\pi r_1 L} + \frac{\ln\left(\frac{r_2}{r_1}\right)}{2\pi \lambda_A L} + \frac{\ln\left(\frac{r_3}{r_2}\right)}{2\pi \lambda_B L} + \frac{1}{h_2 2\pi r_3 L}} \quad [1.24]$$

which can be represented by the equivalent electrical network of Figure 1.16.

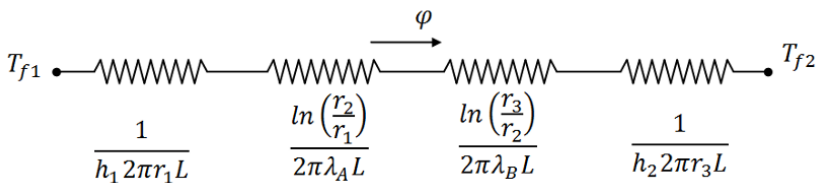


Figure 1.16. Equivalent electrical network of a multilayer hollow cylinder

1.2.2.6. General case

We have just given the value of the thermal resistance for some particular cases of practical interest. More generally, the thermal resistance of a flux tube (see Figure 1.17) is written as:

$$R = \int_{r_1}^{r_2} \frac{dr}{\lambda(r)S(r)} \quad [1.25]$$

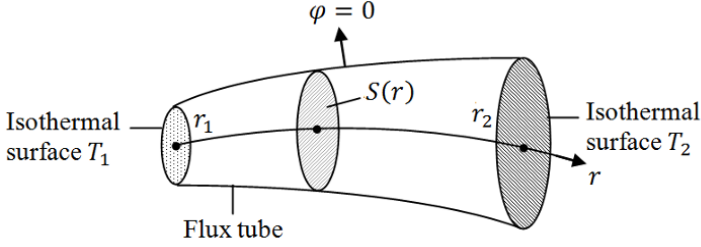


Figure 1.17. Diagram of a flux tube

1.2.3. Conduction in unsteady state

1.2.3.1. Medium at uniform temperature

We will study the transfer of heat to a medium at a uniform temperature (“small body” hypothesis), which is *a priori* contradictory because it is necessary that there is a thermal gradient for heat transfer to occur. This approximation of the medium at uniform temperature may nevertheless be justified in certain cases which will be specified. For example, the quenching of a metal ball which consists of immersing a ball initially at the temperature T_i in a bath of constant temperature T_0 . Assuming that the temperature inside the ball is uniform, which will be all the more true as its size is small and its thermal conductivity is high, we can write the thermal balance of this ball between two points in time as t and $t + dt$:

$$-hS(T - T_0) = \rho cV \frac{dT}{dt} \quad \text{or} \quad \frac{dT}{T - T_0} = -\frac{hS}{\rho cV} dt$$

$$\text{Hence: } \frac{T - T_0}{T_i - T_0} = \exp\left(-\frac{hS}{\rho cV} t\right) \quad [1.26]$$

This solution is shown in Figure 1.18. We note that the grouping $\frac{\rho c V}{h S}$ is homogeneous at a time which will be called τ and is the system's time constant:

$$\tau = \frac{\rho c V}{h S} \quad [1.27]$$

This quantity is fundamental insofar as it gives the order of magnitude of the physical phenomenon at that point in time; we have in fact: $\frac{T-T_0}{T_i-T_0} = \exp\left(-\frac{t}{\tau}\right)$

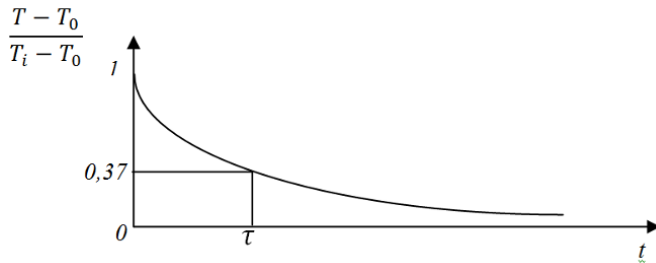


Figure 1.18. Evolution of the temperature of a medium at uniform temperature

It is always interesting in physics to present the results in dimensionless form. Two dimensionless numbers are particularly important in variable regimes:

– The Biot number: $Bi = \frac{\text{Internal thermal resistance}}{\text{External thermal resistance}} = \frac{\ell}{\frac{\lambda S}{h}}$, ℓ is the medium's characteristic dimension, $\ell = R$ for a sphere.

$$\text{Or: } Bi = \frac{h \ell}{\lambda} \quad [1.28]$$

– The Fourier number:

$$Fo = \frac{at}{\ell^2} \quad [1.29]$$

The Fourier number characterizes heat penetration in a variable regime.

The definition of these two dimensionless numbers makes it possible to write the equation of the temperature of the ball in the form:

$$\frac{T-T_0}{T_i-T_0} = \exp(-Bi Fo) \quad [1.30]$$

Knowing the product of the Biot and Fourier numbers makes it possible to determine the evolution of the sphere's temperature. It is generally considered that a system such as $Bi < 0.1$ can be considered to be at a uniform temperature; the criterion $Bi < 0.1$ is called the criterion of "thermal accommodation".

1.2.3.2. Semi-infinite medium

A semi-infinite medium is a wall of sufficiently large thickness so that the perturbation applied to one face is not felt by the other face. Such a system represents the evolution of a wall of finite thickness for a sufficiently short time so that the disturbance created on one face has not reached the other side (true for the point in time that the temperature of the other face does not vary). We will consider, for example, a layer of thickness $1\ \mu\text{m}$ as a semi-infinite medium in the 3ω method!

EXAMPLE.— *Imposed surface temperature.*

Method: integral Laplace transform in time and inversion by tables.

The semi-infinite medium is initially at the uniform temperature T_i (see Figure 1.19). The surface temperature is suddenly maintained at a temperature T_0 ; this boundary condition is called the Dirichlet condition.

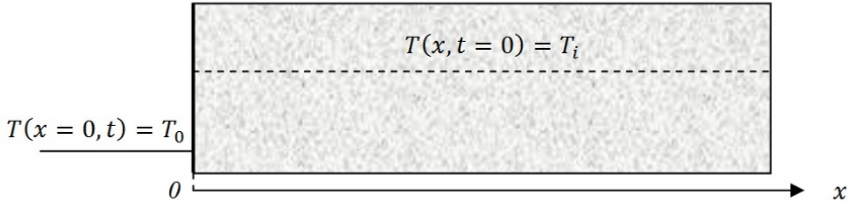


Figure 1.19. *Diagram of semi-infinite medium with imposed surface temperature*

$$\text{The heat equation is written as: } \frac{\partial^2 T}{\partial x^2} = \frac{1}{a} \frac{\partial T}{\partial t} \quad [1.31]$$

$$T(x, 0) = T_i \quad [1.32]$$

$$\text{with boundary conditions: } T(0, t) = T_0 \quad [1.33]$$

$$T(x, t) \rightarrow T_i \text{ when } x \rightarrow \infty \quad [1.34]$$

The following variable change is made: $\bar{T} = T - T_i$

$$\text{Hence: } \frac{\partial \bar{T}}{\partial x} = \frac{\partial T}{\partial x}, \frac{\partial^2 \bar{T}}{\partial x^2} = \frac{\partial^2 T}{\partial x^2} \text{ and } \frac{\partial \bar{T}}{\partial t} = \frac{\partial T}{\partial t}$$

$$\text{The equation [1.30] can thus be written as: } \frac{\partial^2 \bar{T}}{\partial x^2} = \frac{1}{a} \frac{\partial \bar{T}}{\partial t} \quad [1.35]$$

$$\bar{T}(x, 0) = 0 \quad [1.36]$$

$$\text{The boundary conditions become: } \bar{T}(0, t) = T_0 - T_i \quad [1.37]$$

$$\bar{T}(x, t) \rightarrow 0 \text{ when } x \rightarrow \infty \quad [1.38]$$

The Laplace transform of $\bar{T}(x, t)$ with respect to time is written as (see Appendix 4 on integral transformations): $\theta(x, p) = L[\bar{T}(x, t)] = \int_0^\infty \exp(-pt) \bar{T}(x, t) dt$.

$$\text{The Laplace transform of equation [1.35] leads to: } \frac{d^2 \theta}{dx^2} - \frac{1}{a} [p\theta - \bar{T}(x, 0)] = 0$$

with: $\bar{T}(x, 0) = 0$ (which justifies the change of variable).

$$\text{This equation is therefore of the form: } \frac{d^2 \theta}{dx^2} - q^2 \theta = 0 \quad \text{with: } q = \sqrt{\frac{p}{a}}.$$

$$\text{Hence: } \theta(x, p) = A \exp(-qx) + B \exp(qx).$$

The temperature keeps a finite value when $x \rightarrow \infty$, thus $B = 0$ and:

$$\theta(x, p) = A \exp(-qx).$$

$$\text{The Laplace transform of equation [1.37] leads to: } (0, p) = \frac{T_0 - T_i}{p},$$

$$\text{hence: } A = \frac{T_0 - T_i}{p}$$

$$\text{and: } \theta(x, p) = \frac{T_0 - T_i}{p} \exp(-qx).$$

Using the inverse Laplace transform tables presented in Appendix 5 leads to the following result:

$$\frac{T(x, t) - T_0}{T_i - T_0} = \text{erf}\left(\frac{x}{2\sqrt{at}}\right) \quad [1.39]$$

$$\text{where: } \text{erf}(u) = \frac{2}{\sqrt{\pi}} \int_0^u \exp(-t^2) dt.$$

The function *erf* is called the error function (see values in Appendix 6).

EXAMPLE.— *Imposed heat flux at the surface.*

Method: integral Laplace transform in time and inversion by tables.

Consider the same configuration but instead by brutally imposing a heat flux density on the surface of the semi-infinite medium (see Figure 1.20); this boundary condition is called the Neumann condition.

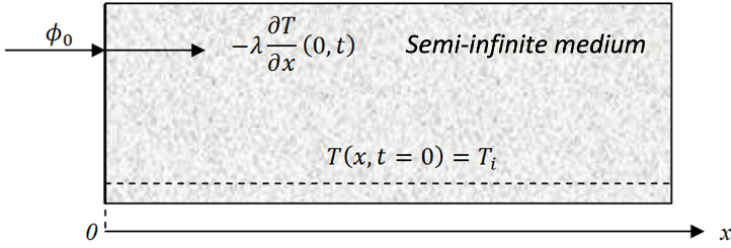


Figure 1.20. *Diagram of the semi-infinite medium with imposed surface flux*

The heat equation is written as: $\frac{\partial^2 T}{\partial x^2} = \frac{1}{a} \frac{\partial T}{\partial t}$ [1.40]

$T(x, 0) = T_i$ [1.41]

with the boundary conditions: $-\lambda \frac{\partial T}{\partial x}(0, t) = \phi_0$ [1.42]

$T(x, t) \rightarrow T_i$ when $x \rightarrow \infty$ [1.43]

Condition [1.42] expresses the conservation of heat flux at the surface of the semi-infinite medium.

The following change of variable is made: $\bar{T} = T - T_i$.

Equation [1.40] can then be written as: $\frac{\partial^2 \bar{T}}{\partial x^2} = \frac{1}{a} \frac{\partial \bar{T}}{\partial t}$ [1.44]

$\bar{T}(x, 0) = 0$ [1.45]

The boundary conditions become: $-\lambda \frac{\partial \bar{T}}{\partial x}(0, t) = \phi_0$ [1.46]

$\bar{T}(x, t) \rightarrow 0$ when $x \rightarrow \infty$ [1.47]

The Laplace transform of equation [1.44] leads to: $\frac{d^2\theta}{dx^2} - q^2\theta = 0$ with: $q = \sqrt{\frac{p}{a}}$.
hence: $\theta(x, p) = A \exp(-qx) + B \exp(qx)$.

The temperature keeps a finite value when $x \rightarrow \infty$, thus $B = 0$ and $\theta(x, p) = A \exp(-qx)$.

The Laplace transform of equation [1.46] leads to: $\frac{\phi_0}{p} = -\lambda \frac{d\theta}{dx}(0, p)$.

Hence: $A = \frac{\phi_0}{\lambda p q}$ and $\theta(x, p) = \frac{\phi_0}{\lambda} \frac{\exp(-qx)}{p q}$.

Using the inverse Laplace transform tables presented in Appendix 5 leads to the following result:

$$T(x, t) - T_i = \frac{2\phi_0}{\lambda} \sqrt{at} \operatorname{ierfc}\left(\frac{x}{2\sqrt{at}}\right) \quad [1.48]$$

where: $\operatorname{ierfc}(u) = \frac{1}{\sqrt{\pi}} \exp(-u^2) - u[1 - \operatorname{erf}(u)]$, this function is tabulated in Appendix 6.

EXAMPLE.— *Imposed heat transfer coefficient.*

Method: integral Laplace transform in time and inversion by tables.

We consider the case where the convective heat transfer coefficient h between the semi-infinite medium and the ambient medium is imposed (see Figure 1.21). This boundary condition is called Newton's condition:

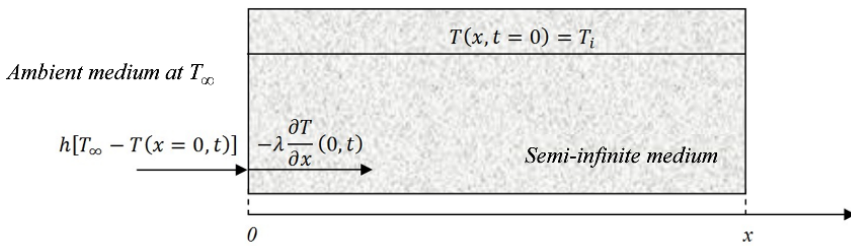


Figure 1.21. *Diagram of the semi-infinite medium with imposed convective transfer coefficient*

The heat equation is written as: $\frac{\partial^2 T}{\partial x^2} = \frac{1}{a} \frac{\partial T}{\partial t}$ [1.49]

$$T(x, 0) = T_i \quad [1.50]$$

with the boundary conditions: $-\lambda \frac{\partial T}{\partial x}(0, t) = h[T_\infty - T(x = 0, t)]$ [1.51]

$$T(x, t) \rightarrow T_i \text{ when } x \rightarrow \infty \quad [1.52]$$

The following change of variable is made: $\bar{T} = T - T_i$.

Equation [1.49] can then be written as: $\frac{\partial^2 \bar{T}}{\partial x^2} = \frac{1}{a} \frac{\partial \bar{T}}{\partial t}$ [1.53]

$$\bar{T}(x, 0) = 0 \quad [1.54]$$

The boundary conditions become: $\lambda \frac{\partial \bar{T}}{\partial x}(0, t) = h[\bar{T}(x = 0, t) - (T_\infty - T_i)]$ [1.55]

$$\bar{T}(x, t) \rightarrow 0 \text{ when } x \rightarrow \infty \quad [1.56]$$

The Laplace transform of equation [1.53] leads to: $\frac{d^2 \theta}{dx^2} - q^2 \theta = 0$ with $q = \sqrt{\frac{p}{a}}$.

Hence: $\theta(x, p) = A \exp(-qx) + B \exp(qx)$.

The temperature keeps a finite value when $x \rightarrow \infty$, thus $B = 0$ and:

$$\theta(x, p) = A \exp(-qx).$$

The Laplace transform of equation [1.55] is written as:

$$\lambda \frac{d\theta}{dx}(0, p) = h\theta(0, p) + h \frac{T_i - T_\infty}{p}$$

or: $-\lambda Aq = hA + h \frac{T_i - T_\infty}{p}$ hence: $A = \frac{h(T_\infty - T_i)}{p(\frac{h}{\lambda} + q)}$

and: $\theta(x, p) = \frac{h}{\lambda} (T_\infty - T_i) \frac{\exp(-qx)}{p(q + \frac{h}{\lambda})}$.

Using the inverse Laplace transform tables presented in Appendix 5 leads to the following result:

$$\frac{T(x, t) - T_\infty}{T_i - T_\infty} = \operatorname{erf}\left(\frac{x}{2\sqrt{at}}\right) + \exp\left(\frac{hx}{\lambda} + \frac{ah^2t}{\lambda^2}\right) \operatorname{erfc}\left(\frac{x}{2\sqrt{at}} + \frac{h\sqrt{at}}{\lambda}\right) \quad [1.57]$$

EXAMPLE.— *Sinusoidal surface temperature, established periodic regime (see Figure 1.22).*

Method: search for a solution with the same frequency as the excitation

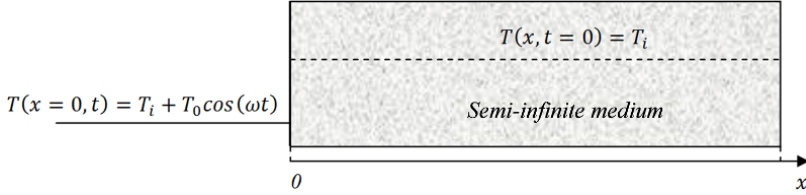


Figure 1.22. *Diagram of a semi-infinite medium with surface-imposed sinusoidal temperature*

The heat equation is written as: $\frac{\partial^2 T}{\partial x^2} = \frac{1}{a} \frac{\partial T}{\partial t}$

with the boundary conditions: $T(x = 0, t) = T_i + T_0 \cos(\omega t)$

$T(x, t) \rightarrow T_i$ when $x \rightarrow \infty$

A steady-state solution is sought for which the temperature field of the medium evolves as follows:

$$T(x, t) = T_i + f(x) \exp(i\omega t)$$

The problem, being linear, makes us consider either the real part or the imaginary part of the solution depending on whether the temperature varies as $\cos(\omega t)$ or $\sin(\omega t)$. The complex function f is a solution of:

$$\frac{d^2 f}{dx^2} - i \frac{\omega}{a} f = 0 \text{ with } f(0) = T_0$$

$$f(x) = A \exp\left(-\sqrt{\frac{i\omega}{a}} x\right) + B \exp\left(\sqrt{\frac{i\omega}{a}} x\right) \text{ with: } \sqrt{\frac{i\omega}{a}} = \sqrt{\frac{\omega}{2a}} (1 + i)$$

The function f must remain finite when $x \rightarrow \infty$, thus $B = 0$ and $f(0) = T_0$ leading to $A = T_0$.

Hence:

$$T(x, t) - T_i = T_0 \exp\left[-\sqrt{\frac{\omega}{2a}} (1 + i)x + i\omega t\right] = T_0 \exp\left(-\sqrt{\frac{\omega}{2a}} x\right) \exp\left[i\left(\omega t - \sqrt{\frac{\omega}{2a}} x\right)\right]$$

or by taking the real part of the solution:

$$T(x, t) = T_i + T_0 \exp\left(-\sqrt{\frac{\omega}{2a}}x\right) \cos\left(\omega t - \sqrt{\frac{\omega}{2a}}x\right) \quad [1.58]$$

NOTES.—

– The amplitude of the oscillations decreases rapidly when moving away from the interface.

– The amplitude of the oscillations also decreases rapidly when the excitation frequency increases: a high frequency excitation applied to the surface of a solid will only change its temperature at a shallow depth.

– Between temperatures T_1 and T_2 of two points with a distance respectively of x_1 and x_2 from the surface, there exists a phase shift equal to $\sqrt{\frac{\omega}{2a}}(x_1 - x_2)$. The knowledge of ω and the temperature measurement within the medium at two points located at known distances x_1 and x_2 from the surface can make it possible to evaluate the thermal diffusivity a .

1.2.4. The quadrupole method

In the following, we will note:

- $\theta(x, p)$ the Laplace transform of the temperature $T(x, t)$ (see Appendix 4).
- $\Phi(x, p)$ the Laplace transform of the heat flow rate $\varphi(x, t)$.

In the following, we will explain the principle of quadrupole modeling and demonstrate the expressions of the most common quadrupole matrices. The expressions of these matrices for other configurations of interest are given in Appendix 7 and [MAI 00].

1.2.4.1. Unidirectional transfer in plane walls

1.2.4.1.1. Single wall

We consider the case of a unidirectional heat transfer in a wall of thickness e , with zero initial temperature: $T(x, 0) = 0$.

The temperature $T(x, t)$ within the wall satisfied the equation: $\frac{\partial^2 T}{\partial x^2} = \frac{1}{a} \frac{\partial T}{\partial t}$ [1.59]

By applying the Laplace transformation to equation [1.59], we obtain:

$$\frac{d^2\theta}{dx^2} = \frac{p}{a}\theta \quad [1.60]$$

Equation [1.60] takes a solution in the form:

$$\theta(x, p) = k_1 \cosh(qx) + k_2 \sinh(qx) \quad [1.61]$$

where: $q = \sqrt{\frac{p}{a}}$

The Laplace transform of the heat flow rate at any point of the wall is written as:

$$\Phi(x, p) = L \left[-\lambda S \frac{\partial T}{\partial x}(x, t) \right] = -\lambda S L \left[\frac{\partial T}{\partial x}(x, t) \right] = -\lambda S \frac{d\theta}{dx}(x, p)$$

This equation makes it possible to express $\Phi(x, p)$ as a function of k_1 , k_2 and x :

$$\Phi(x, p) = -\lambda S q k_1 \sinh(qx) - \lambda S q k_2 \cosh(qx) \quad [1.62]$$

Equations [1.61] and [1.62] can be written for $x = 0$ and $x = e$, in which case we obtain:

$$\begin{aligned} \theta(0, p) &= k_1 & \theta(e, p) &= k_1 \cosh(qe) + k_2 \sinh(qe) \\ \Phi(0, p) &= -\lambda S q k_2 & \Phi(e, p) &= -\lambda S q k_1 \sinh(qe) - \lambda S q k_2 \cosh(qe) \end{aligned}$$

It is possible to eliminate k_1 and k_2 from these four equations, which amounts, for example, to expressing $(\theta(0), \Phi(0))$ as a function of $(\theta(e), \Phi(e))$, in which case we obtain:

$$\begin{bmatrix} \theta(0, p) \\ \Phi(0, p) \end{bmatrix} = \begin{bmatrix} \cosh(qe) & \frac{1}{\lambda q S} \sinh(qe) \\ \lambda q S \sinh(qe) & \cosh(qe) \end{bmatrix} \begin{bmatrix} \theta(e, p) \\ \Phi(e, p) \end{bmatrix} \quad [1.63]$$

$$M = \begin{bmatrix} \cosh(qe) & \frac{1}{\lambda q S} \sinh(qe) \\ \lambda q S \sinh(qe) & \cosh(qe) \end{bmatrix} \text{ is called the quadrupole matrix.}$$

We have the property: $\det(M) = 1$, which makes it possible to establish the reciprocal relation:

$$\begin{bmatrix} \theta(e, p) \\ \Phi(e, p) \end{bmatrix} = \begin{bmatrix} \cosh(qe) & -\frac{1}{\lambda q S} \sinh(qe) \\ -\lambda q S \sinh(qe) & \cosh(qe) \end{bmatrix} \begin{bmatrix} \theta(0, p) \\ \Phi(0, p) \end{bmatrix}$$

An analogy can also be made between the propagation of an unsteady state current and the unidirectional thermal transfer in transitory mode:

Intensity of the electric current $I \rightarrow$ Heat flow rate in Laplace space $\Phi(x, p)$

Electrical potential $U \rightarrow$ Temperature in Laplace space $\theta(x, p)$

Electrical impedance $Z \rightarrow$ Thermal impedance Z

Ohm's law: $U_1 - U_2 = ZI$ is analogous to: $\theta_1 - \theta_2 = Z\Phi$

Kirchhoff's law: $\sum I = 0$ is analogous to: $\sum \Phi = 0$

By means of these notations, the quadrupole relation [1.63] can be represented by the equivalent electrical network of Figure 1.23.

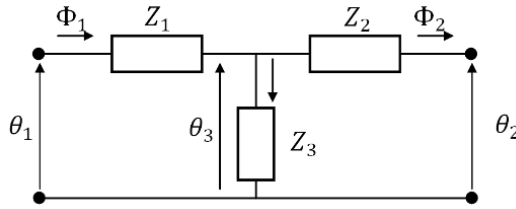


Figure 1.23. *Equivalent electrical network to a single wall in unsteady state*

In the case of a plane wall:

$$Z_1 = Z_2 = \frac{\cosh(qe)-1}{\lambda q S \sinh(qe)} \quad \text{and} \quad Z_3 = \frac{1}{\lambda q S \sinh(qe)}$$

1.2.4.1.2. Wall with a convective exchange

We consider the case of a wall exchanging heat by convection with a fluid (see Figure 1.24).

Equation $\varphi = hS[T_\infty - T(0, t)]$ can also be written as: $T_\infty = \frac{\varphi}{hS} + T(0, t)$ which can be translated in the Laplace space by: $\theta_\infty = \frac{\Phi}{hS} + \theta(x = 0)$.

We can therefore write in quadrupole matrix form:

$$\begin{bmatrix} \theta_\infty \\ \Phi_\infty \end{bmatrix} = \begin{bmatrix} 1 & \frac{1}{hS} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \theta(0, p) \\ \Phi(0, p) \end{bmatrix} \quad [1.64]$$

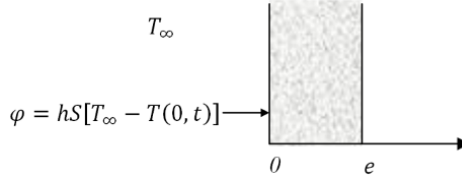


Figure 1.24. *Diagram of a single wall with convective transfer*

The quadrupole relation [1.64] can be represented by the equivalent electrical diagram of Figure 1.25.

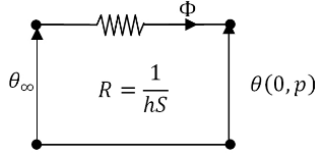


Figure 1.25. *Equivalent electrical network to a convective transfer in unsteady state*

1.2.4.1.3. Contact resistance between two walls

Consider now the case of the heat transfer through a contact resistance R at the interface between two solid media as shown in Figure 1.26.

The heat flow rate is written as $\varphi = \frac{T_{1(x=0)} - T_{2(x=0)}}{R}$ and can also be written as: $T_{1(x=0)} = T_{2(x=0)} + R\varphi$ that we can translate in the Laplace space by:

$$\theta_{1(x=0)} = \theta_{2(x=0)} + R\Phi.$$

We can therefore write in quadrupole matrix form:

$$\begin{bmatrix} \theta_1 \\ \Phi_1 \end{bmatrix} = \begin{bmatrix} 1 & R \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \theta_2 \\ \Phi_2 \end{bmatrix} \quad [1.65]$$

1.2.4.1.5. Semi-infinite medium

It has been demonstrated in the preceding section that the temperature in Laplace space of a semi-infinite medium is written as:

$$\theta(x, p) = A e^{-qx} \quad \text{where: } q = \sqrt{\frac{p}{a}} = \sqrt{\frac{\rho c p}{\lambda}}.$$

We deduce the value of the Laplace transform of the heat flow rate at a point of the semi-infinite medium as:

$$\Phi(x, p) = -\lambda S \frac{d\theta}{dx}(x, p) = \lambda q S A e^{-qx} = \lambda q S \theta(x, p)$$

Φ can therefore also be written as:

$$\Phi = \lambda q S \theta = \lambda \sqrt{\frac{\rho c p}{\lambda}} S \theta = \sqrt{\lambda \rho c} S \sqrt{p} \theta = E S \sqrt{p} \theta$$

where: E is the thermal effusivity.

We can therefore write at every point of a semi-infinite medium:

$$\begin{bmatrix} \theta \\ \Phi \end{bmatrix} = \begin{bmatrix} 1 & \frac{1}{ES\sqrt{p}} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} 0 \\ \Phi \end{bmatrix} = \begin{bmatrix} \theta \\ ES\sqrt{p}\theta \end{bmatrix} \quad [1.66]$$

The quadrupole equation [1.66] can be represented by the equivalent electrical diagram of Figure 1.28.

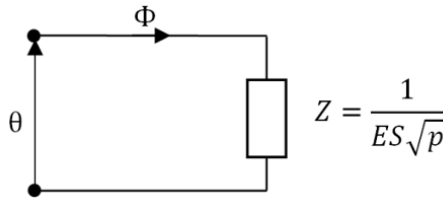


Figure 1.28. Equivalent electrical network to a semi-infinite medium in unsteady state

1.2.4.1.6. Wall at uniform temperature

In the case of a “thin system”, a wall whose thickness and/or thermal conductivity make it possible to consider its temperature as uniform ($Bi < 0.1$, small body hypothesis), the difference between the incoming heat flow rate and the outgoing heat flow rate leaving the system is simply written as:

$$\varphi_1 - \varphi_2 = \rho c V \frac{dT}{dt} \text{ or by applying the Laplace transform: } \Phi_1 - \Phi_2 = \rho c V p \theta.$$

This equation can be expressed in quadrupole form as:

$$\begin{bmatrix} \theta_1 \\ \Phi_1 \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ \rho c V p & 1 \end{bmatrix} \begin{bmatrix} \theta_2 \\ \Phi_2 \end{bmatrix} \quad [1.67]$$

The quadrupole equation [1.67] can be represented by the equivalent electrical network of Figure 1.29.

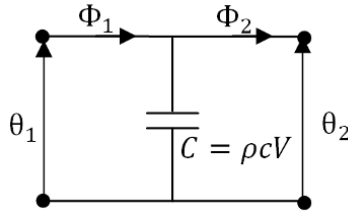


Figure 1.29. *Equivalent electrical network to a medium at uniform temperature in unsteady state*

1.2.4.2. Radial transfer

1.2.4.2.1. Hollow cylinder

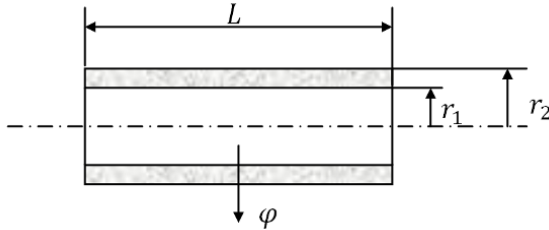


Figure 1.30. *Diagram of a hollow cylinder*

It is shown in the same manner as previously demonstrated [MAI 00] that the temperatures and fluxes in the Laplace space can be connected by a quadrupole equation:

$$\begin{bmatrix} \theta(r_1, p) \\ \Phi(r_1, p) \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta(r_2, p) \\ \Phi(r_2, p) \end{bmatrix}$$

$$A = qr_2[K_1(qr_2)I_0(qr_1) + K_0(qr_1)I_1(qr_2)]$$

$$B = \frac{1}{2\pi\lambda L} [K_0(qr_1)I_0(qr_2) - K_0(qr_2)I_0(qr_1)] \quad [1.68]$$

$$C = 2\pi L \rho c r_1 r_2 p [K_1(qr_1)I_1(qr_2) - K_1(qr_2)I_1(qr_1)]$$

$$D = qr_1[K_1(qr_1)I_0(qr_2) + K_0(qr_2)I_1(qr_1)]$$

I_0 , I_1 , K_0 and K_1 are Bessel's functions (see Appendix 8). The determinant of the quadrupole matrix is equal to 1.

1.2.4.2.2. Semi-infinite hollow cylinder

As in the case of the plane wall, we show that we can write at any point of a semi-infinite hollow cylinder ($r_2 \rightarrow \infty$) [MAI 00]:

$$\begin{bmatrix} \theta \\ \Phi \end{bmatrix} = \begin{bmatrix} 1 & Z \\ 0 & 1 \end{bmatrix} \begin{bmatrix} 0 \\ \Phi \end{bmatrix} = \begin{bmatrix} \theta \\ 2\pi\lambda L \frac{qr_1 K_1(qr_1)}{K_0(qr_1)} \theta \end{bmatrix} \quad [1.69]$$

where $Z = \frac{K_0(qr_1)}{2\pi\lambda L qr_1 K_1(qr_1)}$

The quadrupole equation [1.69] can be represented by the equivalent electrical network of Figure 1.31.

APPLICATION.— *Modeling of the hot wire method.*

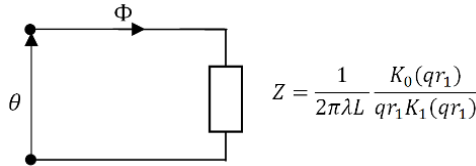


Figure 1.31. *Equivalent electrical network to a semi-infinite medium in unsteady state*

1.3. The thermal properties of a material

In the following, we will recapitulate the various characteristic quantities of a material that have appeared in the heat transfer equations.

1.3.1. Thermal conductivity

The thermal conductivity λ characterizes the resistance to the passage of heat; it is expressed in $\text{W m}^{-1} \text{K}^{-1}$. It is the only thermal property involved in steady-state equations. The values for the most common materials can be found in Appendix 1. In the case of solids, it varies from $0.014 \text{ W m}^{-1} \text{K}^{-1}$ for the superinsulators to $400 \text{ W m}^{-1} \text{K}^{-1}$ for copper ($1500 \text{ W m}^{-1} \text{K}^{-1}$ for diamond). It should be noted that electrical insulators may have higher thermal conductivities than electrical conductors (we have ceramics that have a value of $50 \text{ W m}^{-1} \text{K}^{-1}$ and some stainless steels with a value of $15 \text{ W m}^{-1} \text{K}^{-1}$). For multi-constituent media, it must be known that thermal conductivity is not an additive quantity. There exist a very large number of models of equivalent conductivity to predict the thermal conductivity of a multi-constituent medium as a function of the thermal conductivities of each of the constituents. We will simply present the main ones here. We start with the parallel and series models, since the lowest possible value of the thermal conductivity is given by the series model and the highest by the parallel model [WIE 12]. We will assume in the following that the medium consists of N components of conductivity λ_i and each occupying the volume fraction ε_i .

1.3.1.1. Parallel model

In this model, the different constituents are assumed to be arranged in parallel layers, and the heat flux is parallel to the layers as shown in Figure 1.32.

The equivalent thermal conductivity is given by:

$$\lambda = \sum_{i=1}^N \varepsilon_i \lambda_i \quad [1.70]$$

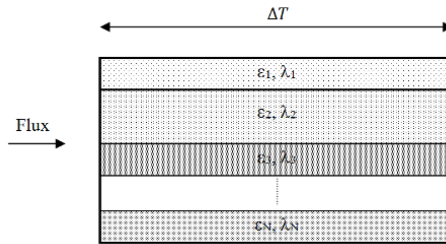


Figure 1.32. Diagram representing the parallel model

The parallel model represents the thermal conductivity of light insulating materials.

1.3.1.2. Series model

In this model, the different constituents are assumed to be arranged in parallel layers, and the heat flux is perpendicular to the layers as shown in Figure 1.33.

The equivalent thermal conductivity is given by:

$$\lambda = \frac{1}{\sum_{i=1}^N \frac{\varepsilon_i}{\lambda_i}} \quad [1.71]$$

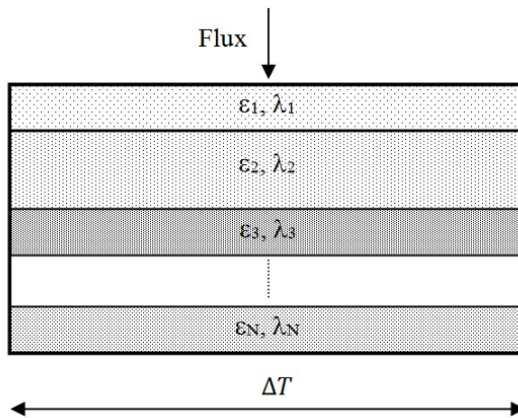


Figure 1.33. Diagram representing the series model

1.3.1.3. Maxwell's model

This simple model is an exact representation of the thermal conductivity of a continuous medium (0) containing spherical particles (1) sufficiently far apart to be able to neglect their mutual interactions [MAX 54]. The thermal conductivity is then given by:

$$\lambda = \frac{\varepsilon_0 \lambda_0 (2\lambda_0 + \lambda_1) + 3\varepsilon_1 \lambda_0 \lambda_1}{\varepsilon_0 (2\lambda_0 + \lambda_1) + 3\varepsilon_1 \lambda_0} \quad [1.72]$$

This model represents well the thermal conductivity of a medium containing inclusions such as lightened concretes, for example.

1.3.1.4. Bruggeman's model

Bruggeman's model [DEV 52] is constructed by adding "progressively" ellipsoidal inclusions of the dispersed phase (1) to the continuous medium (0). The equivalent thermal conductivity λ is deduced from:

$$\left(\frac{\lambda}{\lambda_0}\right)^{A_0} \left(\frac{\lambda_1 - \lambda_0}{\lambda_1 - \lambda}\right) = (1 - \varepsilon_1)^{\frac{1}{3}} \quad [1.73]$$

where ε_1 is the volume fraction of the inclusions and $A_0 = \frac{1-e^2}{e^3} \left[\frac{1}{2} \ln \left(\frac{1+e}{1-e} \right) - e \right]$ with: $e = \sqrt{1 - \frac{b^2}{a^2}}$, a being the semi-major axis and b the semi-minor axis of the ellipsoids.

This model makes it possible to correctly represent the conductivity of a medium containing inclusions (solid or gaseous), for volume fraction values of inclusions higher than those acceptable by Maxwell's model.

1.3.1.5. Special case of a porous medium

A porous medium consists of a hollow solid matrix delimiting pores filled with a gas. The pores are most often assimilated to cylinders of diameter D_i . The conductivity λ_{D_i} of the air contained in the pores depends on the diameter of the pore and the gas pressure according to the equation [LIT 96]:

$$\lambda_{D_i} = \frac{\lambda_0}{1 + C \frac{T}{P D_i}} \quad [1.74]$$

where: λ_0 is the air conductivity ($0.026 \text{ W m}^{-1} \text{ K}^{-1}$ at 300 K);

T is the air temperature (K);

P is the air pressure (Pa);

C is the constant ($6.8 \times 10^{-5} \text{ Pa m K}^{-1}$).

The conductivity of the air at atmospheric pressure in pores with a diameter of $1 \mu\text{m}$ is equal to $0.021 \text{ W m}^{-1} \text{ K}^{-1}$, and only $0.008 \text{ W m}^{-1} \text{ K}^{-1}$ in pores of diameter

100 nm. A material containing a high proportion of nanopores can therefore be superinsulating, i.e. have a thermal conductivity that is lower than that of air at atmospheric pressure (or $\lambda < 0.026 \text{ W m}^{-1} \text{ K}^{-1}$).

1.3.2. Thermal diffusivity

The thermal diffusivity α is expressed in $\text{m}^2 \text{ s}^{-1}$. It characterizes the rate at which a heat flux diffuses into a material. For solids, it varies from $5 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$ for certain rubbers to $3 \times 10^{-4} \text{ m}^2 \text{ s}^{-1}$ for diamond.

It is important to note that it is not correlated with the thermal conductivity: a low conductive material can be very diffusive. For example, air with a low thermal conductivity: $\lambda = 0.026 \text{ W m}^{-1} \text{ K}^{-1}$ has a thermal diffusivity $\alpha = 2,1 \cdot 10^{-5} \text{ m}^2 \text{ s}^{-1}$ which is identical to that of iron with a much higher thermal conductivity: $\lambda = 73 \text{ W m}^{-1} \text{ K}^{-1}$. The thermal diffusivity makes it possible to estimate the time constant of a material of thickness e with:

$$\tau = \frac{e^2}{\alpha} \quad [1.75]$$

The time constant is the time taken to reach a steady state when it exists.

1.3.3. Volumetric heat capacity

It is noted that in all the equations for the heat transfer in solids, the quantities ρ and c never appear separately but only in the form of their product ρc volumetric heat capacity expressed in $\text{J m}^{-3} \text{ K}^{-1}$. This quantity represents the ability of a material to store heat. For solids, it varies from $10^4 \text{ J m}^{-3} \text{ K}^{-1}$ for low density aerogels to $4 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$ for certain steel.

This magnitude is additive for multi-constituent materials:

$$\rho c = \sum_{i=1}^N \varepsilon_i \rho_i c_i \quad [1.76]$$

1.3.4. Thermal effusivity

The thermal effusivity E characterizes the transitory variation in the temperature rise of a surface subjected to a heat flux. It is expressed in $\text{W m}^{-2} \text{ K}^{-1} \text{ s}^{1/2}$. For solids, it varies from $35 \text{ W m}^{-2} \text{ K}^{-1} \text{ s}^{1/2}$ for low density aerogels to $3.6 \times 10^4 \text{ W m}^{-2} \text{ K}^{-1} \text{ s}^{1/2}$ for certain steels.

1.3.5. Conclusion

Although four parameters characterizing the heat transfer within a material have been defined, it is sufficient to measure two parameters, since these parameters are linked by the following equations:

$$a = \frac{\lambda}{\rho c} \quad [1.77]$$

and

$$E = \sqrt{\lambda \rho c} \quad [1.78]$$

Tools and Methods for Thermal Characterization

2.1. Measurement of temperature

Temperature is often considered to be an easily measurable quantity. It is important to remember the following fact in order to avoid over-optimism: “The most accurate thermometer measures only its own temperature”. The causes of uncertainty are mostly less related to the accuracy of the temperature sensor than to the difference between the temperature to be measured and that of the temperature sensor that is used. In order to achieve a precise temperature measurement, it is necessary to combine at least two conditions:

- the heat exchange between the sensor and the area where we want to measure the temperature (surface area of a solid, gas, liquid) must be very large compared to any heat exchanges between the sensor and other surrounding elements: fluid in contact with the sensor if a surface temperature is measured, a radiation source when measuring a gas temperature, for example;

- the presence of the sensor must not modify the temperature field compared to that observed in the absence of a sensor.

2.1.1. Liquid column thermometer

Linear expansion of a liquid as a function of temperature is used. These thermometers are virtually no longer used in thermal metrology.

2.1.2. Thermocouple

The Seebeck effect is used: when the two junctions of two wires of different nature are brought to different temperatures, an electrical potential difference appears, which increases with the difference in temperature (see Figure 2.1).

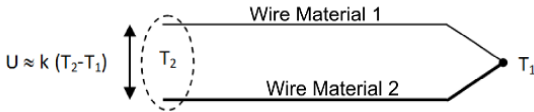


Figure 2.1. Schematic diagram of a thermocouple

For each type of thermocouple, tables give the voltage $U(T)$ delivered when the cold junction is at 0°C and the hot junction at T . The function $U(T)$ can be linearized over reduced temperature intervals as shown in Table 2.1.

The main types of thermocouples and the temperature range in which they can be used are as follows:

Type T:	– 185 to + 300°C	Copper–Constantan
Type K:	0 to 1100°C	Nickel–Nickel Chrome
Type R:	0 to 1600°C	Platinum–Platinum Rhodium
Type G:	20 to 2360°C	Tungsten–Tungsten Rhenium

In the unsteady methods of measuring thermal properties, knowledge of the absolute temperature is of no interest, it is only necessary to measure a rise in temperature between two instants. A reference temperature can be constituted by making two blocks of very effusive material (copper, aluminum, for example), so that their temperatures can be considered constant and uniform during the measurement, given their high heat capacity (see Figure 2.2). A hole of small diameter is drilled in each block and the junction between each wire of the thermocouple and two copper wires is made in the holes in the two blocks. Thus, at the terminals of the two copper wires, we measure the voltage at the terminals of a thermocouple whose cold junction is at the (constant) temperature of the blocks and whose hot junction is at the temperature to be measured. The temperature variation can be deduced by assimilating the temperature curve $= f(\text{voltage})$ to a straight line over small temperature intervals. It is imperative to insulate the blocks electrically (surround them with tape or insulating varnish) and preferably to insulate them thermally (encircle them with polystyrene, for example).

It will be noted, however, that the cold junction compensation achieved in the current thermocouple temperature measuring devices has reached very good stability and can now be used directly.

Real values		Linear model				Parabolic model			
		$U(T) = 0.04189 T$				$U(T) = 4.147 \times 10^{-5} T^2 + 0.003832 T$			
T	$U(T)$	$U(T)_{calc}$	ΔU	T_{calc}	ΔT	$U(T)_{calc}$	ΔU	T_{calc}	ΔT
°C	mV	mV	mV	°C	°C	mV	mV	°C	°C
0	0	0	0	0	0	0	0	0	0
10	0.391	0.419	0.0279	9.33	-0.67	0.387	-0.00365	10.09	0.09
20	0.789	0.838	0.0488	18.84	-1.16	0.783	-0.00601	20.15	0.15
30	1.196	1.257	0.0607	28.55	-1.45	1.187	-0.00908	30.22	0.22
40	1.611	1.676	0.0646	38.46	-1.54	1.599	-0.01185	40.28	0.28
50	2.035	2.095	0.0595	48.58	-1.42	2.020	-0.01532	50.36	0.36
60	2.467	2.513	0.0464	58.89	-1.11	2.448	-0.01851	60.43	0.43
70	2.908	2.932	0.0243	69.42	-0.58	2.886	-0.02240	70.51	0.51
80	3.357	3.351	-0.0058	80.14	0.14	3.331	-0.02599	80.58	0.58
90	3.813	3.770	-0.0429	91.02	1.02	3.785	-0.02829	90.62	0.62
100	4.277	4.189	-0.088	102.10	2.10	4.247	-0.03030	100.65	0.65

Table 2.1. Value of $U(T)$ for a T -type thermocouple (Copper–Constantan)

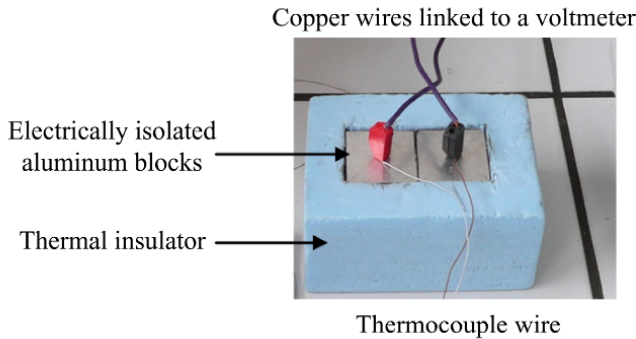


Figure 2.2. Device for making a junction at a constant temperature

The welding of two wires to produce a thermocouple can be carried out using a small spot welder, and for wires of small diameter it is sufficient to just twist them. In this case, the measured temperature is that of the last point of contact between the two wires.

The measurement of a surface temperature is difficult because of the imperfect contact between the weld (and twist) of the wires' extremities and the surface whose temperature is to be measured. There exists always a thermal contact resistance between the two which renders the measurement less accurate. It is preferable to realize a separate contact by applying the extremities of the two wires of the thermocouple on the surface at a distance of a few mm (by means of a piece of adhesive tape if the temperature permits). The electrical contact is then realized by the surface itself, provided that it is conductive (if it is not conductive, then it is covered with a thin layer of conductive silver lacquer). The mean of the temperatures of the two contact points is thus measured by ensuring that there is no contact resistance; the existence of a contact defect is easily detected because it leads to a zero voltage at the extremities of the thermocouple (open circuit).

During measurements in rapid transient mode, it is important that the thermocouple has the lowest possible response time. The thinner the wires, the lower the response time, therefore it is always advantageous to use the thinnest possible wires. Wires with a diameter of less than 0.1 mm on a coil and having a response time of less than 0.1 s are often used.

It will also be advantageous to orient the wires in the direction where the thermal gradient is weakest so as to minimize the fin effects in the wires. For this reason, the type T thermocouple with one of the wires made of copper (very conductive) must be avoided, preferring instead the type K thermocouple.

2.1.3. Thermistor

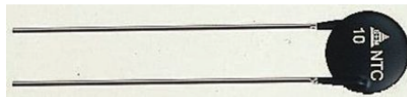


Figure 2.3. *View of a thermistor*

A thermistor is a passive component of semiconductor material. If self-heating by the Joule effect is negligible, its resistance varies with the temperature according to the law:

$$R(T) = R(T_0) \exp \left[B \left(\frac{1}{T} - \frac{1}{T_0} \right) \right] \quad [2.1]$$

Temperatures are expressed in degrees Kelvin, B and T_0 are characteristic constants of the component. As the resistance decreases with temperature, we get what we sometimes call thermistor resistances NTC (for negative temperature coefficient).

The current–voltage characteristic presents, for the weak currents, a linear part, then a plateau, and finally, for the more intense currents, a zone with negative slope corresponding to component's self-heating.

The range of use is -50°C to 200°C .

2.1.4. Platinum resistance

A platinum resistance in the form of a wire whose resistance increases quasilinearly with temperature is used, for example for a resistance Pt100 we have:

$$R(T) = 100 + 0.3864 T \quad [2.2]$$

where: R is the resistance (Ω);

T is the temperature ($^{\circ}\text{C}$).

T	R_{Pt}	$R_{Pt\,calc}$	ΔR	T_{calc}	ΔT
$^{\circ}\text{C}$	Ω	Ω	Ω	$^{\circ}\text{C}$	$^{\circ}\text{C}$
0	100	100.00	0.00	0.00	0.00
5	101.95	101.93	0.02	5.05	0.05
10	103.9	103.86	0.04	10.09	0.09
15	105.85	105.80	0.05	15.14	0.14
20	107.79	107.73	0.06	20.16	0.16
25	109.73	109.66	0.07	25.18	0.18
30	111.67	111.59	0.08	30.20	0.20
35	113.61	113.52	0.09	35.22	0.22
40	115.54	115.46	0.08	40.22	0.22
45	117.47	117.39	0.08	45.21	0.21
50	119.4	119.32	0.08	50.21	0.21
55	121.32	121.25	0.07	55.18	0.18
60	123.24	123.18	0.06	60.14	0.14
65	125.16	125.12	0.04	65.11	0.11
70	127.08	127.05	0.03	70.08	0.08
75	128.99	128.98	0.01	75.03	0.03
80	130.9	130.91	-0.01	79.97	-0.03
85	132.8	132.84	-0.04	84.89	-0.11
90	134.71	134.78	-0.07	89.83	-0.17
95	136.61	136.71	-0.10	94.75	-0.25
100	138.51	138.64	-0.13	99.66	-0.34

Table 2.2. Resistance value of a platinum wire as a function of temperature

The temperature range in which they can be used is from -200°C to $+850^{\circ}\text{C}$.

2.1.5. IR detector

These are optical detectors (photovoltaic) of a few mm² which generate a current when exposed to infrared radiation. They are placed close to the surface whose temperature is to be measured and deliver a signal proportional to the surface temperature if the temperature deviations are small (possible linearization of the radiation flux proportional to T^4). Their advantage is to allow measurement without contact and therefore without disturbance of the surface whose temperature is to be measured. They also have a practically zero response time, which makes it possible to study transient phenomena of a few ms. They need to be maintained at the lowest possible constant temperature by using liquid nitrogen, a Stirling engine or Peltier elements, and their cost is relatively high. Figure 2.4 shows some examples.

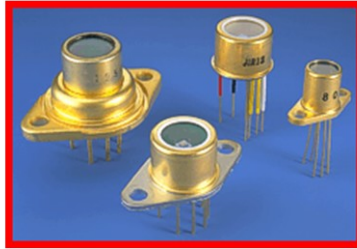


Figure 2.4. View of an IR detector: indium antimonide (InSb) photodiodes sensitive in the wavelength region from 1 to 5.5 μm

2.1.6. IR camera

An infrared camera consists of a matrix of photonic detectors sensitive to radiation emitted in the mid-IR (1.5–20 μm). It therefore does not measure the temperature T_s of a surface but instead the flux received by the detectors. This flux expressed in digital level (DL: digital level) is represented by the following equation if we consider the atmosphere to be perfectly transparent with respect to IR radiation:

$$DL_{mes} = \varepsilon DL(T_s) - (1 - \varepsilon) DL(T_r) \quad [2.3]$$

where: T_s is the target surface temperature;

ε is the surface emissivity;

T_r is the environment temperature.

We thus deduce:

$$DL(T_s) = \frac{1}{\varepsilon} [DL_{mes} - (1 - \varepsilon) DL(T_r)] \quad [2.4]$$

To obtain T_s from DL_{mes} , we must therefore:

know the emissivity of the surface ε ;

know the function $DL(T)$ which is obtained by a calibration.

Calibration is carried out on experimental black bodies whose temperature can be regulated uniformly and precisely.

We note that if $\varepsilon = 1$, then $DL(T_s) = DL_{mes}$. The emitting surface is therefore generally covered with a thin layer of matte black paint of emissivity close to the unit, provided that the presence of paint does not modify the temperature field that needs to be measured. It is observed that the measurement of the absolute value of the temperature using an IR camera remains difficult to achieve with precision.

However, a number of thermal characterization methods do not require knowledge of the absolute value of the temperature but only a quantity proportional to a temperature difference (between two points or between two instants): case of temperature/temperature methods or flash method, for example. In this type of application, the IR camera is a perfect tool.

In fact, if ΔDL_{mes} is the difference of the measured DL corresponding to two temperatures T and $T + \Delta T$ at two points or at two different times and if T_r is constant, we have:

$$DL(T + \Delta T) - DL(T) = \frac{1}{\varepsilon} \Delta DL_{mes}$$

On a temperature range of low amplitude ($<10^\circ\text{C}$), the curve $DL = f(T)$ can be assimilated to a straight line of slope K and we then have:

$$\Delta T = K \Delta DL_{mes}$$

If the amplitude of the temperature range is more extensive, a calibration curve is used, an example of which is shown in Figure 2.5 [ALH 16].

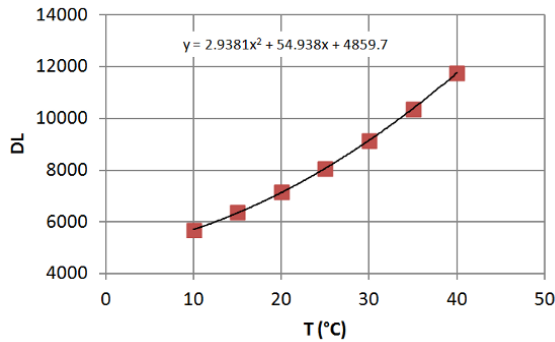


Figure 2.5. Calibration curve of an IR camera using a black body

An IR camera can also measure the distance between two points whose temperatures are measured. Care must be taken because phenomena such as distortion due to optical imperfections of the lens may cause the distance between two pixels not to be the same at all points of the image. It is therefore advantageous to measure the number of pixels corresponding to a known distance (using a ruler, for example) at the very place of the measurement.

It should be noted that these cameras make it possible to measure not a point temperature but a field of a parameter proportional to the temperature. The amount of information to be processed becomes very large and requires the use of high-performance signal processing techniques. The applications are numerous [MAI 10]: detection of defects in plates, field measurement of thermal diffusivity. An example is shown in Figure 2.6.



Figure 2.6. View of an infrared camera: LWIR resolution of 640×480 pixels, low noise < 20 mK, thermal measurements up to 2000°C and video recording at 30 fps

2.1.7. Choice of a measurement method

The choice of a method for measuring the temperature depends on several parameters among which are:

- the frequency at which it is desired to carry out the measurement;
- the geometric configuration: measurement of a surface or internal temperature;
- the nature of the material whose temperature is to be measured: thermal conductor or insulator;
- the type of measurement to be carried out: absolute temperature or temperature increase with respect to a reference.

Table 2.3 presents a summary of the recommendations for choosing a method for measuring the temperature as a function of the measurement conditions. It will be noted that the conductive materials often have a low emissivity and that it is necessary to cover them with an emitter coating in order to be able to measure their temperature by optical means. Its thickness will have to be lower the greater the thermal diffusivity of the material to be coated for transient measurements.

Temperature to be measured		Internal liquid or solid			Conductive surface + Emitter coating			Insulating surface		
Measurement type	Frequency (Hz)	<10	> 10 < 1000	> 1000	<10	> 10 < 1000	> 1000	<10	> 10 < 1000	> 1000
Absolute	Average	S1	S3		S7	S7	S7	S7	S7	S7
	Point	S3	S3		S3, S7	S3, S7	S7	S7	S7	S7
	Field				S5	S5	S5	S5	S5	S5
Variation	Average	S1	S2		S6	S6	S6	S6	S6	S6
	Point	S2	S2		S2, S6	S2, S6	S6	S6	S6	S6
	Field				S4	S4	S4	S4	S4	S4

Table 2.3. Ideal measurement methods depending on the measurement conditions

In Table 2.3:

- S1 platinum resistance;
- S2 thermocouple;
- S3 thermocouple + cold junction measuring device;

S4 IR camera;

S5 IR camera + calibration with black body;

S6 mono optical sensor;

S7 mono optical sensor + calibration with black body.

The following remarks will also be taken into account:

- When measuring the temperature of a gas, it is imperative to protect the sensor from radiation by interposing a perforated screen or by covering the surface with a reflecting material such as aluminum foil. It will also be advantageous to increase convective heat transfer with forced ventilation. It is nonetheless true that the measurement of the temperature of a gas remains delicate in the presence of large amounts of radiation (from walls at high temperature or from the Sun, for example); see examples given in Appendix A.9.

- The measurement of the surface temperature of insulating materials is very difficult to realize:

- they are porous and therefore semi-transparent and an optical measurement may be biased because part of the radiation received by the sensor comes from the material's interior;

- they have a low effusivity and therefore, when they are in contact with thermocouple wires, they are the ones that “impose” their temperature [JAN 16] and the fin effect in the wires may not be negligible.

- The measurement of the surface temperature of metallic materials (highly effusive) by thermocouple is, on the contrary, very precise, and the possibility of welding the thermocouple wires to the surface makes it possible to avoid contact resistances.

2.1.8. Data filtering

Whatever the measuring device, the values of the measured temperatures are always more or less noisy. This is generally the case when measuring with a high acquisition frequency using an IR camera or an optical detector. It may then be tempting to filter the signal using the filtering which is generally proposed by the data acquisition module enabling the recording of the change in temperature.

It should be borne in mind that filtering has two effects:

- it reduces the measurement noise (*a priori* positive effect);
- it shifts the signal in time (negative effect).

The uncertainty due to the measurement noise depends on two parameters:

- the standard deviation of the measurement noise;
- the number of experimental points.

It is therefore possible to process a very noisy signal if the number of points is very high. For example, Figure 2.7 shows the experimental and estimated curves of the temperature as well as the estimation residues of a Flash experiment. Figure 2.7(a) was obtained with filtering at 1 kHz. The number of experimental points of the graph is 40000. It is found that the residues (represented with a shift of 0.05°C for better readability) are “flat” so the model is *a priori* exact. The calculation of the standard deviation of the estimated value due to the measurement noise of the temperature leads to a relatively low value of 0.5%. From the point of view of the estimation’s precision, the filtering of these data is therefore of no interest. This is all the more true as the computing power of the computers makes it possible to process a large number of points in very short calculation times.

The curves obtained with a filtering of 10 kHz (the residuals are shifted by 0.05°C) are shown in Figure 2.7(b). The estimation converges to the same value of the thermal diffusivity (deviation of less than 1.5%) as that estimated with a filtering at 1 kHz.

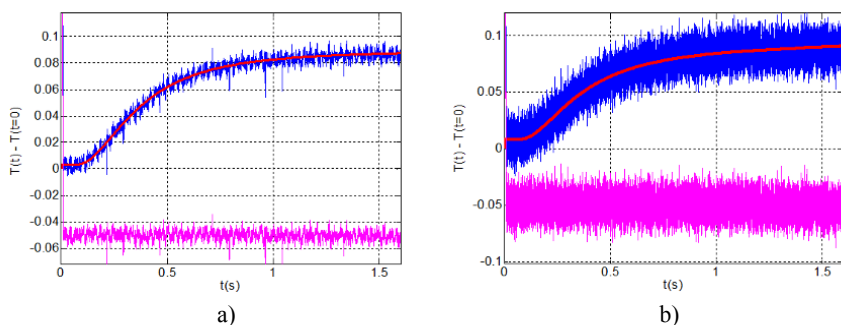


Figure 2.7. Experimental and theoretical thermograms and estimation residues ($\times 10$ and shifted by 0.05°C) of a Flash experiment: (a) filtering at 1 kHz and (b) filtering at 10 kHz. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Data filtering has the effect of shifting the data over time, which may distort the results of estimation. It can be retained as an order of magnitude that the filtering frequency must be greater than $\frac{1000}{t_f}$ (in Hz) where t_f is the duration of the recording used to estimate the parameters. It is possible to use a filtering at 10 Hz for an experiment of duration of 100 s, for example; on the contrary, we will not filter at less than 10 kHz for an experiment of duration of 0.1 s.

2.2. Tools for parameter estimation

2.2.1. Introduction

In this chapter, we present the tools and methods used in the various thermal characterization devices. We will first illustrate the advantage of using a complete model, established in our case, by the application of the thermal quadrupole method. We then present the advantages of carrying out a study of the sensitivity of the measured quantities to the unknown parameters before briefly describing the methods used for estimating the parameters and evaluating the precision of the estimations. We will conclude by presenting the overall methodology chosen for the study of the different methods presented hereafter.

2.2.2. Quadrupole modeling

A number of thermal characterization methods became popular because of the simplicity of the way in which the results were processed, which was a decisive advantage as the calculation methods were more limited than today. In this category, we can classify the hot plate method that allows estimating the thermal effusivity by simple estimation of an experimental temperature curve's slope. The hot wire method for estimating thermal conductivity by a similar method is also included. These simple methods, however, all rely on assumptions that are not always easy to verify such as: negligible inertia of probes, semi-infinite medium. The development of the calculation means now makes it possible to use more complete models using less simplifying hypotheses or makes it possible to verify them more simply.

For example, we will illustrate the advantage of using a complete quadrupole model for the use of thermal measurements of a classical method used by many authors [AND 76, NAG 81, BAC 82, ZHA 93]: the determination of thermal conductivity by the hot wire method. In this method, the temperature evolution of a wire in which a constant heat flux is dissipated is measured, this wire being placed in the material to be characterized.

The vicinity of the heating wire is shown schematically in Figure 2.8.

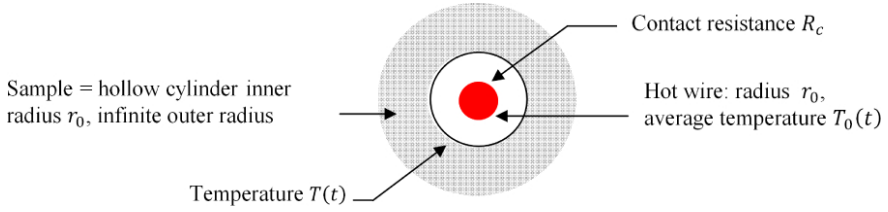


Figure 2.8. Hot wire schematic diagram

The modeling of the system using the quadrupole formalism allows us to write [MAI 00]:

$$\begin{bmatrix} \theta_0 \\ \frac{\varphi_0}{p} \end{bmatrix} = \begin{bmatrix} 1 & \frac{1}{2\pi\lambda_f L q r_0} \frac{I_0(q_f r_0)}{I_1(q_f r_0)} - \frac{1}{\rho c \pi r_0^2 L p} \\ \rho c \pi r_0^2 L p & \frac{q_f r_0}{2} \frac{I_0(q_f r_0)}{I_1(q_f r_0)} \end{bmatrix} \begin{bmatrix} 1 & R_c \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \theta \\ 2\pi\lambda L q r_0 \frac{K_1(q r_0)}{K_0(q r_0)} \theta \end{bmatrix}$$

$$\text{With: } q = \sqrt{\frac{p}{a}} \text{ and } q_f = \sqrt{\frac{p}{a_f}}$$

where: $T_0(t)$ is the average temperature of the hot wire;

$T(t)$ is the temperature of the sample in $r = r_0$;

θ_0 is the Laplace transform of the difference $T_0(t) - T_0(t = 0)$;

θ is the Laplace transform of the difference $T(t) - T_0(t = 0)$;

R_c is the contact resistance at the heating resistance/sample interface;

c is the specific heat of the hot wire;

ρ is the density of the hot wire;

λ is the sample's thermal conductivity;

λ_f is the wire's thermal conductivity;

a is the sample's thermal diffusivity;

α_f is the wire's thermal diffusivity;

p is the Laplace variable;

r_0 is the heating wire's radius;

L is the heating wire's length;

φ_0 is the power dissipated in the wire;

I_0, I_1, K_0, K_1 is Bessel's functions.

$$\text{Where: } \theta_0 = \frac{\varphi_0}{p} \frac{A_0 + (A_0 R_c + B_0)/Z}{C_0 + (C_0 R_c + D_0)/Z} \quad [2.5]$$

$$\text{With: } A_0 = 1; B_0 = \frac{1}{2\pi\lambda_f L q r_0} \frac{I_0(q_f r_0)}{I_1(q_f r_0)} - \frac{1}{\rho c \pi r_0^2 L p}; C_0 = \rho c \pi r_0^2 L p; D_0 = \frac{q_f r_0}{2} \frac{I_0(q_f r_0)}{I_1(q_f r_0)},$$

$$\frac{1}{Z} = 2\pi\lambda L q r_0 \frac{K_1(q r_0)}{K_0(q r_0)}$$

In the classical use of the hot wire method, the hypothesis assumes a thin wire (small r_0) and taking place at "long" times ($p \rightarrow 0$), which allows the use of limited developments of the Bessel functions in the neighborhood of 0:

$$K_0(x) \approx -\ln(x); K_1(x) \approx \frac{1}{x}; I_0(x) \approx 1; I_1(x) \approx \frac{x}{2}$$

$$\text{Leading to: } A_0 = 1; B_0 = 0; C_0 = \rho c \pi r_0^2 L p; D_0 = 1; \frac{1}{Z} = \frac{2\pi\lambda L}{\ln(q r_0)}$$

$$\text{Hence: } \theta_0 = \frac{\varphi_0}{p} \frac{Z + R_c}{mcp(Z + R_c) + 1} \quad [2.6]$$

where m is the mass of the hot wire.

By neglecting the effect of the probe's inertia, the hot wire's temperature is written in Laplace space:

$$\text{Or: } \theta_0 = \frac{\varphi_0}{p} (Z + R_c) \approx \frac{\varphi_0}{p} \left[\frac{\ln\left(r_0 \sqrt{\frac{p}{a}}\right)}{2\pi\lambda L} + R_c \right] \approx \frac{\varphi_0}{p} \left[\frac{\ln(p)}{4\pi\lambda L} - \frac{\ln\left(\frac{r_0}{\sqrt{a}}\right)}{2\pi\lambda L} + R_c \right] \quad [2.7]$$

Using the inverse Laplace transform tables allows us to calculate the temperature $T_0(t)$ at long times:

$$T_0(t) - T_0(0) = \frac{\varphi_0}{4\pi\lambda L} \ln(t) + R_c \varphi_0 - \frac{\gamma \varphi_0}{4\pi\lambda L} - \frac{\varphi_0 \ln\left(\frac{r_0}{\sqrt{a}}\right)}{2\pi\lambda L} \quad [2.8]$$

where $\gamma = 0.57721$ is Euler's constant.

$$\bar{T}_0(t) = T_0(t) - T_0(0) = \frac{\varphi_0}{4\pi\lambda L} \ln(t) + \varphi_0 \left[R_c - \frac{\gamma}{4\pi\lambda L} - \frac{\ln\left(\frac{r_0}{\sqrt{a}}\right)}{2\pi\lambda L} \right] \quad [2.9]$$

Subject to the hypotheses listed above, the $T_0(t) - T_0(0)$ plot as a function of $\ln(t)$ is thus a straight line with slope $\frac{\varphi_0}{4\pi\lambda L}$ whose determination allows estimating the thermal conductivity λ .

The graph in Figure 2.9 represents, for different cases, the deviation (in %) between the slope of the curve $\bar{T}_0(t) = f[\ln(t)]$ calculated between t_0 and $t_0 + 100$ s and the value $\frac{\varphi_0}{4\pi\lambda L}$ obtained for an infinite time. This slope was determined by calculating the temperature $\bar{T}_0(t)$ by inverse Laplace transform of formula [2.5] by the method of Stehfest with 10 terms [STE 70] or of De Hoog [DEH 82], then by derivation with respect to $\ln(t)$. The following values were used in the calculations:

$$\frac{\varphi_0}{L} = 0.5 \text{ W m}^{-1}; \lambda = 0.12 \text{ W m}^{-1} \text{ K}^{-1}; E = 300 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}; R_c = \frac{1}{2\pi r_0 h L};$$

$$\rho c = 2 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$$

The different cases dealt with are as follows:

- a) $r_0 = 0.25$ mm, heating wire of zero mass, zero contact resistance;
- b) $r_0 = 0.25$ mm, $\rho c = 2 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$ and $h = 10000 \text{ W m}^{-2} \text{ K}^{-1}$;
- c) $r_0 = 0.25$ mm, $\rho c = 2 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$ and $h = 5000 \text{ W m}^{-2} \text{ K}^{-1}$;
- d) $r_0 = 0.5$ mm, $\rho c = 2 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$ and $h = 10000 \text{ W m}^{-2} \text{ K}^{-1}$.

The radius values used are of the same order of magnitude as the radius of the hot wire used by some authors. For example, we have noted the use of a 0.7 mm diameter probe to measure the conductivity of fruits and vegetables [LIA 99].

The analysis of Figure 2.9 leads to the following notes concerning the treated case:

– The error due to the “small r_0 ” approximation, represented by curve a) is not negligible: it is of the order of 1 to 2% for the usual operating intervals. This error which increases rapidly with the wire’s diameter (as shown by the difference between curves b) and d) between which only the radius has varied) is due to the approximation:

$$2\pi\lambda L q r_0 \frac{K_1(qr_0)}{K_0(qr_0)} \approx \frac{2\pi\lambda L}{\ln(qr_0)} \quad [2.10]$$

– The error due to the “zero mass probe” approximation leads to errors of several percentage points; this error increases with the contact resistance for the same probe mass as shown by the difference between curves b) and c).

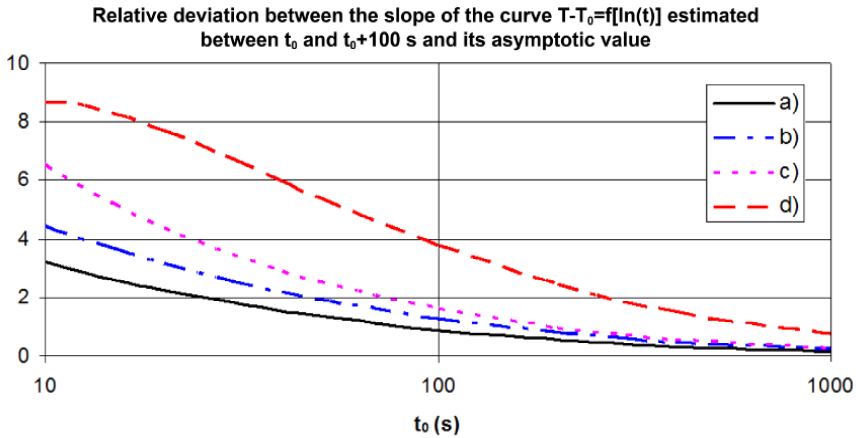


Figure 2.9. Relative deviation (in %) between the slope of the curve $T - T_0 = f[\ln(t)]$ estimated between t_0 and $t_0 + 100$ s and the value $\varphi_0/4\pi\lambda L$. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

The goal of these remarks is not to put a reference method into question in its most common use framework, which is the measurement of thermal properties of materials with heating wires of very small diameter of the order of 0.025 mm [RHA 94], for which the use of simplified formula [2.9] does not induce any significant error. They show, however, that the use of simplified models obtained by limited developments of the expression of temperature in the Laplace space can lead

to non-negligible errors in certain particular cases where probes of larger diameter are used. It is therefore preferable to use a complete model taking into account *inter alia* contact resistances as well as the probe's size and inertia. The application of the quadrupole method makes it possible to establish these complete models in many cases, the Stehfest or De Hoog methods associated with a Newton or Levenberg–Marquardt method of estimating parameters then allows determining the thermal properties with better accuracy than by using a simplified model.

Finally, it will be noted that a “complete” quadrupole model makes it possible to exploit all the points of the thermograms and avoids the often delicate choice of the ideal operating window of the temperature measurements for which the differences between the ideal model and the experimental results are low. This interval is sometimes determined in a rather empirical manner: an estimation of the parameters is carried out over a time interval of constant amplitude whose boundary is varied increasingly from zero. The curves giving the values of the estimated parameters are then plotted as a function of the value of the lower limit of the interval and an interval with a constant value of the parameters is selected as the optimum time interval. The identification of this stage does not seem to be a sufficient guarantee to assert that the estimate is not biased by the failure to take into account the existence of contact resistances and the inertia of the probe and also the existence of a level is not always very noticeable [KUB 00, KUB 02].

2.2.3. Dimensional analysis

We will show from an example what dimensional analysis can bring to the study of a thermal characterization method. Let us consider the case of the flash method shown in Figure 2.10.

The measurement of the temperature $T(t)$ associated with a modeling of the thermal transfers makes it possible to estimate the thermal diffusivity a .

The temperature T depends on the following parameters: $t, a, \lambda, e, q_0, h_1, h_2$.

In this problem, we have eight variables ($T, t, a, \lambda, e, q_0, h_1, h_2$) and four fundamental quantities (L, T, M, θ). We have to choose four variables (basic equations) which cannot give a dimensionless grouping (because we have four fundamental quantities according to the Buckingham π theorem).

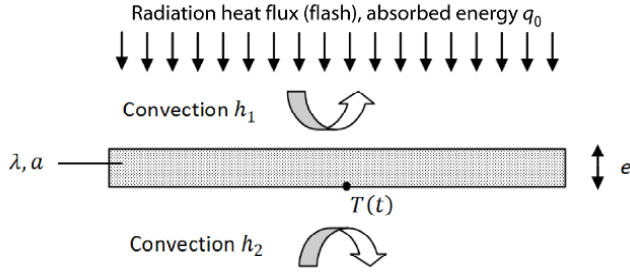


Figure 2.10. Schematic diagram of the flash method

To do this, we must first associate the fundamental quantities with the variables:

– a	$L^2 T^{-1}$
– λ	$M L T^{-3} \theta^{-1}$
– h_1, h_2	$M T^{-3} \theta^{-1}$
– e	L
– q_0	$M T^{-3}$
– t	T
– T	θ

We can choose a , λ , e and q_0 as basic equations and write the remaining four variables T , t , h_1 and h_2 as dimensionless groups:

$$\begin{aligned}
 -\pi_1 &= a^{i_1} \lambda^{j_1} e^{k_1} q_0^{l_1} T \\
 -\pi_2 &= a^{i_2} \lambda^{j_2} e^{k_2} q_0^{l_2} t \\
 -\pi_3 &= a^{i_3} \lambda^{j_3} e^{k_3} q_0^{l_3} h_1 \\
 -\pi_4 &= a^{i_4} \lambda^{j_4} e^{k_4} q_0^{l_4} h_2
 \end{aligned}$$

We still need to write the equation for the dimensions of each group to calculate the values of i , j , k and l by writing that the groups are dimensionless:

$$-[\pi_1] = [L^2 T^{-1}]^{i_1} [M L T^{-3} \theta^{-1}]^{j_1} [L]^{k_1} [M T^{-3}]^{l_1} [\theta]$$

$$\text{Hence: } 2i_1 + j_1 + k_1 = 0; -i_1 - 3j_1 - 3l_1 = 0; j_1 + l_1 = 0; -j_1 + 1 = 0$$

$$\text{Or: } i_1 = 0; j_1 = 1; k_1 = -1; l_1 = -1$$

$$\text{And: } \pi_1 = \frac{\lambda}{q_0 e} T$$

$$-[\pi_2] = [L^2 T^{-1}]^{i_2} [MLT^{-3} \theta^{-1}]^{j_2} [L]^{k_2} [MT^{-3}]^{l_2} [T]$$

$$\text{Hence: } 2i_2 + j_2 + k_2 = 0; -i_2 - 3j_2 - 3l_2 + 1 = 0; j_2 + l_2 = 0; -j_2 = 0$$

$$\text{Or: } i_2 = 1; j_2 = 0; k_2 = -2; l_2 = 0$$

$$\text{And: } \pi_2 = \frac{at}{e^2}$$

$$-[\pi_3] = [L^2 T^{-1}]^{i_3} [MLT^{-3} \theta^{-1}]^{j_3} [L]^{k_3} [MT^{-3}]^{l_3} [MT^{-3} \theta^{-1}]$$

$$\text{Hence: } 2i_3 + j_3 + k_3 = 0; -i_3 - 3j_3 - 3l_3 - 3 = 0; j_3 + l_3 + 1 = 0; -j_3 - 1 = 0$$

$$\text{Or } i_3 = 0; j_3 = -1; k_3 = 1; l_3 = 0$$

$$\text{And: } \pi_3 = \frac{h_1 e}{\lambda}$$

$$-[\pi_4] = [L^2 T^{-1}]^{i_4} [MLT^{-3} \theta^{-1}]^{j_4} [L]^{k_4} [MT^{-3}]^{l_4} [MT^{-3} \theta^{-1}]$$

$$\text{Hence: } 2i_4 + j_4 + k_4 = 0; -i_4 - 3j_4 - 3l_4 - 3 = 0; j_4 + l_4 + 1 = 0; -j_4 - 1 = 0$$

$$\text{Or } i_4 = 0; j_4 = -1; k_4 = 1; l_4 = 0$$

$$\text{And: } \pi_4 = \frac{h_2 e}{\lambda}$$

The solution can therefore take the form:

$$f\left(\frac{\lambda}{q_0 e}, T, \frac{at}{e^2}, \frac{h_1 e}{\lambda}, \frac{h_2 e}{\lambda}\right) = 0$$

The dimensional analysis applied to this case allows us to say, without having solved the equations, that the measurement of the temperature T as a function of time t can *a priori* make it possible to estimate the following groups: $\frac{\lambda}{q_0 e}, \frac{a}{e^2}, \frac{h_1 e}{\lambda}, \frac{h_2 e}{\lambda}$. Since the transfer coefficients h_1 and h_2 are not known precisely, as is the heat flux q_0 absorbed on the heated surface, this method will only make it possible to estimate only the thermal diffusivity a from the identification of the group $\frac{a}{e^2}$.

2.2.4. Study of reduced sensitivity

The principle of reduced sensitivity analysis has been described by several authors [BEC 77, MAI 91, KUR 95, PET 08]. The reduced sensitivity of the temperature with respect to a parameter k_i is equal to $k_i \frac{\partial T}{\partial k_i}$. This quantity represents the variation of T (in °C) induced by a relative variation of k_i of 100 %. The reduced sensitivity thus has the advantage of allowing a direct comparison of the relative influence of the various parameters on the temperature T .

2.2.4.1. Unsteady state

A necessary condition for the separate estimation of the parameters is that the sensitivities of $T(t)$ relative to each of the parameters are decorrelated. This means that there must be no linear relationship of the type:

$$\sum_i C_i \frac{\partial T}{\partial k_i}(t) = 0 \quad [2.11]$$

between the functions $\frac{\partial T}{\partial k_i}(t)$ [BEC 77].

This condition implies that if the sensitivities to two parameters are proportional, then these two parameters cannot be estimated separately.

By way of example, we have calculated the various sensitivities for the temperature T of a hot plate which depends on the parameters:

- E effusivity of the sample;
- R_c contact resistance between the heating element and the sample;
- mc thermal capacity of the heating element.

For the following nominal conditions:

$$E = 500 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}; \quad R_c = 2 \times 10^{-3} \text{ m}^2 \text{ K W}^{-1}; \quad mc = 0.75 \text{ J K}^{-1};$$

$$\frac{\varphi_0}{S} = 500 \text{ W m}^{-2}$$

The Laplace transform of this temperature is written as (see section 4.2.2):

$$\theta(p) = \frac{\varphi_0}{2p} \frac{1 + R_c E S \sqrt{p}}{\frac{mc}{2} p + \left(R_c \frac{mc}{2} p + 1 \right) E S \sqrt{p}} \quad [2.12]$$

The temperature $T(t)$ is deduced by inverse Laplace transformation by applying, for example, the Stehfest method. The different partial derivatives $\frac{\partial T}{\partial k_i}(t)$ can then be computed numerically by:

$$\frac{\partial T}{\partial k_i}(t) = \frac{T(k_0, k_1, \dots, k_i + \epsilon, \dots, k_p, t) - T(k_0, k_1, \dots, k_i, \dots, k_p, t)}{\epsilon} \quad [2.13]$$

Figure 2.11 shows:

- The simulated curve $T(t)$ with these nominal conditions over 500 s.
- The curves $E \frac{\partial T}{\partial E}(t)$, $R_c \frac{\partial T}{\partial R_c}(t)$ and $mc \frac{\partial T}{\partial mc}(t)$ between 0 and 500 s.

And in Figure 2.12 the curves $\frac{E}{R_c} \frac{\partial T}{\partial R_c}(t)$; $\frac{E}{mc} \frac{\partial T}{\partial mc}(t)$ and $\frac{R_c}{mc} \frac{\partial T}{\partial mc}(t)$ between 0 and 500 s.

It is found that the sensitivity at E is strong and uncorrelated to the sensitivities at R_c and at mc because the two ratios $\frac{E}{R_c} \frac{\partial T}{\partial R_c}(t)$ and $\frac{E}{mc} \frac{\partial T}{\partial mc}(t)$ are not constant. It is therefore *a priori* possible to estimate E from the experimental curve $T_{exp}(t)$. It can also be seen that the sensitivities at R_c and at mc are strongly decoupled between 0 and 50 s, so it will be possible to estimate them separately over this time interval. On the contrary, the ratio $\frac{R_c}{mc} \frac{\partial T}{\partial mc}(t)$ varies slightly after 50 s, so it is no longer possible to estimate R_c and mc separately with the portion of the curve $T_{exp}(t)$ corresponding to $t > 50$ s.

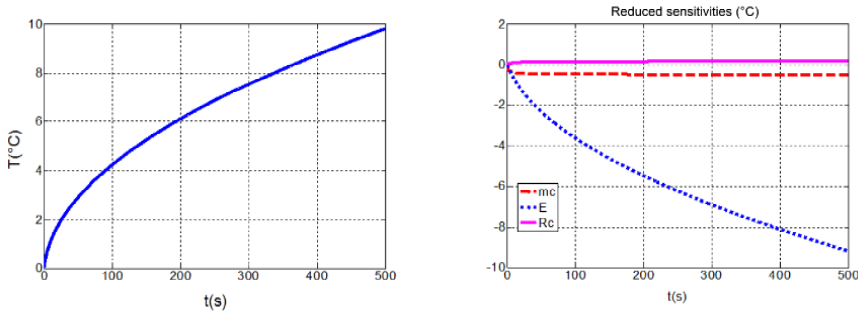


Figure 2.11. Curve $T(t)$ and curves of reduced sensitivity at E , R_c and mc for the hot plate method. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

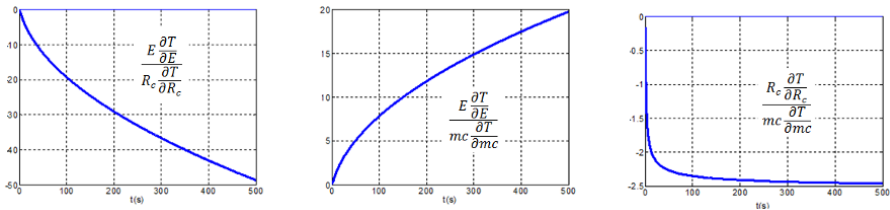


Figure 2.12. Ratio of reduced sensitivity to the three parameters E , R_c and mc as a function of time

In some very particular cases, the two-to-two ratios of the reduced sensitivity are not constant but there exists an equation of the type of equation [2.11] linking the reduced sensitivities of the set of parameters. In the case of our example, we could thus deduce an equation of the type:

$$k_1 E \frac{\partial T}{\partial E} + k_2 mc \frac{\partial T}{\partial mc} + k_3 Rc \frac{\partial T}{\partial Rc} = 0$$

That we can write as: $k_1 \frac{E \frac{\partial T}{\partial E}}{Rc \frac{\partial T}{\partial Rc}} = -k_2 \frac{mc \frac{\partial T}{\partial mc}}{Rc \frac{\partial T}{\partial Rc}} - k_3$

The plot of $\frac{E \frac{\partial T}{\partial E}}{Rc \frac{\partial T}{\partial Rc}}$ as a function of $\frac{mc \frac{\partial T}{\partial mc}}{Rc \frac{\partial T}{\partial Rc}}$ is therefore a straight line in this case. In the opposite case, the sensitivities to the three parameters are decorrelated and the parameters can be estimated separately. This is the case for the hot plate as shown in Figure 2.13.

This representation is, however, only possible in cases where at least one sensitivity is never zero over the entire time interval, in this case it will be chosen as the denominator in the sensitivity ratios.

Otherwise, we can write:

$$E \frac{\partial T}{\partial E} = -\frac{k_2}{k_1} mc \frac{\partial T}{\partial mc} - \frac{k_3}{k_1} Rc \frac{\partial T}{\partial Rc}$$

And determine a couple (k_4, k_5) that minimizes the sum of the quadratic deviations between $E \frac{\partial T}{\partial E}(t)$ and $k_4 mc \frac{\partial T}{\partial mc}(t) + k_5 Rc \frac{\partial T}{\partial Rc}(t)$, then plot $E \frac{\partial T}{\partial E}(t)$ as a function of $k_4 mc \frac{\partial T}{\partial mc}(t) + k_5 Rc \frac{\partial T}{\partial Rc}(t)$. If the plot is close to the line of equation $y = x$, then the sensitivities are correlated and the simultaneous estimation of the three parameters is not possible. We will see an example of the application of this technique in section 4.5.3.

We will remember that the sensitivity study carried out from a complete quadrupole model makes it possible to study among other things the sensitivity of the temperature T to the parameters R_c and mc . It is then possible to determine, as a function of the values of R_c , of mc and the thermal properties of the material, from which instant t_0 the sensitivities to these two parameters become constant. We can then optionally choose to use an ideal model applicable for $t > t_0$ to estimate the other parameters, being assured that the hypotheses leading to its establishment are satisfied.

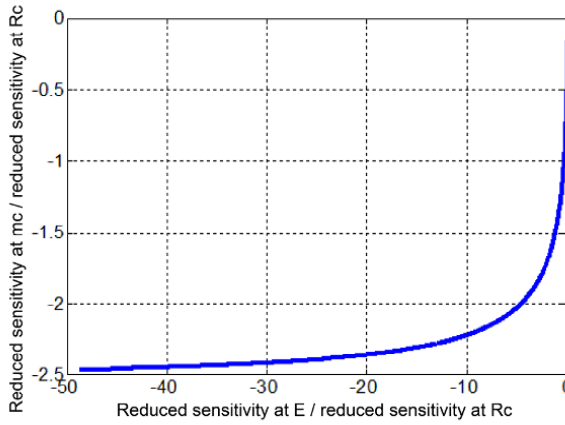


Figure 2.13. *Reduced sensitivities ratios*

2.2.4.2. Steady state

The interest of the reduced sensitivity analysis is not limited to the unsteady-state methods, it can also be applied to the steady-state methods where some parameters are deduced from the measurement of a stationary temperature field $T(x,y,z)$. To illustrate it, we consider the method of measuring the thermal conductivity of a conductive wire shown in Figure 2.14.

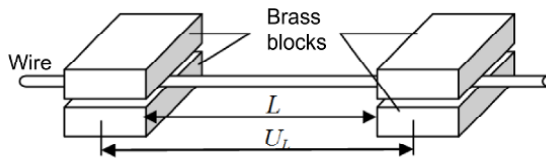


Figure 2.14. *Schematic diagram of the device for measuring the conductivity of a wire*

The two ends of the wire to be characterized are pinned in brass blocks which can be considered to be at constant temperature T_a equal to the ambient temperature. The wires' ends are connected to a current generator which delivers an intensity I_0 in the wire. The electrical potential difference between the two brass blocks is recorded. Its equilibrium value $U(L)$ depends on the wire's length, thermal conductivity and the global coefficient (convection + radiation) of exchange h of the wire with the surrounding environment. To reduce the exchanges by convection, the assembly is placed in an enclosure under a pressure of $3 \cdot 10^{-2}$ mbar.

The wire's average temperature $\bar{T}$ is related to the stationary value of the electric potential difference U_L by: $U_L = R_0 I_0 (1 + \alpha \bar{\theta})$

where: α is the temperature coefficient of the wire's electrical resistance;

$$\bar{\theta} = \bar{T} - T_a;$$

R_0 is the value of the wire's electrical resistance at room temperature T_a . Its value is measured just before heating.

The solution of the heat equation with boundary conditions allowed the derivation of the relationship $\bar{T}(L, \lambda, h)$ where L is the distance between the two brass blocks (wire length). This relationship can then be used to calculate the reduced sensitivity of $\bar{T}(L)$ to λ and to h to analyze the possibility of separately estimating these two parameters from the measurement of $\bar{T}$ for different values of L . We find an example of the result in Figure 2.15. The analysis of this figure makes it possible to draw the following conclusions:

- it is *a priori* possible to estimate λ for values of L less than 0.015m since the sensitivity of $\bar{T}$ to h is negligible over this interval. We will therefore fix its value arbitrarily to estimate only λ from measurements of $\bar{T}$ for several values of L less than 0.015 m;
- we can then estimate h by varying L over an interval [0.015 m; 0.05 m] where the sensitivity of $\bar{T}$ to h is sufficiently high.

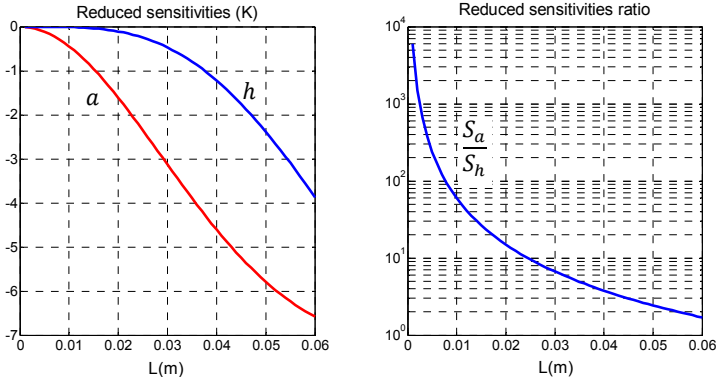


Figure 2.15. Reduced sensitivity at λ and at h for a chromel wire with a diameter of 0.254 mm

2.2.4.3. Conclusion

A preliminary sensitivity study using a complete model will always be carried out to determine the time intervals over which the sensitivities to the parameters are sufficiently decorrelated to allow a satisfactory estimate.

IMPORTANT NOTES.—

– We recall that the reduced sensitivity of temperature T to a parameter represents the variation of T (in°C) induced by a relative variation of k_i of 100 %. Therefore, for a parameter to be estimated, the reduced sensitivity of the temperature T to this parameter must be much greater than the measurement noise.

– If the sensitivity of the temperature to a parameter is negligible with respect to the sensitivity to the other parameters, then it is necessary to fix the value of this parameter at an approximate value and above all not to try to estimate it. Indeed, on the one hand, its sensitivity, although small, can be perfectly correlated with the sensitivity to another parameter and cause the estimation process to diverge and, on the other hand, it is shown that the standard deviations of estimations decrease when the number of parameters to be estimated decreases.

2.2.5. Method for estimating parameters

The problem posed is to estimate the values of p unknown parameters $k_0, k_1, \dots, k_p$ (which can be in our case: λ, E, R_c, mc) by knowing:

– the results of N measurements $T_{exp}(t_i)$ realized at times t_i (or at abscissas x_i in steady state), of one (or even several) temperature T depending on these p parameters;

– the complete (quadrupole) model making it possible to connect the value of T to those of the parameters $k_0, k_1, \dots, k_p$ in the form $T = f(k_0, k_1, \dots, k_p, t)$.

Note that in the applications treated here, the standard deviation on the temperature measurement does not change over time and the measurement errors are decorrelated: the error on the measurement $T_{exp}(t_i)$ does not depend on the other measurements $T_{exp}(t_j)$.

In the following, we will designate $[B]$ as the matrix of the unknown parameters:

$[B] = \begin{bmatrix} k_0 \\ \vdots \\ k_p \end{bmatrix}$ and $[X]$ as the sensitivity matrix defined by:

$$[X] = \begin{bmatrix} \frac{\partial f}{\partial k_0}(t_0) & \cdots & \frac{\partial f}{\partial k_p}(t_0) \\ \vdots & \ddots & \vdots \\ \frac{\partial f}{\partial k_0}(t_N) & \cdots & \frac{\partial f}{\partial k_p}(t_N) \end{bmatrix} \quad [2.14]$$

The problem posed is twofold:

- find the values of $k_0, k_1, \dots, k_p$ such that the curve $T = f(k_0, k_1, \dots, k_p, t)$ best represents the N experimental points $[t_i, T_{exp}(t_i)]$;
- evaluate the precision with which the values of $k_0, k_1, \dots, k_p$ are estimated.

The first problem is solved by minimizing the most common criterion which is the sum of squared differences, or the sum of squares of the distances of the experimental points at the theoretical curve: $\Sigma(B) = \sum_{i=1}^N [T_{exp}(t_i) - T_{mod}(t_i, B)]^2$.

The relationship between T and the various parameters is often complex and very rarely linear, so the method of estimating linear least squares cannot be applied. Newton's or Levenberg–Marquardt's method, which combines Newton's and the gradient methods, is most often used. Newton's method is an iterative method of order 2 [TRI 88] whose principle we will describe.

In Newton's method, matrix $[B]$ of the unknown parameters k_j is computed iteratively by:

$$[B_{n+1}] = [B_n] - \begin{bmatrix} \frac{\partial^2 \Sigma}{\partial k_0^2} & \cdots & \frac{\partial^2 \Sigma}{\partial k_p \partial k_0} \\ \vdots & \ddots & \vdots \\ \frac{\partial^2 \Sigma}{\partial k_0 \partial k_p} & \cdots & \frac{\partial^2 \Sigma}{\partial k_p^2} \end{bmatrix} \begin{bmatrix} \frac{\partial \Sigma}{\partial k_0} \\ \vdots \\ \frac{\partial \Sigma}{\partial k_p} \end{bmatrix} = [B_n] - [\Sigma'']^{-1} [\Sigma'] \quad [2.15]$$

where n is the iteration index.

The matrix $[\Sigma'']$ is called the Hessian of the minimization criterion, with:

$$\frac{\partial \Sigma}{\partial k_j} = -2 \sum_{i=1}^N [T_{exp}(t_i) - T_{mod}(t_i)] \frac{\partial T}{\partial k_j}(t_i)$$

$$\text{And: } \frac{\partial^2 \Sigma}{\partial k_j \partial k_\ell} = 2 \sum_{i=1}^N \frac{\partial T}{\partial k_j}(t_i) \frac{\partial T}{\partial k_\ell}(t_i) - 2 \sum_{i=1}^N [T_{exp}(t_i) - T_{mod}(t_i)] \frac{\partial^2 T}{\partial k_j \partial k_\ell}(t_i)$$

In practice, we neglect the second term compared to the first (Gauss–Newton approximation) and we compute the terms of the pseudo-Hessian by using:

$$\frac{\partial^2 \Sigma}{\partial k_j \partial k_\ell} = 2 \sum_{i=1}^N \frac{\partial T}{\partial k_j}(t_i) \frac{\partial T}{\partial k_\ell}(t_i) \quad [2.16]$$

where $T(t_i)$, $\frac{\partial T}{\partial k_j}(t_i)$ and $\frac{\partial^2 T}{\partial k_j \partial k_\ell}(t_i)$ are calculated to the n^{th} iteration with values $k_{0n}, k_{1n}, \dots, k_{pn}$.

This method is stable only if $[\Sigma]$ is a positive definite matrix and can diverge if the starting values are too far from the solution, hence special care should be taken in choosing the starting values $k_{01}, k_{11}, \dots, k_{p1}$ that may be guided by physical considerations or by a pre-estimation using a simplified model. Newton's method has the advantage of being easily programmable and be pre-programmed in the "solver" that is integrated in Microsoft Excel[®]. Most programming languages offer pre-programmed minimization functions such as "*fminsearch*" and "*fminbond*" functions in Matlab[®].

NOTES.—

– The first derivatives of the temperature (which are usually calculated by inverse Laplace transformation) are rarely expressible in explicit form and are therefore often computed numerically by using:

$$\frac{\partial T}{\partial k_j}(t) = \frac{T(k_0, k_1, \dots, k_j + \varepsilon, \dots, k_p, t) - T(k_0, k_1, \dots, k_j, \dots, k_p, t)}{\varepsilon}.$$

– When the reduced sensitivity to a parameter is very low in the presence of reduced sensitivities to the other parameters, then this parameter must be given a fixed (even biased) value rather than leaving it free in the estimation process.

2.2.6. Evaluation of the estimation error due to the measurement noise

2.2.6.1. Analytical calculation

In the case where the parameters are linked by a linear relationship, i.e. where the functions $\left(\frac{\partial f}{\partial k_j}\right)$ are independent of parameters $k_0, k_1, \dots, k_p$, we can evaluate the standard deviation of the error on the estimation of the parameters $k_0, k_1, \dots, k_p$ due to measurement noise on $T_{exp}(t)$ (null mean random measurement error) using the following formula [BEC 77]:

$$[cov(e_B)] = \sigma T^2 [X^t X]^{-1} = \begin{bmatrix} a_{00} & \cdots & a_{0p} \\ \vdots & \ddots & \vdots \\ a_{p0} & \cdots & a_{pp} \end{bmatrix} \quad [2.17]$$

where σT^2 is the square of the standard deviation of the measurement error on T .

The diagonal terms a_{jj} represent the squares of the standard deviation of the estimation error on k_j , the standard deviation σk_j of the estimation error on k_j due to the random measurement noise on T is therefore calculated by:

$$\sigma k_j = \sqrt{a_{jj}} \quad [2.18]$$

In the cases where the relationship between temperature and unknown parameters is not linear, we can assume that the function $T(k_0, k_1, \dots, k_p, t)$ is linear in relation to $k_0, k_1, \dots, k_p$ on intervals of very small amplitudes around the estimated optimal values. We can thus evaluate the precision with which the unknown parameters were estimated using the above relationships.

2.2.6.2. Statistical calculation (Monte Carlo simulations)

In cases where the inversion of the matrix $[X^t X]$ is problematic, the following method can be adopted:

From a series of N experimental pairs $[t_i, T_{exp}(t_i)]$, we can numerically simulate q ($q > 100$) other series of N pairs in one of the following ways:

$$T_{exp_j}(t_i) = T_{exp}(t_i) + r_i \sigma T$$

where:

– r_i is a random number following a normal distribution with zero mean and standard deviation equal to 1. This number can be generated in Matlab with the instruction: $r_i = randn(1)$;

– σT is the standard deviation of the measurements of T . If we know the uncertainty dT , we shall take $\sigma T = \frac{dT}{3}$ since the number r_i follows a normal law.

We thus simulate measurements with a noise according to a normal centered law representative of most measurements.

After applying this method, we estimate the parameters $k_0, k_1, \dots, k_p$ for each of the simulated q series. We thus get a matrix of the type:

$$[B] = \begin{bmatrix} k_{0_1} & \cdots & k_{0_q} \\ \vdots & \ddots & \vdots \\ k_{p_1} & \cdots & k_{p_q} \end{bmatrix}$$

We can then form the matrix $[eB] = [k_i - k_{i_j}]$ where k_i is the result of the estimation of the i^{th} parameter from the series of experimental points and k_{i_j} is the result of the estimation of this same parameter from the j^{th} series of simulated points.

We then calculate the matrix $[cov(e_B)]$ whose diagonal terms a_{jj} represent the squares of the standard deviation of the estimation error of k_j .

2.2.6.3. Advantage of the calculation

Another technique that can be used to determine whether all parameters can be estimated is as follows:

- parameters are assigned nominal values of the same order of magnitude as those to be measured;
- a simulation of the experimental curve is carried out using the model and these values;
- the simulated curve is noised by adding to each of its points a random measurement noise of standard deviation of the same order of magnitude as that observed on the experimental readings;
- this curve is treated as an experimental curve and both the value of the parameters and the uncertainty on the estimated values due to the measurement noise are estimated;
- we compare the estimated values to the nominal values that should have been found and the standard deviations on each value to conclude as to the possibility of estimating the various parameters.

2.2.7. Other sources of error

We have evaluated the uncertainties on the estimated parameters due to the measurement noise on the temperature $T_{exp}(t)$, the advantage of this evaluation is to allow an optimal choice of the experimental conditions and of the time interval chosen for the estimation. To these errors, we must add the uncertainties due to the errors of measurement of the model's parameters considered as data (flux, surface, etc.) and the errors due to the model itself which sometimes gives only an approximate representation of the reality by neglecting certain physical phenomena.

The main sources of error are:

- the calibration of the measuring device;
- an error on the parameters assumed to be known in the model;

- an error in the model;
- an error due to the inversion process;
- a numerical calculation error of the model.

2.2.7.1. Calibration of the measuring device

The quantities measured are either temperatures or fluxes. We will present a concrete case of a measurement carried out to show the important errors to which a bad calibration can lead to.

Let us therefore consider by way of example a method for measuring the thermal conductivity by comparison with a reference sample whose thermal conductivity λ_{ref} is known with precision.

This flowmetric measurement method is based on the measurement of the heat flow assumed to be 1D on the central zone of a sample placed between two thermostat plates (see Figure 2.16). A flowmeter that consists of thermocouples placed in series delivers a voltage U that, through a calibration, is connected to the value of the heat flux flowing through it. The measurement procedure is as follows:

– A first measurement of U_0 , T_0 and T_1 is carried out with a reference sample of thickness e_{ref} and conductivity λ_{ref} : it makes it possible to determine the coefficient of proportionality k between the heat flux and the voltage:

$$k = \frac{1}{U_0} \frac{\lambda_{ref}}{e_{ref}} (T_0 - T_1).$$

– A second measurement of U , T_0 and T_1 is carried out with the sample to be measured and the thermal conductivity is deduced by: $\lambda = \frac{e k U}{T_0 - T_1}$.

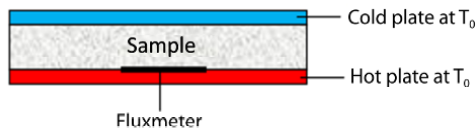


Figure 2.16. Schematic diagram of a fluxmetric measurement of thermal conductivity

We used this device with only one reference sample with the following properties: $e_{ref} = 30 \text{ mm}$ and $\lambda_{ref} = 0.03 \text{ W m}^{-1} \text{ K}^{-1}$ or a thermal resistance $R_{ref} = \frac{e_{ref}}{\lambda_{ref}} = 1 \text{ m}^2 \text{ K W}^{-1}$. Measurement of the coefficient k using this sample under a temperature difference $T_0 - T_1 = 10^\circ\text{C}$ led us to the value $k = 5.40 \times 10^{-3} \text{ W V}^{-1}$.

We then used the device to measure the conductivity of two polyurethane samples of thickness 30 mm and 10 mm and thermal conductivity (identical for the two thicknesses) $\lambda = 0.0315 \text{ W m}^{-1} \text{ K}^{-1}$ measured by the reference method of the guarded hot plate. We obtained the following results:

$$\lambda_{30\text{mm}} = 0.0318 \text{ W m}^{-1} \text{ K}^{-1} \text{ and } \lambda_{10\text{mm}} = 0.0338 \text{ W m}^{-1} \text{ K}^{-1}$$

The value obtained at 10 mm has a deviation of 7.4% from the reference value, whereas the value obtained with the 30 mm thickness sample differs by less than 1% from the reference value.

This is due to the difference in the thermal resistance of the two samples: $R_{30\text{mm}} = 0.95 \text{ m}^2 \text{ K W}^{-1}$ and $R_{10\text{mm}} = 0.32 \text{ m}^2 \text{ K W}^{-1}$. It is found that $R_{ref} \approx R_{30\text{mm}}$, as a result, the heat flux passing through the 30 mm sample is not that different from the one passing through the reference sample whereas the one flowing through the 10 mm sample is three times greater. The measurement deviation is due to the fact that coefficient k of the fluxmeter used is not strictly constant but depends on the value of the flux. In order to carry out an accurate measurement with this device, it would therefore be necessary to have a reference sample of thermal resistance close to that of the sample to be characterized.

The example described makes it possible to draw the following conclusions:

- a calibration error can lead to errors far greater than those due to the measurement noise;
- the measuring devices are accurate only if they are used within the measuring range in which they have been calibrated;
- this last observation is especially true for fluxmeters which have a mostly nonlinear response.

2.2.7.2. Error on known parameters

An error on the value of one or more parameters considered as known may also induce an error on the estimation of the unknown parameters. Let us consider by way of example the case of the flash method schematically represented in Figure 2.17.

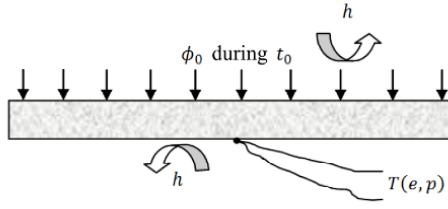


Figure 2.17. Schematic diagram of the flash method

A flux of high intensity and very short duration ($t_0 \approx 1$ ms) is sent on one face of a sample of thickness e which absorbs a heat flux ϕ_0 . The evolution of the temperature $T(e, t)$ of the opposite face in relation to time is recorded. Assuming that the heat transfer is 1D, we show that the Laplace transform $\theta(e, p)$ of this temperature is expressed as:

$$\theta(e, p) = \frac{\phi_0}{C + 2Ah + Bh^2}$$

$$\text{With: } A = \cosh\left(\sqrt{\frac{p}{a}}e\right); B = \frac{1}{\lambda\sqrt{\frac{p}{a}}} \sinh\left(\sqrt{\frac{p}{a}}e\right); C = \lambda \sinh\left(\sqrt{\frac{p}{a}}e\right)$$

which can be written as:

$$\theta(e, p) = \frac{e\phi_0}{\lambda} \frac{1}{\sqrt{\frac{p}{a}}e \sinh\left(\sqrt{\frac{p}{a}}e\right) + 2\frac{he}{\lambda} \cosh\left(\sqrt{\frac{p}{a}}e\right) + \frac{\left(\frac{he}{\lambda}\right)^2}{\sqrt{\frac{p}{a}}e} \sinh\left(\sqrt{\frac{p}{a}}e\right)} = f\left(\frac{e\phi_0}{\lambda}, \frac{he}{\lambda}, \frac{e^2}{a}\right)$$

In this method, we estimate three groups where the latter $\frac{e^2}{a}$ makes it possible to determine the thermal diffusivity a if we know the thickness e by using: $a = \frac{e^2}{\frac{e^2}{a}}$.

This equation shows that an error of 1% on the measurement of e leads to a 2% error on the estimation of a . By way of example, an error of 0.1 mm over a thickness of 3 mm leads to an error of 6.6% on a . If the value of 0.1 mm is excessive for precisely machinable materials such as metals, it is not unrealistic for materials such as wood or concrete.

2.2.7.3. Model error

Methods of measuring thermal properties most often use analytical models based on one or more simplifying hypotheses.

These assumptions are generally true over limited time and/or space intervals and realizing an estimate outside of these intervals can lead to estimation errors. A full study of this aspect is proposed in section 2.2.8.

One of the main sources of errors is due to heat transfer in the heating element not taken into account in the 2D or 3D methods with the hot strip, hot disc, hot wire (if the wire is inserted between two sheets).

EXAMPLE.—*Hot strip.*

Let us consider by way of example the hot strip method in which a “long” heat transfer (2D heat transfer) of width b is inserted between two samples of thickness e and width L ($e > 3b$, $L > 3b$). In this method, the time variation of the rise in temperature in the center of a strip subjected to a flux step is exploited.

Assume that the strip is a thin heating element on which a thermocouple has been attached (see Figure 2.18).



Figure 2.18. *View of a hot strip*

The resistive track has a high thermal conductivity. If the thermal conductivity of the Kapton around the track is $0.3 \text{ W m}^{-1} \text{ K}^{-1}$, it has been calculated that the thermal conductivities of the homogenized strip are $\lambda_z = 0.3 \text{ W m}^{-1} \text{ K}^{-1}$ and $\lambda_x = 2 \text{ W m}^{-1} \text{ K}^{-1}$. We will see later in this book that the model used makes the hypothesis of the medium at uniform temperature that would be obtained with the values: infinite λ_z and null λ_x .

2D numerical simulations were carried out in COMSOL for these two configurations as well as for an intermediate configuration for which $\lambda_z = \lambda_x = 0.3 \text{ W m}^{-1} \text{ K}^{-1}$ (case where the resistive track would be in the direction perpendicular to that shown in Figure 2.18). The results are shown in Figure 2.19.

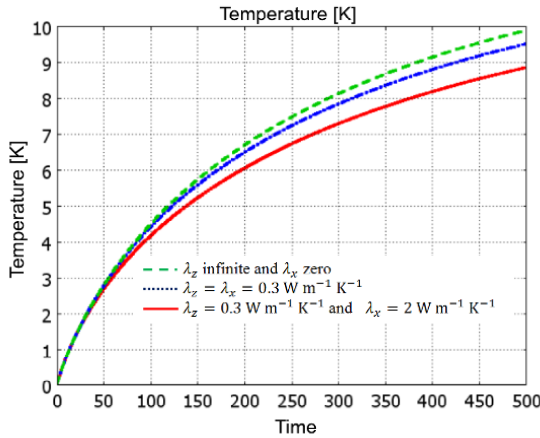


Figure 2.19. Simulated curves for different thermal conductivity values of the hot strip. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

The difference between the two extreme curves is important as soon as the transfer to the center of the strip becomes 2D, which leads to an overestimation of the thermal diffusivity of the sample to be measured if its conductivity is less than $2 \text{ W m}^{-1} \text{ K}^{-1}$. This gap may in some cases reach 10%.

EXAMPLE.— *Hot wire.*

The hot wire is actually a hot strip of very small width. Consider a “hot wire” type device constituted by a resistive track of width $100 \mu\text{m}$ inserted between two Kapton sheets of thickness $150 \mu\text{m}$ and width 100 mm . The wire is then inserted between two samples with a cross-section $100 \times 100 \text{ mm}^2$, with a thickness of 2 cm and a thermal conductivity $\lambda = 0.03 \text{ W m}^{-1} \text{ K}^{-1}$. The sample’s thermal conductivity is inversely proportional to the slope of the curve $T(t) = f[\ln(t)]$ in its linear part. A 2D numerical simulation was performed in COMSOL and the curve’s slope was calculated. The use of this slope leads to an estimate of the sample’s

thermal conductivity $\lambda = 0.0282 \text{ W m}^{-1} \text{ K}^{-1}$, i.e. an error of 6%. It will be noted that this error is negligible for materials with a conductivity greater than $0.03 \text{ W m}^{-1} \text{ K}^{-1}$.

Through these two examples it can be seen that methods based on 2D heat transfer using a flat probe (hot strip, hot disc) may become less accurate especially for insulating materials due to transverse heat transfer in the probe not taken into account.

2.2.7.4. Numerical calculation error of the model

Whatever the method used for solving the heat equation, the simulation of the solution is affected by a calculation error:

- If one solves using the quadrupole method, the calculation of the inverse Laplace transform is done by an approximate numerical method.

- If one solves by separation of variables or by integral transform, one must calculate sums with an infinite number of terms whose truncation also induces an error.

- If one solves numerically by finite differences or by finite elements, the choice of a mesh of a size making it possible to keep the calculation time within reasonable limits again leads to an error.

This source of error rarely taken into account can lead to an additional uncertainty of the order of 0.5%.

2.2.7.5. Error due to the inversion process

Another possible source of error is the inversion method which consists of finding an optimum set of parameters making it possible to minimize the sum of the quadratic deviations between the experimental points and the model curve. This sum may present local minima to which the minimization process can converge if initial values that are too far from actual value are assigned to the parameters to be estimated. This risk will be greater as the signal is noisy and the number of parameters to be estimated is high (thus less decorrelated). In order to minimize the risk of error, it is always advantageous to use a simplified method to obtain an initial value of the parameter(s) to be estimated: for example in the case of the flash method, use the Parker method or the partial times method [DEG 86] to obtain an approximate value of the thermal diffusivity which will be used as initial value in an iterative process based on a more complete quadrupole model.

2.2.8. Validity domain of a model and estimation time interval

2.2.8.1. Numerical simulation

For reasons of simplicity of modeling and to limit the number of parameters to be estimated, it is often necessary to issue one or more simplifying hypotheses, for example:

- 1D heat transfer for flash and hot plate methods;
- semi-infinite medium for hot wire and hot disk;
- negligible probe inertia for some simplified models.

To determine the temporal validity domain of these hypotheses, a solution consists of carrying out a 3D numerical simulation of the complete system without making any simplifying hypothesis. A number of software programs (COMSOL @, FLEXPDE @, etc.) make it possible to perform these simulations in a rather simple way, but the computation time can be long depending on the complexity of the system represented.

For a given system and experimental conditions, two simulations are performed:

- one with the simplified analytical model;
- one with 3D numerical simulation.

By analyzing the difference between the two simulations, the temporal validity domain of the simplified analytical model is determined and the time interval considered for estimating the parameters in this domain is then reduced.

The disadvantage of this method is that it is necessary to know the properties of the material to be measured (*a priori* unknown). This method is also applicable for steady-state methods.

By way of example, this method is applied to the hot plate device shown in Figure 2.20. Since the device is symmetrical, only half of the device will be studied to limit the calculation time. A uniform heat flux $\phi_0 = 150 \text{ W m}^{-2}$ is applied in a heating element of section $100 \times 100 \text{ mm}^2$. By reason of symmetry, the lower part of the probe is considered insulated. The sample placed on the probe is 3 cm thick and of the same cross-section as the probe.

Its thermal properties are as follows:

$$\lambda = 0.15 \text{ W m}^{-1} \text{ K}^{-1} \text{ and } \rho c = 1.5 \times 10^5 \text{ J m}^{-3} \text{ K}^{-1}.$$

Its unheated face is maintained at a constant temperature $T_0 = 20\text{ }^{\circ}\text{C}$. The probe's lateral faces and the sample exchange heat by convection and radiation with the ambient air at temperature $T_a = 20\text{ }^{\circ}\text{C}$.

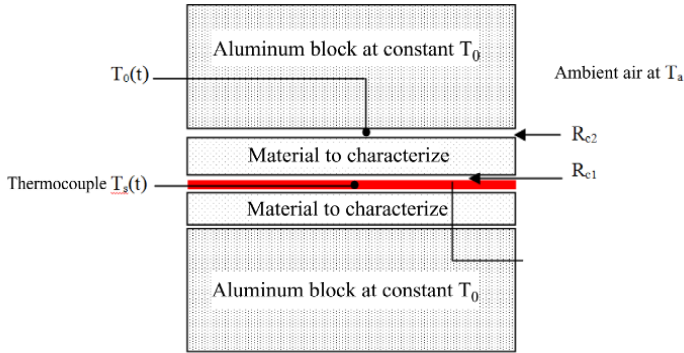


Figure 2.20. Schematic diagram of the experimental hot-plane type device studied

We would like to know until which time we can assume a 1D transfer to the sample's center. For this purpose, a 3D simulation is carried out in COMSOL with an external heat transfer coefficient $h = 10\text{ W m}^{-2}\text{K}^{-1}$ and a second one with $h = 0\text{ W m}^{-2}\text{K}^{-1}$ (to simulate a 1D transfer) to obtain the evolutions of the temperature rise in the center of the heated face in both cases. The plot of the two curves on the same figure makes it possible to determine that the transfer remains 1D at the center for 800 s (see Figure 2.21).

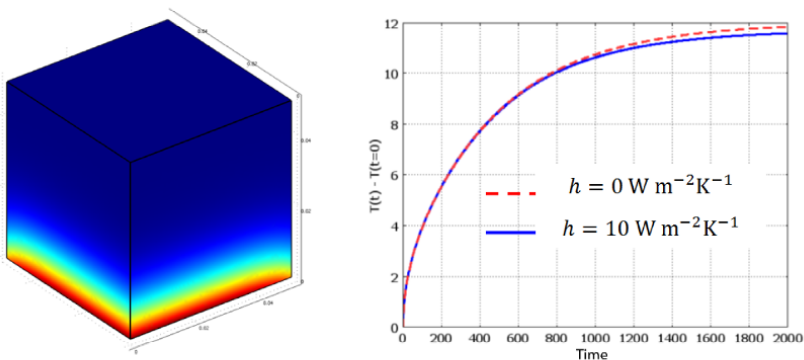


Figure 2.21. Simulated temperature rise with COMSOL for $h = 0\text{ W m}^{-2}\text{K}^{-1}$ and $h = 10\text{ W m}^{-2}\text{K}^{-1}$. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

We would also like to know how long the hypothesis of the semi-infinite medium for the sample is valid. For this purpose, two simulations are carried out in COMSOL:

- a first one with a thickness of 3 cm;
- a second one with a thickness of 4 cm.

The plotting of the curves of the rise in temperature at the center of the heated face obtained by the two simulations makes it possible to determine that the semi-infinite medium hypothesis is valid for 400 s (see Figure 2.22).

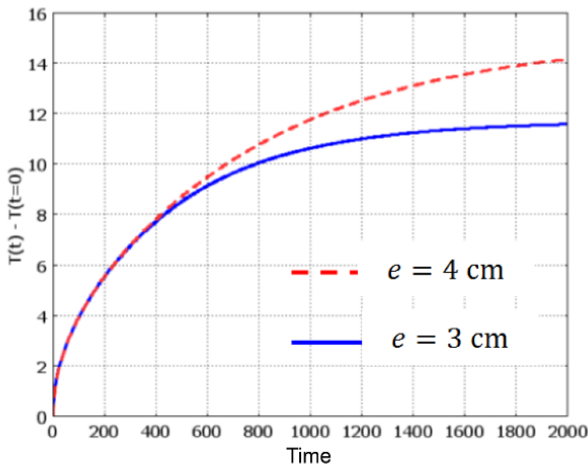


Figure 2.22. Simulated temperature rise with COMSOL for $e = 3$ cm and $e = 4$ cm. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

NOTE.—

A 3D simulation often requires a long calculation time. In the case of a square cross-sectional geometry L , it is possible to reduce the computing times by assimilating the parallelepipeds of square cross-section L to cylinders of radius R and of the same cross-section: $R = \frac{L}{\sqrt{\pi}}$.

The simulated curves of the temperature at the center of the heating element corresponding to the system described above, obtained respectively for a parallelepipedal geometry with a section $100 \times 100 \text{ mm}^2$ and for a cylindrical radius geometry $R = 56.42 \text{ mm}$ are shown in Figure 2.23. It is found that the two curves are superimposed with very small differences.

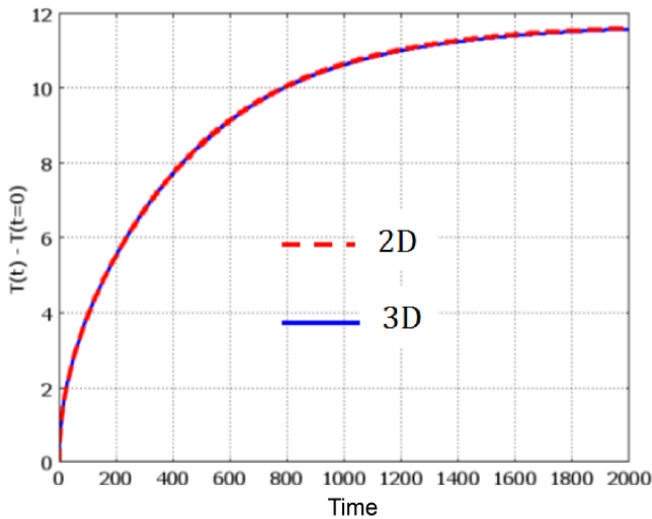


Figure 2.23. COMSOL 3D simulations and equivalent 2D simulations.
For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

In addition to saving time, a 2D simulation can be performed with software that is more common and cheaper than that used to perform 3D simulations.

2.2.8.2. Residues analysis

Estimation residual analysis is a powerful tool for determining the temporal validity domain of the model without requiring prior knowledge of its properties. The principle consists of performing an estimation of the parameters from an experimental temperature reading over a chosen time interval. The temperature is then calculated using the model and the estimated values of the parameters. Finally, the estimation residues (i.e. the difference between the measured values and the estimated values of the temperature) are calculated and represented. The model is valid on the zone where the residues are flat, i.e. centered on the value zero. If the residues are not flat over the entire initial estimation interval, the amplitude will be reduced until the residuals become flat over this interval.

APPLICATION EXAMPLE.—

A hot plate type experiment was carried out using the device shown in Figure 2.20. It will be shown later that, after a certain time, the rise in temperature evolves linearly as a function of the entire time that the hypothesis of the semi-infinite medium remains valid. The measurement of the slope of this linear part

makes it possible to estimate the sample's thermal effusivity. An experimental analysis of the temperature rise is shown in Figure 2.24.

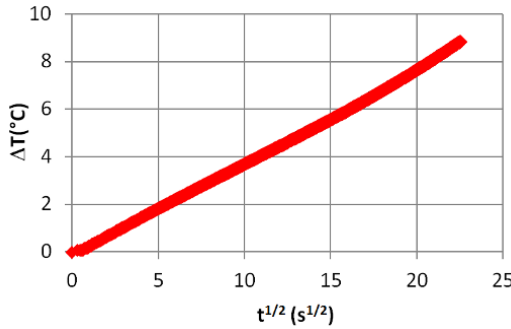


Figure 2.24. *Experimental reading of a curve $\Delta T = f(\sqrt{t})$ during a hot plate experiment*

We start by performing a linear regression over an interval of $[0, 20 \text{ s}^{1/2}]$ for example, we then find equation: $\Delta T_{mod} = 0.3836\sqrt{t} - 0.1173$. The residues are then calculated by: $R(t) = \Delta T_{exp} - 0.3836\sqrt{t} + 0.1173$ and shown in Figure 2.25.

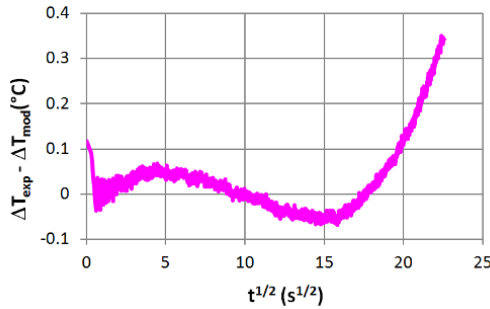


Figure 2.25. *Residuals obtained after estimation over the interval $[0, 20 \text{ s}^{1/2}]$*

The residues are not flat so we have to modify the time interval chosen for the estimation. We find that there is a linear zone between 5 and 15 $\text{s}^{1/2}$ that we will choose for a second estimate. We then obtain: $\Delta T_{mod} = 0.3719\sqrt{t} - 0.0042$. The residues are shown in Figure 2.26.

The residues are flat over the estimation interval therefore the choice was good and the residues indicate that the semi-infinite medium hypothesis is no longer valid

beyond $t = 225$ s. The value $\alpha = 0.3719$ of the slope can be used to estimate the thermal effusivity E .

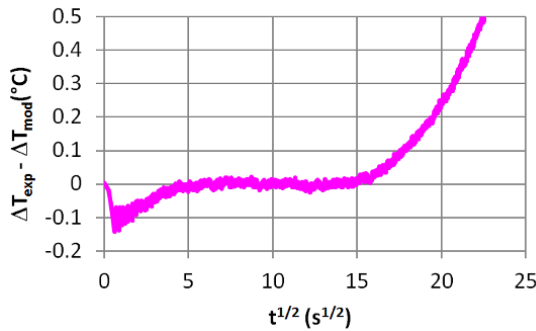


Figure 2.26. Residues obtained after estimation over the interval $[5, 15 \text{ s}^{1/2}]$

IMPORTANT NOTE.—

The analysis of a thermogram may reveal a signal disturbance over a short period of time due to an imperfection of the experimental device. In this case, it is advantageous to remove this time interval from the estimation interval. By way of illustration, Figure 2.27 shows a temperature record during a flash method. There is an abrupt and immediate rise in the temperature of the unheated face as the temperature should begin to increase after a much longer time. This is due to a “leakage” fault in the experimental device which allowed a small fraction of the flash to arrive directly on this face. It can be seen that the signal resumes its initial value after this short disturbance. The lower limit of the estimation interval in this case is equal to 0.01s.

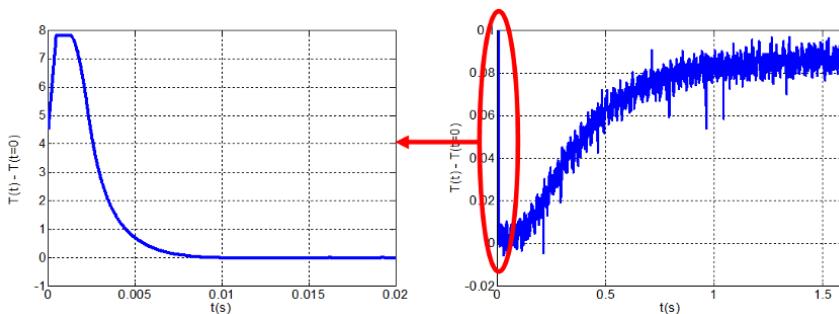


Figure 2.27. Demonstration of an experimental device error on a flash experiment

2.2.9. Choice of the temperature's origin

When the signal is highly noisy, the estimation of the initial temperature (always assumed to be uniform in the sample) may be difficult even with pre-acquisition. It is preferable in this case to estimate it in two steps:

1) Estimate a value by reading the thermogram and subtracting it from all values to obtain a first approximation (up to a constant) of the temperature rise from its initial value. Carry out the estimation of the parameters from the thermogram thus constructed.

2) Use the estimated values as starting values of a new estimate in which a new temperature shift parameter will be added: it is enough in the calculation function to add to the expression of T a constant T_0 that will be considered as a new parameter to be estimated.

The results of the two successive estimation processes on a very noisy signal obtained during an estimation of the thermal diffusivity by the flash method are given in Figure 2.28.

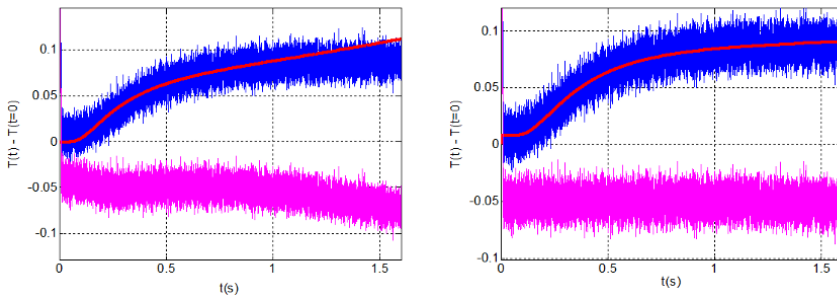


Figure 2.28. Experimental curves, simulated and residues ($\times 10$ and offset by 0.05°C) without, then with initial temperature estimation. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

2.2.10. Conclusion

The diagram of the recommended approach for estimating thermal (or other) parameters from an experiment is shown in Figure 2.29.

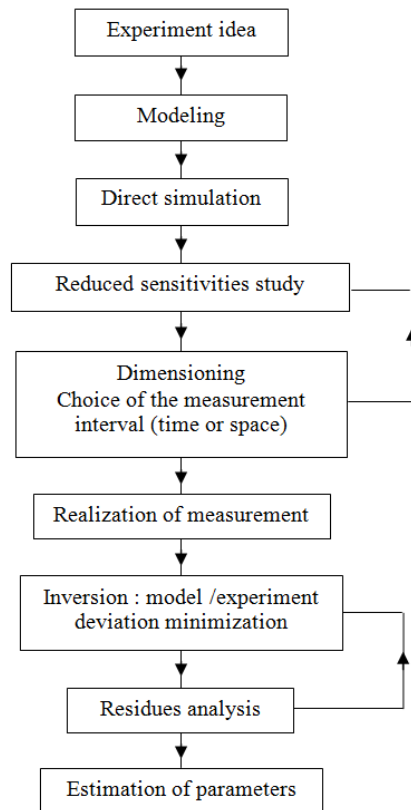


Figure 2.29. Schematic diagram of the approach used for the implementation of a characterization method (thermal)

Steady-state Methods

3.1. Introduction

Steady-state methods can by definition measure only thermal conductivity.

They all rely on the same principle: measure the heat flow rate φ in a heat resistance flux tube R delimited by two isothermal surfaces at temperatures T_0 and T_1 . We deduce the value of R with: $R = \frac{T_1 - T_0}{\varphi}$. Thermal conductivity λ is deduced if the relationship $R(\lambda)$ is established.

They have the disadvantage of relying only on one measurement reading; however, the measurement can be repeated a great number of times. In this case, the uncertainty due to the measurement noise is divided by $\sqrt{N}$.

They are not suitable for materials with high thermal conductivity. We consider the simple case represented in Figure 3.1.

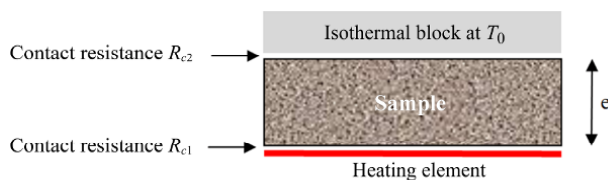


Figure 3.1. Schematic diagram of a device for measuring the thermal conductivity in steady state

A uniform and constant heat flow rate φ is provided on one face of a sample with a thickness e by means of a planar heating element whereas the other face is in contact with an isothermal surface maintained at a constant temperature T_0 . It is assumed that the heat transfer is 1D and the temperature T_1 of the heating element is measured when the steady state is reached.

The thermal resistance between the two temperature measurement points is deduced from:

$$R = \frac{T_1 - T_0}{\varphi} \quad [3.1]$$

This resistance is the sum of three resistances placed in series:

- the thermal resistance $R_e = \frac{e}{\lambda S}$ of the sample;
- the contact resistance R_{c1} between the heating element and the sample;
- the contact resistance R_{c2} between the sample and the surface maintained at T_0 .

A contact resistance can reach a value of $2 \times 10^{-4} \text{ m}^2 \text{ K W}^{-1}$, therefore the thermal resistance of the sample must be at least $4 \times 10^{-2} \text{ m}^2 \text{ K W}^{-1}$ in order for us to be able to neglect the contact resistances.

We can deduce from the above: $\frac{e}{\lambda} > 0.04 \text{ m}^2 \text{ K W}^{-2}$.

For a material of thermal conductivity $\lambda = 10 \text{ W m}^{-1} \text{ K}^{-1}$, a sample of thickness equal to at least 40 cm would be required so that its thermal resistance is 100 times greater than the contact resistance, which is not realistic.

It will therefore be remembered that the steady-state methods only make it possible to measure a thermal resistance and that the value of the thermal conductivity can only be deduced therefrom if it is less than $1 \text{ W m}^{-1} \text{ K}^{-1}$.

3.2. Guarded hot plate

3.2.1. Principle

Guarded hot plate is a reference method for measuring the thermal conductivity of materials with low thermal conductivity, developed and used for many years [GIL 51, WOO 57]. It has been the subject of standards such as: ISO 8302, ASTM C177, DIN EN 12939 and DIN EN 12667, and numerous publications aimed mostly at proposing construction variants or estimating measurement uncertainties [REN 83, HAM 02]. Its schematic diagram is given in Figure 3.2.

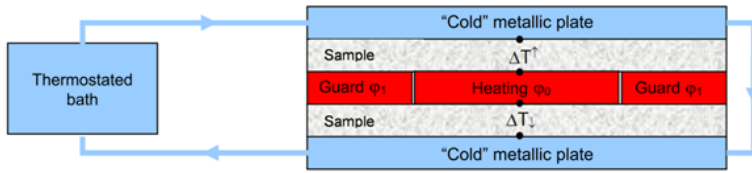


Figure 3.2. Schematic diagram of the guarded hot plate method

The outer plates of highly conductive material (copper, aluminum) are maintained at a constant temperature by circulating a fluid coming from a thermostated bath. A heating plate delivers uniform and constant heating power ϕ_0 which is transmitted to the outer plates through the samples for which the thermal conductivity is to be measured. The 1D transfer is ensured in the central measuring zone by surrounding the heated zone with a guard ring to which a heating power ϕ_1 is supplied such that the temperature of the ring is equal to the temperature of the heating plate. It is thus also ensured that all the heating power ϕ_0 passes through the sample. The heat flux ϕ_1 is greater than the heat flux ϕ_0 to compensate for lateral convective losses. The guard ring and the heating plate are separated by a thin layer of air.

Thermocouples are placed on both sides of the samples to measure temperature and temperature differences $\Delta T_{\downarrow}$ and $\Delta T_{\uparrow}$.

3.2.2. Hypotheses and model

The following hypotheses are made:

- the heat transfer is 1D in the central zone;
- thermal contact resistances are negligible in comparison with the thermal resistance of the sample.

The thermal conductivity can then be calculated by:

$$\lambda = \frac{e \phi_0}{2 S (\Delta T_{\downarrow} + \Delta T_{\uparrow})} \quad [3.2]$$

where S is the surface area of the hot plate.

This method is applicable only to insulators ($\lambda < 1 \text{ W m}^{-1} \text{ K}^{-1}$) for which the contact resistance can be neglected in relation to the material's resistance [DEP 02]. They also make it possible to obtain relatively large temperature differences between the two faces, which can therefore be measured with good accuracy. The result may be biased by lateral losses if the surface of the sample is insufficient relative to the thickness.

3.2.3. Experimental design

There are commercial devices operating on this principle, but their cost is relatively high. Their temperature is usually limited to 200°C [REM 85]. The standard section of the samples required is $500 \times 500 \text{ mm}^2$, the minimum according to our knowledge being $300 \times 300 \text{ mm}^2$. This comparatively complex reference system is *a priori* reserved for laboratories certifying insulating materials. This method is difficult to apply at high temperatures even if there are some devices withstanding up to 800°C .

3.2.4. Practice of the measurement

Conductive grease may be used to reduce contact resistances between samples and plates provided that it does not penetrate the material. This reference method is announced as having accuracy in the order of 2% at moderate temperatures, the predicted accuracy is worse at high temperature (in the order of 10% at 500°C).

3.3. Center hot plate

3.3.1. Principle

Center hot plate is a method adapted to the measurement of the thermal conductivity of reduced size samples whereas the guarded hot plate method requires samples with a cross-section equal to at least $300 \times 300 \text{ mm}^2$. The ideal cross-section of the samples is in the order of $100 \times 100 \text{ mm}^2$ but one can go down to $45 \times 45 \text{ mm}^2$. The thicknesses can go up to 4 cm for the first and up to 2 cm for the second.

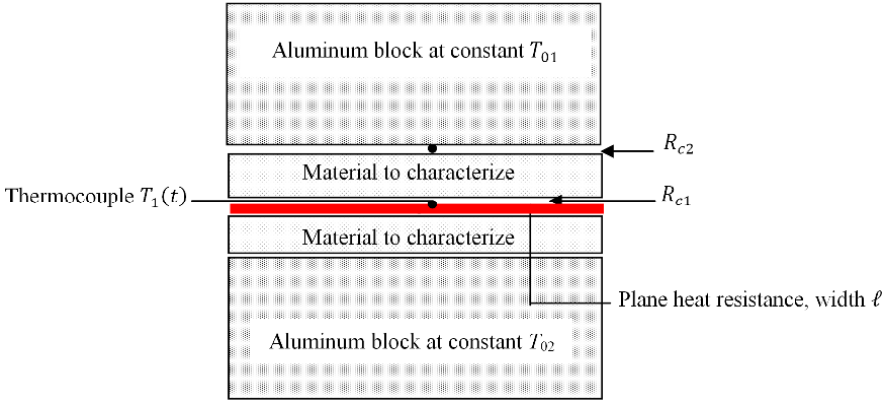


Figure 3.3. *Diagram of the installation of the hot plate method with two samples*

The experimental device shown diagrammatically in Figure 3.3 comprises of:

- a flat heating element of small thickness on which is fixed a type K thermocouple with fine wires inserted between two samples of identical dimensions of the material to be characterized;
- two isothermal aluminum blocks with identical sections to the samples. These hollow blocks are crossed by a fluid coming from a thermostated bath;
- a clamping device to control the pressure and thickness of the material inserted between the aluminum blocks.

The heating element is subjected to a flux step and the temperatures at the center of the heating element and temperatures T_{01} and T_{02} of the aluminum blocks are recorded until they no longer vary.

3.3.2. Hypotheses and model

It is assumed that the heat transfer is 1D (unidirectional) at the center of the sample where the temperature is measured. The heat flow rate φ_0 produced uniformly by the Joule effect in the heating element is transmitted by conduction through the two samples of thermal conductivity λ and of respective thicknesses e_1 and e_2 , therefore:

$$\varphi_0 = \lambda S \left(\frac{T_1 - T_{01}}{e_1} + \frac{T_1 - T_{02}}{e_2} \right) \quad [3.3]$$

It is possible to deduce therefrom the value of the thermal conductivity λ of the sample by:

$$\lambda = \frac{R_{el} I^2}{S} \frac{1}{\frac{\Delta T_1}{e_1} + \frac{\Delta T_2}{e_2}} \quad [3.4]$$

where: R_{el} is the electrical resistance of the heating element;

I is the intensity;

$$\Delta T_i = T_1 - T_{0i}.$$

The hypothesis of 1D transfer at the center is justified if the thickness/width ratio is small. Figure 3.4, where b is the half-width of the samples, shows [JAN 10]:

- the maximum thickness e_{max} not to be exceeded in order to satisfy the 1D transfer hypothesis at the center;
- the thickness e_{min} which it is necessary not to go below so that the contact resistances remain negligible with respect to the thermal resistance of the samples.

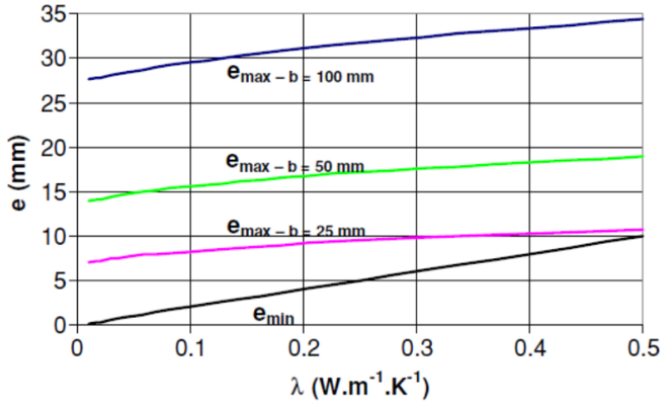


Figure 3.4. Sample thickness limit values for the center hot plate if $T_0 = T_a$ (external temperature) and $h = 10 \text{ W m}^{-2} \text{ K}^{-1}$

It has been shown [JAN 17] that larger thicknesses can be measured provided that a unique optimum value R_{opt} is observed for the ratio: $R = \frac{T_1 - T_a}{T_1 - T_0}$.

The idea is to choose a value T_a between T_0 and T_1 so that the convective exchanges on the lateral faces of the samples are positive over a part of their height and negative on the other. It is thus possible to “straighten” the flux lines in the center as shown in Figure 3.5.

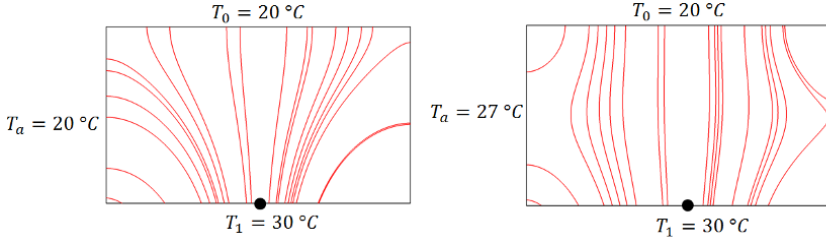


Figure 3.5. Flux lines for two values of T_a

The single value R_{opt} is determined experimentally by using two sets of samples of different thicknesses of the same material of known thermal conductivity λ_0 .

3.3.3. Experimental set-up

Figure 3.6 shows a picture of the experimental device performed at LEMTA, the Laboratory of Energetics, Theoretical and Applied Mechanics. The heating element consists of a resistive track inserted between two Kapton sheets. The thermocouples used are insulated type K thermocouples with an outer diameter of 0.1 mm. To reduce the excessive thickness owing to the thermocouple, the latter is flattened to give it an oblong shape. The thermocouple is attached to the center of the heating element by an aluminum adhesive with a thickness of 0.03 mm and a cross-section of $5 \times 5 \text{ mm}^2$.

Aluminum blocks are covered by a thermostatically controlled fluid and are thick enough (at least 1 cm) so that the temperature is uniform: the same value at all points. Their temperature is measured on a lateral surface in the following manner: a piece of double-sided adhesive of $1 \text{ cm} \times 2 \text{ cm}$ is applied to a lateral surface, the thermocouple is welded to the center and then it is covered by a piece of polystyrene of the same cross-section and of thickness 5 to 10 mm. The purpose of the adhesive is to insulate the thermocouple electrically from the aluminum block which is not insulated because it is traversed by a fluid. Temperature measurement biases have sometimes been found if this precaution is not taken. The purpose of the insulation piece is to make the heat transfer between the thermocouple and the ambient air negligible.

A mechanical device has been designed to allow the vertical displacement of an aluminum plate while remaining always perfectly parallel to the fixed plate. A vernier caliper has been attached to the device to allow measurement of the thickness of the samples in the measurement situation.

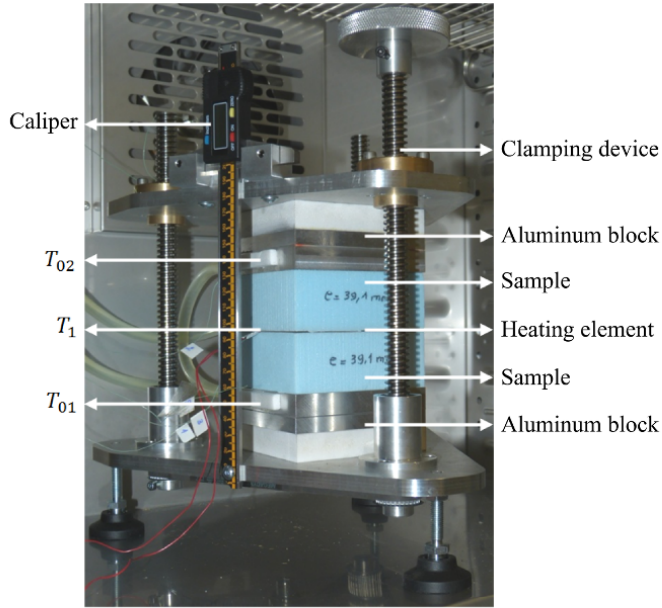


Figure 3.6. *View of the device*

3.3.4. Practice of the measurement

This measurement method is not absolute: it is necessary to have at least one reference sample to carry out a calibration. Indeed, the thermal conductivity is given by the following equation:

$$\lambda = \frac{R_{el}}{S} I^2 \frac{1}{\frac{\Delta T_1}{e_1} + \frac{\Delta T_2}{e_2}} \quad [3.5]$$

Thus, you have to determine the ratio $\frac{R_{el}}{S}$. A measurement of R_{el} by an ohmmeter and the determination of S by measurement of the sides already make it possible to have an approximate value. However, it is preferable to estimate this by performing a measurement with a reference sample of thermal conductivity λ_{ref} of thickness

between the values e_{min} and e_{max} defined in Figure 3.4. From this we deduce the value of $\frac{R_{el}}{S}$:

$$\frac{R_{el}}{S} = \frac{\lambda_{ref}}{l^2} \left(\frac{\Delta T_1}{e_1} + \frac{\Delta T_2}{e_2} \right) \quad [3.6]$$

Figure 3.7 shows the calculated values of $\frac{R_{el}}{S}$ for different values of R from measurements made on samples of extruded polystyrene with a thickness of 9 mm and thermal conductivity $\lambda = 0.0309 \text{ W m}^{-1} \text{ K}^{-1}$.

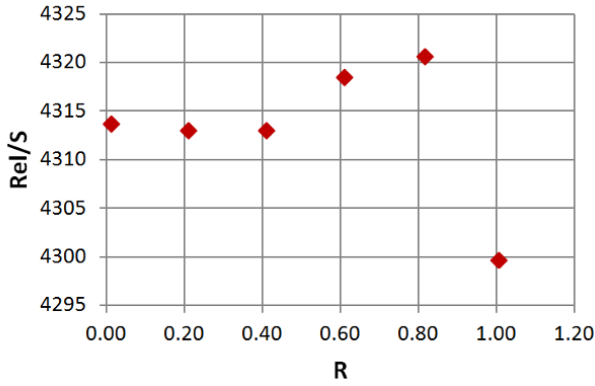


Figure 3.7. Value of the estimated ratio for several values of $R = \frac{T_1 - T_a}{T_1 - T_0}$; measurements carried out on polystyrene of thermal conductivity $\lambda = 0.0309 \text{ W m}^{-1} \text{ K}^{-1}$

The relative standard deviation on the values of $\frac{R_{el}}{S}$ is very small (0.17%), so the average value can be retained as $\frac{R_{el}}{S} = 4313 \text{ } \Omega \text{ m}^{-2}$.

To measure the thermal conductivity of samples of thicknesses greater than e_{max} , R_{opt} needs to be determined. The experimental procedure is as follows:

- the device is placed, with two samples of identical thicknesses of a material of known thermal conductivity, in an enclosure regulated at the temperature $T_a = T_0$;
- the temperature of the thermostatically controlled bath is adjusted so as to obtain the desired value T_0 ;
- the heating power is adjusted so as to obtain $T_1 = T_0 + 10^\circ\text{C}$;
- the values of ΔT_1 and ΔT_2 at equilibrium are noted;

– the setpoint of T_a is increased by 2°C and the procedure is repeated until the value $T_a = T_1$ is reached.

The experimental data are used to calculate λ for each value of T_a with equation [3.5].

The values thus obtained are then represented in a system of axes (λ, R) and we deduce the value of R_{opt} which is the one for which $\lambda = \lambda_{ref}$.

An example of experimental results obtained with extruded polystyrene with thermal conductivity $\lambda = 0.0309 \text{ W m}^{-1} \text{ K}^{-1}$ is shown in Figure 3.8.

Figure 3.8 indicates that the optimum value is $R_{opt} = 0.2$ that is $T_1 - T_a = 0.2 (T_1 - T_0)$. To measure the thermal conductivity of a sample at 20°C , it is necessary to set $T_0 = 15^\circ\text{C}$ and $T_a = 23^\circ\text{C}$, then to adjust the heating power to obtain $T_1 = 25^\circ\text{C}$.

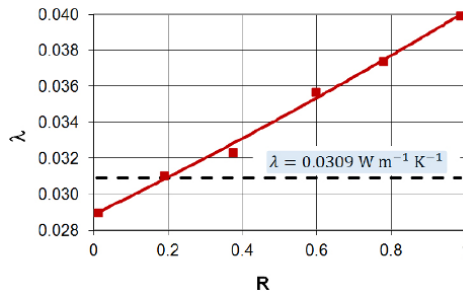


Figure 3.8. Values of λ_{est} as a function of R

NOTES.—

– Values of T_a are varied rather than T_0 or T_1 to erase values of R between 0 and 1 so that the mean temperature of the sample and therefore its thermal conductivity is identical for all the measurements.

– An asymmetrical arrangement can be realized by placing a reference sample on one side of the heating element and the sample to be measured on the other side. This is of particular interest for non-deformable samples such as PVC, for example. The set-up shown in Figure 3.9 will be carried out in which the face of the heating element on which the thermocouple is attached will be in contact with a deformable reference material (e.g. extruded polystyrene), thus avoiding the creation of a strong contact resistance.

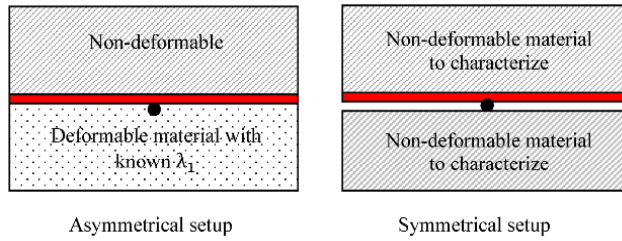


Figure 3.9. *Asymmetrical and symmetrical arrangement schematic diagrams in the case of a non-deformable material to be characterized*

The thermal conductivity λ_2 of the sample with a thickness e_2 is then calculated by:

$$\lambda_2 = \frac{e_2}{\Delta T_2} \left(\frac{R_{el} I^2}{S} - \frac{\lambda_1}{e_1} \Delta T_1 \right) \quad [3.7]$$

This measurement method (symmetric or asymmetric) was used to measure thermal conductivities between $0.022 \text{ Wm}^{-1}\text{K}^{-1}$ and $0.2 \text{ Wm}^{-1}\text{K}^{-1}$ for samples with a cross-section of $100 \times 100 \text{ mm}^2$ and thicknesses varying between 1 and 3 cm with accuracy better than 3% [JAN 17].

3.4. Hot strip

3.4.1. Principle

This is a suitable method for measuring the thermal conductivity of insulating anisotropic samples. This method, which is used in conjunction with the center hot plate method, is used to measure the thermal conductivities λ_x and λ_z (see axis definitions in Figure 3.10). The ideal section of the samples is of the order of $100 \times 100 \text{ mm}^2$ but one can go down to $45 \times 45 \text{ mm}^2$. The thicknesses can go up to 4 cm for the first and up to 2 cm for the second.

The experimental set-up shown in Figure 3.10 is identical to that of the hot plate, except that the heating element here is of a width $2b$ much smaller than the width $2L$ of the samples. It includes:

- a flat thin plane heating element to which is attached a K type thermocouple with fine wires, inserted between two samples of identical dimensions of the material to be characterized;

- two insulated aluminum blocks with identical sections to the samples. These hollow blocks are traversed by a thermal fluid coming from a thermostated bath;
- a clamping device for monitoring the clamping pressure and the thickness of the device is inserted between the aluminum blocks.

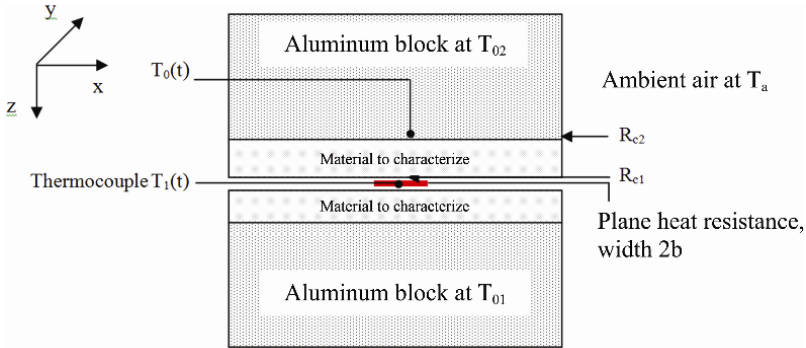


Figure 3.10. Schematic diagram of the set-up of the hot strip method with two samples

The heating element is subjected to a flux step and the temperatures T_1 in the center of the heating element and T_{01} and T_{02} of the aluminum blocks are recorded until they no longer vary.

3.4.2. Hypotheses and model

It is assumed that the heat transfer is 2D at the center of the sample and that the contact resistances are negligible in comparison with the sample's thermal resistance.

The 2D model is therefore based on the following hypotheses:

- the hot strip is long enough so that the heat transfer at its ends does not affect the temperature at the center (independent of the test shown in Figure 3.10);
- the sample is large enough so that the convective transfers on the side faces do not influence the temperature at the center of the hot strip.

Using these hypotheses, for the configuration shown in Figure 3.11, it has been shown that the temperature at the center of the hot strip is written as [JAN 11]:

$$T_1 = T_0 + \frac{b \phi_0}{\sqrt{\lambda_x \lambda_z}} K(u) \quad [3.8]$$

with: $u = \sqrt{\frac{\lambda_x e}{\lambda_z b}}$

where: $\phi_0 = \frac{R_{el} l^2}{s}$

$$K(u) = \frac{2}{\pi} \int_0^\infty \frac{\sin(\omega)}{\omega^2} \tanh(u\omega) d\omega$$

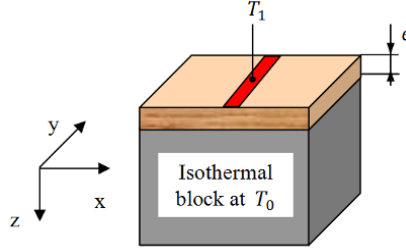


Figure 3.11. Representation of the modeled configuration (cross-section)

The function K can be represented with a precision better than 0.15% for $u \in [0.5; 5]$ by the following polynomial:

$$K(u) = a_0 + a_1 u + a_2 u^2 + a_3 u^3 + a_4 u^4 + a_5 u^5 + a_6 u^6 \quad [3.9]$$

The values of the coefficients a_i determined for $u \in [0.5; 5]$ are given in Table 3.1.

After having measured λ_z by the center hot plate method, for example, the value of λ_x can easily be deduced with a hot strip type measurement by numerical resolution of equation [3.8] with a single unknown parameter λ_x , in which we replace $K(u)$ by the polynomial given by equation [3.9].

a_6	a_5	a_4	a_3	a_2	a_1	a_0
-2.7042×10^{-4}	0.0055375	-0.0475164	0.22443	-0.655252	1.3614	-0.06099

Table 3.1. Values of the polynomial's coefficients

It has been shown that the accuracy is better the smaller the ratio of the width of the hot strip to the thickness of the sample [JAN 11].

In the case of an isotropic material, the thermal conductivity $\lambda = \lambda_x = \lambda_y = \lambda_z$ is calculated by:

$$\lambda = \frac{R_{el}}{S} \frac{e I^2}{T_s - T_0} F(v) \quad [3.10]$$

with: $F(v) = \frac{K(v)}{v}$ and: $v = \sqrt{\frac{e}{b}}$

The function $F(v)$ is shown in Figure 3.12.

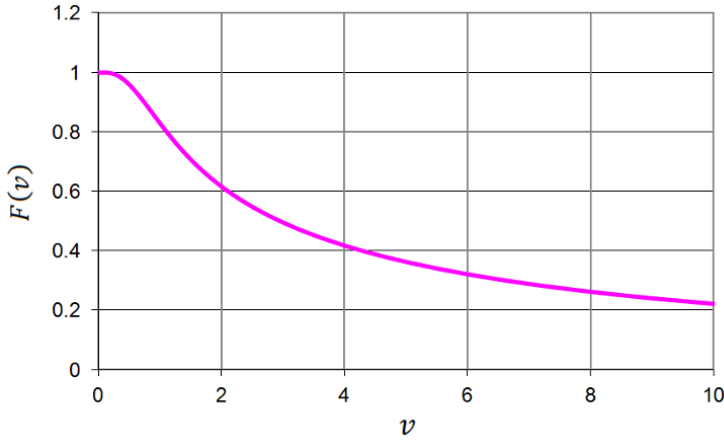


Figure 3.12. Function $F(v) = \frac{K(v)}{v}$

We will define the limits of validity of these assumptions by considering that the measurement is carried out on an isotropic sample of square section having as side the length of the hot strip with a convective exchange coefficient $h = 10 \text{ W m}^{-2}\text{K}^{-1}$ and assuming the blocks to be perfectly isothermal. The width of the hot strip will be fixed to $2b = 6 \text{ mm}$, then to $2b = 12 \text{ mm}$.

For different values of λ , we calculate the values of the sample thicknesses not to be exceeded in order for the estimation error to be kept below 1%. For all these scenarios, a complete 3D simulation is performed to calculate the value T_0 which is then used to estimate λ from equation [3.8]. The relative deviations between the nominal and estimated values are then calculated. The results obtained for a square sample of 40 mm length are given by way of example in Figure 3.13.

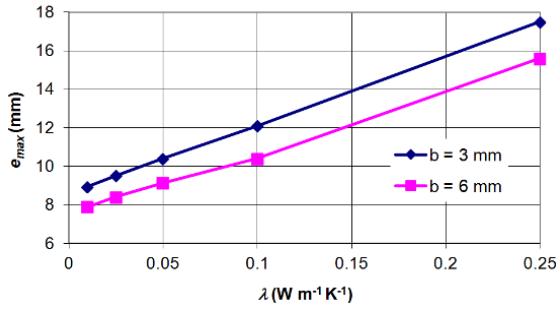


Figure 3.13. Sample thickness $e_{\max}$ corresponding to an estimation error of 1% on λ for a square sample of 40 mm on each side. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

It can be seen that for a hot strip of width 6 mm and length 40 mm, the simplified model is valid for thicknesses up to 9 mm for superinsulators and for more than 15 mm for materials of thermal conductivity in the order of $0.25 \text{ W m}^{-1} \text{ K}^{-1}$. It is therefore not necessary to have samples of very large cross-sections to measure the conductivity of materials whose thickness is less than 10 mm.

NOTE.—

The principle of measurement is to use two experiments with different $\frac{e}{b}$ ratios, an error calculation shows that the best configuration is to choose the two extreme cases $\frac{e}{b} \rightarrow 0$ (i.e. the center hot plate) and $\frac{e}{b} \rightarrow \infty$ (i.e. close to the hot wire). In the case $\frac{e}{b} \rightarrow 0$, we directly measure λ_z , and in the case $\frac{e}{b} \rightarrow \infty$, we directly measure the geometric mean of λ_x and λ_z that is $\lambda_m = \sqrt{\lambda_x \lambda_z}$.

Assuming that the relative errors in the two experiments are identical (the same experimental set-up), we obtain $\lambda_x = \frac{\lambda_m^2}{\lambda_z}$ that is:

$$\frac{\Delta \lambda_x}{\lambda_x} = 2 \frac{\Delta \lambda_m}{\lambda_m} + \frac{\Delta \lambda_z}{\lambda_z} \text{ and consequently: } \frac{\Delta \lambda_x}{\lambda_x} = 3 \frac{\Delta \lambda_z}{\lambda_z}$$

The relative error on λ_x is therefore at least three times greater than that on λ_z . In practice, we are closer to six times than three times, so we cannot expect to get a relative error on λ_x better than 10%.

3.4.3. Experimental set-up

The experimental set-up is identical in all respects to that described in section 3.3.3 with a thermal resistance of small width $2b$ with respect to the width $2L$ of the samples.

3.4.4. Practice of the measurement

This measurement method is not absolute: it is necessary to have at least one reference sample to realize a calibration. Indeed, the thermal conductivity is proportional to the ratio $\frac{R_{el}}{S}$ to be determined beforehand. A measurement of R_{el} by an ohmmeter and the determination of S by measurement of the sides already make it possible to have an approximate value. However, it is preferable to estimate it by carrying out a measurement with an isotropic reference sample of thermal conductivity λ_{ref} of thickness situated with the range of values e_{min} and e_{max} defined in Figure 3.13. From this we deduce the value of $\frac{R_{el}}{S}$:

$$\frac{R_{el}}{S} = \frac{\lambda_{ref}(T_s - T_0)}{eI^2F(v)} \quad [3.11]$$

A study was carried out using two heating elements:

- A heating resistive element (1) with a thickness of 0.25 mm, a length of 44.0 mm and a width of 6.5 mm.
- A heating resistive element (2) of thickness 0.25 mm, length 44.0 mm and width 44.0 mm.

The ratio values $\frac{R_{el}}{S}$ were determined for the two elements by measurement on samples of opacified EPS foam of square cross-section of 44 mm on each side, thickness $e = 5$ mm and thermal conductivity $\lambda = 0.0309 \text{ W m}^{-1} \text{ K}^{-1}$ at 10°C .

Three measurements, shown in Figure 3.14, were then carried out on samples of Ayous, light tropical wood, of dimensions $8 \times 44 \times 44 \text{ mm}^3$.

The measurement results are shown in Table 3.2. The calculated values of the thermal conductivities are presented in Table 3.3, which also includes the values deduced from the measurement of thermal effusivity by the transient hot plate method and the thermal diffusivity by the flash method. The results are in very good agreement and show a strong anisotropy of this material.

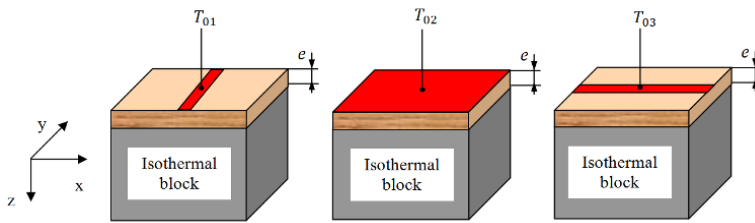


Figure 3.14. *Diagram of realized measurements*

	Measurement 1		Measurement 2		Measurement 3	
Sample dimensions	$8 \times 44 \times 44 \text{ mm}^3$					
Strip dimensions	$6.5 \times 44 \text{ mm}^2$		$44 \times 44 \text{ mm}^2$		$6.5 \times 44 \text{ mm}^2$	
Experiment N	ϕ	T_{01}	ϕ	T_{02}	ϕ	T_{03}
1	188.9	7.63	97.03	7.65	251.2	9.11
2	242.7	9.83	144.9	11.33	294.8	10.77
3	303.2	12.34	202.4	15.6	341.9	12.52

Table 3.2. *Experimental results of ϕ (W m^{-2}) and T_{0i} ($^{\circ}\text{C}$)*

Hot strip			Hot plate + Flash		
λ_z	λ_x	λ_y	λ_z	λ_x	λ_y
0.104	0.094	0.184	0.103	0.103	0.190

Table 3.3. *Estimated values of thermal conductivities ($\text{W m}^{-1} \text{K}^{-1}$) in the three directions*

3.5. Hot tube

3.5.1. Principle

Knowledge of the thermal properties and in particular of the thermal conductivity of bulk products as a function of their moisture is of great interest in several fields, among which are:

- thermal insulation with the current development of natural insulators such as cork, vermiculite or hemp. These insulators are hydrophilic and it is essential to know the evolution of their thermal conductivity as a function of the humidity to predict their *in situ* performance;

– the food industry: energy consumption during operations such as drying, pasteurization, roasting, etc. depends strongly on the thermal properties of the product.

Measurement of the thermal properties of wet materials is a poorly understood field. Measurement methods are based on the measurement of temperature rise resulting from product heating and assume that the vapor transfer induced by heating does not influence the temperature of the product. This hypothesis may lead to erroneous results, in particular for unsteady-state methods. The main method used to measure the thermal conductivity of powders or clusters is the hot wire method, which is a transient method. Moreover, this method is unsuitable for products with a high granulometry due to the small number of contact points between the heating surface (small diameter wire) and the product. The method of the guarded hot plate is a method of measurement for the steady state but poorly adapted to the powders or wet clusters around which a perfect seal must be ensured.

The “Hot Tube” method shown in Figure 3.15 is an absolute steady-state method in which the wire is replaced by a tube. This tube is inserted into another tube of larger diameter maintained at constant temperature. Two discs ensure the centering of the inner tube with respect to the outer tube. The annular space between the two tubes is filled by the product to be characterized. The sealing at both ends which must be perfect for precise measurement is achieved by a plastic film. An electrical potential difference is applied between the two ends of the inner tube traversed by a current of intensity I and in which a release of heat by the Joule effect occurs. Knowledge of the produced heat flow rate φ and of the temperatures T_1 and T_2 of the two tubes, measured at mid-height, makes it possible to estimate the material's thermal conductivity.

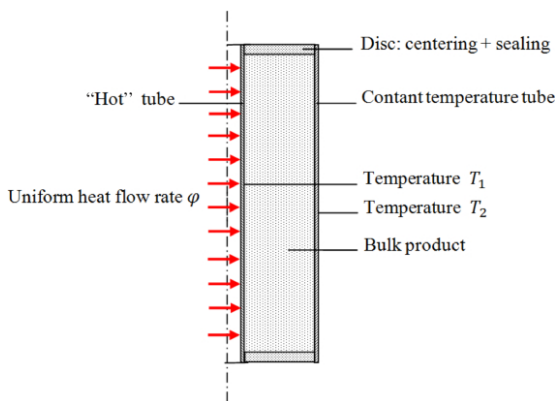


Figure 3.15. Schematic diagram of the hot tube set-up

3.5.2. Hypotheses and model

Modeling is based on the following assumptions:

- the axial temperature gradients are negligible at mid-height of the device (1D radial heat transfer);
- the device is watertight: no transfer of steam to the outside;
- the heat flow rate is uniform on the hot tube;
- the water vapor pressure is uniform in the medium and there is therefore no mass transfer.

Under these conditions, we have the following equation in steady state:

$$\varphi = \frac{2 \pi \lambda L \Delta T}{\ln\left(\frac{R_2}{R_1}\right)} \quad [3.12]$$

The value of the thermal conductivity λ is deduced therefrom by:

$$\lambda = \frac{\varphi \ln\left(\frac{R_2}{R_1}\right)}{2 \pi L \Delta T} \quad [3.13]$$

where:

φ is the heat flow rate produced by the Joule effect in the inner tube;

R_2 is the inner diameter of outer tube;

R_1 is the outer diameter of inner tube;

L is the length of the inner tube traversed by current I ;

ΔT is the temperature difference between the two tubes in steady state.

The heat flow rate is calculated by:

$$\varphi = R_{el} I^2 \quad [3.14]$$

where:

I is the intensity (A);

R_{el} is the electrical resistance of the tube.

The latter can be calculated by:

$$R_{el} = \frac{\rho_e L}{\pi (R_1^2 - R_0^2)} \quad [3.15]$$

where:

ρ_e is the electrical resistivity of the inner tube;

R_0 is the inner tube radius;

R_1 is the outer radius of the inner tube.

3.5.3. Experimental set-up

The patented “Hot Tube” device [JAN 13] is shown in Figure 3.16. It consists of two coaxial tubes:

- an inner tube made of stainless steel with inner radius $R_0 = 4$ mm, outer radius $R_1 = 4.5$ mm and height $L = 380$ mm.

- an outer brass tube with an inner radius $R_2 = 39$ mm, an outer radius $R_3 = 40$ mm and a height $L = 426$ mm.

The outer diameter of the inner tube was chosen to be large enough (9 mm) so that the number of contact points between the grains and the tube is important even for grains with large diameters.

For the set-up at ambient temperature, the outer surface of the outer tube is in direct contact with the air. It exchanges heat with the surrounding environment by both convection and radiation.

For a set-up allowing measurement at different temperatures, the outer tube is in contact with a fluid circulation (see Figure 3.16).

Two K type thermocouples are placed at mid-height respectively on the inner tube to measure T_1 and on the outer tube to measure T_2 . On the outer brass tube, thermocouple wires with a diameter of 0.1 mm are bonded to the outer surface a few millimeters apart to provide a set-up with separate contacts.

Since the internal tube is traversed by a high intensity current (up to 8 A), the temperature measurement is distorted by the passage of this current if the thermocouple is in electrical contact with the tube, the bias can be greater than 2°C and completely distort the measurement of thermal conductivity. The surface of the

heating tube is thus entirely covered with a very thin film of insulating varnish deposited by spraying before fixing the thermocouple (whose hot weld is previously produced by spot welding) using a piece of aluminum foil adhesive of thickness 0.05 mm to promote transfer between the thermocouple and the tube.

The centering of the inner tube is ensured by two hollow discs of expanded PVC with a thickness of 8 mm, an internal diameter of 9 mm and an outer diameter of 78 mm. These two discs placed respectively at the bottom and at the top of the tubes make it possible to maintain the product to be measured in the annular space between the tubes and to isolate it from the external environment.

Two discs made of machinable polyurethane with low thermal conductivity ensure the sealing with respect to the external environment. This sealing can be enhanced by placing a waterproof stretchable film on the outer surface of the discs. An adjusted hole is made in the center of these discs to allow the welded wire to pass from one side to the brass ring and the other to the removable banana plug.

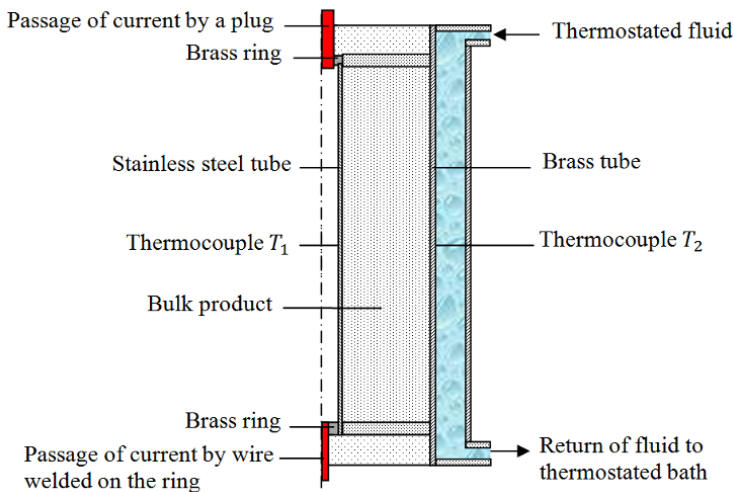


Figure 3.16. Schematic diagram of the hot tube device (Patent Jannot and Degiovanni, 2013)

The electrical resistivity ρ_e ($\mu\Omega \cdot \text{cm}^{-1}$) of 304L stainless steel between 0°C and 77°C is given by:

$$\rho_e = 71.3 + 1.261 \times 10^{-3}T + 7.76 \times 10^{-4} T^2 \quad [3.16]$$

where T is the heating tube temperature ($^{\circ}\text{C}$).

The measured value of the resistance of a stainless steel tube of the same diameter as that used in our device by a four-wire set-up led us to the value: $R = 0.0545\Omega$ for $L = 1005\text{ mm}$ at 26°C . This corresponds to a value of the electrical resistivity $\rho_e = 72.4\text{ }\mu\Omega\cdot\text{cm}^{-1}$ which differs by less than 1% from the calculated value ($\rho_e = 71.9\text{ }\mu\Omega\cdot\text{cm}^{-1}$) from equation [3.16].

We have therefore retained equation [3.16] for the calculation of the tube's electrical resistance.

The thermal conductivity is therefore finally calculated by:

$$\lambda = \frac{\rho_e \ln\left(\frac{R_2}{R_1}\right)}{2\pi^2 (R_1^2 - R_0^2)} \frac{I^2}{\Delta T} \quad [3.17]$$

Considering the dimensions of the device:

$$\lambda = 0.0002574 \left(71.3 + 1.261 \times 10^{-3} T_1 + 7.76 \times 10^{-4} T_1^2 \right) \frac{I^2}{\Delta T} \quad [3.18]$$

The validity of the hypothesis “1D transfer at mid-height of the tube” was verified by performing 3D simulations of the device using the COMSOL software. Simulated temperature T_1 and T_2 values at mid-height were used to estimate the thermal conductivity λ using formula [3.17]. This value was then compared with the nominal value introduced in the simulation. The dimensions of our device have been fixed in such a way that the difference between the two values is less than 1%.

3.5.4. Practice of the measurement

3.5.4.1. Measurement on solids

The experimental procedure is as follows: the annular space between the two tubes is filled with the product to be characterized, it is not necessary to wait for the temperatures T_1 and T_2 to reach an identical stable value. A current of intensity I is then passed through the inner tube made of stainless steel. Temperatures T_1 and T_2 are recorded from the start of heating until their difference reaches a constant value.

The thermal conductivity is deduced from this value ΔT .

A comparative experimental study was carried out with the conventional device of the hot wire on products for which the latter method is suitable, namely dry products with a grain size smaller than one millimeter. Measurements have also

been made with the device of the cylindrical tri-layer. The following products were measured:

- polystyrene beads;
- glass beads;
- dry cork in the form of pellets.

The experimental results obtained by the three methods are given in Table 3.4.

Material	Hot tube					Hot wire	Tri-layer	Max. deviation
	°C	°C	°C	A	W m ⁻¹ K ⁻¹	W m ⁻¹ K ⁻¹	W m ⁻¹ K ⁻¹	%
	T_1	T_2	ΔT	I	λ	λ	λ	
Polystyrene beads	34.7	26.6	8.1	4.03	0.0373	0.0378	0.0364	3.8
Dry cork	34.7	26.8	7.9	4.03	0.0382	0.0391	0.0384	2.4
Glass beads	31.0	26.4	4.8	6.95	0.187	0.185	0.188*	1.1

* The value deduced from a measurement of the diffusivity a by the cylindrical tri-layer method and a measurement of the calorific volume capacity ρc by differential calorimetry.

Table 3.4. *Measured values of thermal conductivity with the Hot Tube, Hot Wire and Cylindrical Tri-layer methods*

The deviations between the values measured by the hot tube and the other devices are less than 5%, which can be considered satisfactory. Indeed, it is difficult to reproduce identically a cluster of beads or grains. The thermal conductivity which depends, to a large extent, on the contacts between the solid elements can thus vary from one filling to another. The discrepancies between the different measured values of the thermal conductivity can therefore be attributed in part to this variability, which makes the value of 5% all the more acceptable.

SPECIAL CASE OF WET MATERIALS.—

The hot tube device is ideally suited for measuring the thermal conductivity of clustered moist materials provided that the following considerations are taken into account:

- if we want to measure the thermal conductivity of a cluster with precise water content, it is necessary to prevent any transfer of water vapor between the material

and the outside. All that is required is to seal the top and bottom of the tube after having introduced the product to be characterized. This can be achieved simply with stretch film, for example.

– if the material is moist, the heating induces mass transfers within the cluster. The time taken to achieve equilibrium is much longer than in the case of a dry material because the mass diffusion coefficient is much lower than the thermal diffusion coefficient. You have to be very patient!

3.5.4.2. Measurement of liquids

A device similar to that described above for bulk products can be used for measuring the thermal conductivity of liquids. The additional constraint to be respected in this case is to place oneself in conditions such that there is no movement of convection within the liquid. For this it is necessary that [HUE 90]:

$$Ra_{\delta} \leq 400 \frac{H}{\delta} \quad [3.19]$$

where: H is the tube height in m;

δ is the thickness of liquid between the two tubes in m;

$$Ra_{\delta} \text{ is Rayleigh's number: } Ra_{\delta} = \frac{cg\beta\Delta T\rho^2\delta^3}{\lambda\mu}.$$

where: c is the heat capacity in $\text{J kg}^{-1} \text{K}^{-1}$;

g is the acceleration in m s^{-2} ($g = 9.81 \text{ m s}^{-2}$);

β is the coefficient of expansion;

ΔT is the temperature difference in $^{\circ}\text{C}$;

ρ is the density in kg m^{-3} ;

δ is the thickness of liquid between the two tubes in m;

λ is the thermal conductivity in $\text{W m}^{-1} \text{K}^{-1}$;

μ is the dynamic viscosity in Pa s .

By way of example, a device of identical design to that produced for the clusters (see Figure 3.17) but having the following dimensional characteristics was produced at LEMTA [HAU 13]:

- an inner tube made of stainless steel with inner radius $R_0 = 2.5$ mm, outer radius $R_1 = 2.75$ mm and height $L = 200$ mm;
- an outer copper tube with inner radius $R_2 = 10$ mm, outer radius $R_3 = 12$ mm and height $L = 200$ mm.

This is illustrated in Figure 3.17.

Equation [3.17] remains valid for the calculation of the thermal conductivity of a liquid. Given the low value of the thermal resistance of the liquid it is necessary to use a high enough intensity in the stainless steel tube to observe a sufficient temperature rise (at least 3°C). For water whose thermal conductivity is of the order of $0.60 \text{ W m}^{-1} \text{ K}^{-1}$, an intensity of the order of 10 A is required.

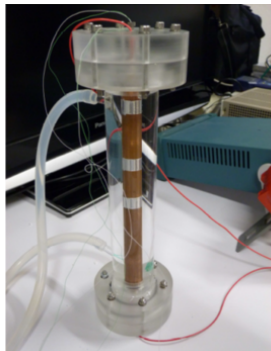


Figure 3.17. *View of a hot tube device for measuring the thermal conductivity of liquids*

This device made it possible to measure the thermal conductivity of water between 10 and 60°C with accuracy better than 3%.

3.6. Cut bar

3.6.1. Principle

This method, also called “divided bar method” or “comparison method”, is a steady-state method for the measurement of the thermal conductivity of solids. It is used mainly for the measurement of the conductivity of rocks and ceramics and also

of metals at high temperature [SAS 84, MCG 14, XIN 14]. It can be implemented in two main versions, the basic schematic diagrams of which are shown in Figures 3.18 and 3.19.

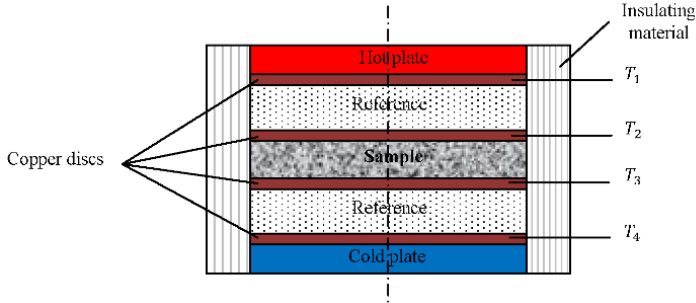


Figure 3.18. Schematic diagram of version 1 of the cut bar method

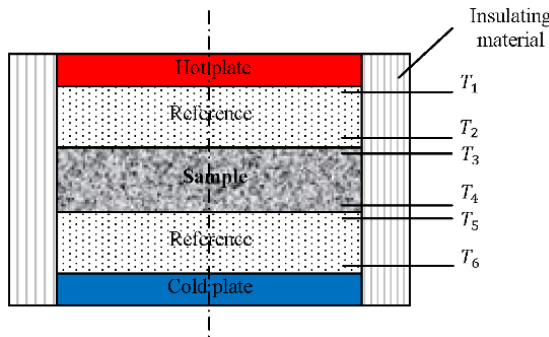


Figure 3.19. Schematic diagram of version 2 of the cut bar method

Temperature measurements are used to estimate both the heat flux ϕ_0 passing through the sample to be characterized and the temperature gradient $\frac{dT}{dx}$ in the same sample. By applying the Fourier law in a steady state and in 1D, we deduce the value of the thermal conductivity of the sample by:

$$\lambda = -\frac{\phi_0}{\frac{dT}{dx}} \quad [3.20]$$

3.6.2. Hypotheses and model

CUT BAR METHOD VERSION 1.–

Knowing the thermal resistances of the reference samples and the values of the temperatures, it is possible to deduce therefrom the value of the heat flux ϕ_0 passing through the system by:

$$\phi_0 = \frac{\lambda_{ref}}{e_{ref}} \left(\frac{T_1 - T_2}{2} + \frac{T_3 - T_4}{2} \right) \quad [3.21]$$

Then the thermal conductivity of the sample by:

$$\lambda = \frac{e \phi_0}{T_2 - T_3} \quad [3.22]$$

The following assumptions were made implicitly to write equations 3.21 and 3.22:

- the heat transfer is unidirectional, the lateral heat losses are negligible compared to the flow passing through the sample;
- the thermal resistances at the interfaces are negligible in comparison with the thermal resistance of the sample to be characterized and those of the reference samples.

We see that these two hypotheses are hardly feasible simultaneously:

- if the resistance of the sample is large (the second hypothesis verified), the heat flux passing through it for a given temperature deviation is small and the lateral losses are no longer necessarily negligible. This method as shown in Figure 3.18 will therefore not apply to thermal insulation;
- if the resistance of the sample is small (first hypothesis verified), the contact resistances are no longer necessarily negligible. This method as shown in Figure 3.18 will therefore not apply to highly conductive materials.

These remarks justify the fact that version 1 is used for rocks having conductivities between $\lambda = 1 \text{ W m}^{-1} \text{ K}^{-1}$ and $\lambda = 5 \text{ W m}^{-1} \text{ K}^{-1}$.

CUT BAR METHOD VERSION 2.–

The only hypothesis is that of the 1D transfer, no hypothesis is made concerning the contact resistances. This version 2 can thus *a priori* allow the measurement of more conductive materials than is possible with version 1. However, it is

unsuitable for the thermal characterization of insulators. The value of the heat flow density passing through the system is calculated by:

$$\phi_0 = \frac{\lambda_{ref}}{e_{ref}} \left(\frac{T_1 - T_2}{2} + \frac{T_5 - T_6}{2} \right) \quad [3.23]$$

Then, the thermal conductivity of the sample is calculated by:

$$\lambda = \frac{e \phi_0}{T_3 - T_4} \quad [3.24]$$

3.6.3. Experimental set-up

In the device shown in Figures 3.18 and 3.19, the hot source is constituted by an electric resistance traversed by a current of adjustable intensity. The cold source is either a metal block traversed by a thermostatic fluid or a Peltier element. Temperature measurements are made with thermocouples made up of very small diameter wires (a few hundredths of a mm).

In version 1 (see Figure 3.18), a groove can be made on the surface of the copper discs to house the thermocouples without creating additional thermal resistances. In version 2 (see Figure 3.19), holes of very small diameters must be made in the samples in order to insert fine thermocouples. Conductive grease or oil at the interfaces will reduce the contact resistance, provided that the samples are not porous since the grease or oil could penetrate the material and modify its properties. Pressure must also be applied to the assembly to reduce contact resistances.

3.6.4. Practice of the measurement

VERSION 1.—

The main source of measurement bias in this version is the existence of neglected contact resistances in the model. It will therefore be necessary to ensure a decent flatness of the surfaces and parallelism between the faces of the same sample. Too large defects cannot be completely compensated by a contact fluid (oil or grease). The advantage of this version is the accuracy of the temperature measurement: it is uniform throughout the copper plate in view of its high thermal conductivity and its measurement by contact is facilitated by the copper's very high effusivity. The maximum uncertainty can be estimated by:

$$\frac{\Delta \lambda}{\lambda} = \frac{\Delta \lambda_{ref}}{\lambda_{ref}} + \frac{\Delta e_{ref}}{e} + \frac{\Delta e}{e_{ref}} + \frac{2\Delta T}{(T_2 - T_3)} + \frac{4\Delta T}{(T_1 - T_2) + (T_3 - T_4)} \quad [3.25]$$

To minimize this uncertainty, the temperature differences must be of the same order of magnitude. It is therefore necessary that the thermal resistances of the reference samples and of the sample to be characterized are also of the same order of magnitude. This therefore requires having *a priori* several reference samples covering the range of thermal resistances to be measured.

By way of indication, considering an uncertainty of 0.05°C on temperature measurements and temperature differences of the order of 10°C in order to be able to consider the constant thermal properties, the last two terms of equation [3.25] lead to a relative uncertainty of 2%. To this value is added the relative uncertainty on λ_{ref} which is of the order of 2%, i.e. a total of more than 4% if the uncertainties on the thicknesses are taken into account.

To estimate the error induced by the non-respect of the 1D transfer hypothesis (which is added to the one calculated by equation [3.25]), the simplest way is to perform a numerical simulation of the whole device in 3D (or rather axisymmetric 2D) by giving the sample to be characterized a conductivity value equal to that measured. The numerical simulation makes it possible to calculate the four temperatures and to deduce a new estimated value of the thermal conductivity λ_1 using equation [3.22]. The comparison between the values of λ_0 and λ_1 allows the estimation of the value and the direction of the measurement error due to the non-respect of the 1D hypothesis.

By way of example, we used the COMSOL software to simulate the device represented in Figure 3.18 by considering samples with a diameter of 40 mm, surrounded by an insulator with a thickness of 20 mm and thermal conductivity $\lambda = 0.04 \text{ W m}^{-1} \text{ K}^{-1}$. The characteristics of the samples for the different cases tested are shown in Table 3.5. The configuration is simulated where the heating resistor and the cold source are set so that: $T_1 - T_4 = 60^{\circ}\text{C}$. For each configuration, two cases will be studied:

- temperature of the cold face equal to the ambient temperature: all the points of the device are at a temperature higher than the ambient air;
- ambient temperature equal to the mean temperature of the hot and cold sources in order to straighten the flow lines in the sample to be characterized, as shown by the COMSOL simulation results presented in Figure 3.20.

The results show that the accuracy of the measurement is significantly better in the case where $T_a = \frac{T_1 + T_4}{2}$ with an average error of the order of 3% (see Table 3.6). It is also observed that the error is less when the three samples (references and unknown) have the same thermal conductivity.

On the example discussed, it is advantageous to carry out a 3D numerical simulation of the device, which makes it possible to choose the best configuration and to predict the accuracy of the measurement. It also shows that without this preliminary study, the relative error on the estimation of the thermal conductivity due to the non-respect of the 1D hypothesis can quickly reach or even exceed 10%. It should not be forgotten that this uncertainty is compounded by equation [3.25] which can be greater than 4%.

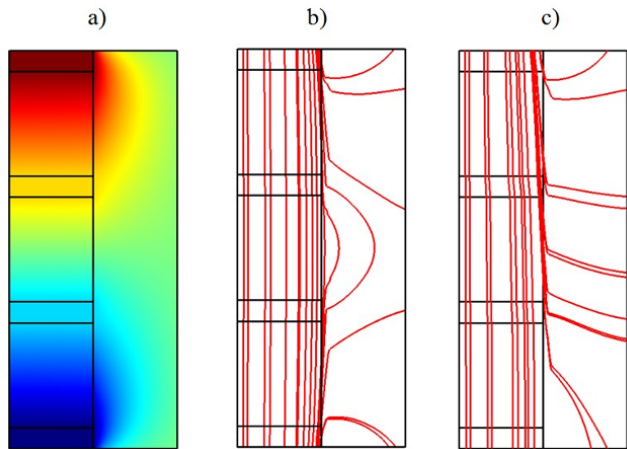


Figure 3.20. COMSOL simulation results: a) temperature field, b) flow lines with $T_a = \frac{T_1 + T_4}{2}$, c) flow lines with $T_4 = T_a$. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Case N°		Reference		Material to measure		Copper	
	$T_1 - T_a$	e_{ref}	λ_{ref}	e	λ	e_{Cu}	λ_{Cu}
	°C	mm	W m ⁻¹ K ⁻¹	mm	W m ⁻¹ K ⁻¹	mm	W m ⁻¹ K ⁻¹
1	60	25	2.5	25	1	5	400
2	60				2.5		
3	60				5		
4	30				1		
5	30				2.5		
6	30				5		

Table 3.5. Characteristics of the samples for the different tested cases

Case N°	$T_1 - T_a$	$T_2 - T_a$	$T_3 - T_a$	$T_4 - T_a$	λ_1	Error
			°C	°C	$\text{W m}^{-1} \text{K}^{-1}$	%
1	60	11.88	46.76	0	1.10	10.3
2	60	18.09	37.53	0	2.61	4.3
3	60	21.91	32.70	0	5.70	14.0
4	30	15.95	-16.95	-30	1.03	3.0
5	30	9.71	-9.72	-30	2.61	4.3
6	30	5.88	-5.90	-30	5.12	2.3

Table 3.6. Results of numerical simulations of the cut bar version 1 method

VERSION 2.—

In this version, temperatures are measured by fine thermocouples inserted into holes made directly in the reference samples and in the sample to be measured. There are two disadvantages:

- an uncertainty on the axial and vertical positions of each thermocouple;
- temperature bias due to possible contact resistances at the end of the thermocouple and a fin effect in the wires.

On the contrary, the problem of the contact resistances no longer exists except for temperature differences $(T_1 - T_2)$, $(T_3 - T_4)$, and $(T_5 - T_6)$ and may be small if the resistances of the samples are small compared to the thermal contact resistances (in the case of highly conductive metals).

Cases 1 to 6 referenced in Table 3.5 were simulated in 3D in COMSOL in the version 2 of the cut bar method. Temperatures were calculated at 2.5 mm from the interfaces, the thickness of the samples being 25 mm. The distance between two measurement points in the same sample is therefore 20 mm. The results are reported in Table 3.7.

Case N°	$T_1 - T_a$	λ_1	Error
	°C	$\text{W m}^{-1} \text{K}^{-1}$	%
1	60	1.10	6.6
2	60	2.61	3.0
3	60	5.70	1.8
4	30	1.03	6.7
5	30	2.61	2.5
6	30	5.12	1.7

Table 3.7. Results of numerical simulations of the cut bar version 2 method

It should be noted that in this version the temperature varies slightly between the center and the periphery of the samples. The values reported in Table 3.7 were calculated at the center of the samples. Between a measurement at the center ($r = 0$) and a measurement offset by 1 cm ($r = 1\text{cm}$), an increase in the error in the order of 1% is noted for the studied cases.

Assuming that the distances between the thermocouples in the reference samples are perfectly known and that the position uncertainty of the thermocouples is 0.2 mm in the sample (an uncertainty of 0.4 mm over the distance separating them), an uncertainty of 2 to 3% is added to that calculated in Table 3.7.

So for version 2 as for version 1, it is necessary to carry out 3D numerical simulations of the device to evaluate the uncertainty on the estimated value of the thermal conductivity and to choose the optimal configuration due mainly to:

- failure to comply with hypothesis 1D;
- the uncertainty about the positioning of the six thermocouples.

It should not be forgotten that the measurement uncertainty thus calculated is added to the uncertainty due to errors on the measurements of the various quantities and which can be calculated by the following equation:

$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta\lambda_{ref}}{\lambda_{ref}} + \frac{\Delta e}{e} + \frac{\Delta e_{ref}}{e_{ref}} + \frac{2\Delta T}{(T_3 - T_4)} + \frac{4\Delta T}{(T_1 - T_2) + (T_5 - T_6)} \quad [3.26]$$

This formula is similar to formula [3.25] and therefore the conclusions are the same as for version 1, with in particular an uncertainty that can be greater than 4%.

EXAMPLE OF MEASUREMENT INSTALLATION.—

We will describe a practical measurement installation for engineering students designed by DIDATEC (PCT 010 study bench for conduction).

The bench includes a device for creating a longitudinal flow in a cylindrical sample. Two samples isolated laterally by Teflon are available:

- one stainless steel, diameter of 25 mm and length 30 mm;
- one brass, diameter of 25 mm and length 30 mm.

The samples are placed between two brass cylinders ($\lambda = 110 \text{ W m}^{-1} \text{ K}^{-1}$) of 25 mm diameter isolated by Teflon, one heated by the Joule effect and the other cooled by water circulation.

A total of nine temperatures are recorded (thermocouple type K): three on the hot bar, three on the sample and three on the cold bar. The power injected by the Joule effect is measured. The thermocouples are spaced 10 mm apart. Their positions are $x = 0, 10, 20, 30, 40, 50, 60, 70, 80$ mm.

Figure 3.21 shows the experimental bench.



Figure 3.21. *Cut bar type experimental bench*

In Figure 3.22, we give the results obtained by least squares identification of the nine measurements with a 1D conduction model taking into account the contact resistances (MATLAB program) for the brass sample and Figure 3.23 for the stainless steel sample.

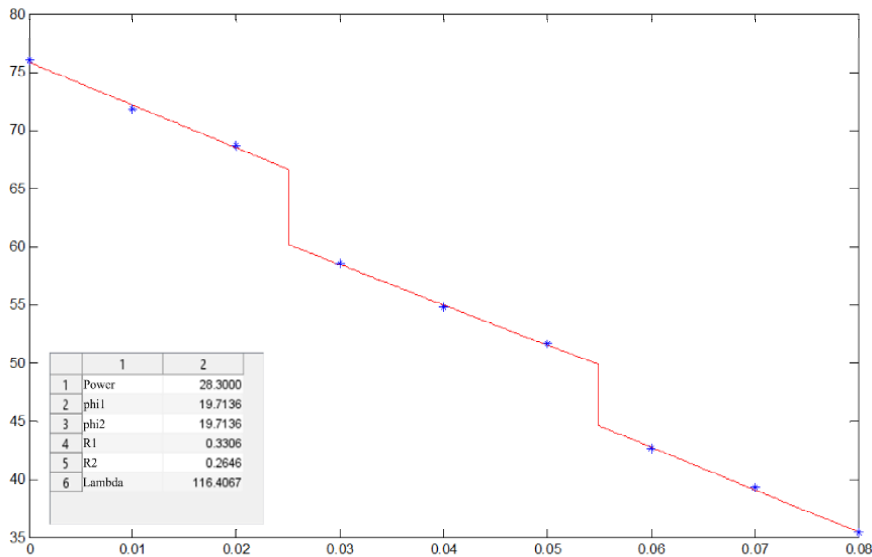


Figure 3.22. *Results for the brass sample*

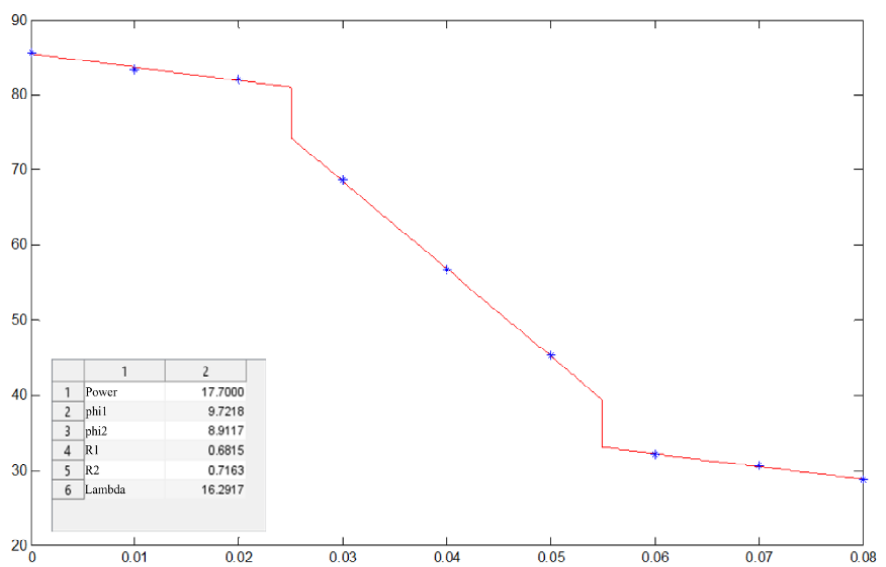


Figure 3.23. Results for the stainless steel sample

Flux/Temperature Transient Methods

4.1. Introduction

By definition, steady-state measurement methods only measure thermal conductivity λ since it is the only property that appears in steady-state heat transfer equations. Knowledge of a second property necessary for thermally characterizing a material must therefore be realized by a transient or periodic type method. This second property can be:

- Thermal effusivity E
- Thermal diffusivity a
- Volumetric heat capacity ρc

It is recalled that the knowledge of two thermal properties makes it possible to deduce the other two by means of equations:

$$a = \frac{\lambda}{\rho c}$$
$$E = \sqrt{\lambda \rho c}$$
[4.1]

4.2. Infinite hot plate

4.2.1. Principle

This is an absolute transient method used to measure the thermal effusivity of a fairly wide range of consolidated or unconsolidated solids. In the classical version of this method, a flat thin heating element is inserted between two samples of the

material to be characterized (see Figure 4.1). A thermocouple placed at the center of the heating element allows the temperature to be measured. Starting from a situation of initial thermal equilibrium, a heat flux step is applied to the heating element and the rise in temperature over time is recorded. The comparison of the experimental data with a model makes it possible to estimate the thermal effusivity of the material constituting the two samples.

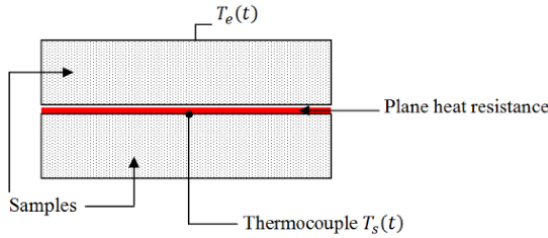


Figure 4.1. Schematic diagram of the hot plate method

4.2.2. Hypotheses and model

In this method, the following hypotheses are assumed:

- the samples are considered as semi-infinite media during the time of measurement;
- the heat transfer is 1D at the center of the sample;
- the thermal resistance of the heating element is zero.

4.2.2.1. Complete model

The heat equation is written in the sample:

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{a} \frac{\partial T}{\partial t}$$

with boundary conditions: $T(x, 0) = T_s(0) = T_i$

$$T(\infty, t) = T_i$$

$$\frac{T_s(t) - T(0, t)}{R_c} = -\lambda \frac{\partial T(0, t)}{\partial x}$$

$$\frac{\phi_0}{2} = \frac{(mc)_S}{2S} \frac{dT_s}{dt} + \frac{T_s(t) - T(0, t)}{R_c}$$

- where: $(mc)_s$ Product of mass and specific heat of the heating element (J K^{-1})
- ϕ_0 heat flux produced in the heating element (W m^{-2})
- R_c contact resistance at sample/heating interface ($\text{K m}^2 \text{W}^{-1}$)
- $T(x, t)$ temperature within the sample ($^\circ\text{C}$)
- S surface area of the heating element (m^2).

The last two equations reflect the conservation of the heat flux at the surface of the semi-infinite medium on the one hand and at the level of the heating element on the other.

The following variable is changed: $\bar{T} = T - T_i$

hence: $\frac{\partial \bar{T}}{\partial x} = \frac{\partial T}{\partial x}$, $\frac{\partial^2 \bar{T}}{\partial x^2} = \frac{\partial^2 T}{\partial x^2}$ and $\frac{\partial \bar{T}}{\partial t} = \frac{\partial T}{\partial t}$

The heat equation can then be written as:

$$\frac{\partial^2 \bar{T}}{\partial x^2} = \frac{1}{a} \frac{\partial \bar{T}}{\partial t}$$

and the boundary conditions become:

$$\bar{T}(x, 0) = 0$$

$$\bar{T}(\infty, t) = 0$$

$$\frac{\bar{T}_s(t) - \bar{T}(0, t)}{R_c} = -\lambda \frac{\partial \bar{T}(0, t)}{\partial x}$$

$$\frac{\phi_0}{2} = \frac{(mc)_s}{2S} \frac{d\bar{T}_s}{dt} + \frac{\bar{T}_s(t) - \bar{T}(0, t)}{R_c}$$

The Laplace transform of the heat equation leads to: $\frac{d^2 \theta}{dx^2} - \frac{1}{a} [p\theta - \bar{T}(x, 0)] = 0$ with $\bar{T}(x, 0) = 0$

Hence: $\theta(x, p) = A \exp(-qx) + B \exp(qx)$, where the temperature retains a finite value when x approaches infinity; therefore, $B = 0$ and we deduce that: $\theta(x, p) = A \exp(-qx)$ with: $A = \theta(0, p)$.

The Laplace transforms of the boundary equations are written as:

$$\frac{\theta_s(p) - \theta(0,p)}{R_c} = -\lambda \frac{\partial \theta(0,p)}{\partial x} = \lambda \sqrt{\frac{p}{a}} \theta(0,p) = E\sqrt{p} \theta(0,p)$$

$$\frac{\phi_0}{2p} = \frac{(mc)_s}{2S} p \theta_s(p) + \frac{\theta_s(p) - \theta(0,p)}{R_c}$$

By eliminating $\theta(0,p)$ from these two equations, we get the following result:

$$\theta_s(p) = \frac{\phi_0}{2p} \frac{1 + R_c E \sqrt{p}}{\frac{(mc)_s}{2S} p + \left(1 + R_c \frac{(mc)_s}{2S} p\right) E \sqrt{p}} \quad [4.2]$$

The unknown parameters to be experimentally determined are:

- the sample's effusivity E ;
- the thermal contact resistance R_c between the probe and the sample;
- the probe's thermal capacitance $(mc)_s$.

4.2.2.2. Simplified model

For long times ($p \rightarrow 0$), this expression becomes:

$$\theta_s(p) = \frac{\phi_0}{2p} \frac{1 + R_c E \sqrt{p}}{\frac{(mc)_s}{2S} p + \left(1 + R_c \frac{(mc)_s}{2S} p\right) E \sqrt{p}} \approx \frac{\phi_0}{2Ep^{3/2}} \frac{1 + R_c E \sqrt{p}}{1 + \frac{(mc)_s}{2ES} \sqrt{p}} \approx \frac{\phi_0}{2Ep^{3/2}} \left(1 + R_c E \sqrt{p}\right) \left[1 - \frac{(mc)_s}{2ES} \sqrt{p}\right]$$

$$\text{Or: } \theta_s(p) = \frac{\phi_0}{2Ep^{3/2}} \left[1 + \left(R_c E - \frac{(mc)_s}{2ES}\right) \sqrt{p}\right] = \frac{\phi_0}{2Ep^{3/2}} + \frac{\phi_0}{2p} \left(R_c - \frac{(mc)_s}{2E^2 S}\right)$$

where:

$$T_s(t) - T_s(0) = \frac{\phi_0}{E\sqrt{\pi}} \sqrt{t} + \frac{\phi_0}{2} \left(R_c - \frac{(mc)_s}{2E^2 S}\right) \quad [4.3]$$

The plot of $T_s(t) - T_s(0)$ as a function of $\sqrt{t}$ is therefore a straight line of slope $\alpha = \frac{\phi_0}{E\sqrt{\pi}}$ whose determination makes it possible to calculate the thermal effusivity E by:

$$E = \frac{\phi_0}{\alpha\sqrt{\pi}} \quad [4.4]$$

The probe's inertia and contact resistance do not influence the temperature at long times. To apply this estimation method, it is necessary to make sure that the semi-infinite medium hypothesis remains valid over the chosen estimation interval. It should be noted that this method of estimating the thermal effusivity E does not require knowing the heating element's thermal capacity $(mc)_s$.

4.2.2.3. *Validity limits of models*

The method's limits are mainly related to the semi-infinite medium and 1D transfer hypothesis. It has been shown [BAL 11] that in the most favorable case where the probe and the sample have the same surface, the upper bound of the estimation interval must be less than the following two values:

Limit of the semi-infinite medium

$$t_1 = \left(\frac{1+0.04 E}{-1.5+0.2 E} \right) \frac{e^2}{a} \quad [4.5]$$

Limit of the 1D transfer

$$t_2 = (2 \times 10^{-5} E + 0.0669) \frac{b^2}{a} \quad [4.6]$$

where: a thermal diffusivity

b Probe's width

e sample's thickness

E thermal effusivity

The hot plate method will therefore be difficult to implement for very diffusive materials for which the times t_1 and t_2 are going to be very small. Moreover, it is also not very suitable for very light materials for which the temperature remains sensitive to the probe's thermal inertia for several tens of seconds. The method based on measuring the slope of the curve $T_s(t) = f(\sqrt{t})$ can therefore only be applied after a long time for which it is then possible that the semi-infinite medium hypothesis can no longer be verified. The complete model does not take full account of the transfers in the probe considered as a body at uniform temperature, which remains a convenient approximation. This may result in residues that are slightly off-centered from zero at the very first instants of heating.

4.2.3. Experimental design

This method can be implemented in an extremely simple manner using a flat (rectangular or circular) heating resistor of sufficient surface area (at least 25 cm²). A thermocouple made of fine wires (diameter < 0.1 mm) is fixed at the center of this resistor. Figure 4.2 shows a resistor with a surface area of 10.25 × 10.25 cm². The two wires of the thermocouple can also be separated and placed at the center of the resistor at a distance of about 5 mm from each other and then covered with an aluminum adhesive film that will ensure a good contact between the thermocouple and the resistor, the electrical contact between the two wires of the thermocouple (with separate contacts in this case) and a homogenization of the temperature in the thermocouple's vicinity.

The implementation of the method also requires a stabilized power supply and a device for recording the voltage delivered at the thermocouple's terminals. A recording time scale of 0.1 s is usually sufficient.

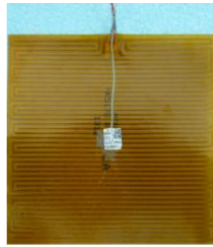


Figure 4.2. View of a hot plate probe

4.2.4. Practice of the measurement

This relatively simple assembly makes it possible to evaluate the value of the thermal effusivity E with good accuracy. The main source of uncertainty is the value of the heat flux ϕ_0 . It is calculated by:

$$\phi_0 = \frac{R_{el}}{S} I^2 \quad [4.7]$$

The measurement of the current I flowing through the heating element is accurate, but the value of the ratio $\frac{R_{el}}{S}$ must be calibrated. For this purpose, an experiment is carried out with two identical samples of known effusivity E_{ref} and

the value of this $\frac{R_{el}}{S}$ is calculated using the simplified model which has the advantage of not depending on the heating element's heat capacity $(mc)_s$:

$$\frac{R_{el}}{S} = \frac{\alpha E_{ref} \sqrt{\pi}}{l^2} \quad [4.8]$$

The heat capacity $(mc)_s$ of the heating element must then be estimated. For this purpose, a measurement is made with two samples of identical thickness of a light insulating material (polystyrene, for example). With this type of material, the sensitivity of the temperature to $(mc)_s$ is maximum and decorrelated from the sensitivity to the other parameters and the contact resistances can be neglected. Equation [4.2] thus becomes:

$$\theta_s(p) = \frac{\phi_0}{2p} \frac{1}{\frac{(mc)_s}{2S}p + E\sqrt{p}} \quad [4.9]$$

The value of $\frac{R_{el}}{S}$ having previously been determined, using equation [4.9], we estimate the following parameters: $(mc)_s$ and E over a time period such that the medium is semi-infinite. The figure shows an example of an experimental result obtained with a heating element with a square cross-section with side of 102.5 mm and a thickness of 0.32 mm inserted between two samples of extruded polystyrene with a thickness of 30 mm. The estimation of the parameters has led to the value of $(mc)_s = 3.7 \text{ J K}^{-1}$ leading to a value of its heat capacity of: $\rho c = \frac{(mc)_s}{es} = 1.10 \times 10^6 \text{ J K}^{-1} \text{ m}^{-3}$, which corresponds well to the order of magnitude characteristic for plastic materials. The reduced sensitivity curves shown in Figure 4.3 show that this experiment makes it possible to estimate the heat capacity of the heating element because the two sensitivities are not proportional and therefore decorrelated.

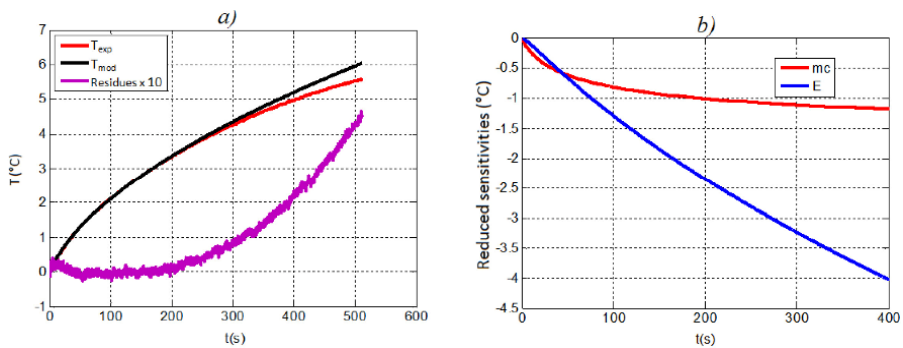


Figure 4.3. a) Experimental thermograms and models obtained in a symmetrical plate experiment with polystyrene of 30 mm thickness; b) reduced sensitivity of the temperature to the parameters. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Then, the calibrated heating element can be used to measure the effusivity of materials with unknown properties. An experiment is carried out in which the temperature rise in the center of the heating element is recorded as soon as the heating starts.

The model temperature $T_s(t)$ can be calculated by applying the Stehfest method (or any other inverse Laplace transform algorithm) to equation [4.2]. A minimization algorithm can then be used to estimate the values of parameters E and R_c minimizing the sum of the quadratic deviations between the theoretical curve and the experimental points. This estimation method makes it possible to take into account all the points of the recording realized.

An example of an experimental thermogram obtained with two samples of PVC foam with a thickness of 6 mm is shown in Figure 4.4. It can be seen that between 30 and 80 s, the curve representing $T_s(t) - T_s(0)$ as a function of $\sqrt{t}$ can be assimilated to a line whose slope is obtained by linear regression: $\alpha = 1.0638 \text{ K s}^{-1/2}$. With $\frac{R_{el}}{s} = 4000 \text{ } \Omega \text{ m}^{-2}$ and $I = 0.280 \text{ A}$, by applying formula [4.4], a first value of the thermal effusivity is deduced therefrom: $E = 166.5 \text{ W m}^2 \text{ K}^{-1} \text{ s}^{1/2}$.

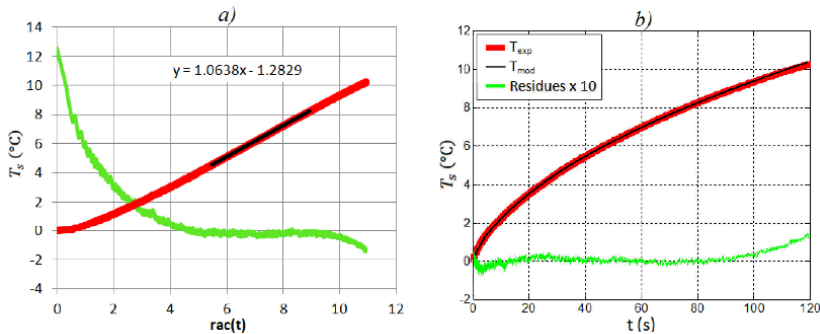


Figure 4.4. a) Experimental curve $T_s(t)$ and linear regression of the curve $T_s = f[\sqrt{t}]$ between 30 and 80 s; b) experimental curve $T_s(t)$ and complete model (estimation between 0 and 80 s). For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

A more precise value can then be estimated between 0 and 60 s as follows:

- initial values of the parameters E (estimated using equation [4.4]) and R_c are chosen;
- a numerical method (Stehfest for example) of inversion of the Laplace transform is used to calculate the difference $T_s(t) - T_s(0)$ from formula [4.2].

A minimization algorithm is used to determine the value of the parameters E and R_c minimizing the difference between the experimental points and the inversion curve of [4.2].

In the case of the example studied, we find: $E = 162.5 \text{ W m}^2 \text{ K}^{-1} \text{ s}^{1/2}$. This value is very similar to that found using the simplified model (difference of 2.4%).

4.2.5. Asymmetric setup

The assembly studied up to now and shown in Figure 4.1 is a symmetrical assembly that leads to a simplification of the modeling and the calculations. However, it has two disadvantages for certain materials:

- If the materials are undeformable, the presence of the thermocouple wires between the heating probe and the material's surface will impose a minimum distance (equal to the diameter of the wires) between the two and cause a fairly large and asymmetrical contact resistance.
- In certain particular cases, it may be difficult to produce two samples of identical properties and surface conditions, in particular in the case of certain building materials whose properties are to be measured for a given water content.

A solution for these two disadvantages is to use an asymmetrical assembly in which one of the samples is replaced by a deformable insulator of known thermal effusivity (see Figure 4.5). For example, polyurethane foam is used by placing the thermocouple on the face of the probe in contact with the foam. The deformable character of the foam will enable the thermocouple to be inserted therein and not to create any substantial contact resistance. Moreover, this contact resistance has a much lower importance if it is on the side of an insulating material.

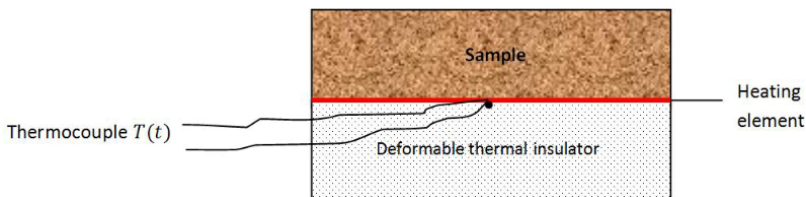


Figure 4.5. Schematic diagram of the asymmetric hot plate method with a semi-infinite sample

All the analysis developed on the symmetrical hot plate remains applicable by means of these modifications:

– Equation [4.2] should be replaced by the following equation:

$$\theta_s(p) = \frac{\phi_0}{p} \frac{1}{\frac{\frac{(mc)_s p + \left[1 + R_c \frac{(mc)_s}{S}\right] E \sqrt{p}}{1 + R_c E \sqrt{p}} + \frac{E_i \sqrt{p}}{1 + R_{ci} E_i \sqrt{p}}} \quad [4.10]$$

with: θ_s Laplace transform of the temperature rise of the probe

R_c contact resistance at the interface heat resistance/material to be characterized

R_{ci} contact resistance at interface heat resistance/known insulating material

m mass of thermocouple + heat resistance

c specific heat of thermocouple + heat resistance

E thermal effusivity of the material to be characterized

E_i thermal effusivity of the known insulating material

p Laplace variable

S surface of the heating element

ϕ_0 heat flux dissipated in the heat resistance.

– The approximate equation [4.9] valid at long times must be replaced by the following equation:

$$T_s(t) - T_s(0) = \frac{2\phi_0}{(E+E_i)\sqrt{\pi}} \sqrt{t} + \phi_0 \left[\frac{R_c E^2 + R_{ci} E_i^2}{(E+E_i)^2} - \frac{(mc)_s}{(E+E_i)^2 S} \right] \quad [4.11]$$

The plot of $T_s(t) - T_s(0)$ as a function of $\sqrt{t}$ is therefore at long times a straight line of slope $\frac{2\phi_0}{(E+E_i)\sqrt{\pi}}$, whose determination makes it possible to estimate the thermal effusivity of the sample knowing the thermal effusivity E_i of the insulator:

$$E = \frac{2\phi_0}{\alpha\sqrt{\pi}} - E_i \quad [4.12]$$

This method was applied to the measurement of the thermal diffusivity of PVC. Figure 4.6(a) shows the experimental and theoretical curves as well as the estimation residues obtained. Figure 4.6(b) shows the experimental curve as a function of $\sqrt{t}$.

Using the slope of the curve $T = f(\sqrt{t})$ leads to: $E = 495 \text{ W m}^{-2} \text{ K}^{-1} \text{ s}^{1/2}$. The use of the complete model leads to: $= 505 \text{ W m}^{-2} \text{ K}^{-1} \text{ s}^{1/2}$. The difference is in the order of 2%.

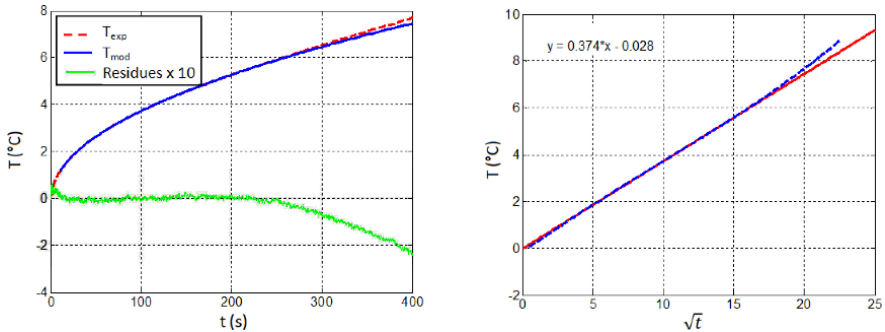


Figure 4.6. Experimental thermograms and models obtained in an asymmetric hot plate experiment with 10 mm-thick PVC and 30 mm-thick polystyrene. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

EXAMPLE.— *Application to the characterization of pasty or pulverulent materials.*

This device can also be used to measure the thermal effusivity of pasty or pulverulent materials by adapting it according to the diagram in Figure 4.7 [DAM 16].

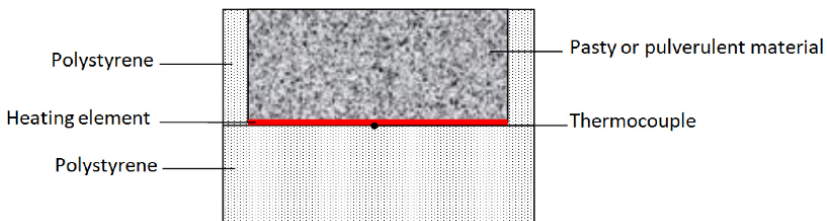


Figure 4.7. Cross-section schematic diagram of an asymmetrical hot plate device suitable for pasty or pulverulent materials

The curve of the temperature rise at the center of the heating resistor is used to estimate the thermal effusivity during the time when the sample can be considered as a semi-infinite medium. This measurement must be coupled to a calorimetric measurement to estimate thermal diffusivity and conductivity.

4.3. Asymmetric hot plate

4.3.1. Measuring temperature

A variant of the conventional hot plate method described above was used to measure the thermal effusivity and conductivity of heavy construction materials [BAL 13]. At the experimental level, this method differs from the classical method by the following three points (see Figure 4.8):

- The assembly is asymmetric: the heating element is placed between the sample to be characterized and a sample (1) of an insulator of known thermal properties.
- The unheated face of the sample is in contact with a sample (2) identical to sample (1).
- The “rear” faces of the insulating samples are maintained at a constant temperature by contact with aluminum blocks (possibly thermostated)

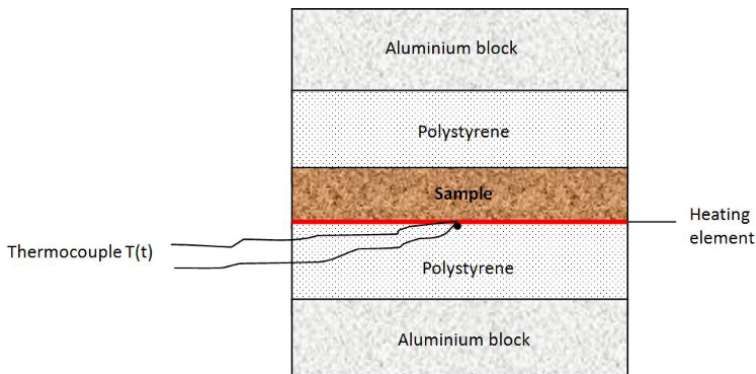


Figure 4.8. Schematic diagram of the finite asymmetric hot plate setup

The setup is asymmetrical in order to reduce the contact resistance as explained in section 3.3.4.

At the modeling level, we no longer assume the hypothesis of a semi-infinite medium for the samples but we always assume a 1D heat transfer at the center.

$$\begin{bmatrix} \theta_0 \\ \Phi_{01} \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ mcp & 1 \end{bmatrix} \begin{bmatrix} 1 & R_c \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A_e & B_e \\ C_e & D_e \end{bmatrix} \begin{bmatrix} A_i & B_i \\ C_i & D_i \end{bmatrix} \begin{bmatrix} 0 \\ \Phi_3 \end{bmatrix},$$

$$\begin{bmatrix} \theta_0 \\ \Phi_{02} \end{bmatrix} = \begin{bmatrix} A_i & B_i \\ C_i & D_i \end{bmatrix} \begin{bmatrix} 0 \\ \Phi_2 \end{bmatrix},$$

assuming:
$$\begin{bmatrix} A_0 & B_0 \\ C_0 & D_0 \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ mcp & 1 \end{bmatrix} \begin{bmatrix} 1 & R_c \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A_e & B_e \\ C_e & D_e \end{bmatrix} \begin{bmatrix} A_i & B_i \\ C_i & D_i \end{bmatrix}$$

with: $A_e = D_e = ch(q e), B_e = \frac{1}{\lambda S q} sh(q e), C_e = \lambda S q sh(q e), q = \sqrt{\frac{p}{a}},$

$$A_i = D_i = ch(q_i e_i), B_i = \frac{1}{\lambda_i S q_i} sh(q_i e_i), C_i = \lambda_i S q_i sh(q_i e_i), q_i = \sqrt{\frac{p}{a_i}},$$

where:

θ_0 Laplace transform of the temperature at the center of the heating element

θ_1 Laplace transform of the temperature at the center of the unheated face

Φ_{01} Laplace transform of the heat flow rate towards the sample

Φ_{02} Laplace transform of the heat flow rate towards the insulator

Φ_1 Laplace transform of the heat flow rate on the unheated face of the sample

Φ_3 Laplace transform of the heat flow rate on the unheated face of the insulator

e thickness of the sample (m)

e_i thickness of the insulator (m)

S surface area of the heating element and sample (m²)

R_c thermal contact resistance at sample / heating element interface (KW⁻¹)

m mass of the heating element (kg)

c heat capacity of the heating element (J kg⁻¹K⁻¹)

λ thermal conductivity of the sample (Wm⁻¹K⁻¹)

λ_i thermal conductivity of the insulator (Wm⁻¹K⁻¹)

a thermal diffusivity of the sample (m²s⁻¹)

a_i thermal diffusivity of insulator (m^2s^{-1})

p Laplace parameter

we can deduce:

$$\Phi_{01} = \theta_0 \frac{D_0}{B_0} \quad \text{and:} \quad \Phi_{02} = \theta_0 \frac{D_i}{B_i},$$

$$\text{with:} \quad \Phi_0 = \frac{\varphi_0}{p} = \Phi_{01} + \Phi_{02},$$

where φ_0 is the heat flow rate produced by Joule's effect in the heating element.

Therefore:

$$\theta_0 = \frac{\varphi_0}{\left(\frac{D_i}{B_i} + \frac{D_0}{B_0}\right)} \quad [4.13]$$

The energy balance between the heating element and the unheated face of the sample can be written as:

$$\begin{bmatrix} \theta_0 \\ \Phi_{01} \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ mcp & 1 \end{bmatrix} \begin{bmatrix} 1 & R_{c1} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A_e & B_e \\ C_e & D_e \end{bmatrix} \begin{bmatrix} \theta_2 \\ \Phi_1 \end{bmatrix},$$

setting:

$$\begin{bmatrix} A_2 & B_2 \\ C_2 & D_2 \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ mcp & 1 \end{bmatrix} \begin{bmatrix} 1 & R_{c1} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A_e & B_e \\ C_e & D_e \end{bmatrix},$$

finally:

$$\theta_1 = \theta_0 \left(D_2 - \frac{D_0}{B_0} B_2 \right) \quad [4.14]$$

Temperatures θ_0 and θ_1 in equations [4.13] and [4.14] are obtained in Laplace space. The inverse Laplace transformation allowing us to deduce the values of T_0 and T_1 is realized numerically using Stehfest or De Hoog algorithms.

The principle of the method is to estimate the values of unknown parameters E , ρc , R_{c1} and mc minimizing the sum of the quadratic deviations $\sum_{i=1}^N [T_{exp}(t_i) - T_{mod}(t_i, E, \rho c, R_{c1}, mc)]^2$ between the experimental and theoretical curves

calculated from equations [4.13] and [4.14]. The number of parameters can be reduced to 3 by carrying out an experiment with a material of known properties to estimate mc , which depends only on the heating element.

This method has been applied to the measurement of the thermal properties of a 10 mm-thick PVC sample. The experimental and theoretical curves of the temperature $T_0(t)$ as well as the estimation residuals and the reduced sensitivity curves obtained are presented in Figure 4.9. The sensitivity curve shows that the hypothesis of the semi-infinite medium is valid up to 200 s for the PVC and that it is therefore necessary to go beyond that to be able to estimate the thermal conductivity λ .

For PVC, we obtained: $\lambda = 0.183 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 1.37 \times 10^6 \text{ J K}^{-1} \text{ m}^{-3}$.

These values differ by less than 4% of the values deduced from a flash diffusivity measurement and a measurement of thermal capacity carried out by differential scanning calorimetry.

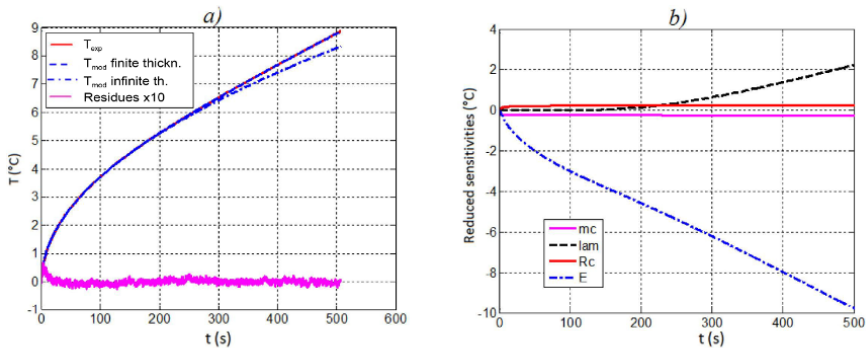


Figure 4.9. a) Experimental thermograms and models obtained in an asymmetric hot plate experiment with 10 mm-thick PVC and b) reduced sensitivities. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

4.3.2. Measurement of two temperatures

The finite asymmetric hot plate method makes it possible to estimate thermal diffusivity and thermal conductivity provided that the hypothesis of the 1D transfer to the center is verified [JAN 04, OSS 17]. Sensitivity to thermal conductivity becomes non-zero only from the time t_1 when the semi-infinite medium hypothesis

is no longer valid. Moreover, convective transfers on lateral surfaces occasionally make the transfer 3D from time t_2 . If time t_1 is greater than time t_2 , then it will be impossible to estimate the sample's thermal conductivity since the heat transfer including at the center will be 3D before the temperature is sensitive to thermal conductivity.

Figure 4.10 shows the temperature T_0 rise at the center of the heated face of a 30 mm-thick earth brick sample during an asymmetric hot plate measurement. Estimation residues diverge from 300 s, indicating that 3D effects appear at this moment. At this time, the reduced sensitivity curves of Figure 4.10(b) indicate that the temperature is still sensitive only to thermal effusivity. It will therefore not be possible to estimate the conductivity λ or heat capacity ρc of this sample using a single-temperature measurement.

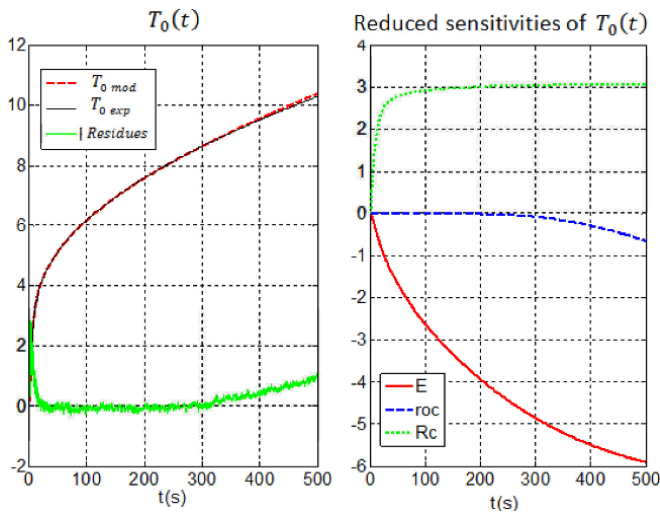


Figure 4.10. a) Temperature T_0 of the heated face of the sample and b) reduced sensitivities during a measurement of the asymmetrical hot plate type. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

The idea on which the two-temperature method is based is that the temperature T_1 of the unheated face of the sample is sensitive to a thermal parameter other than effusivity much earlier than the heated face. It can be seen in Figure 4.11 that the temperature of the unheated face is sensitive to λ and therefore to ρc as soon as $t = 100$ s, long before the hypothesis 1D is no longer valid.

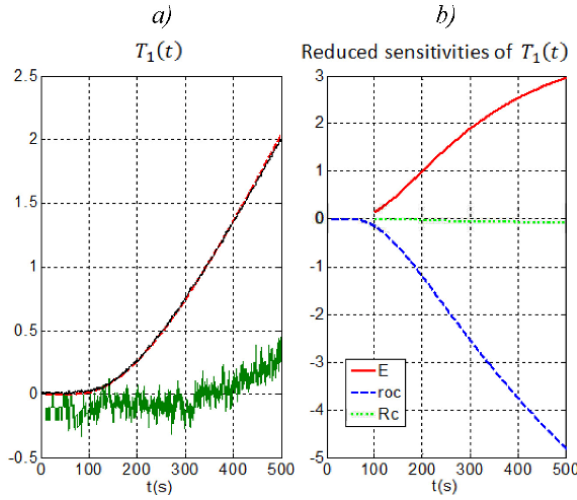


Figure 4.11. Temperature of the unheated face of the sample and reduced sensitivities during a measurement of the finite asymmetrical hot plate type.
For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

We can therefore proceed as follows:

- use the temperature curve of the heated face to estimate E and R_c at “short time intervals”;
- consider the known E and R_c and use the temperature of the unheated face to estimate ρc and therefore λ for the interval $[0, t_2]$. The time t_2 is estimated from the residual analysis: it is the maximum value of the upper limit of the estimation interval making it possible to obtain “flat” residuals.

In the example considered, we obtained the following values by applying this method:

On $[0.300\text{s}]$: $\lambda = 0.856 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 1.276 \times 10^6 \text{ J K}^{-1} \text{ m}^{-3}$.

On $[0.400\text{s}]$: $\lambda = 0.857 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 1.289 \times 10^6 \text{ J K}^{-1} \text{ m}^{-3}$.

Values weakly change when you change the estimation interval, which is a good indicator of the estimation's accuracy. Figure 4.12 shows the two experimental and simulated temperatures as well as the estimation residues ($\times 10$ and offset by 0.1°C).

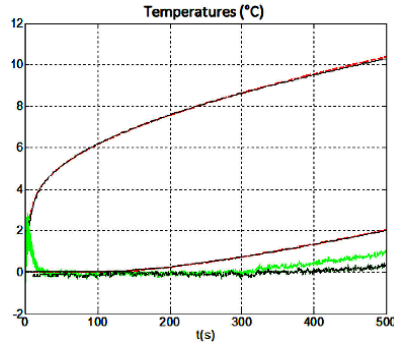


Figure 4.12. *Temperatures of both sides of sample and estimation residues ($\times 10$ and offset by 0.1°C) during a finite asymmetric hot plate measurement*

4.4. Hot wire

4.4.1. Principle

It is a transient measurement method used to measure the thermal conductivity of a fairly wide range of materials (solid, liquid or gaseous). It should be noted, however, that it is not suitable for anisotropic solids.

4.4.1.1. One-wire method

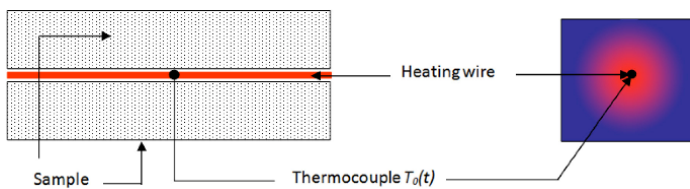


Figure 4.13. *Schematic diagram of the device of the hot wire method.*

A heating wire is placed between the surfaces of two samples of the material to be characterized (see Figure 4.13). A constant heat flow rate step ($\dot{\varphi} = 0$ if $t < t_0$ and $\dot{\varphi} = \dot{\varphi}_0$ if $t \geq t_0$) is applied to the heating wire and the temperature change of this wire is recorded. During the time when the perturbation does not reach the samples' other faces, that is to say where the semi-infinite medium hypothesis is

valid, it can be considered that the transfer to the center of the sample around the wire is radial. The 2D modeling of this heat transfer makes it possible to calculate the evolution of the temperature at the sample's center. A method of estimating parameters is used to calculate the values of:

- the thermal conductivity λ of the sample;
- the thermal capacitance mc of the probe + heat resistance;
- the contact resistance at the probe/sample interface,

which minimize the gap between theoretical and experimental curves.

The hot wire can also be used to measure the thermal conductivity of fluids (liquids or gases), in which case it is simply immersed in the fluid away from the walls.

4.4.1.2. Two-wire method

A variant involves placing a second parallel wire in the sample and in recording its temperature. The measurement of this second temperature makes it possible to estimate the thermal diffusivity of the sample.

4.4.2. Hypotheses and model

4.4.2.1. One-wire method

The vicinity of the heating wire is shown in Figure 4.14.

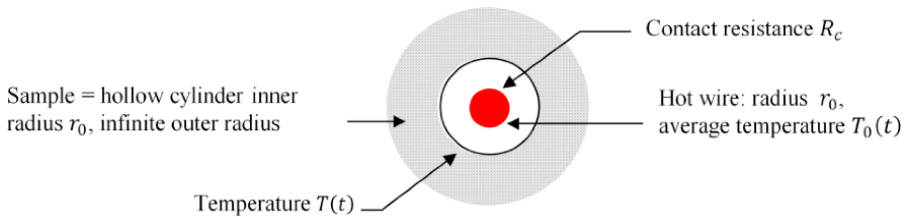


Figure 4.14. Schematic diagram of heat transfer in the vicinity of the hot wire

The heat equation is written for the sample as:

$$\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} = \frac{1}{a} \frac{\partial T}{\partial t}$$

The equation of heat is written for the wire as:

$$\frac{\partial^2 T_s}{\partial r^2} + \frac{1}{r} \frac{\partial T_s}{\partial r} + \frac{p_0}{\lambda_s} = \frac{1}{a_s} \frac{\partial T_s}{\partial t}$$

where p_0 is the power density generated in the wire by Joule's effect with the following initial and boundary conditions:

$$\text{at: } t = 0 : T_s(r, 0) = T(r, 0) = T_i$$

$$\text{at: } r = 0 : \frac{\partial T_s(0, t)}{\partial r} = 0$$

$$\text{at: } r = r_0 : \lambda_s \frac{\partial T_s(r_0, t)}{\partial r} = \lambda \frac{\partial T(r_0, t)}{\partial r}$$

$$\frac{T_s(r_0, t) - T(r_0, t)}{R_c} = \lambda \frac{\partial T(r_0, t)}{\partial r}$$

$$\text{at: } r \rightarrow \infty : T(\infty, t) = T_i$$

The measured temperature is the average temperature of the wire:

$$T_0(t) = \frac{1}{\pi r_0^2} \int_0^{r_0} 2\pi r T_s(r, t) dr$$

This problem can be solved using the Laplace transform. The quadrupole formalism allows writing an equation between the transform θ_0 of the average temperature T_0 of the wire, the transform of the total power released in the wire $\frac{\varphi_0}{p}$ (with $\varphi_0 = p_0 \pi r_0^2 L$) and the transform of the surface temperature of the wire (here in $r = r_0$). The associated quadrupole is given in Maillet *et al.* [MAI 00]. This quadrupole is written as:

$$\begin{bmatrix} A_0 & B_0 \\ C_0 & D_0 \end{bmatrix} = \begin{bmatrix} 1 & \frac{1}{2\pi\lambda_s L q_s r_0} \frac{I_0(q_s r_0)}{I_1(q_s r_0)} - \frac{1}{\rho_s c_s \pi r_0^2 L p} \\ \rho_s c_s \pi r_0^2 L p & D_0 = \frac{q_s r_0}{2} \frac{I_0(q_s r_0)}{I_1(q_s r_0)} \end{bmatrix}$$

$$\text{with: } q_s = \sqrt{\frac{p}{a_s}}.$$

In the case of the small body hypothesis for the wire (r_0 small and λ_s large), the quadrupole becomes: $A_0 = 1$; $B_0 = 0$; $C_0 = \rho_s c_s \pi r_0^2 L p$; $D_0 = 1$

Hence, the total system:

$$\begin{bmatrix} \theta_0 \\ \frac{\varphi_0}{p} \end{bmatrix} = \begin{bmatrix} A_0 & B_0 \\ C_0 & D_0 \end{bmatrix} \begin{bmatrix} 1 & R_c \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \theta \\ \frac{\theta}{z} \end{bmatrix}$$

with: $\frac{1}{z} = 2\pi\lambda L q r_0 \frac{K_1(qr_0)}{K_0(qr_0)}$ or: $q = \sqrt{\frac{p}{a}}$

where:

$T_0(t)$ average temperature of the hot wire

$T(t)$ temperature of the sample at $r = r_0$

θ_0 Laplace Transform of the difference $T_0(t) - T_i$

θ Laplace Transform of the difference $T(t) - T_i$

R_c contact resistance at heating resistance / sample interface

a_s wire's thermal diffusivity

c_s wire's specific heat

ρ_s wire's density

λ_s wire's thermal conductivity

λ sample's thermal conductivity

a sample's thermal diffusivity

p Laplace variable

r_0 heating wire's radius

L heating wire's length

φ_0 power dissipated in the heating resistor

I_0, I_1, K_0, K_1 Bessel's functions

Hence,

$$\theta_0 = \frac{\varphi_0}{p} \frac{A_0 + (A_0 R_c + B_0)/Z}{C_0 + (C_0 R_c + D_0)/Z} \quad [4.15]$$

4.4.2.1.1. Simplified model

If we consider a fine (r_0 small) wire and if we take a long time ($p \rightarrow 0$), we can use the limited developments of the Bessel functions in the neighborhood of 0:

$$K_0(x) \approx -\ln(x); K_1(x) \approx \frac{1}{x}; I_0(x) \approx 1; I_1(x) \approx \frac{x}{2}$$

which lead to (see section 2.2.2):

$$\bar{T}_0(t) = T_0(t) - T_i = \frac{\varphi_0}{4\pi\lambda L} \ln(t) + \varphi_0 \left[R_c - \frac{\gamma}{4\pi\lambda L} - \frac{\ln\left(\frac{r_0}{\sqrt{a}}\right)}{2\pi\lambda L} \right] \quad [4.16]$$

where $\gamma = 0.57721$ is Euler's constant.

The plot of $\bar{T}_0(t)$ as a function of $\ln(t)$ is therefore a straight line of slope $\frac{\varphi_0}{4\pi\lambda L}$, whose determination makes it possible to estimate the thermal conductivity λ . The inertia of the probe and the contact resistance do not influence the temperature at long time intervals. To apply this estimation method, it is necessary to make sure that the semi-infinite medium hypothesis remains valid over the chosen estimation interval. As the measurement noises on the temperature values at the different measurement times are constant and uncorrelated, the linear least squares method is used to estimate the slope.

NOTE.—

The heat produced in the wire propagates in two directions Ox and Oy . It is shown that if the material is anisotropic, the slope of the line $\bar{T}_s(t) = f[\ln(t)]$ is equal to $\frac{\varphi_0}{4\pi\sqrt{\lambda_x \lambda_y} L}$. The hot wire method does not therefore make it possible to measure the conductivity in only one direction in this case.

4.4.2.2. Two-wire method

It is possible to write for two wires situated at distances r_1 and r_2 from the center of the heating wire:

$$\begin{bmatrix} \theta(r_1, p) \\ \Phi(r_1, p) \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta(r_2, p) \\ \Phi(r_2, p) \end{bmatrix}$$

$$A = qr_2[K_1(qr_2)I_0(qr_1) + K_0(qr_1)I_1(qr_2)]$$

$$B = \frac{1}{2\pi\lambda L} [K_0(qr_1)I_0(qr_2) - K_0(qr_2)I_0(qr_1)]$$

$$C = 2\pi L\rho cr_1r_2p[K_1(qr_1)I_1(qr_2) - K_1(qr_2)I_1(qr_1)]$$

$$D = qr_1[K_1(qr_1)I_0(qr_2) + K_0(qr_2)I_1(qr_1)]$$

The semi-infinite medium hypothesis makes it possible to write:

$$\begin{bmatrix} \theta(r_2, p) \\ \Phi(r_2, p) \end{bmatrix} = \begin{bmatrix} \theta(r_2, p) \\ 2\pi\lambda L \frac{qr_1K_1(qr_1)}{K_0(qr_1)}\theta(r_2, p) \end{bmatrix}$$

We can deduce:

$$\theta(r_1, p) = H(p) \times \theta(r_2, p)$$

Hence:

$$H(p) = qr_2[K_1(qr_2)I_0(qr_1) + K_0(qr_1)I_1(qr_2)] + [K_0(qr_1)I_0(qr_2) - K_0(qr_2)I_0(qr_1)] \frac{qr_1K_1(qr_1)}{K_0(qr_1)} \quad [4.17]$$

It is found that the function $H(p)$ only depends on the thermal diffusivity a via $q = \sqrt{\frac{p}{a}}$. By recording the two temperatures $T(r_1, t)$ and $T(r_2, t)$, it is possible to deduce therefrom the transfer function $F(t, a) = L^{-1}[H(p, a)]$, and therefore, the value of the thermal diffusivity a .

This method falls within the category of transient “temperature/temperature” methods, which will be studied in detail in Chapter 5.

4.4.3. Experimental setup

The hot wire may be constituted of:

- a resistive strip of very small width and thickness inserted between two Kapton sheets and on which a thermocouple consisting of two thin tapes (copper and constantan, for example, for a thermocouple T) is placed;

- a wire or a resistive strip whose known temperature coefficient makes it possible to deduce the mean temperature knowing the value of its resistance measured by a 4-wire method.

In the first type of strip, thermal transfer in Kapton sheets is not taken into account in the model, which can lead to a source of error for insulating materials whose thermal conductivity may be 10 times lower than that of the Kapton.

In the second type of strip, edge effects and heat losses through the electrical wires are not taken into account.

4.4.4. Practice of the measurement

4.4.4.1. Choice of time interval for estimation

The first problem is to know the heating time during which the semi-infinite medium hypothesis is valid. For this purpose, the temperature change $\bar{T}(e, t)$ over a radius equal to the thickness of the sample ($r = e$) is calculated using the parameters estimated over an arbitrary time. If the calculated temperature $\bar{T}(e, t)$ differs from the initial temperature $\bar{T}(e, 0)$ by more than 0.1°C , the estimation calculation of the parameters is resumed over a shorter time.

Given that we simply seek to estimate the time during which the medium remains semi-infinite, we can only do so in the simple (and most unfavorable) case where the contact resistance and the thermal capacitance of the probe assembly and heating resistor are zero and the probe's radius r_0 is very low. It is shown that, during the time when the medium is semi-infinite, we can calculate $\theta(e, p)$ by:

$$\theta(e, p) = \frac{\varphi_0}{\left(c + \frac{D}{Z}\right)p} \quad [4.18]$$

with:

$$C = \rho_s c_s 2\pi r_0 L e p [K_1(qr_0)I_1(qe) - K_1(qe)I_1(qr_0)]$$

$$D = qr_0 [K_0(qe)I_1(qr_0) - K_1(qr_0)I_0(qe)]$$

$$\frac{1}{Z} = 2\pi\lambda L q r_0 \frac{K_1(qr_0)}{K_0(qr_0)}$$

$\bar{T}(e, 0)$ is computable by applying the Stehfest or De Hoog method to formula [4.18].

This formula is valid all the time that temperature $\bar{T}(e, 0)$ has not varied. It can therefore be used to determine the instant from which $\bar{T}(e, 0)$ will start to vary but will not be able to determine the evolution $\bar{T}(e, 0)$ after this moment.

EXAMPLE.—

Determination of the thermal conductivity of carbide with a hot wire of length 5 cm delivering a heat flow rate equal to 3.03 W. Figures 4.15 and 4.16 represent the graph of the experimental temperature $\bar{T}_0(t)$ obtained with a sample of thickness 2.5 cm, this being recorded every 0.05 s. We have included on this same graph:

– the temperature $\bar{T}(e, t)$ of the unheated face calculated with formula [4.18] taking as the value of the thermal effusivity $E = 5000 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$;

– the temperature $\bar{T}_0(t)$ calculated with a simplified model at long times after estimation of the thermal conductivity by linear regression of the curve $\bar{T}_0(t) = f[\ln(t)]$ between the times t_1 and t_2 (detail below).

Figure 4.15 corresponds to an estimate between 20 and 80 s leading to a value $\lambda = 10.1 \text{ W m}^{-1} \text{ K}^{-1}$. Figure 4.16 corresponds to an estimate between 2 and 20 s leading to a value $\lambda = 19.7 \text{ W m}^{-1} \text{ K}^{-1}$. The graph of the calculated temperature $\bar{T}(e, t)$ as well as the shape of the experimental curve $\bar{T}_0(t)$, which undergoes a slope modification at $t = 20 \text{ s}$ shows us that the first estimate of λ (between 20 and 80 s) is not valid because it is performed on times when the semi-infinite medium hypothesis is no longer valid (the perturbation has reached the other side). The value of λ obtained by linear regression between 2 and 20 s is obtained under satisfactory conditions: linearity zone of the curve $\bar{T}_0 = f[\ln(t)]$ and constant temperature of the non-heated face. The uncertainty on this estimate comes mainly from the uncertainty on the temperature: the estimation is carried out on a temperature rise of the order of 1°C , while the uncertainty on the temperature is of the order of 0.05°C . This shows the limitations of the hot wire method for estimating high thermal conductivities.

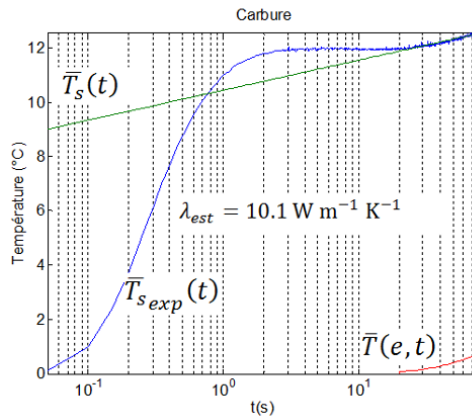


Figure 4.15. Hot wire thermogram for carbide estimation between 20 and 80 s

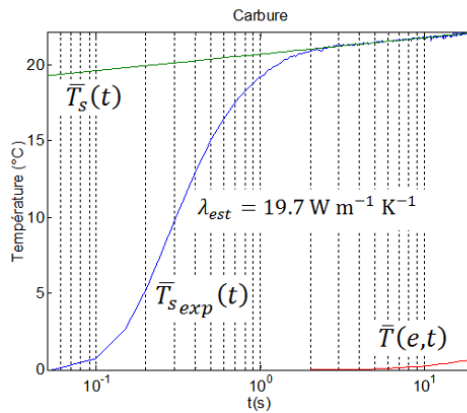


Figure 4.16. Hot wire thermogram for carbide estimation between 2 and 20 s

In the case of liquids or gases, measurement by the hot wire method is valid only in the absence of convection. This is in general true at the very first instants of heating, the temporal limit of the validity of this hypothesis being able to be determined by analysis of the estimation residues: the time interval over which the residuals are flat and centered on the null value.

This method can be used at high temperatures of up to more than 1500°C. [ZHA 93].

4.5. Flash 1D

4.5.1. Principle

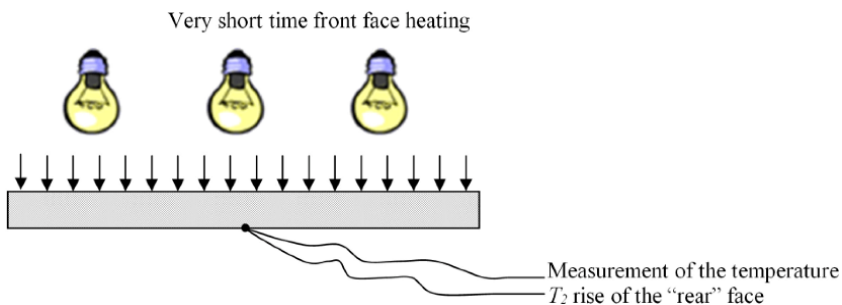


Figure 4.17. Schematic diagram of the flash method

This method makes it possible to estimate the thermal diffusivity of solids. Its block diagram is described in Figure 4.17. On one of the faces of a sample with parallel faces, a heat flux of high power is sent for a very short time, and the temperature rise of the opposite face is recorded. A model of the heat transfer in the sample allows us to propose several methods of estimating the thermal diffusivity from the experimental thermogram. The simplicity of some of these methods has made the flash method very popular, but a number of experimental precautions must be followed to achieve good accuracy.

A measurement setup requires the definition of:

- a sample holder adapted to the desired temperature and pressure range;
- a source of surface heating;
- a system for measuring and recording the surface temperature;
- a thermal model associated with heat transfer;
- a method of identification.

For a detailed presentation of the method and its applications, as well as the associated bibliography, the reader can refer to Hay *et al.* [HAY 02].

4.5.1.1. A brief history

The “rear face flash” method is the oldest method in transient mode and mostly used for the measurement of the thermal diffusivity of solids.

– The story started in 1961 with a publication by Parker [PAR 61]. As shown in Figure 4.18, the heat source is a flash tube of 400 J (pulse time of a few milliseconds), and the measurement system consists of a Chromel–Alumel thermocouple with separate contacts, an amplifier, an oscilloscope and a Polaroid device. The model used is 1D with a Dirac source and without loss, the method of identification uses the half-rise time measured on the photograph with the aid of a pantograph. The diffusivity is given by:

$$a = \frac{t_{1/2}^* e^2}{t_{1/2}} \quad [4.19]$$

where $t_{1/2}^* = 0.139$.

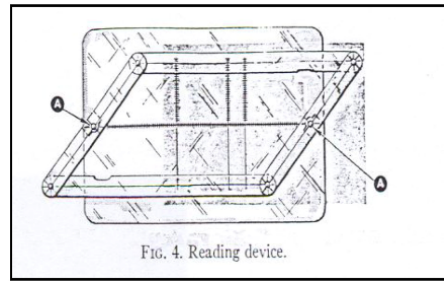
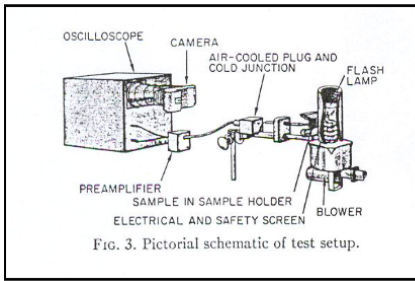


Figure 4.18. Experimental device used by Parker

– The 1960s saw the development of technology in particular in the United States. The sources of heat have diversified with the use of lasers and electron guns. The models have evolved taking into account the thermal losses and the pulse length, but the methods of identification have changed little, the corrected half-rise time $t_{1/2}^*$ is used to take into account the length of the pulse, the losses and the non-homogeneity of the impulse. For example, concerning the length of the pulse, $t_{1/2}^*$ is calculated as a function of the length of the adimensional flash $t_f^* = \frac{a t_f}{e^2}$ for two pulse shapes: slot and triangle (Figure 4.19). Regarding losses, $t_{1/2}^*$ is calculated as a function of the Biot number (Figure 4.20).

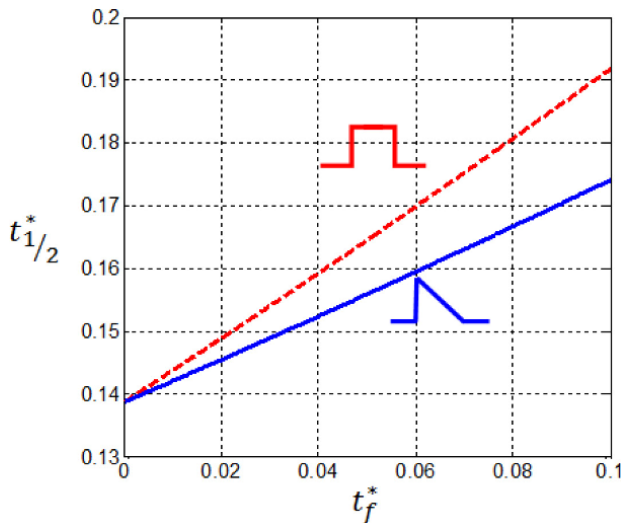


Figure 4.19. Correction of flash length

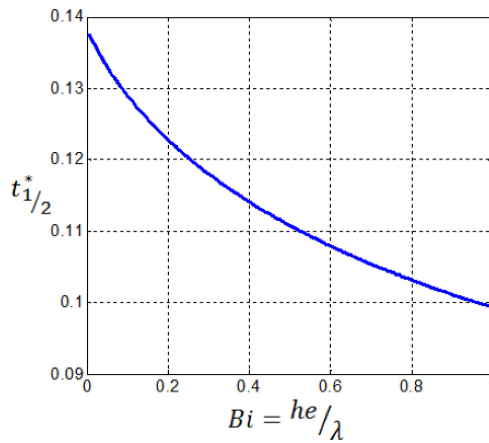


Figure 4.20. *Correction of heat losses*

– France and Great Britain took over during the 1970s with notable developments at all levels:

- 3D analytical modeling with losses;
- new identification methods identifying losses on the thermogram;
- new measurement and acquisition techniques, semiconductor thermocouples, galvanometric and then potentiometric recorders;
- extensions at high and low temperatures (from liquid nitrogen to more than 1000°C).

Other applications of the method are also apparent, such as the measurement of thermal contact resistances or the characterization of surface layers.

– The 1980s saw the appearance of temperature measurements without contact and the beginning of the use of minimization methods. It was not until the 1990s that progress was made that was still valid today:

- mastered use of inverse methods for identification;
- digital acquisition systems;
- the generalization of non-contact measurements (infrared camera, for example).

Numerous extensions have emerged, such as applications to semi-transparent media, anisotropic materials, liquids, as well as evolutions towards ultra-fast thermal or non-destructive testing.

4.5.2. Hypotheses and models

Whoever the authors are, the following hypotheses are used:

- uniformity of the absorbed radiative flux $\phi_0(t)$ over the entire surface of the sample;
- temperature is uniform and equal to ambient temperature at $t = 0$;
- taking into account the thermal losses around the measurement sample by exchange coefficients that are constant on each face.

The first two hypotheses must be verified in the experimental set-up because it is extremely difficult to know the spatial shape of the flux in the case of a non-uniform flux as well as the temperature distribution in the sample if the latter is not at thermal equilibrium with the environment. For the latter hypothesis, the use of constant exchange coefficients in unsteady state is an approximation, but there is currently no alternative solution; knowing moreover, that, in this problem, the taking into account of the losses is of the second order, experiments show that the approximation is justified.

4.5.2.1. The 3D model

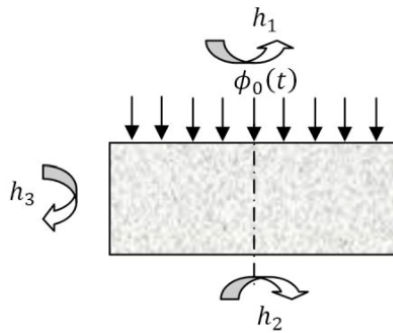


Figure 4.21. Schematic diagram of fluxes

In this model, the losses are characterized by three exchange coefficients, h_1 , h_2 and h_3 , the flux absorbed by a variable flux density $\phi_0(t)$ and the temperature detection is realized at a distance R_m from the center. The problem is in the form of cylindrical symmetry, so we use the equation of heat in cylindrical coordinates:

$$\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2} = \frac{1}{a} \frac{\partial T}{\partial t}$$

Boundary and initial conditions: $\lambda \frac{\partial T(r, 0, t)}{\partial z} = h_1 [T(r, 0, t) - T_i] - \phi_0(t)$

$$-\lambda \frac{\partial T(r, e, t)}{\partial z} = h_2 [T(r, e, t) - T_i]$$

$$\frac{\partial T(0, z, t)}{\partial r} = 0$$

$$-\lambda \frac{\partial T(R, z, t)}{\partial r} = h_3 [T(R, z, t) - T_i]$$

$$T(r, z, 0) = T_i$$

The problem, being linear, has different methods of resolution:

- separation of the space and time variables, then separation of space variables;
- Laplace transform on time, then separation of space variables;
- Laplace transform on time, then transformed integral over space.

By way of example, the solution obtained by the second method is written as [JAN 16]:

$$\theta(r, z, p) = \sum_{n=1}^{\infty} F_n J_0(\alpha_n r) \{ \beta_n \cosh[\gamma_n(e - z)] + H_2 \sinh[\gamma_n(e - z)] \} \quad [4.20]$$

where:
$$F_n = \frac{2\Phi_0(p) \frac{e}{\lambda}}{\omega_n \left(1 + \frac{\omega_n^2}{H_3^2} \right) J_1(\omega_n) [(\beta_n^2 + H_1 H_2) \sinh(\beta_n) + \beta_n (H_1 + H_2) \cosh(\beta_n)]}$$

The ω_n being the solutions of the transcendent equation

$$J_0(\omega_n) = \frac{\omega_n J_1(\omega_n)}{H_3} \quad [4.21]$$

solved numerically.

In addition, with:

$$\Phi_0(p) = L[\phi_0(t)],$$

$$H_1 = \frac{h_1 e}{\lambda}, H_2 = \frac{h_2 e}{\lambda}, H_3 = \frac{h_3 R}{\lambda}$$

$$\gamma^2 = \frac{p}{a} + \alpha^2, \quad \omega = \alpha R, \quad \beta = \gamma e.$$

A hundred terms is sufficient to calculate $\theta(r, z, p)$. Then, we compute $T(r, z, t)$ by numerically transforming the inverse Laplace (see Appendix 5).

The temporal form of the flux is taken into account by a simple product in the Laplace space:

$$\theta(r, z, p) = \Phi_0(p) \times \theta_D(r, z, p, h_1, h_2, h_3, e, R, R_m, \lambda, a)$$

where θ_D is the solution in the Laplace space obtained for a Dirac pulse for which $\Phi_0(p) = 1 \text{ J m}^{-2}$.

Therefore, the solution is given by a product of convolution in real space:

$$T(r, z, t) = \phi_0(t) \otimes T_D(r, z, t, h_1, h_2, h_3, e, R, R_m, \lambda, a)$$

where: $T_D(r, z, t) = L^{-1}[\theta_D(r, z, p)]$.

We can therefore reduce the problem to its solution T_D to the Dirac obtained by positing $\Phi_0(p) = 1$, it will suffice subsequently to convolute the solution by the exact form of the impulse (which will have to be acquired).

4.5.2.2. Reduction of the model

Here, we will study the correlations that exist between certain parameters in order to reduce the number of parameters to be estimated. In this case, the impulse is of very short duration, so that $\Phi_0(p) = q_{01}$:

The measured temperature is of the form:

$$T_2(t) = T(t, z = e, r = R_m) = q_{01} f(h_1, h_2, h_3, e, R, R_m, \lambda, a, t)$$

The 9 parameters ($q_{01}, h_1, h_2, h_3, e, R, R_m, \lambda, a$), where R_m is the measurement radius, that appear in the solution are not independent, thus there are no 9 degrees of freedom. As we have seen previously, the problem must be adimensioned by making a dimension parameter of temperature appear, one of time dimension and parameters without dimension, for example:

$$T_2(t) = \beta f\left(H_1, H_2, H_3, \frac{t}{\alpha}, \gamma, \delta\right)$$

$$\text{with: } H_1 = \frac{h_1 e}{\lambda}, H_2 = \frac{h_2 e}{\lambda}, H_3 = \frac{h_3 R}{\lambda}, \alpha = \frac{e^2}{a}, \beta = \frac{q_{01}}{\rho c e}, \gamma = \frac{R}{e}, \delta = \frac{R_m}{e}$$

In the general case, the problem contains 7 parameters.

As the identification problem is nonlinear, it is possible that, for some of the parameter values, some are almost correlated. It is the sensitivity study that can

demonstrate it. This is the case here where most often the 3 Biot numbers H_i are strongly correlated, so we must fix a Biot number or a relation between them. The right solution is to take $h_3 = 0$, which makes the problem 1D and thus removes 3 Parameters ($\frac{h_3 R}{\lambda}$, $\frac{R}{e}$, $\frac{R_m}{e}$) and, in particular R_m , the radial position of the thermometer which is a very difficult parameter to know.

In Figure 4.22, the case of an insulating material at ambient temperature was treated with $\gamma = \frac{R}{e} = 2$. We give the 3D simulation, the sensitivities to the 3 Biot numbers H_i , to $\alpha = \frac{a}{e^2}$ and $\beta = \frac{e q_{01}}{\lambda}$. It is found that the 3 Biot numbers H_i are almost correlated. Figure 4.23 shows the identification of the previous thermogram (established by a 3D model and considered a “numerical” experiment) by a 1D model with $h_1 = h_2 = h$ and $h_3 = 0$. It is found that the residuals are good, and the error on the value of the diffusivity obtained is of the order of 1% (see Table 4.1). Figures 4.24 and 4.25 are the results for the same sample with $\gamma = \frac{R}{e} = 1$; in this case, the Biot numbers are not sufficiently correlated for the 1D model to be adequate.

Figures 4.26, 4.27, 4.28, and 4.29 show the results in the case of a conductor at 1000 K (high values of h_1 and h_2). In this case, even for $\gamma = \frac{R}{e} = 1$, the 1D model gives good results.

		Case no. 1	Case no. 2	Case no. 3	Case no. 4	Case no. 5
λ	$\text{W m}^{-1} \text{K}^{-1}$	0.1	0.1	10	10	0.1
ρc	$\text{J m}^{-3} \text{K}^{-1}$	2×10^6	2×10^6	3×10^6	3×10^6	2×10^6
R	m	0.01	0.005	0.01	0.005	0.005
e	m	0.005	0.005	0.005	0.005	0.005
h_1	$\text{W m}^{-2} \text{K}^{-1}$	10	10	200	200	200
h_2		5	5	190	190	190
h_3		10	10	200	200	200
h_i		8.8	18	197	395	1077
a_s	$\text{m}^2 \text{s}^{-1}$	5.00×10^{-8}	5.00×10^{-8}	3.33×10^{-6}	3.33×10^{-6}	5.00×10^{-8}
a_i		4.93×10^{-8}	4.79×10^{-8}	3.305×10^{-6}	3.29×10^{-6}	4.42×10^{-8}
a^*		4.96×10^{-8}	4.89×10^{-8}	3.315×10^{-6}	3.32×10^{-6}	5.08×10^{-8}

Table 4.1. Simulated properties (in 3D) and identification results (in 1D) (h_i identified exchange coefficient, a_s simulated diffusivity, a_i diffusivity identified by the 1D model, a^* diffusivity identified by the fin model)

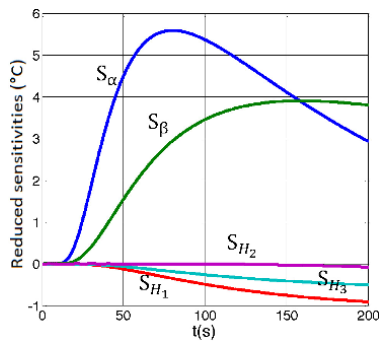


Figure 4.22. Sensitivity curves (case 1)

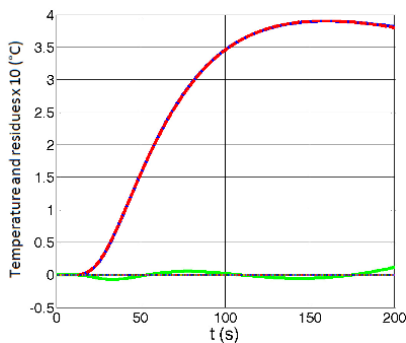


Figure 4.23. Temperature and residues $\times 10$ (case 1)

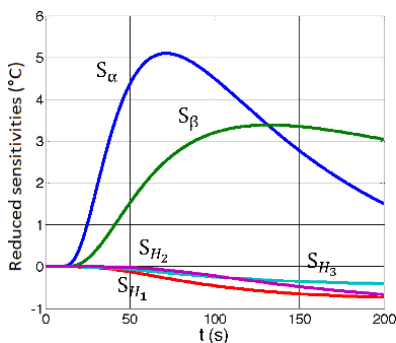


Figure 4.24. Sensitivity curves (case 2)

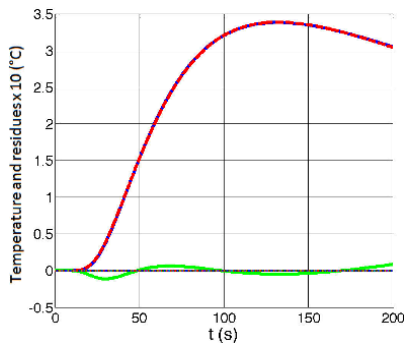


Figure 4.25. Temperature and residues $\times 10$ (case 2)

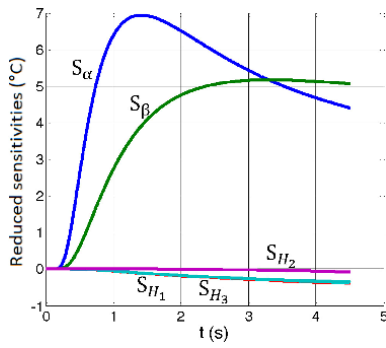


Figure 4.26. Sensitivity curves (Case 3)

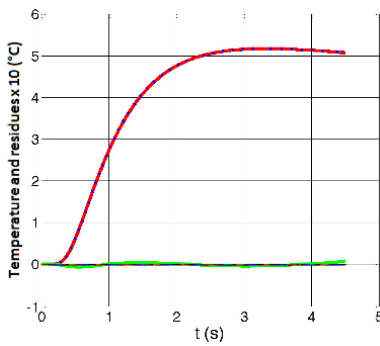


Figure 4.27. Temperature and residues $\times 10$ (Case 3)

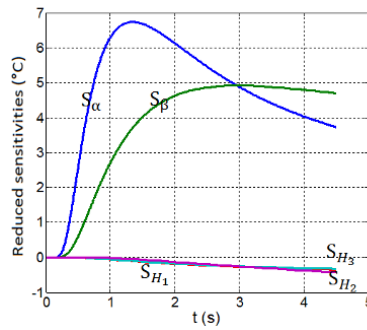


Figure 4.28. Sensitivity curves (Case 4)

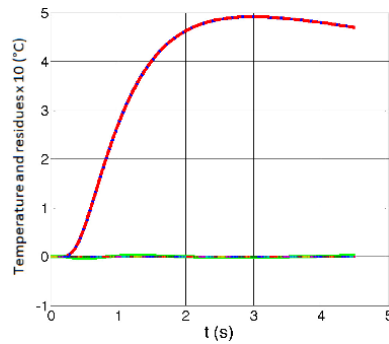


Figure 4.29. Temperature and residues $\times 10$ (Case 4)

We have also shown an extreme case (Case 5), with a ratio $\gamma = \frac{R}{e} = 1$ and values of H_i in the order of 10 (Figures 4.30, 4.31 and Table 4.1). In this case, the 1D model is not suitable for identification. Nevertheless, in the hot plate modeling, it has been shown (fin approximation) that the lateral losses can be taken into account in the 1D model, replacing the Laplace variable by the grouping of $p + \frac{haP_e}{\lambda S}$ where:

- P_e perimeter of the sample
- S surface of the heated face.

However, this formulation leads to an overestimation of the influence of the lateral losses on the measurement of the temperature in the center of the rear face, which is all the more accurate as the thermal conductivity is high.

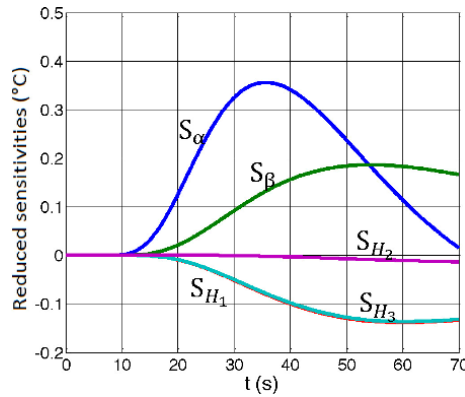


Figure 4.30. Sensitivity curves (case 5)

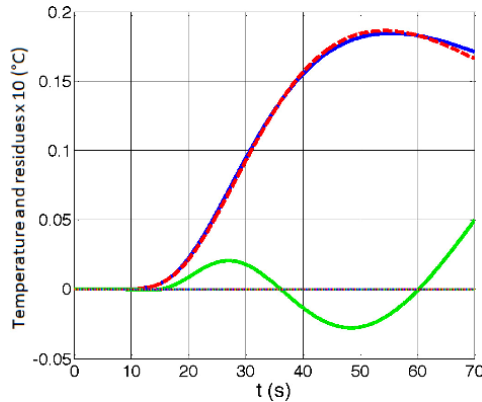


Figure 4.31. Temperature and residues $\times 10$ (case 5)

In summary, by choosing a sample of radius at least equal to 2 times its thickness, it is possible to neglect in all cases the influence of lateral convective losses and to use a unidirectional identification model. In the other cases, if a model error of 2% is acceptable, the modified 1D model can be used with the fin approximation.

4.5.2.3. The 1D model

In the 1D case, we can use the quadrupole method, after the Laplace transform over time. The equivalent electrical diagram is given in Figure 4.32.

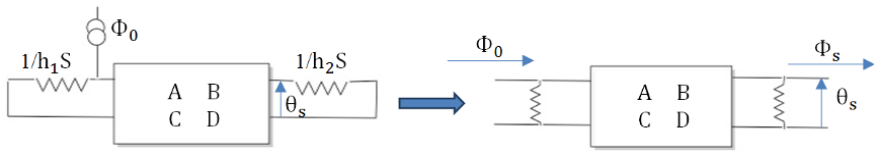


Figure 4.32. Equivalent electrical diagram

We can write:
$$\begin{bmatrix} \theta_e \\ \Phi_e \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ h_1 & 1 \end{bmatrix} \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} 1 & 0 \\ h_2 & 1 \end{bmatrix} \begin{bmatrix} \theta_s \\ \Phi_s \end{bmatrix}$$

with: $\Phi_e = \Phi_0(p)$ and $\Phi_s = 0$

We can write:
$$\begin{bmatrix} \theta_e \\ \Phi_0 \end{bmatrix} = \begin{bmatrix} A_T & B_T \\ C_T & D_T \end{bmatrix} \begin{bmatrix} \theta_s \\ 0 \end{bmatrix}$$

hence:
$$\theta_s = \frac{\Phi_0}{C_T}$$

with: $C_T = C + A(h_1 + h_2) + B h_1 h_2$

$$C_T = \lambda \sqrt{\frac{p}{a}} sh\left(\sqrt{\frac{p}{a}} e\right) + (h_1 + h_2) ch\left(\sqrt{\frac{p}{a}} e\right) + \frac{h_1 h_2}{\lambda \sqrt{\frac{p}{a}}} sh\left(\sqrt{\frac{p}{a}} e\right)$$

To find the number of parameters of the problem, we must adimension the formula with:

– a “temperature” parameter $\frac{e \Phi_0(p)}{\lambda} = K$

– a “time” parameter $\frac{e^2}{a} = \alpha$

– two dimensionless parameters $h_1 \frac{e}{\lambda} = H_1$ and $h_2 \frac{e}{\lambda} = H_2$

We have a problem with four independent parameters:

$$\theta_s(p) = \frac{K}{\sqrt{\alpha p} sh(\sqrt{\alpha p}) + (H_1 + H_2) ch(\sqrt{\alpha p}) + \frac{H_1 H_2 sh(\sqrt{\alpha p})}{\sqrt{\alpha p}}} \quad [4.22]$$

This solution can also be put in the form:

$$\theta_s(p) = \Phi_0(p) \times \theta_D(p)$$

where $\theta_D(p)$ is the Laplace transform of the rise in temperature $T_D(t)$ of the unheated face of the sample in the case of the Dirac pulse (case where $\Phi_0(p) = 1 \text{ J m}^{-2}$):

$$\theta_D(p) = \frac{\frac{e}{\lambda} \times 1}{\sqrt{\alpha p} \operatorname{sh}(\sqrt{\alpha p}) + (H_1 + H_2) \operatorname{ch}(\sqrt{\alpha p}) + \frac{H_1 H_2 \operatorname{sh}(\sqrt{\alpha p})}{\sqrt{\alpha p}}} \quad [4.23]$$

4.5.2.4. Taking into account the shape and length of the pulse

It can be seen from equation [4.22] that, to calculate the temperature $T_2(t)$, it is necessary to know the value of $\phi_0(t)$ (or of $\Phi_0(p)$), which is not possible to identify on the experimental curve $T_2(t)$. It is therefore necessary either to measure directly the pulse or to make a hypothesis about its form.

4.5.2.4.1. General case

The shape of the pulse $I(t)$ is recorded together with the temperature $T_2(t)$ of the unheated face. A simple infrared diode or a photovoltaic cell can be used for this purpose. The heat flux $\phi_0(t)$ absorbed on the surface of the sample is proportional to this pulse:

$$\phi_0(t) = C I(t) \quad [4.24]$$

If we call $T_D(t)$ the solution in response to a Dirac (numerically computable by inverse Laplace transformation of equation [4.23], see Appendix 5), the response to the pulse $I(t)$ is given by the following convolution product:

$$T_2(t) = T_D(t) \otimes C I(t) = \int_0^t C I(\tau) T(t - \tau) d\tau \quad [4.25]$$

The parameters to be identified in this case are therefore: $C \frac{e}{\lambda}$, $\frac{e^2}{a}$, $h_1 \frac{e}{\lambda}$ and $h_2 \frac{e}{\lambda}$. An example will be given in section 4.5.6.

4.5.2.4.2. Special cases

The shape of the pulse and therefore of the absorbed flux $\phi_0(t)$ is known, for example (see Figure 4.33):

– a gating pulse (Taylor model) obtained by cutting off a continuous flow: halogen lamp or laser

$$\phi_0(t) = \frac{q_{01}}{t_f} [1 - \gamma(t - t_f)] \quad [4.26]$$

– a triangle (Cape model) obtained by a flash lamp with a long pulse length

$$\phi_0(t) = \frac{2q_{01}}{t_f} \left(1 - \frac{t}{t_f}\right) [1 - \gamma(t - t_f)] \quad [4.27]$$

– an exponential (Larson model) obtained by a flash lamp of short pulse length or by a pulse laser

$$\phi_0(t) = \frac{q_{01}}{\tau^2} t \exp\left(-\frac{t}{\tau}\right) \quad [4.28]$$

where γ is the Heaviside function: $\gamma(t - t_f) = 0$ if $t \leq t_f$ and $\gamma(t - t_f) = 1$ if $t > t_f$.

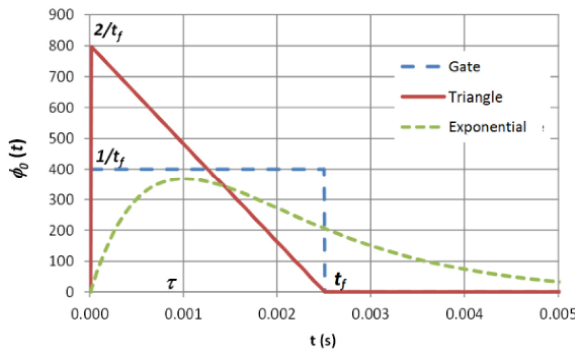


Figure 4.33. Forms considered for pulse. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Knowing the shape of the pulse, it is possible to know its Laplace transform and thus to replace the convolution product in real space by a product in Laplace space:

$$T_2(t) = L^{-1} \left[\Phi_0(p) \frac{\frac{e}{\lambda}}{\sqrt{\alpha p} \operatorname{sh}(\sqrt{\alpha p}) + (H_1 + H_2) \operatorname{ch}(\sqrt{\alpha p}) + \frac{H_1 H_2 \operatorname{sh}(\sqrt{\alpha p})}{\sqrt{\alpha p}}} \right] \quad [4.29]$$

For the three pulse shapes described above:

Gating pulse

$$\Phi_0(p) = \frac{q_{01}}{p} \frac{1 - \exp(-t_f p)}{t_f} \quad [4.30]$$

Triangle

$$\Phi_0(p) = \frac{2q_{01}}{t_f} \left[\frac{1}{p} - \frac{1 - \exp(-t_f p)}{t_f p^2} \right] \quad [4.31]$$

Exponential

$$\Phi_0(p) = \frac{q_{01}}{\tau^2} \frac{1}{\left(p + \frac{1}{\tau^2}\right)} \quad [4.32]$$

t_f or τ are generally known by comparison of the recording of the illumination with one of the forms described by equations [4.30] to [4.32], the unknown parameters still to be estimated are in this case:

$$\frac{eq_{01}}{\lambda}, \frac{e^2}{a}, h_1 \frac{e}{\lambda} \text{ and } h_2 \frac{e}{\lambda}.$$

4.5.2.4.3. Simple case

The barycenter t_b of the pulse is defined by:

$$t_b = \int_0^\infty I(t) t \, dt \quad [4.33]$$

The calculation gives $t_b = \frac{t_f}{2}$ for the gate, $t_b = \frac{t_f}{3}$ for the triangle and $t_b = 2\tau$ for the exponential.

In the case where $t_b < 0.002 \frac{e^2}{a}$, the pulse can be considered as infinitely short and there is no flash length correction to be performed. The temperature is given by:

$$T_2(t) = L^{-1} \left[\frac{\frac{eq_{01}}{\lambda}}{\sqrt{\alpha p} \operatorname{sh}(\sqrt{\alpha p}) + (H_1 + H_2) \operatorname{ch}(\sqrt{\alpha p}) + \frac{H_1 H_2 \operatorname{sh}(\sqrt{\alpha p})}{\sqrt{\alpha p}}} \right] \quad [4.34]$$

In the case where $0.002 \frac{e^2}{a} < t_b < 0.03 \frac{e^2}{a}$, first-order correction is sufficient [DEG 86]. It is sufficient to consider the barycenter of the pulse as the origin of the times, that is, to replace t by $(t - t_b)$ when recording the temperature.

In both cases, the parameters to be estimated are: $\frac{eq_{01}}{\lambda}, \frac{e^2}{a}, h_1 \frac{e}{\lambda}$ and $h_2 \frac{e}{\lambda}$.

4.5.3. Methods for the estimation of diffusivity

To estimate the thermal diffusivity from the thermogram recorded on the non-irradiated face, the set of measurement points is used by comparing the thermogram with the previous theoretical model. The comparison is done by minimizing the sum of the quadratic deviations between the experimental points $T_{2exp}(t_i)$ and the theoretical points $T_{2mod}(t_i)$ (Levenberg–Marquardt or simplex method, for example).

However, since the identification problem is highly nonlinear, the minimization is done by iteration and it is important to initialize the process by the use of values of the unknown parameters which are closest to the sought-after values. For this purpose, we can use the approximate methods developed during the period between 1960 and 1980 (all these methods assume an infinitely short pulse duration).

4.5.3.1. Parker's method [PAR 61]

This method is based on the hypothesis of zero heat losses, which reduces the problem to two unknowns $\frac{e^2}{a}$ and $\frac{\Phi_0}{\rho c e_0}$, two points of the curve are then sufficient to determine the two parameters, the maximum of the temperature and half-rise time $t_{1/2}$.

The thermal diffusivity is then calculated simply from the time $t_{1/2}$ required for the rise in temperature $T_2(t)$ of the rear face to be equal to half the maximum temperature rise reached, T_{2M} , as represented in Figure 4.34.

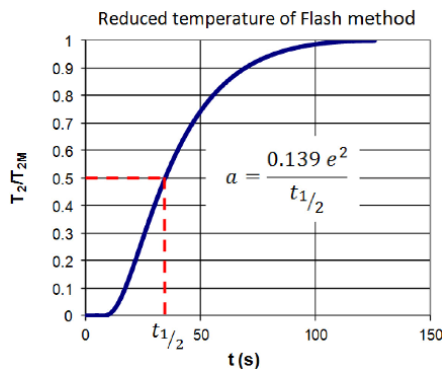


Figure 4.34. Illustration of Parker's method

We thus deduce:

$$a = \frac{0.139 e^2}{t_{1/2}} \quad [4.35]$$

4.5.3.2. Partial times method [DEG 77]

This method takes Parker's idea by assuming equal losses $h_1 = h_2$, and here are three unknown parameters to be identified, so we need at least three points from the curve.

Its principle is based on the use of four points of the thermogram shown in Figure 4.35.

The thermal diffusivity is obtained by the following formulas [DEG 77]:

$$a = \frac{e^2}{t_{5/6}} \left(1.131 - 1.222 \frac{t_{2/3}}{t_{5/6}} \right) \quad [4.36]$$

$$a = \frac{e^2}{t_{5/6}} \left[0.954 - 1.581 \frac{t_{1/2}}{t_{5/6}} + 0.588 \left(\frac{t_{1/2}}{t_{5/6}} \right)^2 \right] \quad [4.37]$$

$$a = \frac{e^2}{t_{5/6}} \left[0.818 - 1.708 \frac{t_{1/3}}{t_{5/6}} + 0.885 \left(\frac{t_{1/3}}{t_{5/6}} \right)^2 \right] \quad [4.38]$$

An average of the three values obtained is generally used. This method is more precise than Parker's method because it takes into account convective losses but has the disadvantage of using only a very small number of points of the thermogram.

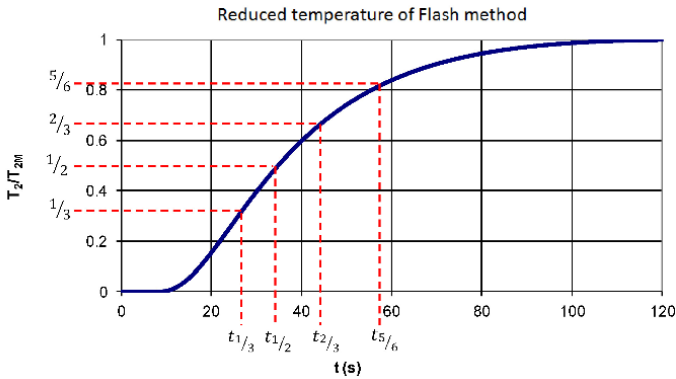


Figure 4.35. Schematic diagram representing partial times

4.5.3.3. Temporal moments method [DEG 86]

This is an improvement on the previous method. It uses all the points of the thermogram in the estimation method through the calculation of integrals. The thermal diffusivity is calculated by:

$$a = e^2 \frac{F(M_{-1})}{M_0}$$

with: $F(x) = 0.08548 - 0.314 (0.5486 - x) + 0.5 (0.5486 - x)^{2.63}$ for $x > 0.27$

$$F(x) = -0.08519 + 0.305x \quad \text{for } x > 0.44$$

where M_0 and M_{-1} are the partial moments of order 0 and -1 defined by:

$$M_0 = \int_{0.1}^{0.8} \frac{T_2(t)}{T_{2m}} dt \quad \text{and} \quad M_{-1} = \int_{0.1}^{0.8} \frac{1}{t} \frac{T_2(t)}{T_{2m}} dt$$

The integrals in the calculation of M_0 and M_{-1} are computed numerically from the set of points of the thermogram.

The partial time moments method also makes it possible to estimate the Biot number: $Bi = \frac{he}{\lambda}$.

4.5.3.4. Estimation using the complete model

As we have already said, the best method is the experimental thermogram/theoretical model comparison by nonlinear least squares (as for all unsteady state methods).

It has been shown that a 1D model was satisfactory in most cases, but it remains to determine the identification interval and the number of exchange coefficients to be determined.

To do this, we have to calculate:

- the sensitivities: $S_K = K \frac{\partial T}{\partial K}$ $S_\alpha = \alpha \frac{\partial T}{\partial \alpha}$ $S_{H_1} = H_1 \frac{\partial T}{\partial H_1}$ $S_{H_2} = H_2 \frac{\partial T}{\partial H_2}$
- the correlations;
- the standard deviations;
- the residues.

For example, consider the following case a) (simulation with 4 parameters):

$$\lambda = 0.1 \text{ W m}^{-1} \text{ K}^{-1}, \rho c = 2 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}, \varphi_0 = 5 \times 10^4 \text{ W m}^{-2}, e = 5 \text{ mm}, \\ h_1 = 10 \text{ W m}^{-2} \text{ K}^{-1}, h_2 = 5 \text{ W m}^{-2} \text{ K}^{-1}.$$

Figure 4.36 gives the theoretical thermogram and Figure 4.37 the sensitivities to the four parameters.

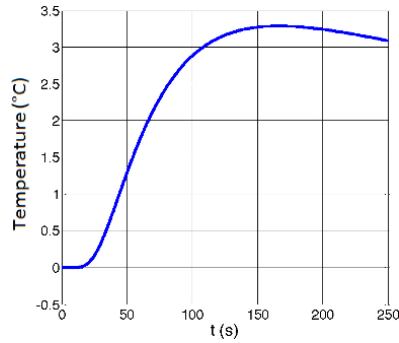


Figure 4.36. *Rear face temperature*

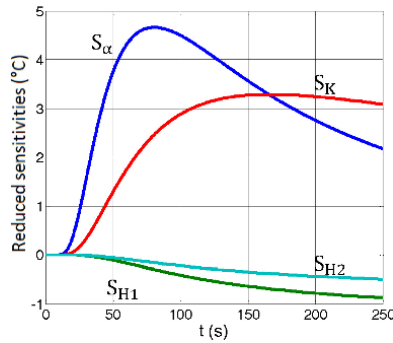


Figure 4.37. *Reduced sensitivities*

It can be seen that the maximum sensitivity to the diffusivity is situated approximately at the $\frac{1}{2}$ rise time and decreases rapidly after the maximum. This shows that it is unnecessary to go very far after the maximum of the thermogram, especially as losses become more important as time increases.

For the number of exchange coefficients to be identified, we must look at the correlations between the parameters. We have estimated the values of coefficients k_1 , k_2 , k_3 , k_4 and k_5 , which minimize the following sums: $\sum_i (S_{H_1} - k_1 S_{H_2})^2$, $\sum_i (S_\alpha - k_2 S_K)^2$, $\sum_i (S_\alpha - k_3 S_{H_2})^2$ and $\sum_i (S_{H_1} - k_4 S_{H_2} - k_5 S_\alpha)^2$. The correlation between the parameters is all the stronger as the curves shown in Figure 4.38 are close to the linear equation $y = x$.

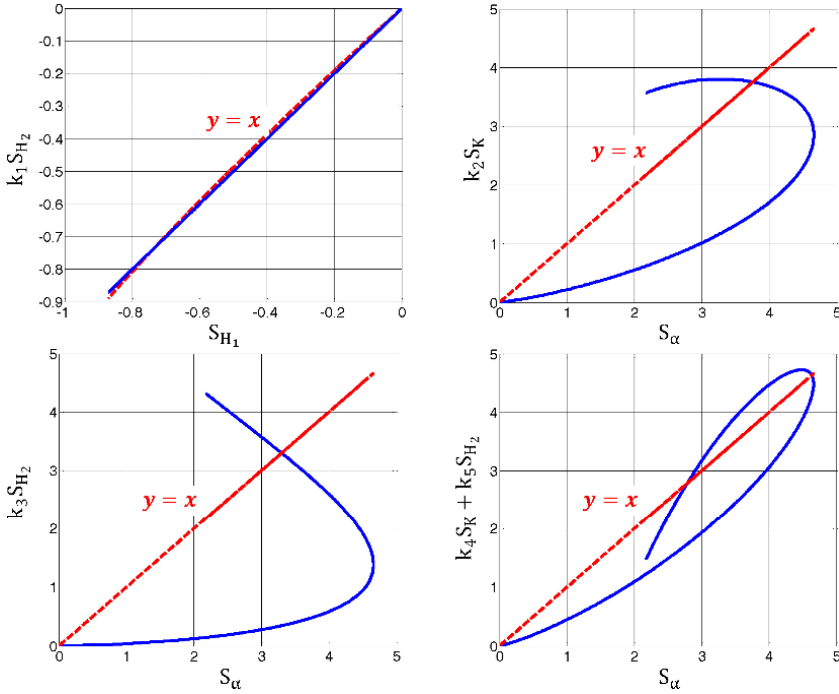


Figure 4.38. Correlations of parameters 2 to 2 and 3 to 3

We observe that H_1 and H_2 are correlated, which is often the case. For the Biot numbers to be uncorrelated, the exchange coefficients must be extremely different, which is rarely the case in practice. We have also simulated an extreme case b) with $h_1 = 100 \text{ W m}^{-2}\text{K}^{-1}$ and $h_2 = 5 \text{ W m}^{-2}\text{K}^{-1}$ and have identified the 2 cases with a model with 3 parameters ($h_1 = h_2$) (see Figure 4.39). The residue analysis confirms the results of the sensitivity study: they are very low in case a), which confirms the very strong correlation of H_1 and H_2 , and they are much worse in case b) indicating that H_1 and H_2 are not sufficiently correlated so that we can make the hypothesis $H_1 = H_2$.

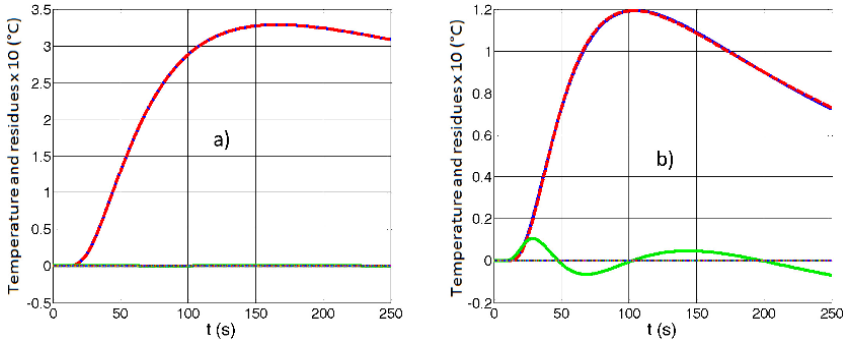


Figure 4.39. Simulated flash thermograms with four parameters and then estimated with three parameters and estimation residues a) $h_1 = 10 \text{ W m}^{-2}\text{K}^{-1}$ and $h_2 = 5 \text{ W m}^{-2}\text{K}^{-1}$; b) $h_1 = 100 \text{ W m}^{-2}\text{K}^{-1}$ and $h_2 = 5 \text{ W m}^{-2}\text{K}^{-1}$

To confirm these results, we performed two simulations of the temperature T_2 of the unheated face of the sample by assuming $h_1 \neq h_2$. We then added a Gaussian measurement noise of standard deviation σT to the simulated values. These simulated and noisy thermograms were then treated as experimental data by making two successive estimations:

- E1: estimation of the 4 parameters α , K , H_1 and H_2
- E2: estimation of the 3 parameters α , K , H_1 by making the hypothesis $H_1 = H_2$.

Recall that: $K = \frac{e \Phi_0(p)}{\lambda}$; $\alpha = \frac{e^2}{a}$; $H_1 = h_1 \frac{e}{\lambda}$ and $H_2 = h_2 \frac{e}{\lambda}$.

The standard deviations (due to measurement noise on the temperature) on the estimated parameters were then calculated using the covariance matrix as explained in section 2.2.6.

– Case a):

$q_0 = 5000 \text{ J m}^{-2}$; $a = 5.0 \times 10^{-8} \text{ m}^2\text{s}^{-1}$; $h_1 = 10 \text{ W m}^{-2}\text{K}^{-1}$ and $h_2 = 5 \text{ W m}^{-2}\text{K}^{-1}$, 2000 measurement points and σT the noise's standard deviation (in K).

$$\text{E1 : } \frac{\sigma K}{K} = 0.698 \sigma T; \frac{\sigma \alpha}{\alpha} = 0.139 \sigma T; \frac{\sigma H_1}{H_1} = 42.5 \sigma T; \frac{\sigma H_2}{H_2} = 26.0 \sigma T$$

$$\text{E2 : } \frac{\sigma K}{K} = 0.135 \sigma T; \frac{\sigma \alpha}{\alpha} = 0.047 \sigma T; \frac{\sigma H}{H} = 0.263 \sigma T$$

Estimated values: $q_0 = 49918 \text{ J m}^{-2}$; $a = 5.004 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$; $h_1 = 7.37 \text{ W m}^{-2} \text{ K}^{-1}$

Model error (bias) (four-parameter simulation identified by a three-parameter model)

$$\frac{\sigma_m K}{K} = 0.000016; \quad \frac{\sigma_m \alpha}{\alpha} = 0.0000056; \quad \frac{\sigma_m H}{H} = 0.000031$$

– Case b):

$q_0 = 5000 \text{ J m}^{-2}$; $a = 5.0 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$; $h_1 = 100 \text{ W m}^{-2} \text{ K}^{-1}$ and $h_2 = 5 \text{ W m}^{-2} \text{ K}^{-1}$, 2000 measurement points and σT the noise's standard deviation.

$$\text{E1: } \frac{\sigma K}{K} = 2.97 \sigma T; \quad \frac{\sigma \alpha}{\alpha} = 0.411 \sigma T; \quad \frac{\sigma H_1}{H_1} = 6.4 \sigma T; \quad \frac{\sigma H_2}{H_2} = 4.5 \sigma T$$

$$\text{E2: } \frac{\sigma K}{K} = 0.568 \sigma T; \quad \frac{\sigma \alpha}{\alpha} = 0.144 \sigma T; \quad \frac{\sigma H}{H} = 0.406 \sigma T$$

Estimated values: $q_0 = 31215 \text{ J m}^{-2}$; $a = 5.37 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$; $h_1 = 25.5 \text{ W m}^{-2} \text{ K}^{-1}$

Model error (bias) (four-parameter simulation identified by a three-parameter model)

$$\frac{\sigma_m K}{K} = 0.0016; \quad \frac{\sigma_m \alpha}{\alpha} = 0.00050; \quad \frac{\sigma_m H}{H} = 0.0014$$

We conclude that, in the case of estimation E1, the standard deviations on H_1 and H_2 are very large in comparison with the other standard deviations, which is another way of showing that the parameters at H_1 and H_2 are correlated. The standard deviations on α and K are lower for E2 than for E1, which confirms that in the case of a correlation between two parameters, we have to be fixed to improve the quality of the estimation of the other parameters.

In agreement with the residues, σ_m of case b) is 100 times greater than that of case a).

4.5.3.5. Practical conclusion

To conclude, in most cases we will use the 1D model with three parameters ($h_1 = h_2$), the estimation will be done by nonlinear least squares and we will take into account the shape of the pulse, if necessary, by a convolution product.

4.5.4. Experimental setups

4.5.4.1. Measurement bench

A measurement bench requires defining:

- a source of surface heat;
- a system for measuring and acquiring the surface temperature;
- an enclosure and a sample holder adapted to the desired temperature and pressure range.

4.5.4.1.1. Realization of the surface heat source

It is necessary to obtain a very high power illumination for a short time; the different sources used depend on the type of sample to be analyzed. Two important constraints regardless of the source are:

- the heat flux absorbed by the sample must be as uniform as possible;
- the heat flux must only arrive on the front side (the illumination must not reach the lateral faces and the rear face by reflection).

Going from the longest to the shortest durations, we find:

- the halogen lamp with a duration of illumination that can reach several tens of seconds to obtain a sufficient amount of radiated flux (power up to 1000 W);
- the flash lamps fed by the discharge of a battery of capacitors, the illumination times can vary between 100 μ s and 10 ms, for energies comprised between 100 J and 5000 J;
- continuous CO₂ lasers whose illumination times are greater than 100 ms, for powers of the order of 100 to 200W;
- pulsed lasers whose durations can be extremely short, a few nanoseconds for ultra-fast thermal applications (power up to 100 J).

For reflector samples, a thin layer of matt black paint is coated on the front face.

In some cases it will be necessary to correct the shape of the flash if its duration is too long to be assimilated to a Dirac, for that one will record the flash by an infrared cell.

4.5.4.1.2. Measurement of the temperature of the rear face

The rise in temperature of the rear face is generally low (of the order of 1°C); two methods are used to measure a surface temperature, either a measurement by contact or without contact:

- With contact, it is necessary to use a thermocouple having a high thermoelectric power; if possible (between -160°C and 100°C) bismuth telluride (Bi_2Te_3) having a thermoelectric power of $360\ \mu\text{V.K}^{-1}$ at 20°C and $400\ \mu\text{V.K}^{-1}$ at 80°C .

Whatever the nature of the thermocouple, the “separate contacts” method must be implemented, the ends of the two wires of the thermocouple do not touch each other, they are held by pressure in contact with the rear face at two points near the center of the sample and a few millimeters apart. The electrical contact between the two wires of the thermocouple is ensured by the rear face of the sample. If the sample is not electrically conductive, the rear face of the sample is covered by a very thin layer of silver lacquer.

- Without contact, measurement can also be carried out using an infrared optical detector that measures a magnitude proportional to the heat flux emitted by this face. This flux can be considered proportional to the rise in temperature for the very small variations measured. The response time of a non-contact measurement is much lower than that of a thermocouple and its use becomes mandatory in the case of a very diffuse and thin material whose flash thermogram can be extremely short. It is important to verify that it is the surface temperature that is measured; in the case of semi-transparent infrared materials, a deposit will be required (for example, $1\ \mu\text{m}$ of titanium).

4.5.4.1.3. Preparation of samples

For reflector samples, a thin layer of matt black paint is coated on the front face.

For electrical insulator samples with contact measurement, the rear face is covered with a thin layer of silver lacquer.

For semitransparent samples with non-contact measurement, the rear face is covered with a metallic deposit.

4.5.4.1.4. Measurement chain

In practice, any good acquisition system will be sufficient, it is only necessary to ensure the lowest measurement noise and a bandwidth adapted to the duration of the measurement.

4.5.4.2. Example of setups and measurements

4.5.4.2.1. Example 1

This is the example of a conventional apparatus with, for the pulse of the flash tubes and for detectors, semiconductor thermocouples with separate contacts. The double enclosure makes it possible to fix the temperature by a circulation of fluid (Figure 4.40).

The characteristics of the device presented in Figure 4.40 are as follows:

- flash: three flashing lamps, 4 J cm^{-2} , duration around 6 ms;
- enclosure: temperature from -10°C to 100°C , under vacuum;
- sample: diameter up to 40 mm;
- sensor: Bi_2Te_3 (sensitivity: $360 \mu\text{V K}^{-1}$);
- acquisition system: amplifier with filter + numerical oscilloscope 12 bits, 4000 points recording.

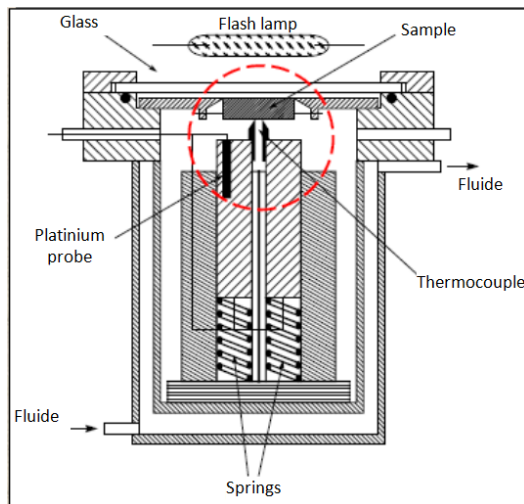


Figure 4.40. Example of a flash diffusivimeter

A “Protherm” software was developed at LEMTA to identify the thermal diffusivity from the recording of the variation in the temperature of the rear face. Figure 4.41 shows an example of the result obtained by Protherm for a plaster

sample of 8 mm thickness, the figure gives the experimental curve, the identified theoretical curve and the residues ($\times 10$), and the diffusivity obtained is $2.9 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$.

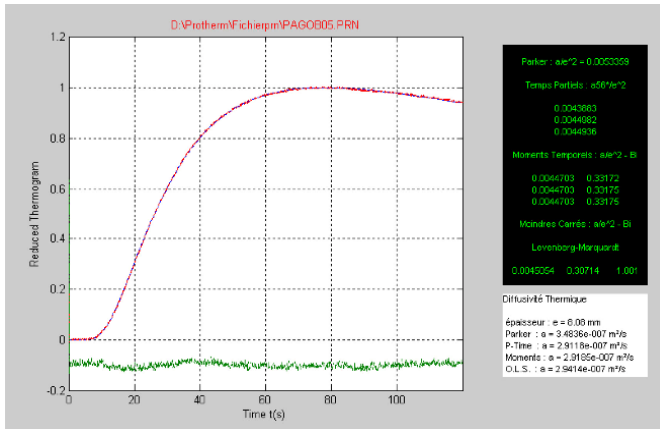


Figure 4.41. Experimental and modeled curves and estimation results given by Protherm software

4.5.4.2.2. Example 2

In this example, an illumination of duration of 30 s was performed using a 500 W halogen lamp on a sample of stabilized earth brick of 5 cm thickness. The representation of the experimental curve [MEU 04] and the theoretical curve plotted using the parameters estimated by least squares are given in Figure 4.42(a). The theoretical curve was calculated using equations [4.29] and [4.30] with $t_f = 30 \text{ s}$ (slot pulse). The estimated diffusivity value is $a = 5.25 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$.

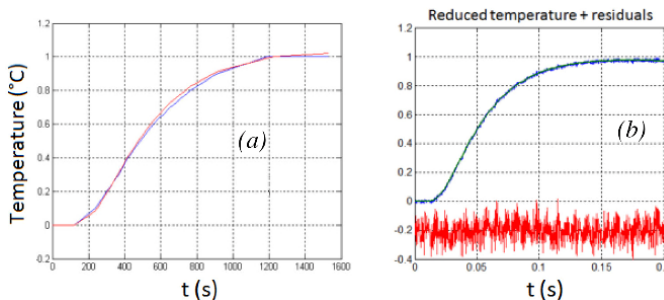


Figure 4.42. Experimental and theoretical thermograms (a) earth brick, (b) carbon

4.5.4.2.3. Example 3

In this example, an illumination of a duration of 1 ms was achieved using a flash lamp on a carbon sample of thickness 6.58 mm. The temperature was measured by an optical detector with a very short response time. The representation of the experimental curve and the theoretical curve plotted using the parameters estimated by least squares is given in Figure 4.42(b). The value of the estimated diffusivity is: $\alpha = 1.14 \times 10^{-4} \text{ m}^2 \text{ s}^{-1}$.

4.5.4.2.4. Example 4

Example 4 is the measurement of the thermal diffusivity of an aluminum nitride sample (AlN). The classical method is not applicable because of the length of the pulse, which can no longer be considered a Dirac and partly because of the semi-transparent character of the material.

To make the surfaces of the samples opaque to the visible and the infrared, we deposit on each face a titanium layer of $1 \mu\text{m}$.

The comparison of a complete model and a reduced model allows us to show that the influence of the titanium layers and the losses are negligible. The model used for the identification is thus a 1D single-layer model without loss.

As far as the pulse is concerned, we have used an analytic representation, which allowed us to work in the Laplace space and replace the convolution with a simple product.

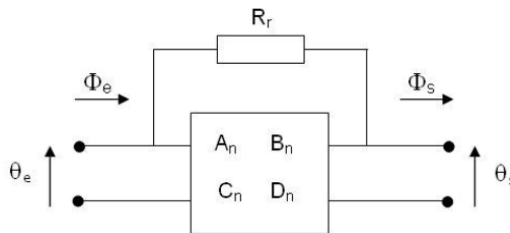


Figure 4.43. *Electrical equivalent model*

Figure 4.43 gives the model representation in electrical analogy where the quadrupole (A_n , B_n , C_n , D_n) represents the conductive transfer and the resistance R_r the radiative transfer.

By calling A, B, C, D the resulting matrix, we get:

$$\begin{bmatrix} \theta_e \\ \Phi_e \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta_s \\ \Phi_s \end{bmatrix}$$

where : $\Phi_s = 0 \rightarrow \theta_s = \frac{\Phi_e}{c}$ (no loss)

with: $C = \frac{C_n + 2C_r(A_n - 1)}{1 + C_r B_n}$ and: $C_r = \frac{1}{R_r}$

The pulse is represented by an exponential: $\phi_0(t) = \frac{q_{01}}{\tau^2} t \exp\left(-\frac{t}{\tau}\right)$, which gives in the Laplace space:

$$\Phi_e = \Phi_0(p) = \frac{q_{01}}{\tau^2} \frac{1}{\left(p + \frac{1}{\tau}\right)}$$

In addition, consequently:

$$\theta_s = \frac{q_{01}(1 + C_r B_n)}{C_n + 2C_r(A_n - 1)} \frac{1}{\tau^2 \left(p + \frac{1}{\tau}\right)^2} \quad [4.39]$$

with:

$$C_r = \frac{1}{R_r}; A_n = \cosh\left(\sqrt{\frac{p}{a}} e\right); B_n = \frac{1}{\lambda \sqrt{\frac{p}{a}}} \sinh\left(\sqrt{\frac{p}{a}} e\right); C_n = \lambda \sqrt{\frac{p}{a}} \sinh\left(\sqrt{\frac{p}{a}} e\right)$$

Equation [4.39] can also be written as:

$$\theta_s = \frac{q_{01} \left[1 + \frac{C_r}{\lambda \sqrt{\frac{p}{a}}} \sinh\left(\sqrt{\frac{p}{a}} e\right) \right]}{\lambda \sqrt{\frac{p}{a}} \sinh\left(\sqrt{\frac{p}{a}} e\right) + 2C_r \left[\cosh\left(\sqrt{\frac{p}{a}} e\right) - 1 \right]} \frac{1}{\tau^2 \left(p + \frac{1}{\tau}\right)^2} = \frac{\frac{e q_{01}}{\lambda} \left[1 + C_r \frac{e}{\lambda \sqrt{\frac{p}{a}}} \sinh\left(\sqrt{\frac{p}{a}} e\right) \right]}{e \sqrt{\frac{p}{a}} \sinh\left(\sqrt{\frac{p}{a}} e\right) + 2C_r \frac{e}{\lambda} \left[\cosh\left(\sqrt{\frac{p}{a}} e\right) - 1 \right]} \frac{1}{(p\tau + 1)^2}$$

This expression gives rise to three adimensional parameters to be identified:

$$\frac{e q_{01}}{\lambda}, \quad \frac{C_r e}{\lambda} \quad \text{and} \quad \frac{a}{e^2}$$

The length of the pulse τ is measured by identification of the flash with the chosen form.

The pulse is obtained by flash tubes (energy up to 3000 J), and, for detection, an infrared mono detector (InSb) cooled by a Sterling motor is used. After amplification, the signal is sent to an acquisition chain (digital Oscilloscope Picoscope) and processed on a computer. The shape of the pulse is recorded simultaneously at the temperature by an infrared diode; Figure 4.44 gives an example of a recording.

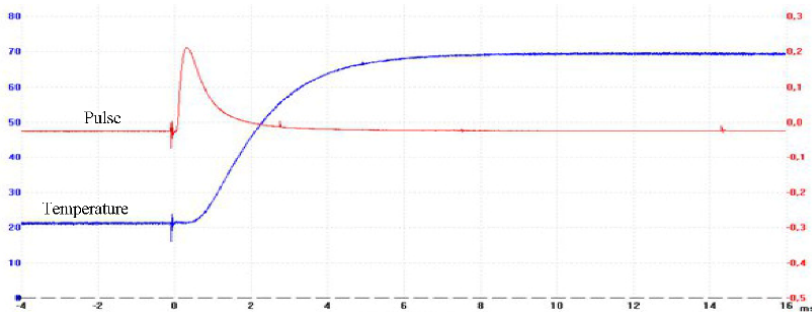


Figure 4.44. Example of a recording; the temperature in blue and the pulse in red.
For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

After identifying τ , the experimental temperature curve is compared with the theoretical least squares curve (Levenberg–Marquardt).

If we look at Figure 4.45, we see that the residuals obtained are not perfectly flat, which means that the model used is not perfectly in line with reality; it would be necessary to build a more sophisticated radiation conduction coupled model.

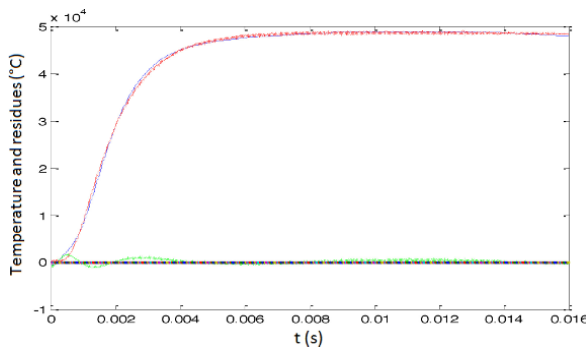


Figure 4.45. Thermogram, identified curve and residues

4.5.4.2.5. Example 5

Example 5 is a case of ultra-fast heat transfer, measuring the thermal diffusivity of diamond:

- the flash is a pulsed laser at 1054 nm – 4 J for a duration of 20 ns;
- the sample has a maximum diameter of 10 mm;
- the detector is an infrared HgCdTe detector with a bandwidth of 500 MHz;
- the acquisition chain consists of a 500 MHz oscilloscope and a 50 MHz amplifier.

Figure 4.46 is a photograph of the measurement system.

Figure 4.47 shows the thermogram and the identified curve.



Figure 4.46. High-speed heat transfer setup

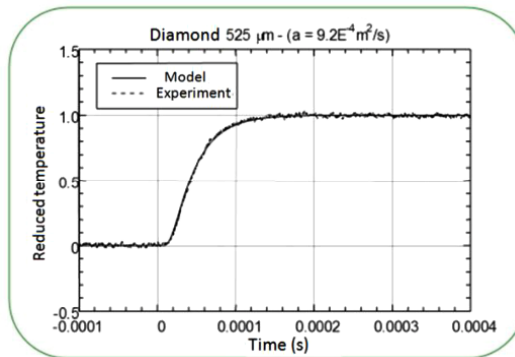


Figure 4.47. Thermogram and identified curve

4.5.4.2.6. Limitations of the flash method:

In choosing the setup, we must take into account a certain number of criteria that condition the validity of the experiment according to the nature of the sample:

$$-\frac{e^2}{a} > \frac{10}{f}, \text{ where } f \text{ is the bandwidth of the acquisition chain (in Hz);}$$

$$-\frac{e^2}{a} > 400 t_d, \text{ where } t_d \text{ is the response time of the temperature detectors;}$$

$-\frac{e^2}{a} > 500 t_b$, where t_b is the energy barycenter of the flash: in this case, we can be satisfied with the approximation $\phi_0(t) = \phi_{01}$ without performing flash length correction.

On the other hand, the flash method relies on two conditions:

- the luminous flux sent on the front face is absorbed by the surface and does not “penetrate” into the sample;
- a quantity proportional to the rise in temperature is measured on the rear face.

These two conditions exclude very light materials and porous media from the field of application. Indeed, it is very difficult to measure the surface temperature of a very light medium; the solution of the thermocouple is inapplicable in this case. In the case of a porous medium, the flash sent to the front face is not completely absorbed at the surface level; a part of it penetrates the mass of the sample through the porosity and distorts the measurement. In these two cases, the trilayer method developed specifically for these types of materials is to be preferred to the flash method.

4.6. Flash 3D

4.6.1. Principle and history

4.6.1.1. Principle

It is a transient-type method that allows the measuring of the values of the thermal diffusivity according to the three directions of space for an anisotropic material.

The experiment involves perturbing a sample of small dimensions (typically $40 \times 40 \times 3 \text{ mm}^3$) in such a way as to create a 3D transfer and to measure several temperatures on the opposite side to the perturbation (see Figure 4.48). The excitation is performed by a laser at the sample's center and the response of the temperature field on the rear face is measured by an infrared camera. The excitation is generally performed so that the temperature variations induced within the material are low.

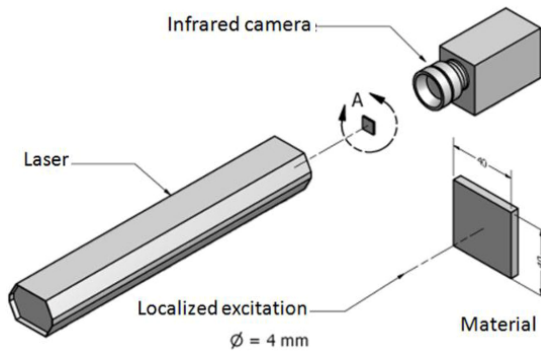


Figure 4.48. *Principle of the experiment*

This method was initiated to enable the measurement of the thermal properties of anisotropic materials in the three directions of space. These materials are usually available in thin plates so that it is not possible to cut three samples with sufficient cross-section area in the three directions of space. Thus it is not possible to use a 1D method to measure their thermal properties.

In all that follows, it will be assumed that the plate is cut in the principal directions of anisotropy.

As for the classic flash, a measurement setup requires defining:

- a sample holder adapted to the desired temperature and pressure range;
- a source of surface heat which in this case will be a laser;
- a system for measuring and acquiring the surface temperature which in this case will be an infrared camera;
- a thermal model associated with heat transfer;
- a method of identification.

4.6.1.2. *A brief history*

Taylor and Donaldson [TAY 75] first proposed extending the flash method for the characterization of anisotropic materials by subjecting the sample to localized excitation.

A theoretical and experimental improvement of the experiment was proposed by Lachi [LAC 91]. However, the implementation is difficult, since the measurements (2 or 3 temperatures) are made by contact; the method is very sensitive to the positioning of the thermocouples and to the size and shape of the excitation.

Major progress was made using infrared cameras for temperature measurements, and it was necessary to wait for improvements in sensitivity and spatial accuracy of the cameras. Philippi and Batsale [PHI 94] showed that, using an infrared camera and a Fourier transform in space, it is possible to get rid of the spatial form of the flux (pulsed in time).

Finally, starting from 2001, the team of B. Rémy at LEMTA [SOU 13] proposed to modify the estimation function, which made it possible to measure, by a single experiment and on a small sample, the thermal longitudinal and transverse diffusivities. This has been made possible thanks to the significant and recent progress made on infrared cameras and lasers.

4.6.2. Hypotheses and model

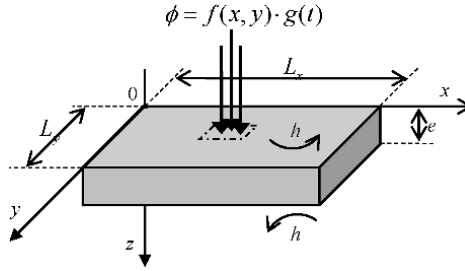


Figure 4.49. Problem modeling

We consider an opaque, parallelepipedic sample subjected to a pulse-type excitation on its front face (see Figure 4.49). The applied heat flux, of any shape in space, can then be written in the form:

$$\phi(x, y, t) = f(x, y) g(t)$$

At the initial time, the system is in thermal equilibrium with the surrounding medium at temperature T_{ext} assumed to be constant, as well as the conductivities of the material along its directions of anisotropy λ_x , λ_y and λ_z (orthotropic material) over the interval temperature at which the experiment takes place. Considering a material of small thickness compared with its other dimensions, the losses on the lateral surfaces can be neglected.

Given all these hypotheses and by setting $T^* = T - T_{ext}$, the heat equation is written as:

$$\lambda_x \frac{\partial^2 T^*}{\partial x^2} + \lambda_y \frac{\partial^2 T^*}{\partial y^2} + \lambda_z \frac{\partial^2 T^*}{\partial z^2} = \rho c \frac{\partial T^*}{\partial t}$$

with the initial condition: $T^*(x, y, z, t = 0) = 0$

and the boundary conditions: $\lambda_z \frac{\partial T^*}{\partial z} \Big|_{z=0} = h_0 T^*_{z=0} - \phi(x, y, t)$

$$\lambda_z \frac{\partial T^*}{\partial z} \Big|_{z=e} = -h_e T^*_{z=e}$$

$$\lambda_x \frac{\partial T^*}{\partial x} \Big|_{x=0} = 0 \quad \lambda_x \frac{\partial T^*}{\partial x} \Big|_{x=L_x} = 0$$

$$\lambda_y \frac{\partial T^*}{\partial y} \Big|_{y=0} = 0 \quad \lambda_y \frac{\partial T^*}{\partial y} \Big|_{y=L_y} = 0$$

To solve this problem, we will perform a double Fourier transform in space and a Laplace transform in time:

$$\theta(\alpha_n, \beta_m, z, p) = \frac{1}{L_x L_y} \int_{x=0}^{x=L_x} \int_{y=0}^{y=L_y} \int_{t=0}^{\infty} T^* \cos(\alpha_n x) \cos(\beta_m y) \exp(-p t) dt dy dx$$

where: p is the Laplace variable

$$\alpha_n = \frac{n \pi}{L_x} \text{ where } n \text{ is a natural number}$$

$$\beta_m = \frac{m \pi}{L_y} \text{ where } m \text{ is a natural number}$$

After transformation, the source is written as: $\Phi(\alpha_n, \beta_m, p) = F(\alpha_n, \beta_m) G(p)$, with:

$$F(\alpha_n, \beta_m) = \frac{1}{L_x L_y} \int_{x=0}^{x=L_x} \int_{y=0}^{y=L_y} f(x, y) \cos(\alpha_n x) \cos(\beta_m y) dy dx$$

$$G(p) = \int_{t=0}^{\infty} g(t) \exp(-p t) dt$$

After transformation, the heat equation becomes:

$$-(\alpha_n^2 \lambda_x + \beta_m^2 \lambda_y + \rho c p) \Theta + \lambda_z \frac{d^2 \Theta}{dz^2} = 0$$

and the boundary conditions become:

$$\lambda_z \left. \frac{d\Theta}{dz} \right|_{z=0} = h_0 \Theta_{z=0} - F(\alpha_n, \beta_m) G(p)$$

$$\lambda_z \left. \frac{d\Theta}{dz} \right|_{z=e} = -h_e \Theta_{z=e}$$

By setting:

$$\gamma = \sqrt{\frac{p}{a_z} + \alpha_n^2 \left(\frac{\lambda_x}{\lambda_z} \right) + \beta_m^2 \left(\frac{\lambda_y}{\lambda_z} \right)} = \sqrt{\frac{p}{a_z} + \alpha_n^2 \left(\frac{a_x}{a_z} \right) + \beta_m^2 \left(\frac{a_y}{a_z} \right)}$$

The system of equations is finally written in the transformed space as:

$$\frac{d^2\Theta}{dz^2} = \gamma^2 \Theta$$

$$\lambda_z \left. \frac{d\Theta}{dz} \right|_{z=0} = h_0 \Theta_{z=0} - F(\alpha_n, \beta_m) G(p)$$

$$\lambda_z \left. \frac{d\Theta}{dz} \right|_{z=e} = -h_e \Theta_{z=e}$$

In this experiment, the temperature fields on the rear face ($z = e$) are of particular interest.

The solution is written as:

$$\Theta(\alpha_n, \beta_m, z = e, p) = \frac{\frac{e}{\lambda_z} F(\alpha_n, \beta_m) G(p)}{\gamma e \operatorname{sh}(\gamma e) + \left(\frac{h_0 e}{\lambda_z} + \frac{h_e e}{\lambda_z} \right) \operatorname{ch}(\gamma e) + \left(\frac{h_0 e}{\lambda_z} \frac{h_e e}{\lambda_z} \right) \frac{\operatorname{sh}(\gamma e)}{\gamma e}} \quad [4.40]$$

This solution seems difficult to use, and it is even more difficult if we use inverse transforms in space and time.

We will present only a particular case, where the flux pulse is short enough to be assimilated to a Dirac.

4.6.3. Identification method

Several constraints do not make it possible to carry out the identification directly on the harmonics given by the expression [4.40].

The most important one is the fact that it is very difficult to obtain the Laplace transform of the measured temperatures because of the problems of truncation of the temperature signal, the latter never returning to the initial temperature in the measurement time.

On the other hand, the large number of parameters to be identified results in a badly posed inverse problem (the number of usable harmonics being quite low, always less than 8).

To reduce the number of parameters, we assume that all the exchange coefficients have the same value. Indeed, it is shown that, when the exchange coefficients are close (whatever their value), they are correlated. On the other hand, we will separate the problem of identification into three problems each containing few parameters to be identified (3 or 2). Finally, we will perform a Laplace on equation [4.40], hence the harmonics used:

$$\Theta(\alpha_n, \beta_m, z = e, t) = \mathcal{L}^{-1}(\Theta(\alpha_n, \beta_m, z = e, p)) \quad [4.41]$$

$$\Theta(\alpha_n, \beta_m, z = e, p) = \frac{\frac{e}{\lambda_z} F(\alpha_n, \beta_m)}{\gamma e \operatorname{sh}(\gamma e) + 2\left(\frac{h e}{\lambda_z}\right) \operatorname{ch}(\gamma e) + \left(\frac{h e}{\lambda_z}\right)^2 \frac{\operatorname{sh}(\gamma e)}{\gamma e}} \quad [4.42]$$

4.6.3.1. First step

We will use the mean temperature, that is the solution for the harmonics $\alpha_n = 0$ and $\beta_m = 0$. Solution [4.42] shows that we obtain the same solution as for the 1D flash problem, so a classical identification (see section 4.5) will enable us to identify, under good conditions, the diffusivity a_z , the exchange coefficient h and a parameter related to the quantity of energy.

The estimated parameters will therefore be: e^2/a_z ; $h e/\lambda_z$; $e F(0,0)/\lambda_z$

4.6.3.2. Second and third steps

Given previous remarks (interest in decreasing the number of parameters to be identified), we will consider the transverse diffusivity a_z and the Biot number as perfectly identified. The estimation of the diffusivities a_x and a_y will be done through the identification functions, and it is demonstrated that:

$$\ln \left[\frac{\Theta(\alpha_n, \beta_m, e, t)}{\Theta(0, 0, e, t)} \right] = \ln \left[\frac{F(\alpha_n, \beta_m)}{F(0, 0)} \right] - (\alpha_n^2 a_x + \beta_m^2 a_y) t \quad [4.43]$$

For a_x , only harmonics $\theta(\alpha_n, \beta_m = 0, z = e, t)$ are considered, and there are only two parameters to be identified:

$$\frac{a_x}{a_z}; \frac{e}{\lambda_z} F(\alpha_n, 0)$$

Similarly, for the diffusivity a_y , only harmonics $\theta(\alpha_n = 0, \beta_m, z = e, t)$ are considered as well as the parameters:

$$\frac{a_y}{a_z}; \frac{e}{\lambda_z} F(0, \beta_m)$$

In practice, only 8 to 10 harmonics for each diffusivity will be usable and often under poor conditions.

Finally, at the end of the procedure, we will obtain a dozen estimates of the diffusivities. However, these will not all be as precise (the harmonics are not of the same quality), and we will realize a weighted average of these.

These different estimates are then weighted by their variances according to the Gauss–Markov equation:

$$a_x = \left[\sum_i \left(\frac{1}{\text{var}(a_{x,i})} \right) \right]^{-1} \sum_i \left[\frac{1}{\text{var}(a_{x,i})} a_{x,i} \right] \quad [4.44]$$

4.6.3.3. Validation of the method

In order to verify the correct functioning of the identification, we performed numerical simulations of the problem using the 3D FlexPDE® software for several types of samples. We give as an example the results obtained on a material of carbon fiber and an insulating material (PVC) (see Figures 4.50 and 4.51):

Dimensions: $L_x = 40$ mm, $L_y = 40$ mm, $e = 3$ mm

Properties:

Carbon:

$$a_x = 3.8 \times 10^{-6} \text{ m}^2 \text{ s}^{-1}, a_y = 4.5 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}, a_z = 4.5 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}, \\ \rho c = 2.6 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$$

PVC:

$$a_x = 1.2 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}, a_y = 1.2 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}, a_z = 1.2 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}, \\ \rho c = 1.58 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$$

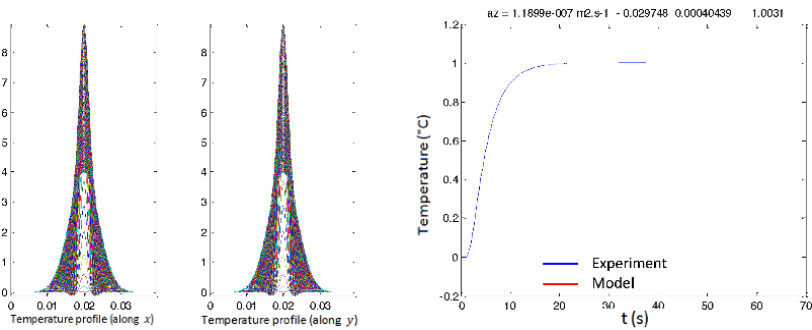


Figure 4.50. Temperature profiles and average thermogram FlexPDE® 3D simulation on PVC material. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

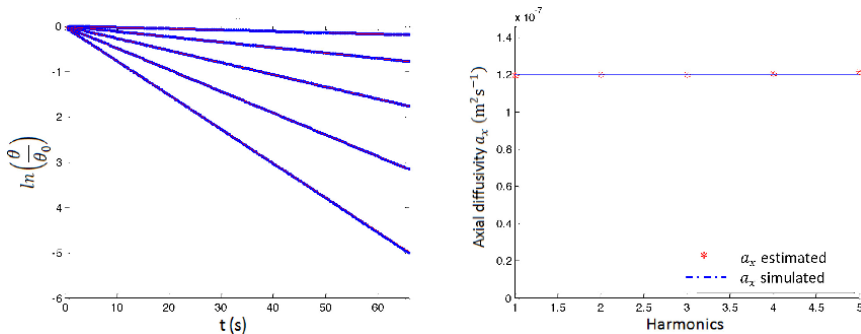


Figure 4.51. Identification functions and estimated axial diffusivity (harmonics 2,4 ... 10) FlexPDE® 3D simulation on PVC material. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Figure 4.50 shows, for PVC, the temperature profiles in x , that is the mean temperature in y for each time as a function of x ; in the same way, the profiles in y (average temperature in x for each time) and the average temperature of the unheated face as a function of time.

Figure 4.51 shows the identification function, that is, the function defined by equation [4.43] and the axial thermal diffusivity estimated by harmonics 2, 4, 6, 8 and 10.

PVC		Carbon	
<i>Simulated diffusivities</i>	<i>Identified diffusivities</i>	<i>Simulated diffusivities</i>	<i>Identified diffusivities</i>
$a_x = 1.2 \times 10^{-7}$	$a_x = 1.2 \times 10^{-7} (\pm 2\%)$	$a_x = 3.8 \times 10^{-6}$	$a_x = 3.8 \times 10^{-6} (\pm 4\%)$
$a_y = 1.2 \times 10^{-7}$	$a_y = 1.2 \times 10^{-7} (\pm 2\%)$	$a_y = 4.5 \times 10^{-7}$	$a_y = 4.5 \times 10^{-7} (\pm 3\%)$
$a_z = 1.2 \times 10^{-7}$	$a_z = 1.19 \times 10^{-7}$	$a_z = 4.5 \times 10^{-7}$	$a_z = 4.46 \times 10^{-7}$

Table 4.2. *Estimates of thermal diffusivity ($m^2 s^{-1}$) on PVC and carbon*

The results presented in Table 4.2 show the feasibility of the method of identification.

4.6.4. Example of an experimental setup

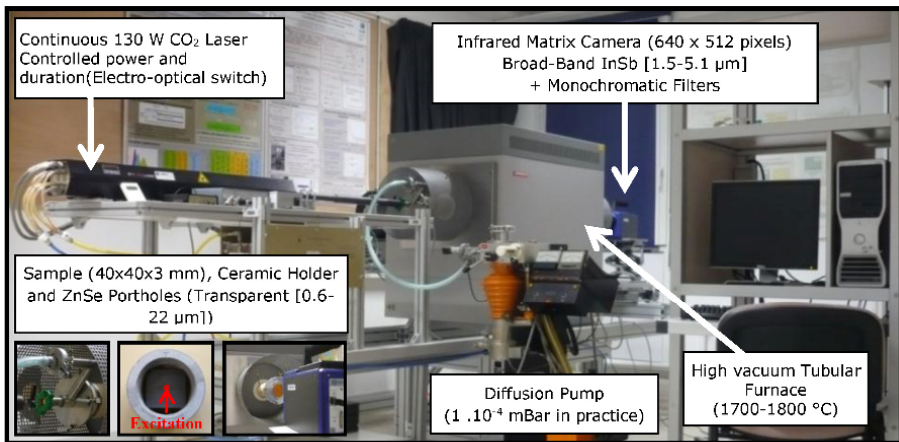


Figure 4.52. *Benchmark for high-temperature thermal diffusivities (LEMTA, 2010)*

Figure 4.52 shows a high-temperature experimental device containing:

- a vacuum tube furnace for measurements up to 1800°C;
- a continuous CO₂ laser of 130 W, for which power and duration can be controlled;
- an InSb matrix infrared camera (1.5 to 5.1 μm);
- a diffusion pump (10^{-4} mbar);
- a sample holder for square samples of 40 mm $\times$ 40 mm;
- a number of accessories such as filters, transparent ZnSe portholes in the 1.5 to 5.1 μm band of the camera.

Figure 4.53 shows the temperature records of the back face as a function of space coordinates for different times on the titanium sample at 60°C.

Figure 4.54 shows the thermogram of the mean temperature of the back face with the estimated curve and residuals, and this thermogram allows the identification of the transverse diffusivity (a_z).

Figure 4.55 shows two examples of the estimation of the axial diffusivity on harmonics 4 and 5. The experimental harmonic, the estimated harmonic and the residuals are found in the figures. If the flash was perfectly centered, the odd harmonics would be zero (which explains the small amplitude of the odd harmonic and therefore a weak signal/noise). These harmonics make it possible to identify the axial diffusivity (a_x).

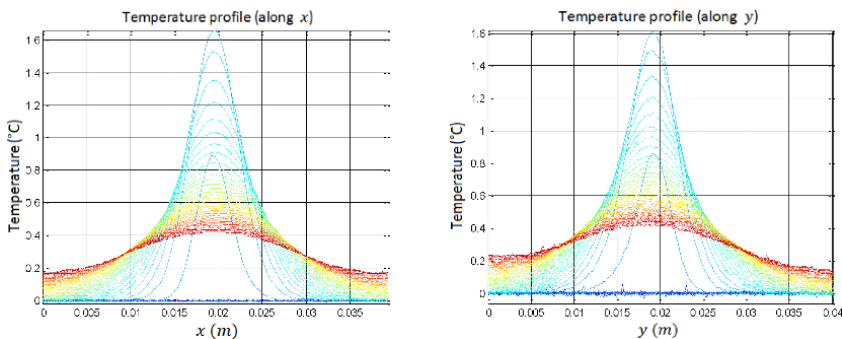


Figure 4.53. Temperature profiles on an isotropic titanium sample at 60°C.
For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

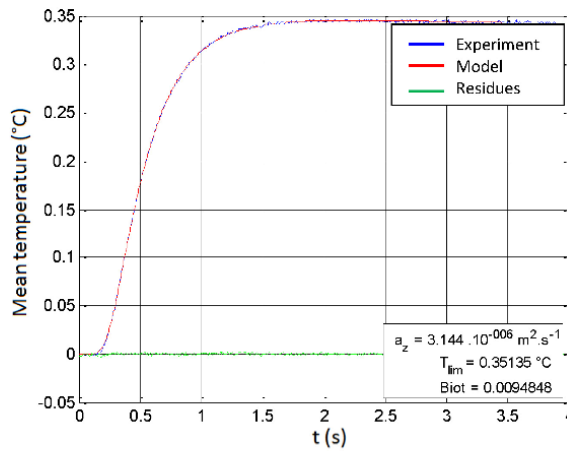


Figure 4.54. Estimation of the transverse diffusivity a_z on a titanium sample at 60°C. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

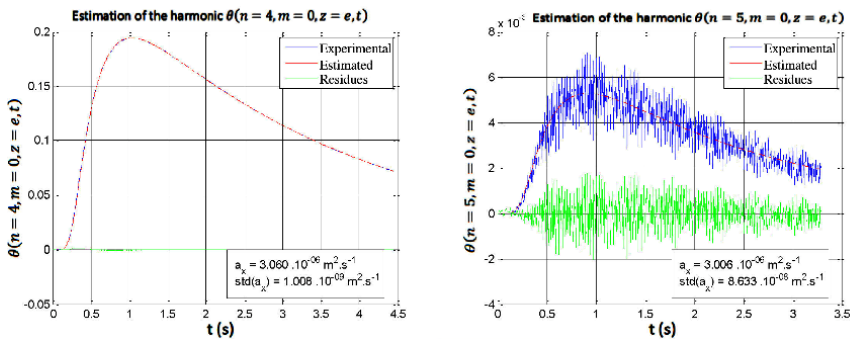


Figure 4.55. Examples of harmonic axial diffusivity a_x estimation, harmonic by harmonic on an isotropic Titanium sample at 60°C. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Figure 4.56 shows the rear face temperature records as a function of space coordinates for different times on the titanium sample at 800°C.

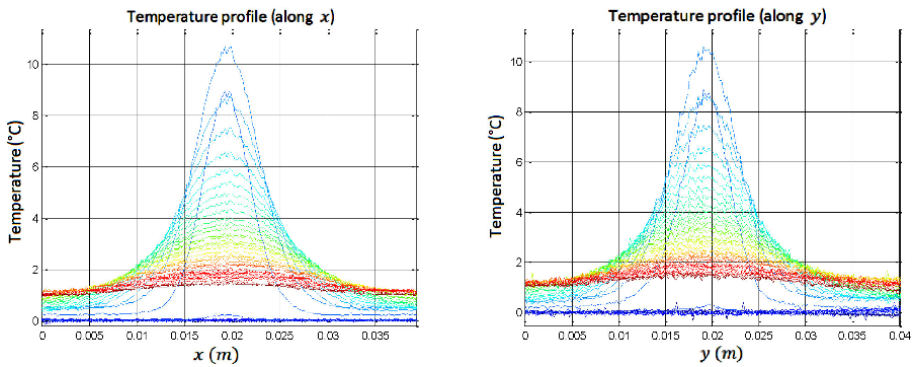


Figure 4.56. Temperature profiles on an isotropic Titanium sample at 800°C.
For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Figure 4.57 gives the results obtained on titanium between 20°C and 1000°C; the isotropy of the material is clearly observed.

Figure 4.58 shows the rear face temperature records as a function of space coordinates for different times on the carbon sample at room temperature and under vacuum. There are significant differences between the x-profiles and the y-profiles as a result of the strong anisotropy of the material.

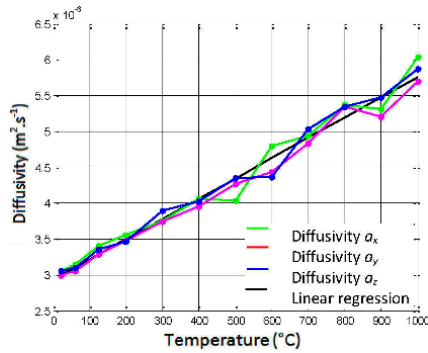


Figure 4.57. Thermal diffusivity of an isotropic Titanium sample between 20°C and 1000°C. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

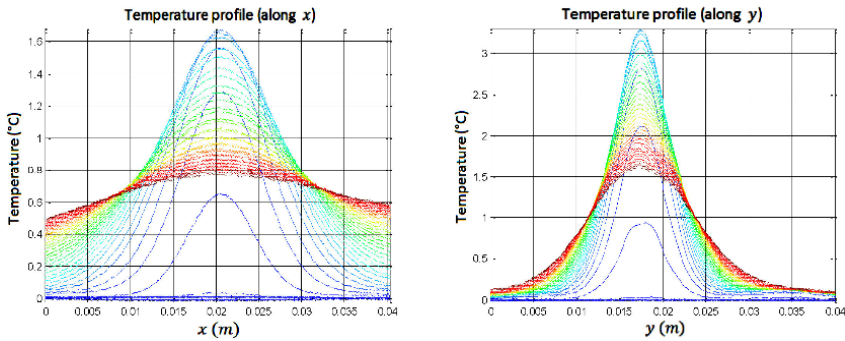


Figure 4.58. Temperature profiles on an anisotropic carbon sample at room temperature and under vacuum. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

4.6.5. Practice of the measurement

Implementing a 3D flash experiment is particularly difficult, so this technique will be reserved for specific cases when other techniques cannot be applied. For example, thin-plate materials at high temperature may be used.

4.6.5.1. Identification implementation

Before describing the difficulties associated with the method, we will explain the procedure for obtaining the curves of Figures 4.53 to 4.58.

Equation [4.40] must be compared with the measurement, and the temperature measurement is a function of x , y and t and the magnitude $\Theta(\alpha_n, \beta_m, z = e, p)$ is a function of α_n , β_m and p . As already mentioned, it is not possible to carry out the Laplace transformation on the experimental thermogram; on the other hand, a Fourier transform in cosine is applicable.

In practice, we record 10^3 to 10^4 infrared images as a function of time, each image consisting of approximately 10^5 pixels (that is 10^5 temperature measurements on the sample's unheated face).

The first step involves averaging the 10^5 temperatures on each image and thus obtaining the mean temperature of the rear face as a function of time (example in Figure 4.54). It is shown that this thermogram is strictly identical to a 1D thermogram (three parameters to be identified including α_z), we compare it to the

inverse Laplace transform of the expression [4.42] in which we considered $\alpha_n = \beta_m = 0$.

The second step is to average the temperatures on the y -axis; we obtain for each image (each time) the temperature as a function of x (see Figure 4.53, for example). On each of the temperature profiles (at each time), a digital Fourier transform is performed in cosine; for example, for the harmonic n at time t_i :

$$F_n(t_i) = \sum_{p=1}^m V_{p,i} \cos\left(\frac{n\pi}{L_x} x_p\right)$$

where $V_{p,i}$ is the average temperature in y at dimension x_p (position of pixel p) for the time t_i (image i).

L_x is the sample's dimension in x

m is the number of pixels on the x axis.

The curves of Figure 4.55 (for harmonics, $n = 5$ and $n = 6$) are obtained.

These curves are compared with the inverse Laplace transform of expression [4.42] where: $\alpha_n = \frac{n\pi}{L_x}$ and $\beta_m = 0$ for the harmonic n . There are two unknown parameters to identify including $\frac{a_x}{a_z}$.

The third step is identical to the second, by permuting x and y . It allows identifying the parameter $\frac{a_y}{a_z}$.

4.6.5.2. Analysis of difficulties

Most difficulties are related to the use of the camera as a measuring device. The calibration curves provided by the manufacturer are no longer accurate enough (see Figure 4.59), the non-uniformity of the responses of each pixel (which requires a pixel-by-pixel calibration) must be corrected, and each camera has one or more parasitic frequencies to be corrected (see Figure 4.60).

Other problems are to be taken into account, such as the estimation of the initial time or the detection of the sample's edges.

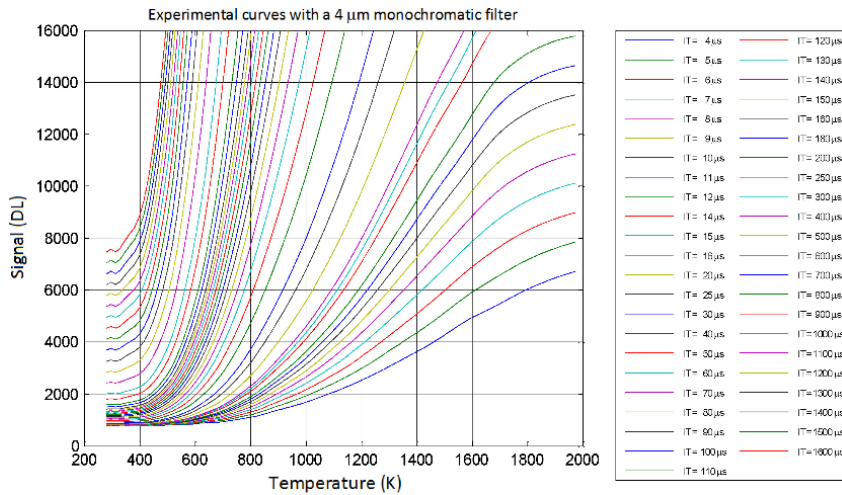


Figure 4.59. Reconstructed calibrations for the camera with 4 μm monochromatic filter. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

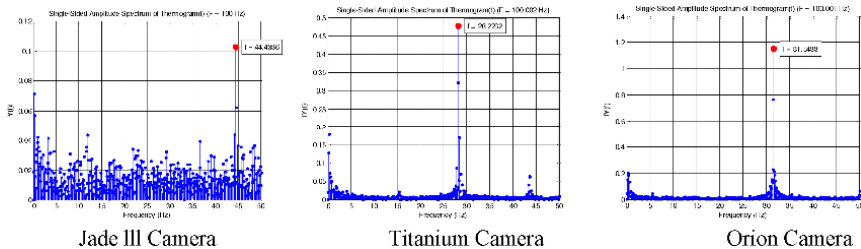


Figure 4.60. Parasitic frequencies on three camera models

Finally, a problem that is difficult to solve (hence the setup under vacuum) is the taking into account of the coupling with air. Indeed, when the thermal conductivity of the material is low, the surrounding air serves as a short circuit for transfers in the plane of the sample (this phenomenon does not exist for 1D transfer). This problem results in identified diffusivities that vary strongly with the harmonic (the biggest errors are on the first harmonics). Figure 4.61 shows experimental results obtained on a sample of Stumatite. Figure 4.62 confirms the nature of the problem by simulating a sample of PVC. Nevertheless, if we know the cause, we do not know how to correct the errors, the only solution is to work under vacuum.

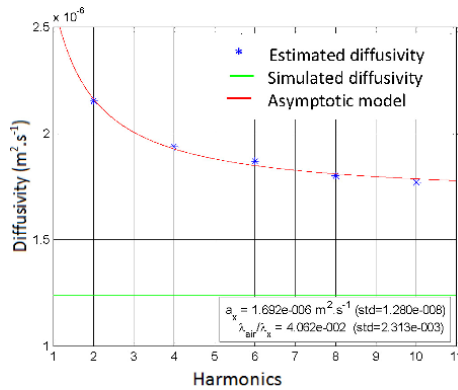


Figure 4.61. Identification of diffusivity, experiment with Stumatite. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

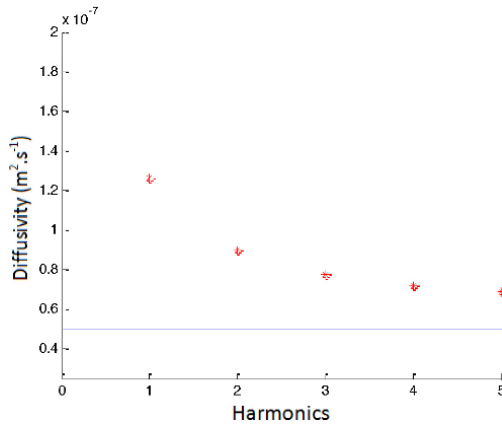


Figure 4.62. Identification of diffusivity on simulation with coupling (PVC)

4.7. Hot disc

4.7.1. Principle

The hot disc method is a thermal characterization method for materials of the “transient plane source” type studied and used by many authors [GUS 94, BOH 00, MAL 04, HE 05].

The heating element is in the form of a disk consisting of a double coil of a resistive material inserted between two thin sheets of an electrical insulator (see Figure 4.63). The double-resistive coil allows us to both uniformly heat the disk and measure the average temperature rise $\bar{T}(t)$. A gated pulse flux is applied to the disk inserted between two samples of the material to be tested and the temperature signal is recorded in transient mode. A modeling of the 2D axisymmetric heat transfer associated with a parameter estimation method makes it possible to determine the values of the thermal conductivity λ and the thermal diffusivity a , which minimize the sum of the quadratic deviations between the experimental and the theoretical curve. The application of this method requires that the semi-infinite medium hypothesis remains valid for the samples tested during measurement.

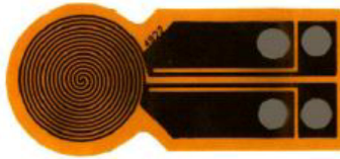


Figure 4.63. *View of a hot disc*

4.7.2. Hypotheses and model

All the presented models make the following hypotheses:

- The two samples between which the hot disc is inserted behave as semi-infinite media for the duration of the measurement.
- The continuous double coil is assimilated to a set of m coils of different diameters equally distributed between the center and the periphery.

4.7.2.1. Model “without probe”

Considering a heating disk consisting of a double coil of a resistive material spaced uniformly and neglecting the mass of the probe and the contact resistance, it has been shown that the mean temperature increase of the disk subjected to heat flow rate φ_0 can be written as [GUS 94]:

$$\bar{T}(t) = \frac{\varphi_0}{\pi^{\frac{3}{2}} r_0 \lambda} D_m(\tau) \quad [4.45]$$

with: $\tau = \sqrt{\frac{a t}{r_0^2}}$

where: a sample's thermal diffusivity
 λ sample's thermal conductivity
 r_0 probe's radius.

In addition:

$$D_m(\tau) = [m(m+1)]^{-2} \int_0^\tau d\sigma \sigma^{-2} \left[\sum_{l=1}^m \sum_{k=1}^m k \exp\left(-\frac{l^2 + k^2}{4m^2 \sigma^2}\right) I_0\left(\frac{lk}{2m^2 \sigma^2}\right) \right] \quad [4.46]$$

where: m is the number of coils of the heating element.

If the space between the coils becomes very small, this solution tends to:

$$D_\infty(\tau) = \int_0^\tau d\sigma \sigma^{-2} \int_0^1 v dv \int_0^1 u du \exp\left(-\frac{u^2 + v^2}{4\sigma^2}\right) I_0\left(\frac{uv}{2\sigma^2}\right) \quad [4.47]$$

A polynomial expression of $D_m(\tau)$ valid for $\tau > 0.3$ has been proposed in the form [JAN 07]:

$$D_m(\tau) = b_0 + b_1 \tau + b_2 \tau^2 + b_3 \tau^3 + b_4 \tau^4 + b_5 \tau^5 + b_6 \tau^6 \quad [4.48]$$

The values of the coefficients of the different polynomials are given in Table 4.3.

	$m = 5$	$m = 8$	$m = 10$	$m = 16$	$m = 25$	$m = 32$	$m = \infty$
b_1	0.9596	0.90557	0.9141	0.9386	0.9564	0.9638	0.9967
b_2	-1.0872	-0.5393	-0.4942	-0.4812	-0.4837	-0.4861	-0.4986
b_3	1.5965	0.1211	-0.03290	-0.1322	-0.1703	-0.1828	-0.2257
b_4	-1.9920	-0.08165	0.1206	0.2561	0.3123	0.3319	0.4006
b_5	1.3271	0.1215	-0.003580	-0.08452	-0.1178	-0.1295	-0.1708
b_6	-0.3437	-0.04620	-0.01610	0.002431	0.009822	0.01243	-0.02165

* The value of b_0 cannot be calculated numerically in the neighborhood of 0 (infinite limits)

Table 4.3. Values of coefficients* of polynomials $D_m(\tau)$

This “ideal” solution has been established for a probe of negligible mass and a zero contact resistance R_c between the probe and the sample. In order to take account of the contact resistance, it has been hypothesized that after a “certain time”

the effect of the contact resistance is to cause a constant temperature shift with respect to the ideal model [GUS 94]. We thus write:

$$\bar{T}(t) = \frac{\varphi_0}{\pi^2 r_0 \lambda} D_m(\tau) + R_c \varphi_0 \quad [4.49]$$

This solution is valid on the assumption that the sample can be considered as a semi-infinite medium during the time t_f of the measurement, that is, $d > \sqrt{2 a t_f}$, where d is the smallest distance between the probe and the external surface of the sample and t_f the duration of the measurement [LOG 95]. Again, it would be necessary to know the thermal diffusivity a in order to apply this criterion, so that we can rather determine this time t_f by analyzing the residuals: we will choose for t_f the time from which the residuals are no longer centered on zero.

This simplified model does not fully take into account the influence of the probe's mass and the thermal contact resistance between the probe and the sample. For the estimation of the parameters, the first points of the thermogram ($t < t_1$) are therefore not taken into account. The time t_1 is estimated empirically [BOH 00]: several successive estimates are obtained over intervals $[t, t_2]$, t_2 being constant and chosen, such that $\tau_2 = \sqrt{\frac{a t_2}{r_0^2}}$ is equal to 1.0 while t is gradually increased starting at $t = 0$ s. It should be noted that this process can only be an iterative one because we do not know at the beginning the value of a necessary for the calculation of τ_2 . It can be seen that, starting from a certain value t_1 of time t , the estimated values of the parameters vary slightly and the interval $[t_1, t_2]$ is retained for the estimation of the parameters. This method of taking into account the probe's mass and the contact resistance remains empirical, and therefore, a source of potential error.

4.7.2.2. Quadrupole model "with probe"

Consider now a probe of thermal capacitance mc and a contact resistance R_c between the probe and the sample.

We can write in quadrupole form:

$$\begin{bmatrix} \theta(p) \\ \Phi(p) \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ mcp & 1 \end{bmatrix} \begin{bmatrix} 1 & R_c \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \theta_{se}(p) \\ \Phi_{se}(p)/Z_c \end{bmatrix}$$

where: θ Laplace transform of the probe's mean temperature

Φ Laplace transform of the probe's heat flow rate

θ_{se} Laplace transform of the sample's mean surface temperature

Φ_{se} Laplace Transform of the heat flow rate at the sample's surface.

$$\text{Hence: } \theta(p) = \frac{\varphi_0}{p} \frac{Z_c + R_c}{mcp + mcpR_c + 1} \quad [4.50]$$

It has been shown [JAN 07] that Z_c can be calculated with good precision by:

$$Z_c = \frac{2}{\pi r_0 \lambda} I_m(p) \quad [4.51]$$

$$\text{with: } I_m(p) = c_0 + \frac{1+d_1\sqrt{p}}{e_0+e_1\sqrt{p}+e_2p} \quad [4.52]$$

m	5	8	10	16	25	32
d_1	6.8559	0.7223	0.6458	0.5999	0.5759	0.5631
e_0	10.3827	5.2843	5.1859	5.0855	5.0134	4.9796
e_1	29.4882	6.0943	5.7153	5.3948	5.2233	5.1490
e_2	30.2988	3.2558	2.8785	2.6027	2.4525	2.3806

Table 4.4. Value of the coefficients of the polynomials representing the functions $I_m(p)$

4.7.2.3. Sensitivity study

The reduced sensitivities of $\bar{T}(t)$ to the thermal diffusivity a , to the thermal conductivity λ , to the thermal capacitance of the probe mc and the contact resistance R_c can be calculated using equations [4.50] to [4.52] for the calculation of I_m then of $\bar{T}$, by:

$$X_i \frac{\partial \bar{T}}{\partial X_i} = 1000[\bar{T}(1,001X_i) - \bar{T}(X_i)]$$

where X_i can represent a , λ , mc or R_c .

The mean temperature is calculated by applying an inverse Laplace transformation to equation [4.52] using the Stehfest or De Hoog method.

The following experimental conditions were considered to calculate the reduced sensitivities of $\bar{T}(t)$:

– Kapton probe with a thickness of 0.2 mm and a radius $r = 6.401$ mm with 16 double coils, Kapton thermal properties: heat capacity $c = 1.09 \times 10^6$ J kg⁻¹ K⁻¹, density $\rho = 1420$ kg m⁻³ [LUA 06];

– recording of 200 measurement points with a minimum time step of 0.015 s.

Figure 4.64 shows the calculated reduced sensitivities for $R_c = 1.56^\circ\text{C W}^{-1}$, thus $SR_c = 2 \times 10^{-3}^\circ\text{C m}^2 \text{W}^{-1}$, calculated for different materials. It is found that when the diffusivity a and thermal conductivity λ increase, the sensitivities to the thermal diffusivity a and the thermal conductivity λ decrease strongly and become significantly lower than the sensitivities to the thermal contact resistance R_c and to the thermal capacitance of the probe mc .

The hot disc method cannot be used to accurately estimate high thermal diffusivities in the case where the estimation method is based on the use of a simplified model that does not take into account the transient effects of the thermal contact resistance R_c and the thermal capacitance mc of the probe.

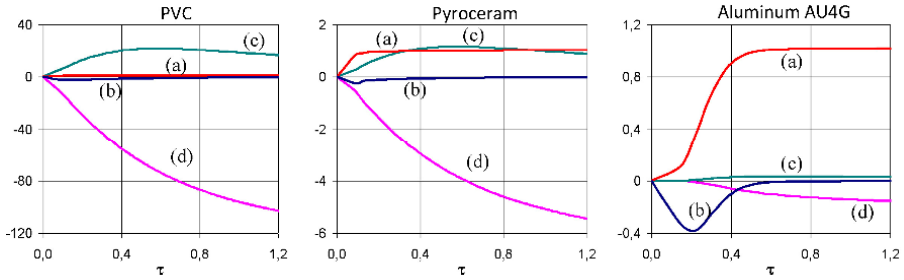


Figure 4.64. Reduced sensitivity curves for λ (d), a (c), R_c (a) and mc (b) for different materials: PVC: $\lambda = 0.21 \text{ W m}^{-1} \text{K}^{-1}$, $a = 1.3 \times 10^{-7} \text{ m}^2 \text{s}^{-1}$.
Pyroceram: $\lambda = 3.98 \text{ W m}^{-1} \text{K}^{-1}$, $a = 1.89 \times 10^{-6} \text{ m}^2 \text{s}^{-1}$.
Aluminum AU4G: $\lambda = 138.5 \text{ W m}^{-1} \text{K}^{-1}$, $a = 5.88 \times 10^{-5} \text{ m}^2 \text{s}^{-1}$

It can be seen that the sensitivities to the contact resistance R_c and to the thermal capacitance of the probe mc only become constant for $\tau > 0.3$. In this example, a simplified model can only be used for estimating the parameters for $\tau > 0.3$, which is consistent with the operating interval recommended elsewhere: $\tau \in [0.3, 1]$ [BOH 00]. The sensitivity to thermal diffusivity a passes through a maximum for $\tau = 0.5$ before decreasing, while the sensitivity to thermal conductivity λ increases continuously.

Finally, we note that the sensitivity to the thermal conductivity λ is always significantly higher than the sensitivity to the thermal diffusivity a , and the estimation error for λ will thus be lower than that for a .

4.7.3. Experimental setup

The heating element consists of a double coil of platinum inserted between two thin electrical insulators. It is inserted between two large samples (semi-infinite medium hypothesis) of the material to be characterized. Pressure can be applied to the assembly to reduce the contact resistance by taking the precaution to check that this pressure has no influence on the value of the electrical resistance. When thermal equilibrium is reached (temperature of the heating element stable and equal to that of the ambient medium), a current of constant intensity I in the heating element is imposed by means of a current generator and the resistance $R(\bar{T})$ is measured by means of a Wheatstone bridge.

The mean temperature value $\bar{T}$ is deduced from the measured value of the heating element's resistance using the following equation:

$$R(\bar{T}) = R_{\bar{T}_0} [1 + \alpha(\bar{T} - \bar{T}_0)] \quad [4.53]$$

where α is the temperature coefficient of the resistance ($\alpha = 3.851 \times 10^{-3} \text{K}^{-1}$ for Platinum).

The heat flow rate φ_0 of equation [4.49] is calculated by:

$$\varphi_0 = \frac{R(\bar{T}_0)I^2}{2} \quad [4.54]$$

By neglecting the variation in $R(\bar{T})$ over time for the flux calculation, this hypothesis does not allow using a large flux, therefore limiting the rise in temperature and consequently the signal-to-noise ratio. It is therefore detrimental in terms of the estimation's accuracy.

4.7.4. Experimental study

Several series of measurements were carried out with three hot disc sensors of respective radii (manufacturer's data):

- 3.189 mm with 8 double coils (5 measurements);
- 6.403 mm with 16 double coils (3 measurements);

– 9.79 mm with 10 double coils (3 measurements).

The tested material was titanium TA5V, whose properties were measured by several methods:

– Hot plate with 2 temperature measurements: $\lambda = 6.61 \text{ W m}^{-1} \text{ K}^{-1}$ and $a = 2.70 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$

– Flash: $a = 2.75 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$

– Differential calorimetry: $\rho c = 2.32 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$.

The parameters R_c , mc , a and λ were estimated by minimizing the sum of the quadratic deviations between the experimental curve and the theoretical curve obtained by the quadrupole model with, respectively: $m = 8$, $m = 16$ and $m = 10$ for the three probes. All the recorded values (200) of $\bar{T}(t)$ were taken into account in this sum. In the model, it has been shown that the coefficient c_0 intervening in equation [4.52] of $I_m(p)$ only causes a constant shift of the curve $\bar{T}(t)$. It has been verified that its value has no influence on the optimal solution found, so it was taken equal to zero. The results obtained with the three probes are shown in Table 4.5.

		a	λ	ρc	R_c	mc
		$\text{m}^2 \text{s}^{-1}$	$\text{W m}^{-1} \text{K}^{-1}$	$\text{J m}^{-3} \text{K}^{-1}$	K W^{-1}	J K^{-1}
$r_0 = 3.189 \text{ mm};$ $m = 8$	Mean	2.69×10^{-6}	6.52	2.43×10^6	5.11	0.0034
	Standard deviation	4.4 %	1.3 %	4.2 %	4.4 %	17.3 %
$r_0 = 6.403 \text{ mm};$ $m = 16$	Mean	2.72×10^{-6}	6.95	2.56×10^6	1.92	0.022
	Standard deviation	4.0 %	0.82 %	3.3 %	7.3 %	2.6 %
$r_0 = 9.79 \text{ mm};$ $m = 10$	Mean	2.84×10^{-6}	6.74	2.38×10^6	0.79	0.091
	Standard deviation	5.0 %	1.2 %	3.8 %	3.2 %	3.5
Flash		2.75×10^{-6}				
Hot plate		2.70×10^{-6}	6.61			
Calorimeter				2.32×10^6		

Table 4.5. Estimated parameter values with three hot disc probes and three other methods on titanium TA6V

The mean values obtained from the thermal properties are close (maximum deviation of the order of 5%) to those measured by the hot plate, flash and calorimetry methods. There is a slight difference between the results obtained with the different probes with a deviation from an average value that does not exceed 5%.

An example of an experimental curve obtained on titanium TA6V under the following conditions is given in Figure 4.65: $\varphi = 0.7 \text{ W}$, recording of 200 points with a time step of $dt = 0.027 \text{ s}$. This figure also shows the residues of estimates whose values are small, although the first points have higher values. This can be explained by the fact that the temperature variation is very rapid at short time intervals and that a slight uncertainty over time can cause a large temperature variation. With the exception of the first 5 points, the standard deviation of the differences between the theoretical curve and the experimental curve is 0.0035°C .

It should be noted that the maximum value $\bar{T}_{max}$ of the hot disc temperature would be only 3.1°C in the absence of contact resistance, whereas it was measured at 6.4°C . The gap would have been even greater with a more effusive material. In this case, if a measured value of $\bar{T}_{max}$ of 1°C is set as a criterion, this may correspond to a very low increase in temperature which does not result in contact resistance and lead to an inaccurate estimate.

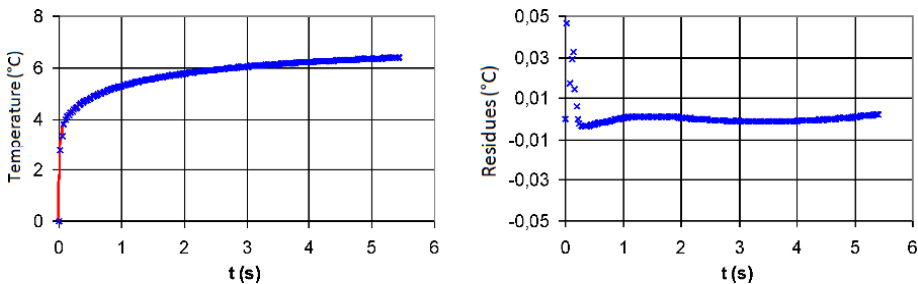


Figure 4.65. Hot disc temperature and estimation residues for a test on titanium TA6V

An improvement in the estimate could be obtained using $I_m(p)$ functions which are not calculated but identified from experiments on reference materials with precisely known thermal properties. This could make it possible to take into account the phenomena neglected in the theoretical model that led to the calculation of the function $D(\tau)$, in particular the influence of the width of the coils considered to be infinitely small in the model. Indeed, heat transfers within the Kapton in the probe's

plane are neglected. In fact, these transfers will have the effect of rendering the behavior of the probe more similar to the behavior of a disk heated uniformly over its entire surface corresponding to an infinite number of coils.

The value of the radius could be estimated in the same way.

4.7.4.1. *Evaluation of the estimations' accuracy*

The uncertainties on the estimated values of the thermal conductivity λ and the thermal diffusivity a originating from:

- measurement errors of the probe's flow rate φ_0 and radius r_0 ,
- the temperature's measurement noise $\bar{T}(t)$,
- model errors due to errors on coefficients d_i and e_i of $I_m(p)$,

were estimated [JAN 07].

It has been shown:

- that a relative error of 1% on the heat flow rate φ_0 induces a relative error of 1% on the thermal conductivity λ and a zero error on the thermal diffusivity a ;
- that a relative error of 1% on the radius r_0 induces a relative error of 1% on the thermal conductivity λ and a relative error of 2% on the thermal diffusivity a .

To conclude, the main sources of errors are the uncertainties on the probe's radius r_0 and coefficients d_i and e_i of the model. The cumulative errors even in the case of an optimal choice of experimental conditions lead to relative standard deviations of the order of 2% for the thermal conductivity and 5% for the thermal diffusivity a .

NOTE.—

Another source of error may be due to the fact that we consider the heat flow rate $\varphi_0 = \frac{R(\bar{T}_0)I^2}{2}$ to be strictly constant. Its instantaneous value $\varphi_0 = \frac{R(t)I^2}{2}$ can be taken into account by calculating the mean temperature $\bar{T}$ by:

$$\bar{T}(t) = L^{-1} \left[\frac{\varphi_0}{p} \frac{Z_c + R_c}{mcp + mcpR_c + 1} \right] = \frac{R(t)I^2}{2} \otimes L^{-1} \left[\frac{Z_c + R_c}{mcp + mcpR_c + 1} \right] \quad [4.55]$$

where $\otimes$ is the convolution product.

4.8. Hot strip

4.8.1. Principle

The hot strip method, shown schematically in Figure 4.66, involves using a rectangular electrical resistor called a hot strip on which a thermocouple of small diameter wires is fixed. The temperature rise $T_{cs}(t)$ is measured at the center of the resistor, which avoids having to take into account thermal losses by the electrical wires at one end of the resistor [JAN 04].

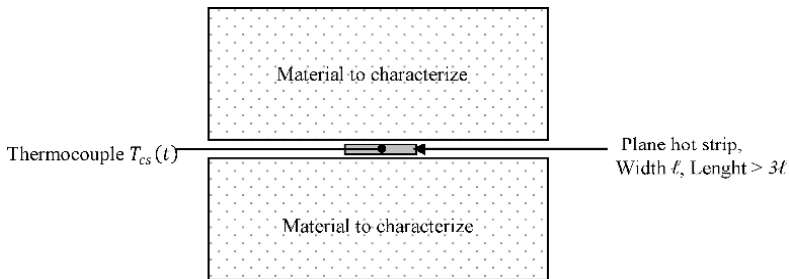


Figure 4.66. Setup schematic diagram of the hot strip method

A variant involves using a resistor whose temperature coefficient is known in order to measure its mean temperature and, in this case, the thermal losses at the ends must be considered. This second variant will be studied in the chapter concerning the 3ω method, which uses the same type of probe.

In the studied variant, the hot strip is inserted between two flat surface samples of the material to be characterized, the dimensions of the sample being such that the disturbance caused by the heat flow rate step ($\varphi = 0$ if $t < t_0$ and $\varphi = \varphi_0$ if $t > t_0$) imposed on the probe does not reach any of its faces during the duration of the measurement (semi-infinite medium hypothesis). The length/width ratio of the resistor is chosen, so that the heat transfer at the center of the strip can be considered 2D for a sufficiently long time. The thermogram corresponding to the beginning of the heating (during the time t_1 where the heat transfer in the center of the probe remains unidirectional) is used to estimate the thermal effusivity by a hot plate method. A complete modeling of bidirectional transfers in the samples associated with a parameter estimation method allows the use of the long time duration thermogram to estimate the thermal conductivity.

The advantages of this method are the low cost of the probe and an estimation method based on a relatively simple modeling of the temperature at the center of the strip:

- monodimensional during the time t_1 for the estimation of the thermal effusivity (see hot plate method);
- bidimensional between times t_1 and t_2 for the estimation of the thermal conductivity.

4.8.2. Hypotheses and model

4.8.2.1. Quadripolar model

The notations used are defined in Figure 4.67. The following assumptions are made:

- the length of the strip is large enough so that the transfer can be considered 2D at its center;
- conduction transfer is neglected according to Ox in the strip;
- the internal resistance of the strip in the direction Oy is zero (thin system).

The temperature increase $T_s(x, t)$ at a point on the strip and the rise in temperature $T(x, y, t)$ in the sample satisfy the following equation during time t_2 where the transfer at this point remains bidirectional (infinite hot strip):

$$\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} = \frac{1}{a} \frac{\partial T}{\partial t}$$

with the initial condition:

$$T(x, y, 0) = T_s(x, 0) = T_i$$

and boundary conditions:

$$\text{if } x \leq b: \quad \frac{T_s(x, t) - T(x, 0, t)}{R_c} = -\lambda \frac{\partial T(x, 0, t)}{\partial x}$$

$$\text{and:} \quad \frac{\phi_0}{2} = \frac{(mc)_s}{2S} \frac{dT_s}{dt} + \frac{T_s(x, t) - T(x, 0, t)}{R_c}$$

$$\text{if } x > b: \quad -\lambda \frac{\partial T(x, 0, t)}{\partial x} = 0$$

$$\frac{\partial T(0, y, t)}{\partial x} = 0 \quad \text{by symmetry}$$

$T(L, y, t) = 0$ hypothesis of the semi-infinite medium according to Ox

$T(x, e, t) = 0$ hypothesis of the semi-infinite medium according to Oy

where ϕ_0 is the heat flux produced in the heating element (W m^{-2}).

In the following, the temperature rise T_{cs} in the center of the strip is denoted by $T_{cs}(t) = T_s(0, t)$

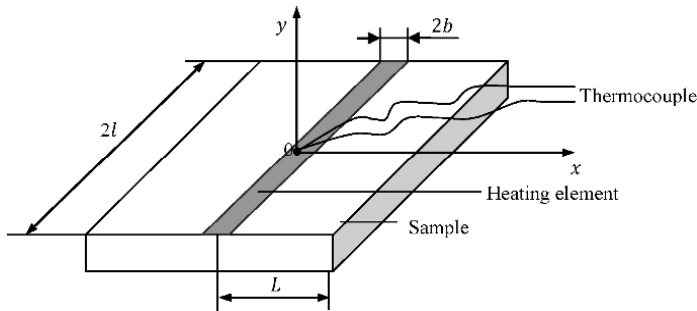


Figure 4.67. Schematic diagram of hot strip device

By successively applying a Laplace transform and then a finite Fourier transformation (see Appendix 4) in cosine between $y = 0$ and $y = L$ to the heat equation, we obtain:

$$\frac{\partial^2 \theta(x, y, p)}{\partial x^2} + \frac{\partial^2 \theta(x, y, p)}{\partial y^2} = \frac{p}{a} \theta(x, y, p)$$

$$\text{then: } \frac{d^2 \theta_c(n, y, p)}{dy^2} = \left(\frac{p}{a} + \frac{n^2 \pi^2}{L^2} \right) \theta_c(n, y, p)$$

$$\text{where: } \theta_c(n, y, p) = \int_0^L \theta(x, y, p) \cos(\alpha_n x) dx \quad \text{with: } \alpha_n = \frac{n\pi}{L}; n \geq 0$$

The general solution of this equation is written in the form:

$$\theta_c(n, y, p) = C_1 \cosh(q_n y) + C_2 \sinh(q_n y) \quad \text{with: } q_n = \sqrt{\frac{p}{a} + \frac{n^2 \pi^2}{L^2}}$$

Using the quadrupole formalism [LAD 98], we can write in matrix form at the center of the probe considered as a thin system (no thermal gradient in the thickness):

$$\begin{bmatrix} \theta_{cs}(n, 0, p) \\ \Phi_{cs}(n, 0, p) \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ (mc)_s p & 1 \end{bmatrix} \begin{bmatrix} 1 & R_c \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \theta_c(n, 0, p) \\ \lambda q_n S \theta_c(n, 0, p) \end{bmatrix}$$

where : $(mc)_s$ product mass $\times$ heat capacity of the heating element (J K^{-1})

R_c contact resistance at sample / heating interface ($\text{K m}^2 \text{W}^{-1}$)

S heating element area (m^2)

We can deduce:

$$\theta_{cs}(n, 0, p) = \frac{1 + \lambda R_c q_n}{(mc)_s p (1 + \lambda R_c q_n) + \lambda q_n} \frac{\sin(\alpha_n b)}{\alpha_n} \frac{\phi_0}{p} \quad [4.56]$$

By inverse Fourier transformation in cosine, we obtain:

$$\theta_s(0, 0, p) = \frac{1}{L} \theta_{cs}(0, 0, p) + \frac{2}{L} \sum_{n=1}^{\infty} \theta_{cs}(n, 0, p)$$

In addition, the temperature rise $T_{cs}(t)$ at the center of the strip can be deduced by Laplace's inverse transformation by the Stehfest or De Hoog method (see Appendix 5).

NOTE.—

This model is not strictly accurate. Indeed, to arrive at the solution proposed by the Laplace and Fourier transform, it was necessary to add the following limiting condition:

$$0 = \frac{(mc)_s}{2S} \frac{dT_s}{dt} + \frac{T_s(x, t) - T(x, 0, t)}{R_c} \quad \text{and} \quad \frac{T_s(x, t) - T(x, 0, t)}{R_c} = -\lambda \frac{\partial T(x, 0, t)}{\partial x} \quad \text{if } x > b$$

This amounts to considering that the whole surface of the sample is covered by a layer of surface mass heat capacity $\frac{(mc)_s}{S}$, in which a heat flux ϕ_0 is produced only between $-b$ and $+b$. The consequence is that this model will slightly underestimate the temperature in the strip.

4.8.2.2. Simplified model

In the case where the contact resistance and the mass of the heating resistor and the thermocouple are neglected, it is possible to write [CAR 59]:

$$T_{cs}(t) = \frac{2 \phi_0 \sqrt{at}}{\lambda \sqrt{\pi}} \left[\operatorname{erf} \left(\frac{b}{2 \sqrt{at}} \right) - \frac{b}{2 \sqrt{\pi at}} E_i \left(\frac{b^2}{4at} \right) \right] \quad [4.57]$$

with: $E_i(-x) = \int_x^\infty \frac{e^{-t}}{t} dt$

The term $\frac{2 \phi_0 \sqrt{at}}{\lambda \sqrt{\pi}}$ represents the evolution of the temperature of a hot plate if we neglect its mass and contact resistance.

4.8.2.3. Case of an anisotropic material

We consider the case of a material with two different thermal conductivities: $\lambda_x = \lambda_y$ and $\lambda_x \neq \lambda_z$. The determination of the three thermal properties necessary for the characterization of the material requires two different measurements and is schematically shown in Figure 4.68.

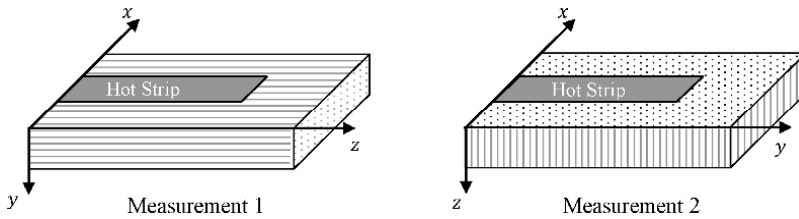


Figure 4.68. Schematic diagram of the two hot strip measurements for the characterization of an anisotropic material

In measurement 1, formula [4.56] remains applicable considering $E = E_x = \sqrt{\lambda_x \rho c}$ and $\lambda = \lambda_x$ that this first experiment will make it possible to estimate. In the case of measurement 2, formula [4.56] is applicable if we compute q_n by:

$$q_n = \sqrt{\frac{p}{a_z} + \frac{\lambda_x}{\lambda_z} \frac{n^2 \pi^2}{L^2}} \quad [\text{GOB 04}] \quad \text{where: } a_z = \frac{\lambda_z}{\rho c}$$

Having determined λ_x by measurement 1, measurement 2 makes it possible to determine E_z and λ_z . A small difference between the values of the product ρc estimated by the two experiments, $(\rho c)_1 = \frac{E_x^2}{\lambda_x}$ and $(\rho c)_2 = \frac{E_z^2}{\lambda_z}$, validates the measurements.

4.8.2.4. Sensitivity study

The complete model was used to calculate the sensitivity of the temperature rise $T_{cs}(t)$ at the center of the hot strip to the variations of the parameters E , λ , R_c and mc . The objectives of this sensitivity study carried out on three different materials are:

- to estimate the time t_1 during which the temperature $T_{cs}(t)$ evolves like that of a hot plate;
- to verify that between 0 and t_1 the sensitivities of $T_{cs}(t)$ to the parameters E , R_c and mc are decorrelated and that these parameters are therefore estimable with good accuracy over this interval;
- to check that the sensitivity to the parameter λ is sufficiently high between t_1 and t_2 to allow an accurate estimation thereof.

Calculations were made for a 1.2 cm wide hot strip for the following materials:

- Extruded polystyrene: $\lambda = 0.037 \text{ W m}^{-1} \text{ K}^{-1}$, $E = 42.4 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$
- PVC: $\lambda = 0.21 \text{ W m}^{-1} \text{ K}^{-1}$, $E = 510 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$
- Concrete: $\lambda = 1.4 \text{ W m}^{-1} \text{ K}^{-1}$, $E = 1683 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$.

The following values were used for this study: $mc = 0.2 \text{ J K}^{-1}$; $S R_c = 0.002 \text{ m}^2 \text{ K W}^{-1}$. The results are shown in Figure 4.69.

For the extruded polystyrene, a hot plate behavior is observed for about $t_1 = 30 \text{ s}$; however, the sensitivity to mc is strong during this period and almost proportional to the sensitivity to E . It will therefore be difficult to estimate E with precision between 0 and t_1 in this case. A broader strip should be used to evaluate the measurements over a time interval $[0, t_1]$ in which the sensitivities of $T_{cs}(t)$ to the parameters mc and E are sufficiently decorrelated. A recording time greater than 180 s will then be necessary to obtain a variation of $T_{cs}(t)$ sufficiently sensitive to λ to allow a good estimate.

For PVC, a hot plate behavior is observed for approximately $t_1 = 25 \text{ s}$ with a high sensitivity at E and not correlated with the sensitivity to the other parameters. The sensitivity to λ is also sufficient for an accurate estimation after determination of the other parameters.

For concrete, the hot plate type behavior for a half width band $b = 0.6 \text{ cm}$ is reduced to a time $t_1 = 6 \text{ s}$ during which the sensitivities to the parameters E , R_c and mc are weakly decorrelated, thus their estimation will be difficult and inaccurate.

The increase in the width at $b = 1$ cm makes it possible to obtain a time t_1 of the order of 15 s and a decorrelation of the parameters making the estimation of E possible between 0 and t_1 while ensuring a good sensitivity at λ between t_1 and 180 s.

In conclusion to this sensitivity study, it appears that the proposed method is applicable to the characterization of materials with low thermal diffusivity such as PVC or concrete with measurement times less than 180 s. Low effusivity materials such as polystyrene can also be thermally characterized, but using a strip of sufficient width to observe a hot plate type behavior with good decorrelation of the parameters E , R_c and mc . In this case, the measurement time must be greater than 180 s to allow an accurate estimation of the parameter λ .

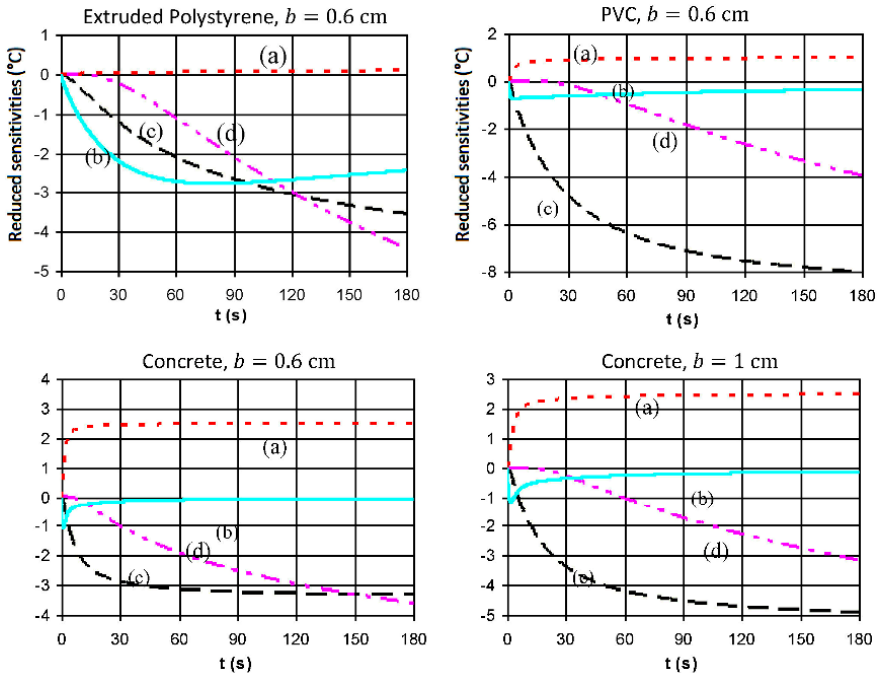


Figure 4.69. Sensitivity of $T_{cs}(t)$ to parameters E , λ , R_c and mc for different materials: a) $R_c \frac{\partial T_{cs}}{\partial R_c}$; b) $mc \frac{\partial T_{cs}}{\partial mc}$; c) $E \frac{\partial T_{cs}}{\partial E}$; d) $\lambda \frac{\partial T_{cs}}{\partial \lambda}$

4.8.3. Experimental setup

This method can be implemented in an extremely simple and inexpensive way by using a rectangular planar heating resistor of length equal to at least 3 times the

width. A thermocouple made of fine wires (diameter <0.1 mm) is fixed at the center of this resistor. A width of 1.2 cm is sufficient for materials with low thermal diffusivity (plastics, wood), and a width of the order of 2.5 cm is necessary for materials that are more diffusive (stone, sandstone, etc.). The smallest dimension of the sample must be greater than 1.5 times the total width of the tape. The method leads to probe and sample dimensions that are too high for the most diffusive materials (metals) for which it is poorly adapted.

A calibration of the probe is necessary from a material of known thermal properties on which several tests are carried out. The useful surface S of the probe is that which makes it possible to recover the known value of the effusivity. The half-width b of the probe is then determined: it is that which makes it possible to recover the known value of the conductivity.

The implementation of the method also requires a stabilized power supply and a device for recording the voltage delivered by the thermocouple or directly by the temperature. Recording 180 seconds after the start of heating in most cases allows for good accuracy. A time step of 0.1 s for recording the temperature gives satisfactory results.

4.8.4. Practice of the measurement

4.8.4.1. Methods for estimating parameters

4.8.4.1.1. Thermal effusivity

During the time t_1 where the transfer of heat to the center of the strip remains unidirectional, the temperature at the center of the strip evolves like that of a hot plate, that is to say in Laplace space:

$$\theta_{cs}(p) = \frac{\phi_0}{2p} \frac{1+R_c E \sqrt{p}}{\frac{(mc)_s}{2S} p + \left(1+R_c \frac{(mc)_s}{2S} p\right) E \sqrt{p}} \quad [4.58]$$

In addition, by inversion at “long times”:

$$\text{Hence } T_{cs}(t) - T_s(0) = \frac{\phi_0}{E\sqrt{\pi}} \sqrt{t} + \frac{\phi_0}{2} \left(R_c - \frac{(mc)_s}{2E^2} \right) \quad [4.59]$$

The simulated representation using formulas [4.56] and [4.58] shows the evolution of the temperature at the center of a hot strip with characteristics: $R = 61.9 \Omega$, $L = 5.3$ cm, $b = 0.6$ cm used on PVC at a voltage $U = 6$ V considering a contact resistance $R_c = 0.001 \text{ K W}^{-1}$ and a zero mass probe. It can be seen that the two curves coincide until $t_1 = 25$ s.

A first approximated value of the thermal effusivity E is estimated from the slope α of the experimental line $T_{cs}(t) = f(\sqrt{t})$ determined by linear regression between times t_0 (chosen such that the sensitivity of $T_{cs}(t)$ to mc stabilized, or about 5 s for PVC in Figure 4.69) and t_1 . The result obtained is then used as the initial value for estimation by the Newton method of the parameters E , R_c and mc , which minimize the sum of the quadratic deviations between the experimental curve $T_{cs}(t)$ and the curve representing the values calculated by inversion of the formula [4.58] by the Stehfest method.

It should also be noted that a slight offset of the thermocouple from the center of the probe will not affect the precision of the measurement but will simply modify the time during which the hypothesis of unidirectional transfer can be assumed.

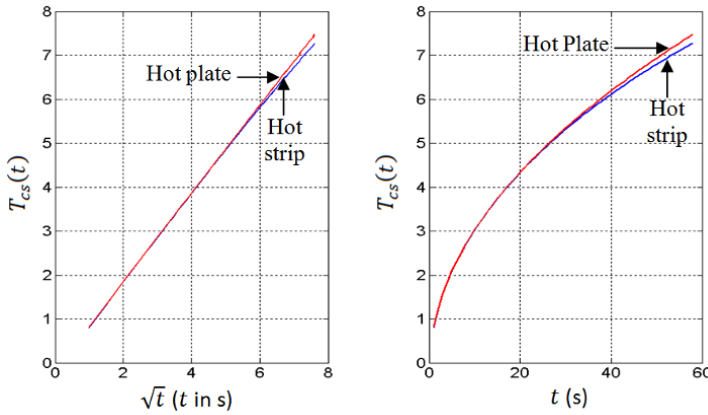


Figure 4.70. Comparison of simulated hot strip and hot plate thermograms

4.8.4.1.2. Thermal conductivity

The temperature of a hot strip of long length tends asymptotically towards that of the hot wire [LAD 98]. By neglecting the effect of the inertia of the probe, the temperature of the hot wire is written in Laplace space:

$$\theta_{cs}(p) = \frac{\phi}{p} \left[R_c + \frac{K_0(qr_0)}{2\pi\lambda L q r_0 K_1(qr_0)} \right] \quad [4.60]$$

Moreover, by inversion at long times:

$$T_{cs}(t) = \frac{\phi_0}{4\pi\lambda L} \ln(t) + \phi_0 \left[R_c - \frac{\gamma}{4\pi\lambda L} - \frac{\ln\left(\frac{r_0}{\sqrt{a}}\right)}{2\pi\lambda L} \right] \quad [4.61]$$

The time after which the hot strip can be assimilated to a hot wire is however too long for this asymptotic behavior to be used in practice for the estimation of the thermal conductivity. The graph in Figure 4.71 represents the slope of the curve $T_{cs}(t) = f[\ln(t)]$ calculated for a hot wire of radius 0.25 mm and for a hot strip of dimensions 5.3 cm $\times$ 1.2 cm and of thermal capacitance $mc = 0.2 \text{ J K}^{-1}$ subjected to the same linear heat flux. The slope for the hot wire was determined by calculating the temperature $T_{cs}(t)$ by inverting the relation [4.60] by the Stehfest method and then by derivation with respect to $\ln(t)$. The slope for the hot strip was determined by calculating the temperature $T_{cs}(t)$ by equation [4.56] of the complete model.

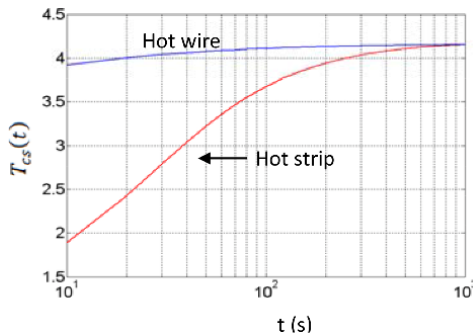


Figure 4.71. Calculated slope of the curves $T_{cs}(t) = f[\ln(t)]$ as a function of time for a hot strip with a width of 1.2 cm and for a hot wire with a diameter of 0.5 mm on a PVC sample

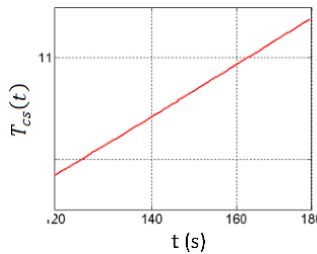


Figure 4.72. Calculated curve $T_{cs}(t)$ at the center of a hot strip with a width of 1.2 cm, shown in semi-logarithmic scale between 120 and 180 s for PVC

A first approximated value of the thermal conductivity λ is estimated from the slope $\beta \approx \frac{\phi}{4\pi\lambda L}$ of the experimental line $T_{cs}(t) = f[\ln(t)]$ determined by linear regression between 120 and 180 s (see Figure 4.72). The result obtained is then used as the initial value for an estimate by the Newton method of the value of λ , which

minimizes the sum of the quadratic deviations between the experimental curve $T_{cs}(t)$ and the curve representing the values calculated by the formula [4.56]. In this formula, the values of E , R_c and mc estimated between 0 and t_1 are considered as data.

4.8.4.2. Hot strip dimensions

The width $2b$ of the resistor must be such that the time t_1 during which the transfer is unidirectional at the center of the heating resistor is sufficient to allow a satisfactory estimate of the effusivity E .

This time (by default) can be estimated in the case of a zero mass + resistance probe and a zero contact resistance using equation [4.56]. The time at the end of which the temperature at the center of the hot strip has not varied by more than 1% with respect to that of the hot plate subjected to the same flux density is given by the equation:

$$\operatorname{erf}(X) - \frac{x}{\sqrt{\pi}} E_i(-X^2) = 0.99 \quad [4.62]$$

with: $X = \frac{b}{2\sqrt{at}}$. The solution of equation [4.62] is: $X = 1,31$.

From this, the value of the width $2b$ of the hot strip is deduced such that the temperature at the center of the hot strip has not varied by more than 1% with respect to that of the hot plate after a time t_1 by:

$$2b = 4X\sqrt{at_1} \quad [4.63]$$

It will be noted that this width depends only on the thermal diffusivity of the material. This formula also makes it possible to estimate the upper bound t_1 of the estimation interval of effusivity E if we have a first estimate of λ and E .

The length L of the resistor must be chosen so that the heat transfer at the center of the hot strip remains bidirectional during the time t_2 of the measurement. This length can be estimated in excess by the formula [4.63] in which t_1 is replaced by t_2 by considering that it is the dimension necessary for the transfer to be assimilated to that of a hot plate during t_2 and gets:

$$L = 4X\sqrt{at_2} \quad [4.64]$$

It has also been verified that an increase in the length at fixed width does not modify the sensitivity of $T_{cs}(t)$ with respect to effusivity and conductivity. We shall therefore choose the smallest possible length, namely that given by formula [4.64].

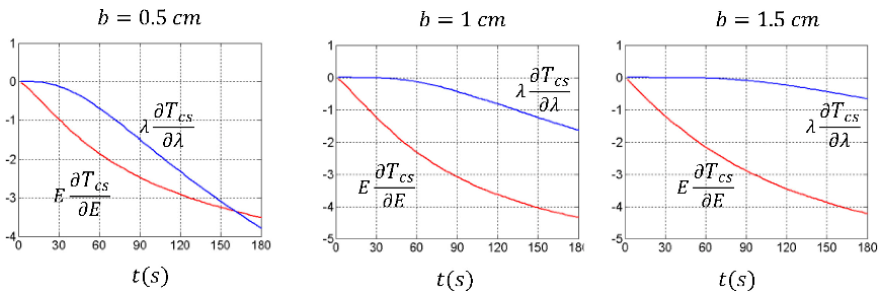


Figure 4.73. Sensitivity of $T_{cs}(t)$ to thermal effusivity E and thermal conductivity λ for several values of b for extruded polystyrene

On the other hand, an increase in width to fixed length increases the sensitivity to effusivity E and decreases the sensitivity to conductivity λ as shown using an example, the curves of Figure 4.73 representing, for extruded polystyrene, the sensitivity of $T_{cs}(t)$ of a probe of length 5 cm with parameters E and λ for 3 different widths: 1 cm, 2 cm and 3 cm. As effusivity is estimated during time t_1 when the sensitivity of $T_{cs}(t)$ to conductivity is negligible, then it is preferable to maximize the sensitivity to conductivity and thus to choose the smallest acceptable width given by formula [4.63].

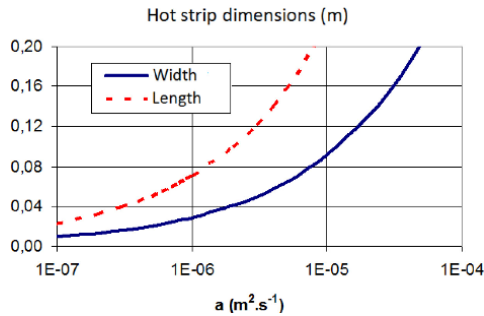


Figure 4.74. Dimensions of the hot strip as a function of the effusivity of the tested material. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Figure 4.74 shows the minimum dimensions (calculated by the relations [4.63] and [4.64]) of the hot strip presenting at its center a hot plate type of behavior for 30 s and a 2D transfer for 180 s. In order to remain in limited sample sizes (dimensions < 0.1 m), the effusivity of the material to be tested should not exceed $2 \times 10^{-6} \text{ m}^2 \text{ s}^{-1}$, which sets the limits of this method.

4.8.4.3. Measurement results

An experimental study was carried out using a MINCO electric heating element HK913P with electrical resistance $61.7 \, \Omega$ and dimensions $5.3 \, \text{cm} \times 1.2 \, \text{cm}$. A K type thermocouple with a diameter of $0.05 \, \text{mm}$ was attached to one side of the resistor. A flux step was applied to the resistor placed between two samples of the material to be characterized and the temperature of the thermocouple was recorded for $180 \, \text{s}$ with a time step of $0.1 \, \text{s}$ by an ALMEMO 2290-4 apparatus with a resolution of 0.1°C . Three materials were tested: PVC, PCTFE and polyethylene. The surface area of the samples was $4 \, \text{cm} \times 6 \, \text{cm}$ and their thickness $1.5 \, \text{cm}$. They have been selected for their low thermal effusivity ensuring a hot plate type of behavior of the temperature at the center during a time t_1 that is sufficiently long given the width of the heating element used. The density values of the three materials were measured at the mercury volumenometer [TAL 04]. Their specific heat was measured in a SETARAM TG-DSC111 differential calorimeter. Five measurements were taken on each sample. An example of an experimental curve $T(t) = f(\sqrt{t})$ between 0 and $60 \, \text{s}$ and $T_{cs}(t) = f[\ln(t)]$ between $120 \, \text{s}$ and $180 \, \text{s}$ is shown in Figure 4.75. These curves were obtained with the hot strip described above subjected to an electrical voltage of $5.6 \, \text{V}$ and placed between two samples of PVC.

It can be noted that, between 5 and $25 \, \text{s}$, the curve $T_{cs}(t) = f(\sqrt{t})$ is almost a straight line. This makes it possible to estimate a first value of the thermal effusivity: $E = 525.0 \, \text{J m}^{-2} \text{K}^{-1} \text{s}^{-1/2}$.

An estimate of the parameters E , R_c and mc by Newton's method (integrated with the solver of a very common spreadsheet) applied to the complete model leads to the following result: $E = 547.7 \, \text{J m}^{-2} \text{K}^{-1} \text{s}^{-1/2}$; $S R_c = 0.0025 \, \text{m}^2 \text{K W}^{-1}$ and $mc = 0.31 \, \text{J K}^{-1}$.

The complete 2D model was then used by considering these values of E , R_c and mc as data. An estimate by minimizing the sum of the quadratic deviations between the experimental and theoretical curves between 0 and $180 \, \text{s}$ led to the following value: $\lambda = 0.215 \, \text{W m}^{-1} \text{K}^{-1}$. The experimental curve as well as the curves obtained by the complete 1D models of the hot plate and 2D model of the hot strip are represented in Figure 4.75. A very good concordance is observed, and the summary of the experimental results obtained by the various methods is presented in Table 4.6.

The values of the thermal capacitance mc estimated by the hot strip method on the one hand and by the joint measurement of the density with a mercury volumenometer and the specific heat with a differential calorimeter on the other hand are in good agreement (difference $< 6.2\%$) for the materials tested. The standard deviations of the estimated values of thermal effusivity E and thermal conductivity λ are also quite low ($< 3.6\%$).

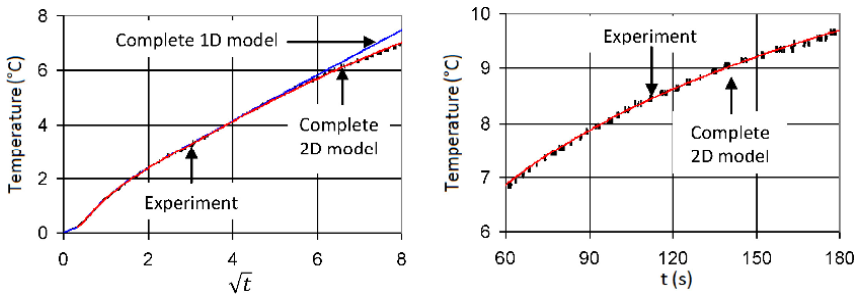


Figure 4.75. *Experimental and theoretical curves obtained with a 1.2 cm wide hot strip on polyethylene*

The method has been experimentally validated for materials with low thermal diffusivity: $\alpha < 2 \times 10^{-7} \text{ m}^2\text{s}^{-1}$, typical values for plastics and wood and even for bricks [EDW 69]. The sensitivity study and the calculation of the dimensions of the hot strip allowing an accurate estimation of the thermal properties have shown the limits of this method, not adapted to the materials of higher thermal diffusivity ($> 2 \times 10^{-6} \text{ m}^2\text{s}^{-1}$) that would require hot strip dimensions and too large samples.

		PVC	PCTFE	Polyethylene
	Time interval:			
	For the estimation of E	0 – 30 s	0 – 40 s	0 – 25 s
	For the estimation of λ	30 – 180 s	40 – 180 s	25 – 180 s
Hot strip	$E \text{ (J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}\text{)}$	530.4	692.8	888.6
	Standard deviation (%)	(1.7)	(1.4)	(3.4)
	$\lambda \text{ (W m}^{-1} \text{ K}^{-1}\text{)}$	0.214	0.228	0.456
	Standard deviation (%)	[4.2]	(3.0)	(3.6)
	$\rho c = \frac{E^2}{\lambda} \text{ (kJ m}^{-3} \text{ K}^{-1}\text{)}$	1315	2105	1732
	$R_c \text{ (W K}^{-1}\text{)}$	3.1	2.58	3.18
	Standard deviation (%)	(23.1)	(13.0)	(29.3)
	$mc \text{ (J K}^{-1}\text{)}$	0.249	0.172	0.208
	Standard deviation (%)	(23.0)	(13.5)	(21.0)
Volumenometer	$\rho_0 \text{ (kg.m}^{-3}\text{)}$	1459	1782	929
DSC	$c_0 \text{ (J kg}^{-1} \text{ K}^{-1}\text{)}$	930	1112	1830
	$\rho_0 c_0 \text{ (kJ m}^{-3} \text{ K}^{-1}\text{)}$	1357	1981	1700
Deviations	$100 \frac{\rho_0 c_0 - \rho c}{\rho c} \text{ (%)}$	3.1	6.2	1.9

Table 4.6. *Experimental results obtained with hot strip*

4.8.4.4. Conclusion

Without being a reference method, this method based on a simple device to be implemented makes it possible to rapidly obtain the thermal properties of a solid with the following reservations:

- The hypothesis of a negligible heat transfer in the plane of the probe is not necessarily satisfied for the insulating materials: the thermal conductivity will be overestimated in this case.

- For this hypothesis to be verified, it is necessary to avoid any contribution from the trace of the resistive track (metallic and therefore of high thermal conductivity) to an increase in the conductivity of the probe (see Figure 4.76) in the direction Ox .

- The value b of the half-width of the strip can hardly be obtained from a direct measurement (see Figure 4.77), so it is preferable to estimate it from the measurement of the thermal conductivity of a reference material.

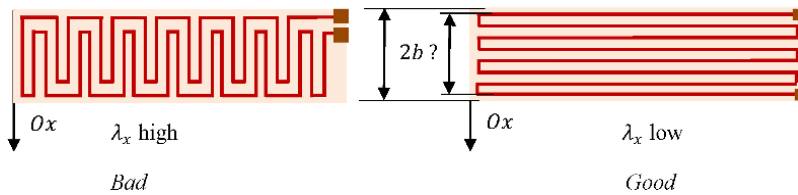


Figure 4.76. Schematic of the optimal configuration of the resistive track

4.9. 3ω Method

4.9.1. Principle

The 3ω method is a method of measuring the thermal conductivity under periodic conditions designed for the characterization of thin layers (<0.1 mm). Its schematic diagram is given in Figure 4.77. A layer of thickness e_0 and width $2b$ (hereinafter referred to as the strip) of a known electrical resistivity material is deposited on the thin layer of thickness e_1 to be characterized. This thin layer of thickness e_1 is itself supported by a substrate of great thickness. A sinusoidal current of pulsation ω is passed in the strip, which causes the production of a heat flow by Joule's effect. The resistive strip also acts as a thermometer: the variation of the voltage $U(t)$ on its central part is measured (see Figure 4.79), which makes it possible to deduce its mean temperature rise knowing the initial value R_{el0} of its resistance and its coefficient of temperature α_R .

The modeling of the heat transfer in the strip + thin film system makes it possible to establish a theoretical expression $U_{mod}(t)$ as a function of the parameters of the system particularly the thermal conductivity λ in the thin layer. This last parameter is estimated by minimizing the sum of the quadratic deviations between the model curve and an experimental curve.



Figure 4.77. *Diagram of a typical device for the 3ω method*

4.9.2. Hypotheses and model

A simplified model has been developed by Cahill with the following simplifying assumptions [CAH 90]:

- the thin layer is very large with respect to the strip, in particular its thickness is assumed to be infinite;
- the thermal capacity and thermal resistance of the hot strip are zero;
- the thermal contact resistance between the hot strip and the thin layer is negligible;
- heat transfer on the upper side of the strip is negligible;
- the power delivered by the strip is constant.

The increase in temperature in the strip is proportional to the dissipated heat power, which comprises a continuous part and a sinusoidal part:

$$\Delta T(t) = \Delta T_{DC} + \Delta T_{AC} \cos(2\omega t + \psi)$$

where ψ is the phase difference between the current and the temperature. The resistance of the strip varies linearly with the temperature, and hence, we can write:

$$R_{el}(t) = R_{el0}(1 + \alpha_R \Delta T) = R_{el0} [1 + \alpha_R (\Delta T_{DC} + \Delta T_{AC} \cos(2\omega t + \psi))]$$

where: α_R strip temperature coefficient (K^{-1}).

R_{el0} electrical resistance of the strip at the initial temperature T_0 .

The voltage drop in the strip can be expressed by:

$$U(t) = R_{el0}I_0(1 + \alpha_R\Delta T_{DC})\cos(\omega t) + \frac{1}{2}R_{el0}I_0\alpha_R\Delta T_{AC}\cos(\omega t + \psi) + \frac{1}{2}R_{el0}I_0\alpha_R\Delta T_{AC}\cos(3\omega t + \psi)$$

The third harmonic of the tension has an expression as:

$$U_{3\omega}(t) = \frac{1}{2}R_{el0}I_0\alpha_R\Delta T_{AC}\cos(3\omega t + \psi) \quad [4.65]$$

It can be extracted from the experimental signal by a synchronous amplifier and can also be calculated by modeling the amplitude of the temperature rise ΔT_{AC} , which is a function of the thermal properties of the thin layer.

Cahill has established the expression of the elevation of the average temperature of the strip:

$$\Delta T_{AC} = \frac{P_0}{\pi\lambda bL_z} \int_0^\infty \frac{\sin^2(ub)}{ub} \frac{1}{\sqrt{u^2 + \frac{2i\omega}{a}}} du \quad [4.66]$$

where: P_0 power produced in the strip by Joule's effect

λ thermal conductivity of the thin layer

a thermal diffusivity of the thin layer

L_z strip length.

The depth of penetration of heat into the layer can be estimated by:

$$e_p = \sqrt{\frac{a}{2\omega}} \quad [4.67]$$

This value must remain less than the thickness of the thin layer, so that the hypothesis of the semi-infinite medium is verified.

If this value is much greater than half the width of the strip, equation [4.66] can be written in an approximate way:

$$\Delta T_{AC} = \frac{P_0}{\pi \lambda L_z} \left[\ln \left(\sqrt{\frac{2\omega}{a}} b \right) + \eta - i \frac{\pi}{4} \right] \quad [4.68]$$

where η is a constant. It was later shown that $\eta = 0.923$ [DUQ 04]. These authors have proposed an exact solution of [4.66] in the form:

$$\Delta T_{AC} = \frac{P_0}{4\pi \lambda L_z} \text{Meijer } G \left(\left[\left[0, \frac{1}{2} \right], [] \right], \left[[0, 0], \left[-\frac{1}{2}, -1 \right] \right], \frac{i\omega b^2}{a} \right) \quad [4.69]$$

In the domain of validity of equation [4.68], the plot of the real part of ΔT_{AC} as a function of $\ln(\omega)$ is therefore a straight line of slope $\frac{P_0}{\pi \lambda L_z}$ whose determination makes it possible to estimate the thermal conductivity λ of the thin layer, knowing the linear power $\frac{P_0}{L_z}$ dissipated in the wire by Joule's effect.

Many other analytical or semi-analytical models with fewer restrictive hypotheses than Cahill have been developed: [GUR 08, ZON 11, DIN 14]. The last one makes no hypothesis but reaches a level of complexity that can make us prefer a numerical resolution, especially since the problem is only 2D.

4.9.3. Experimental setup

The thin layer to be characterized is deposited on a substrate which may be silicon. In order to limit the neglected convective heat transfer in the previously established equations, the device is placed under vacuum. A synchronous amplifier can be used to measure the amplitude and phase shift values of the third harmonic $U_{3\omega}$ of the voltage across the strip (see Figure 4.78). The electrical circuit allows the fundamental component (1ω) to be eliminated by adjusting the variable resistor R_{elV} .

The strip is made of one of the following materials: aluminum or platinum, more rarely gold or copper. The characteristic dimensions found in the literature range from 10 μm to 50 μm for the width and from 100 to 500 nm for the thickness.

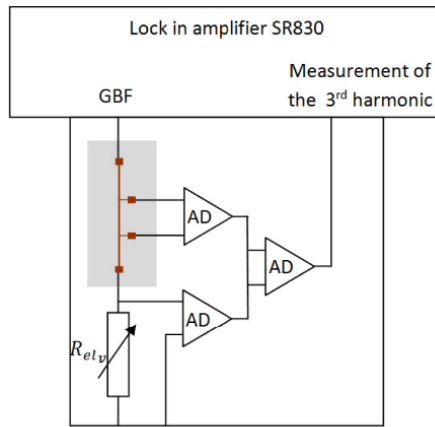


Figure 4.78. Example of an electrical circuit diagram for the 3ω method (GBF: Low-Frequency Generator, AD: Differential Amplifier)

4.9.4. Practice of the measurement

Several measurements of the amplitude of the third harmonic $U_{3\omega}$ of the voltage at the terminals of the strip are made for different values of the pulse 3ω . We then plot the graph of the real part of $U_{3\omega} = f[\ln(\omega)]$, the linear part whose slope β is estimated on this graph. The thermal conductivity λ of the thin layer is then estimated using relations [4.65] and [4.68] by:

$$\lambda = \frac{P_0 R_{el0} I_0 \alpha_R}{2\pi \beta L_z} \quad [4.70]$$

where $\frac{P_0}{L_z}$ is the linear power dissipated by Joule's effect in the strip.

Figure 4.79 shows (in semi-logarithmic scale) the amplitude of the 3ω component of temperature, calculated by different methods, as a function of frequency f [DIN 14]. A linear zone limited to the intermediate frequencies is clearly visible:

- At low frequencies, the limit f_{min} is imposed by the thickness of the sample: when the frequency decreases, the depth of penetration increases and, below a certain value, the semi-infinite medium hypothesis is no longer verified. This effect will occur at higher frequencies as the layer is thin.

- At high frequencies, these are the effects of the width and mass of the strip, not taken into account in equation [4.68], which explain the nonlinearity of the curve

beyond f_{max} . This effect will occur at lower frequencies for a wider or thicker strip, thus reducing the linearity area of the curve.

It can thus be seen that the value of f_{min} is high for a layer of very small thickness. In order for the measurement of the thermal conductivity of such a layer to be possible f_{max} must be even higher, which makes it necessary to use a strip of very small width and of very small thickness. There are technical limitations: the strips used in the literature have a minimum width $2b = 10 \mu\text{m}$ and a minimum thickness $e_s = 50 \text{ nm}$.

Moreover, when the frequency becomes greater than 10^4 Hz , the signal-to-noise ratio becomes low and the measurement is very uncertain. This is another limit of the method because the lower the thickness e of the layer to be characterized, the more high-frequency measurements would have to be made.

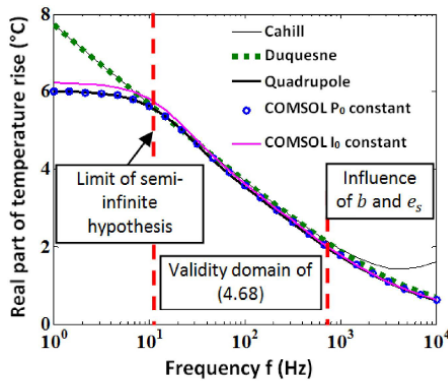


Figure 4.79. Real part of the 3ω component of the temperature calculated by different methods. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

The following results will be taken from these remarks:

- It seems difficult to measure the thermal conductivity of layers of thickness less than $1 \mu\text{m}$ if $\lambda \approx 1 \text{ W m}^{-1} \text{ K}^{-1}$; we can expect it to fall to 100 nm if $\lambda \approx 100 \text{ W m}^{-1} \text{ K}^{-1}$.

- Equation [4.69] allows a more precise estimate than equation [4.68] for the high frequencies.

These remarks can be modulated according to the thickness and the width of the strip, its thermal properties, thickness and thermal properties of the thin layer. It is therefore highly recommended to carry out a 2D numerical simulation of the measuring device for a nominal value λ_0 of the thermal conductivity of the thin layer (equal to that obtained by a first estimate from an experimental curve), a curve $\bar{T} = f[\ln(\omega)]$. This curve can then be treated as an experimental result (called a “numerical experiment”) to estimate a value λ_{est} is of the thermal conductivity of the layer using equation [4.68] or equation [4.69]. The difference between the values λ_{est} and λ_0 will make it possible to determine on which frequency interval these two equations make it possible to obtain a sufficiently precise estimate. Finally, a new estimate will be made from the experimental curve by choosing the frequency interval thus determined.

This choice is important as the example given in Figure 4.80 shows. It can be seen that the estimation error can exceed 20% even in areas where the curve appears to be comparable to a straight line if the right frequency range is not chosen.

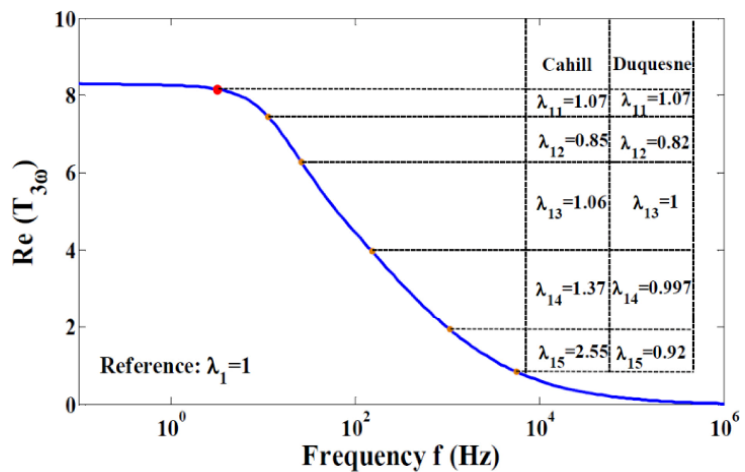


Figure 4.80. Thermal conductivity λ ($W\ m^{-1}\ K^{-1}$) estimated by the Cahill and Duquesne models over different frequency intervals

4.10. Calorimetry

The purpose of calorimetry is to measure the specific heat of materials (solid or liquid). It is the oldest of the thermal characterization methods of which it has become a discipline in its own right [MAX 91]. It is now perfectly controlled and the commercial devices currently on the market are able to obtain very high-quality measurements with an accuracy of the order of 2 to 5% for temperatures up to

700°C. It is therefore useless to want to mount its own device for this range of applications. The measurement of the heat capacity above 1000°C is much less well controlled and therefore the uncertainties on the measured values are much higher.

We are not going to present a course on calorimetry in the following, but simply state the basic principles and precautions to be taken to achieve good measurements. For more information, see the following references: [CLA 05, LEG 06, LEG 11].

A description of the operating principle of the two most commonly used types of calorimeters is given below.

4.10.1. Differential calorimeter

A differential calorimeter consists of two cells: a reference cell and a measurement cell, assumed to be at the same temperature as a calorimetric block (see Figure 4.81). The cells are connected to the calorimetric block by fluxmeters of low thermal resistance. In fact, these fluxmeters are mounted in opposition to measure the difference $\Delta\varphi$ between the heat flow rate φ_m exchanged between the block and the measurement cell and the heat flow rate φ_r exchanged between the block and the reference cell. The signal delivered by the fluxmeters is a voltage $U = k\Delta\varphi$, a calibration is necessary to establish the value of the constant k .

The cells are the most “identical” possible (same mass), so that the value $\Delta\varphi_b$ is as low as possible in the absence of a sample in the measuring cell. An inert gas sweeps the cells to avoid condensation and to obtain reproducible convective exchanges.

In order to measure the heat capacity of a material at temperature T , a temperature plateau at $T_1 = T - \frac{\Delta T}{2}$ is programmed and then a temperature ramp from $T_1 = T - \frac{\Delta T}{2}$ to $T_2 = T + \frac{\Delta T}{2}$ and, finally, a new stabilization plateau at $T_2 = T + \frac{\Delta T}{2}$. The duration of the bearings must be sufficient to ensure a stabilization of the value of $\Delta\varphi$.

The value of $\Delta\varphi$ is recorded during the execution of these three sequences for three different scenarios:

- 1) an experiment (a “blank”) with the empty measuring cell: measurement of $\Delta\varphi_b$;
- 2) an experiment with the measuring cell containing a mass m_r of a material of known specific heat c_r : measurement of $\Delta\varphi_r$;

3) an experiment with the measuring cell containing a mass m_e of a material of specific heat c_e to be measured: measurement of $\Delta\phi_e$.

Recordings of the type shown in Figure 4.82 are obtained.

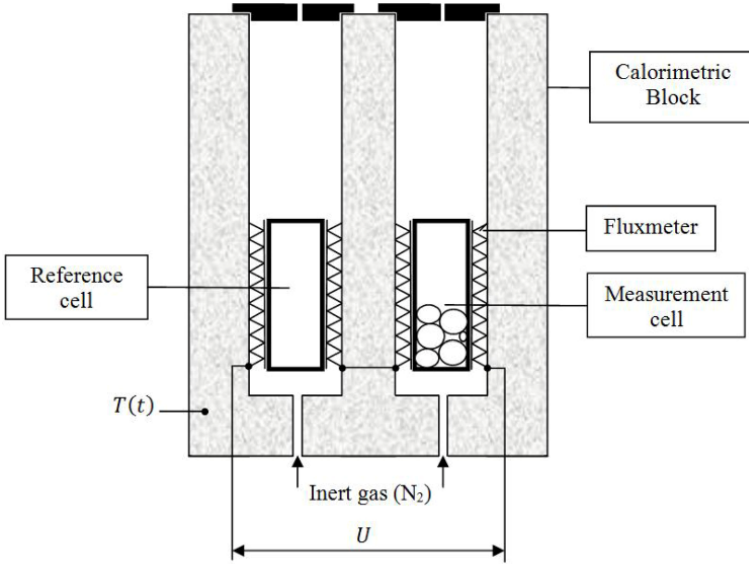


Figure 4.81. Diagram of a differential calorimeter

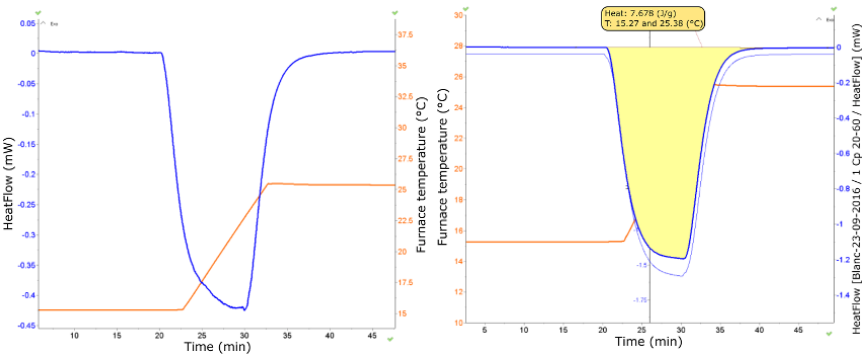


Figure 4.82. Recording of $\Delta\phi$ and $T(t)$ in a DSC experiment: a) blank; b) calibration check with sapphire

The integration of the difference U of $\Delta\varphi_r$ and $\Delta\varphi_b$ between the two stages makes it possible to calculate the constant k in the following way:

$$k = \frac{m_r c_r \Delta T}{\int_{T_1}^{T_2} U dT}$$

This is the calibration phase of the fluxmeter to be repeated regularly and before any new measurement campaign.

The integration of the difference of $\Delta\varphi_e$ and $\Delta\varphi_b$ between the two temperature steps makes it possible to calculate the heat quantity $Q_e = k \int_{T_1}^{T_2} U dT$ that had to be supplied to the mass m_e of sample to raise its temperature of ΔT , and we deduce its heat capacity by:

$$c_e = \frac{Q_e}{m \Delta T} \quad [4.71]$$

4.10.2. Drop calorimeter

This calorimeter is mainly used for high-temperature measurements. Its operating principle is illustrated in Figure 4.83. A sample of mass m_e is introduced into a lock at constant temperature ($T_0 \approx 25^\circ\text{C}$), where it is maintained for a time sufficient to reach thermal equilibrium (phase a). It is then dropped into a cell in contact with a block maintained at a constant temperature $T_1 > T_0$ (phase b). A fluxmeter placed around the cell makes it possible to measure the flow of heat exchanged between the block and the cell. From the time of the drop, this heat flow is recorded until the temperature of the sample reaches the value T_1 (phase c). The integration of the heat flow rate over the duration of the heating makes it possible to calculate the enthalpy required to change the mass m_e from the temperature T_0 to the temperature T_1 . In the absence of phase change, the average specific heat of the sample is deduced between T_0 and T_1 as before.

The measurements are repeated for several values of T_1 so as to obtain an experimental curve $Q_e = f(T)$, from which we can look for a polynomial representation. It is then easy to deduce the value of the heat capacity by derivation of this expression:

$$c_e = \frac{1}{m} \left(\frac{\partial Q_e}{\partial T} \right) \quad [4.72]$$

It is assumed that the duration of the steps is sufficiently long for the sample-cell assembly to be at a uniform temperature at the end.

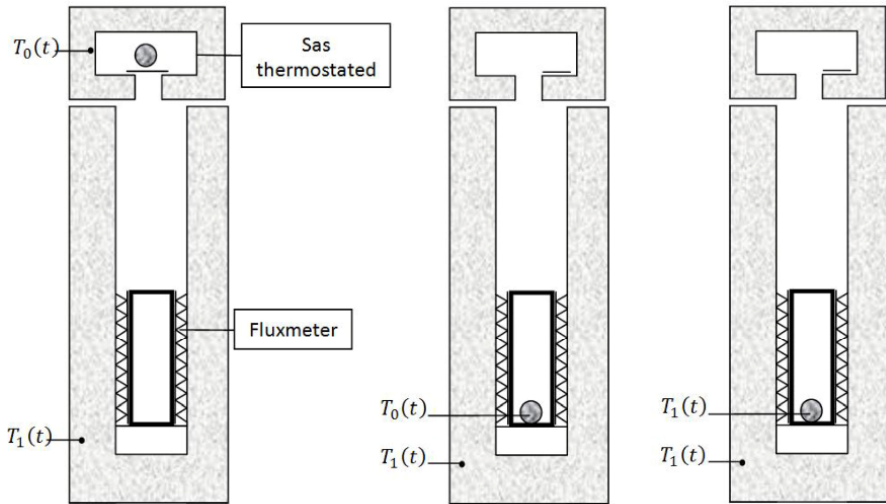


Figure 4.83. *Diagram of a drop calorimeter*

Transient Temperature/ Temperature Methods

5.1. Introduction

Why do we use the Temperature (T_1)/Temperature (T_2) methods, i.e. identifying a transmittance, rather than the conventional Source (q)/Temperature (T) methods, i.e. identifying an impedance?

At first sight, the conventional method requires only one measurement T_2 and therefore the statistical uncertainty on the measurement will depend only on the measurement noise on T_2 , whereas the Temperature/Temperature method requires the measurement of two temperatures (T_1) and (T_2), and therefore noise on (T_1) and (T_2); without analysis, it would appear that the classical method is better.

In fact, the T/T method makes it possible to eliminate a certain number of parameters in the identification as will be seen hereafter and it is demonstrated that the noise on (T_1) does not intervene practically in the calculation of the standard deviations: the convolution performed on (T_1) is equivalent to an integration, which reduces the effect of the measurement noise in large proportions, see [MAI 13].

To show in a simple way the interest of the T/T methods we illustrate the case of a uniform source (the geometry is arbitrary, Cartesian, cylindrical or spherical, but the transfer is 1D) on a homogeneous medium (characterized by two parameters, for example, $\frac{\alpha}{e^2}$ and $\frac{\epsilon}{\lambda}$). The medium is thermally connected to the outside by a system S_f for the face receiving the source and by a system S_r for the face opposite to the source (these systems can be quite arbitrary, from the simple coefficient of exchange

to several media with or without contact resistance). The number of parameters of the system S_f is called n_f and the number of parameters of the system S_r is called n_r . The exterior is taken as the origin of the temperatures ($T_e = 0$).

Figure 5.1 shows the overall system.

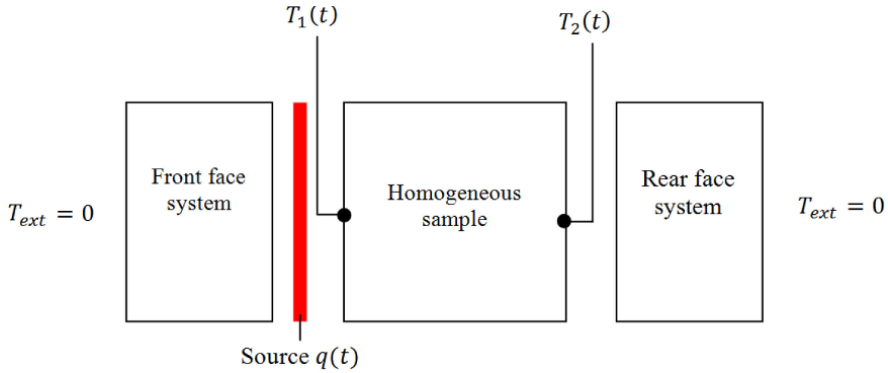


Figure 5.1. *Schematic diagram of the overall system*

After performing a Laplace transform on time, each element (the medium, the systems S_f and S_r) is a quadrupole; Figure 5.2 represents the global system after the Laplace transform. In the figure is shown:

- Q the Laplace transform of the source $q(t)$ (in W/m^2);
- Φ_1 the Laplace transform of the heat flow rate on the front face;
- θ_1 the Laplace transform of the temperature of the front face;
- θ_2 the Laplace transform of the temperature of the rear face.

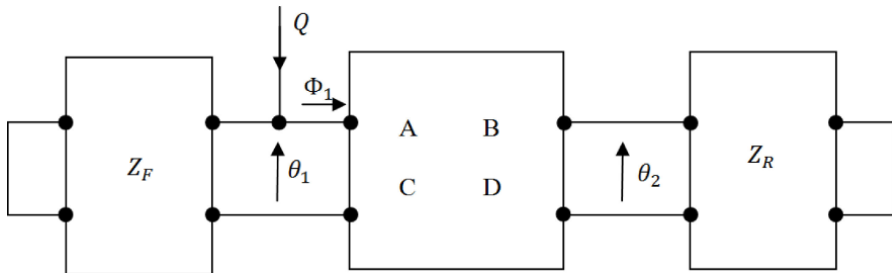


Figure 5.2. *Representation of the equivalent schematic diagram after Laplace transformation*

In the conventional estimation, the heat source $q(t)$ is assumed to be known, namely: $\theta_2 = Z_1 Q$ or $q(t) = q_0 f(t)$, $f(t)$ being known (with $F = L[f(t)]$), i.e. $\theta_2 = Z_2 F$.

The impedance to be identified (Z_1 or Z_2) comprises the two parameters of the medium ($\frac{a}{e^2}$ and $\frac{e}{\lambda}$) or ($\frac{a}{e^2}$ and $q_0 \frac{e}{\lambda}$) and n_f and n_r parameters of the two systems (front and rear):

$$N = n_f + n_r + 2$$

In the case of the T/T identification, i.e. $\theta_2 = W \theta_1$, the parameters of the front system do not intervene and the number of parameters of the medium is now only 1, it is not necessary to transform a temperature into a flux. The transmittance W to be identified comprises one parameter of the medium ($\frac{a}{e^2}$) and the n_r parameters of the rear system, namely:

$$N = n_r + 1$$

We might wonder if it would not be interesting to measure the temperature on the front face before measuring the flow on the front face, i.e. $\theta_2 = Z_3 \Phi_1$.

There are two drawbacks to this approach: the first one comes from fluxmeters themselves which are rarely linear and require difficult calibrations; the second one is that we add a parameter that needs to be identified (we must pass from a flux to a temperature). The impedance to be identified Z_3 comprises the two parameters of the medium ($\frac{a}{e^2}$ and $\frac{e}{\lambda}$) and the n_r parameters of the rear system, namely:

$$N = n_r + 2$$

EXAMPLE.— *Conventional flash.*

Refer to section 4.5 for a description of the method. The equivalent electrical diagram after Laplace transformation is shown in Figure 4.32.

The impedance Z and the Laplace transform θ_2 of the temperature are related by:

$$Z = \frac{q_0}{C + (h_1 + h_2)A + h_1 h_2 B}$$

$$\text{In reduced variables: } Z = \frac{K}{\sqrt{\frac{p}{\alpha}} \sinh\left(\sqrt{\frac{p}{\alpha}}\right) + (H_1 + H_2) \cosh\left(\sqrt{\frac{p}{\alpha}}\right) + \frac{H_1 H_2}{\sqrt{\frac{p}{\alpha}}} \sinh\left(\sqrt{\frac{p}{\alpha}}\right)}$$

The impedance depends on four parameters ($n_f + n_r + 2 = 1 + 1 + 2 = 4$):

$$K = q_0 \frac{e}{\lambda}; \quad H_1 = \frac{h_1 e}{\lambda}; \quad H_2 = \frac{h_2 e}{\lambda}; \quad \alpha = \frac{a}{e^2}$$

For the transmittance: $\theta_2 = W \theta_1$

where: $W = \frac{1}{A + h_2 B}$

In reduced variables: $W = \frac{1}{\cosh\left(\sqrt{\frac{p}{\alpha}}\right) + \frac{H_2}{\sqrt{\frac{p}{\alpha}}} \sinh\left(\sqrt{\frac{p}{\alpha}}\right)}$

The transmittance depends only on two parameters ($n_r + 1 = 1 + 1 = 2$):

$$H_2 = \frac{h_2 e}{\lambda}; \quad \alpha = \frac{a}{e^2}$$

EXAMPLE.— *Three-layer method.*

See section 5.2 for a description of the method. The equivalent electrical diagram after Laplace transformation is shown in Figure 5.3.

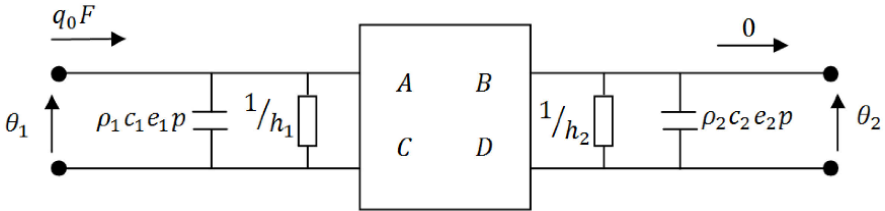


Figure 5.3. Representation of the three-layer equivalent diagram after Laplace transformation

The impedance Z and the Laplace transform θ_2 of the temperature are related by: $\theta_2 = Z F$

where: $Z = \frac{q_0}{C + (h_1 + \rho_1 c_1 e_1 p + h_2 + \rho_2 c_2 e_2 p)A + (h_1 + \rho_1 c_1 e_1 p)(h_2 + \rho_2 c_2 e_2 p)B}$

or, in reduced variables:

$$Z = \frac{K}{\sqrt{\frac{p}{\alpha}} \sinh\left(\sqrt{\frac{p}{\alpha}}\right) + \left(H_1 + C_1 \frac{p}{\alpha} + H_2 + C_2 \frac{p}{\alpha}\right) \cosh\left(\sqrt{\frac{p}{\alpha}}\right) + \frac{(H_1 + C_1 \frac{p}{\alpha})(H_2 + C_2 \frac{p}{\alpha})}{\sqrt{\frac{p}{\alpha}}} \sinh\left(\sqrt{\frac{p}{\alpha}}\right)}$$

The impedance depends on six parameters ($n_f + n_r + 2 = 2 + 2 + 2 = 6$):

$$K = q_0 \frac{e}{\lambda}; H_1 = \frac{h_1 e}{\lambda}; H_2 = \frac{h_2 e}{\lambda}; \alpha = \frac{a}{e^2}; C_1 = \frac{\rho_1 c_1 e_1}{\rho c e}; C_2 = \frac{\rho_2 c_2 e_2}{\rho c e}$$

For the transmittance: $\theta_2 = W \theta_1$

$$\text{where: } W = \frac{1}{A + (h_2 + \rho_2 c_2 e_2 p) B}$$

$$\text{or, in reduced variables: } W = \frac{1}{\cosh\left(\sqrt{\frac{p}{\alpha}}\right) + \frac{H_2 + C_2 \frac{p}{\alpha}}{\sqrt{\frac{p}{\alpha}}} \sinh\left(\sqrt{\frac{p}{\alpha}}\right)}$$

The transmittance depends only on three parameters ($n_r + 1 = 2 + 1 = 3$):

$$H_2 = \frac{h_2 e}{\lambda}; \alpha = \frac{a}{e^2}; C_2 = \frac{\rho_1 c_1 e_1}{\rho c e}$$

5.2. Planar three-layer

5.2.1. Principle

Conventional measurement methods are sometimes poorly adapted to the determination of the thermal conductivity of very light insulating materials. The transient contact methods using plane heat sources (hot disc [GUS 91, HE 05], hot wire [NAG 81], hot plate [ZHA 93], hot strip [HAM 03, JAN 04]) do not allow for a good estimation of the thermal conductivity in the case of very light insulators for the following reasons:

- the capacity and thermal resistance of probes (heterogeneous, generally consisting of a metal filament inserted between two plastic films) which are not known with precision are often taken into account in a simplified way in the models;
- the sensitivity of the measured temperature to this capacity is all the stronger as the heat capacity of the insulation is low;
- the thermal conductivity of the probe is greater than that of the insulation which implies that the longitudinal heat transfer (parallel to the contact probe/sample surface) in the probe that is not taken into account in the models cannot become negligible thus biasing estimates.

The flash method [DEG 86] is also difficult to apply for the following reasons:

- insulators are often semi-transparent with respect to flash radiation;
- the measurement of surface temperature on a very light material is difficult to realize with precision;
- the exchanges on the irradiated surface are often very different from the exchanges on the opposite surface (significant temperature differences).

It therefore becomes essential to sandwich the material between two conductive metal plates in order to avoid the first two disadvantages; a device based on this principle has already been used for liquids [RÉM 06]. In the case of very light materials, it is demonstrated that the sensitivity to thermal conductivity of the temperature of the unheated face is highly correlated with the sensitivity to losses, and that the sensitivity to thermal diffusivity is very low. The three-layer method allows estimation of the thermal conductivity of very light insulating materials.

Its principle is given in Figure 5.4. The sample to be characterized is inserted between two conductive metal plates of the same cross-section as the sample. A thermal excitation is applied to one face so as to raise the temperature thereof and simultaneously the temperature changes of the two plates are recorded. Figure 5.5 shows a typical evolution of the temperatures of the two plates. The transfer function F is then estimated between these two temperatures defined by:

$$T_2(t) = F(t, \lambda, \rho c, h) \otimes T_1(t) \quad [5.1]$$

After modeling the transfer function F , the values of the parameters $(\lambda, \rho c, h)$ which minimize the sum of the quadratic deviations between the temperature $T_{2\text{exp}}(t)$ and the temperature $T_{2\text{mod}}(t) = F(t, \lambda, \rho c, h) \otimes T_{1\text{exp}}(t)$ are estimated. The minimization method used is that of Levenberg–Marquardt.

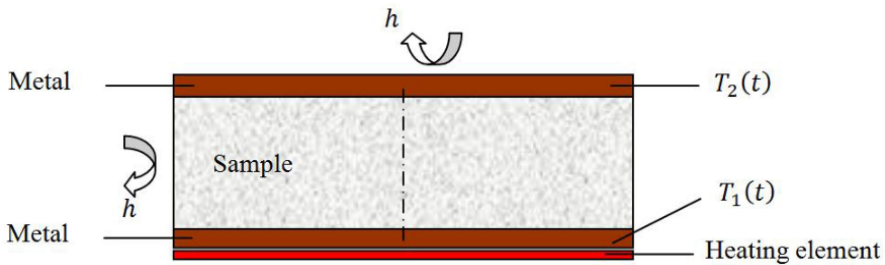


Figure 5.4. Schematic diagram of a three-layer set-up

One of the advantages of the method is that it is not sensitive to heat transfer on the heated face; in the case of a convective transfer and considering the important temporal variations of the temperature on this face, the hypothesis of a constant convection coefficient and especially equal to that of the unheated face is not completely satisfied.

Compared to the limits of the contact methods and of the flash method for measuring the thermal conductivity of light insulators, the proposed method has the following advantages:

- the temperature measurements made on a good conductive metal are precise;
- the thermal capacity of the two discs (homogeneous) is known precisely and accurately taken into account in the model;
- longitudinal heat transfer in the discs is integrated into the model (uniform temperature limit condition).

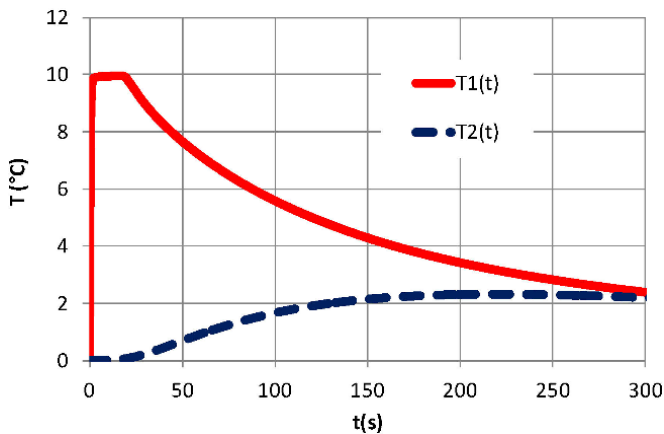


Figure 5.5. Typical temperature changes $T_1(t)$ and $T_2(t)$ of the two plates

5.2.2. Hypotheses and model

5.2.2.1. Hypotheses

- Uniform temperature in metal discs.
- Negligible contact resistances between sample and metal discs.
- Convective transfer coefficient constant and identical on all faces.

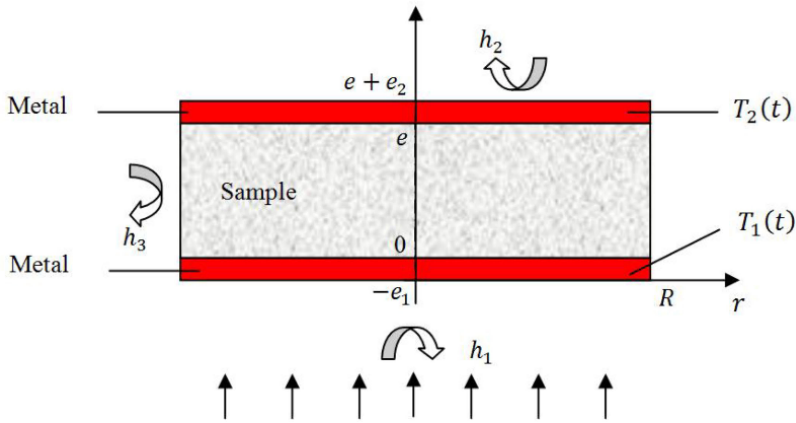


Figure 5.6. Schematic diagram of a three-layer experiment

5.2.2.2. Model

The cylindrical device shown in Figure 5.6 is modeled in cylindrical 2D. The resolution is done by a Laplace transform on time and separation of the space variables. For details of the modeling and its solution, see [JAN 09].

The transfer function $H(p)$ of the system is written as:

$$H(p) = \frac{\theta(e,p)}{\theta(0,p)} = \sum_{n=1}^{\infty} \frac{1}{A+B H_2} \quad [5.2]$$

$$\text{with: } A = \cosh(\beta_n) ; B = \frac{1}{\beta_n} \sinh(\beta_n) \quad [5.3]$$

$$\beta_n = \sqrt{\frac{pe^2}{a} + \left(\frac{e}{R}\right)^2} \omega_n^2 \quad [5.4]$$

$$\text{where } \omega_n \text{ is a solution of: } \omega J_1(\omega) = H_3 J_0(\omega) \quad [5.5]$$

$$\text{with: } H_2 = \frac{e}{\lambda} \left(h + \rho_2 c_2 e_2 p + \frac{2he_2}{R} \right) \quad [5.6]$$

$$H_3 = \frac{hR}{\lambda} \quad [5.7]$$

Equation [5.2] is true whatever the boundary condition on the heated brass disc, especially if the heated brass disc is in contact with an insulator rather than convection with ambient air.

5.2.2.3. Consideration of conduction in ambient air

The previous model implicitly assumes that the heat flux lost by the hot surfaces is transmitted to the ambient air without affecting the other surfaces. In fact, a part of this lost flux can be transmitted by conduction to the unheated brass plate according to the diagram in Figure 5.7 and contribute to the elevation of its temperature.

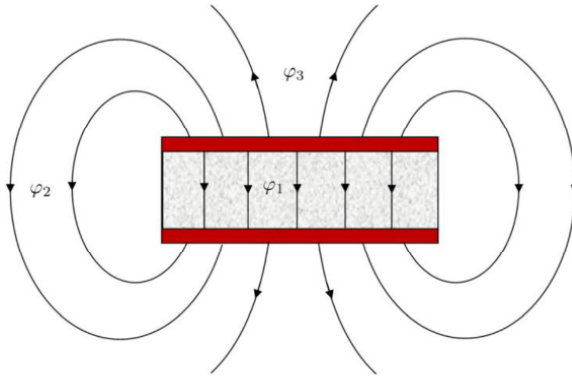


Figure 5.7. *Diagram of the thermal coupling between the two plates by the ambient air*

This phenomenon of heat transfer in parallel by conduction in ambient air can be represented by the quadrupole diagram in Figure 5.8, and it will be more important when the thermal resistance of the sample to be measured is large.

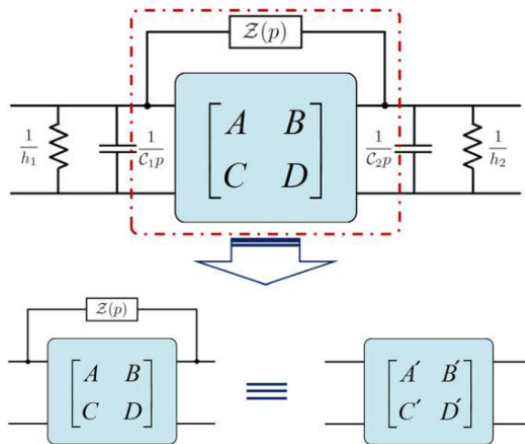


Figure 5.8. *Quadrupole representation of thermal impedance in parallel*

It has been shown [BAH 14] that this impedance can be represented by a pure resistance R and that the expression of the transfer function retains the same form as in equation [5.2] by replacing A by A' and B by B' given by:

$$A' = \frac{A + \frac{B}{R}}{1 + \frac{B}{R}}, \quad B' = \frac{B}{1 + \frac{B}{R}} \quad [5.8]$$

The value of this resistance in parallel is estimated experimentally.

5.2.2.4. Sensitivity study

The reduced sensitivity of the transfer function $H(p)$ was calculated for a three-layer system with the following characteristics:

Brass: $e_2 = 0.4 \text{ mm}$, $D = 40 \text{ mm}$, $\lambda = 110 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 3.2 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$.

Samples:

Low density superinsulator: $\lambda = 0.02 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 1.0 \times 10^4 \text{ J m}^{-3} \text{ K}^{-1}$, $e = 5 \text{ mm}$ and $e = 10 \text{ mm}$.

Polystyrene: $\lambda = 0.03 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 5 \times 10^4 \text{ J m}^{-3} \text{ K}^{-1}$, $e = 10 \text{ mm}$.

Cellular concrete: $\lambda = 0.15 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 3 \times 10^5 \text{ J m}^{-3} \text{ K}^{-1}$, $e = 10 \text{ mm}$.

The reduced sensitivity curves obtained are shown in Figure 5.9.

The following conclusions can be drawn from the figure:

- for the same material, the sensitivity to the heat capacity increases with the thickness of the sample and the sensitivity to thermal conductivity decreases;
- for the same sample thickness, the sensitivity to the heat capacity increases with the heat capacity of the sample;
- the sensitivity to heat capacity is very low for very light materials and will not be measurable by this method for this type of materials; it is sufficient to estimate the heat capacity of lighter insulators such as polystyrene or cellular concrete;
- the sensitivity to thermal conductivity is high in all cases and decorrelated to sensitivities to the convection coefficient and thermal diffusivity, conductivity is therefore *a priori* estimable by this method for all types of insulators.

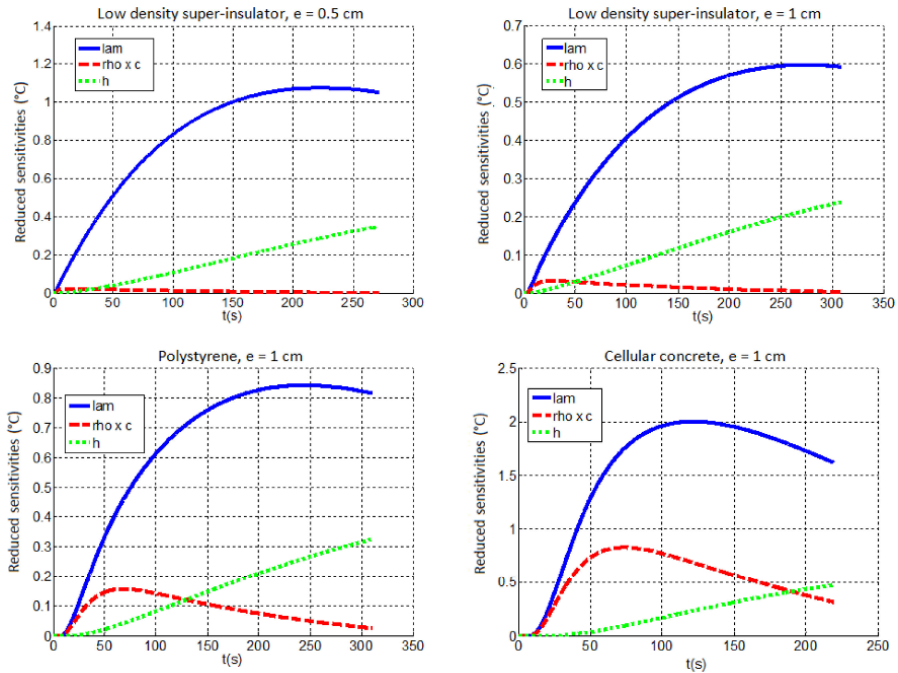


Figure 5.9. Reduced sensitivity curves of temperature $T_2(t)$ calculated by equation $T_{2_{mod}}(t) = F(t, \lambda, \rho c, h) \otimes T_{1_{exp}}(t)$ for different samples. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

5.2.3. Experimental set-up

The section of the sample can be a square or a disc. On deformable materials, which are therefore difficult to machine, it is often easier to cut square-section samples than cylindrical samples. If a square section of side b is used, it has been verified that the cylindrical model presented can be used for estimation by fixing the radius R at the value $R = \frac{b}{\sqrt{\pi}}$. The experimental set-up used in LEMTA consists of two plates of brass 1 mm thick and area $45 \times 45 \text{ mm}^2$. Figure 5.10 shows a view of this device. The thickness of the brass plates was chosen to be equal to 1 mm (thus relatively high) in order to favor the sensitivity to the thermal conductivity λ at the expense of the sensitivity to the volume heat capacity ρc , the latter being measurable with good accuracy per DSC. A sample of the material to be characterized, having the same cross-section as the two brass plates, is inserted between them. Two K type thermocouples with a wire diameter of 0.05 mm are attached to the outside of each brass plate with an aluminum adhesive. The bottom plate is in direct contact with a plane heating resistor of the same section placed on an insulating block. Pressure is

applied to the upper face by means of three points made of PVC or wood (materials chosen for their low thermal conductivity) with very small contact area. The rest of the top surface exchanges by natural convection and radiation with the ambient air.

For better control and reproducibility of measurements, it is preferable to equip the installation with a force sensor and a displacement sensor.

The choice of a gate of a few seconds produced by a flat resistance rather than a very brief flash sent by a lamp (initially tested set-up) is motivated by several reasons:

- the rise in temperature of the heated face must have a speed compatible with the response time of the thermocouple;
- the plane resistance makes it possible to more easily exert pressure on the metal plates, thus making it possible to minimize the contact resistances;
- a small part of the flash can reach the side surfaces by reflection; this inconvenience is avoided with a plane-heating resistor.

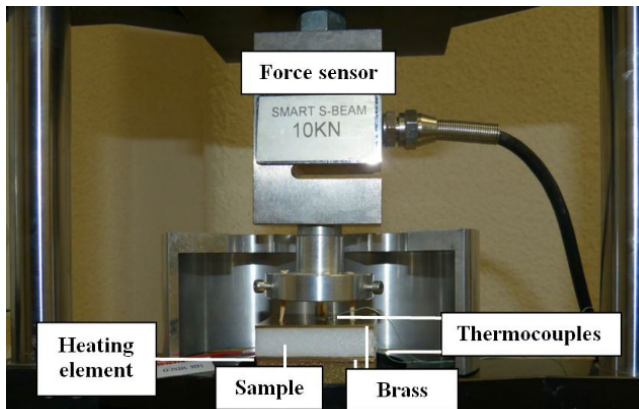


Figure 5.10. *View of the LEMTA three-layer device*

5.2.4. Practice of the method

The sample to be characterized is inserted between the two plates and pressure is applied to the tips in order to prevent the sample from slipping. Care should be taken not to apply excessive pressure which would change the thickness and conductivity of the sample.

A heat flow rate is applied to the heating resistor by modulating it so that the rise in temperature of the front face does not exceed 10°C. This heat flow rate is canceled as soon as the temperature rise of the rear surface reaches at least 2°C and the temperature record is allowed to continue for a time at least equal to twice that required for the temperature $T_2(t)$ to reach its maximum.

The mass m_2 is measured on the sample's unheated surface, which makes it possible to deduce the value of the product $\rho_2 c_2 e_2$, which appears in equation [5.2]:

$$\rho_2 c_2 e_2 = \frac{m_2 c_2}{S}$$

where: S is the surface area of the brass plate (m^2);

c_2 is the specific heat of the brass ($\text{J m}^{-3} \text{K}^{-1}$).

For example, in Table 5.1, measurements are made on different materials for which the thermal conductivity has also been measured by another method (λ_{ref}). The table presents the result of a first estimate made by neglecting the heat flux by conduction in the ambient air ($R = \infty$) and by a second one made by considering an optimum value which minimizes the deviations between the reference values and the values measured by the three-layer method ($R = 1.1$). This value of R depends on too many parameters to be able to be estimated by the calculation, and this calibration phase is essential and must be carried out for any new set-up. It can be seen that by considering this optimum value of R , the deviations between the measured values and the reference values (themselves known with an uncertainty of at least 2%) are quite satisfactory.

It is important to note that the air coupling is no longer negligible as soon as the resistance $\frac{e}{\lambda}$ of the medium is greater than $0.1 \text{ m}^2 \text{W}^{-1} \text{K}^{-1}$.

		$R = 0 \text{ m}^2 \text{W}^{-1} \text{K}^{-1}$		$R = 1.1 \text{ m}^2 \text{W}^{-1} \text{K}^{-1}$		
	e	λ	$\Delta\lambda$	λ	$\Delta\lambda$	λ_{ref}
	mm	$\text{W m}^{-1} \text{K}^{-1}$	%	$\text{W m}^{-1} \text{K}^{-1}$	%	$\text{W m}^{-1} \text{K}^{-1}$
Silica aerogel	5.08	0.0179	29	0.0134	-3	0.0138
Polystyrene	3.35	0.0359	11	0.0328	1	0.0325
	5.75	0.0396	18	0.0344	2	0.0336
	10.26	0.0423	29	0.0329	1	0.0327
PVC foam	5.95	0.0685	6	0.0646	0	0.0648

Table 5.1. Values of thermal conductivities estimated by the three-layer method

Figure 5.11 shows the experimental and simulated curves of $T_2(t)$ obtained in a three-layer experiment on a 10.2 mm thick polystyrene sample as well as the reduced sensitivity curves.

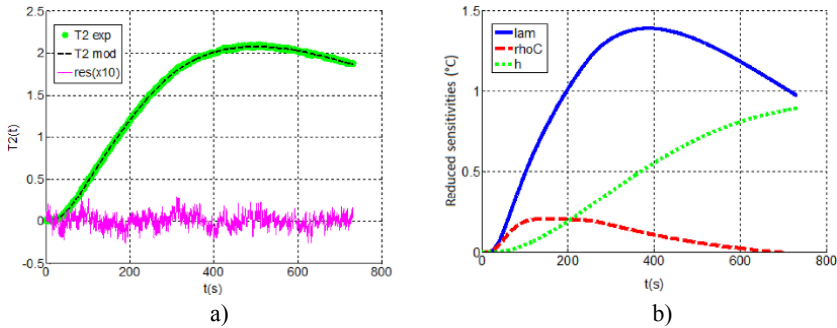


Figure 5.11. a) Experimental and simulated curve of $T_2(t)$ for a sample of polystyrene with a thickness of 10.2 mm; b) reduced sensitivity of $T_2(t)$. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

5.3. Cylindrical three-layer

5.3.1. Principle

Knowledge of the thermal properties of clusters or powders is important in many fields: thermal insulation by filling with a bulk material (cork, vermiculite and cellulose), cooling or heating of pulverulent food products.

The measurement of the thermal conductivity of clusters or powders can be carried out by the hot wire method with certain limits related to the density of the material to be characterized:

- the wire's small diameter (less than 1 mm) means that the number of probe/product contact points is small, so it can only be used for materials of very small particle size;
- like all heated probe methods, it is poorly suited to very low density materials [COQ 06].

The heat capacity can be measured by a differential calorimeter. The hot disc method allows the two parameters to be estimated *a priori* simultaneously but with the following important restrictions:

- the small size of the probe (a few millimeters in diameter) means that the number of probe/product contact points is small, so it can only be used for materials of very small particle size;

– the measurement of diffusivity is less accurate than that of conductivity [GUS 91];

– like the hot wire method, it is poorly adapted to materials with very low density [GUS 94].

The cylindrical three-layer set-up, combined with the appropriate model and estimation method, allows these two thermal properties to be measured simultaneously using a single experiment [JAN 13].

The three-layer cylindrical set-up schematically shown in Figure 5.12 consists of two coaxial tubes:

– a stainless steel inner tube of inner radius $r_0 = 4$ mm, outer radius $r_1 = 4.5$ mm and height 38 cm;

– an outer brass tube of inner radius $r_2 = 39$ mm, outer radius $r_3 = 40$ mm and height 38 cm.

The external diameter of the inner tube was chosen to be large enough (9 mm) so that the number of contact points between the grains and the tube is important even for grains of large diameters.

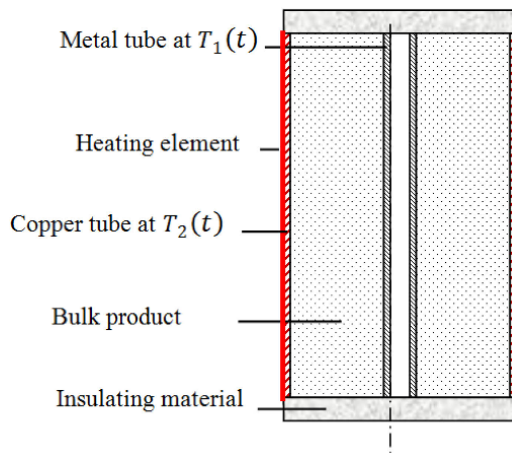


Figure 5.12. Schematic diagram of the three-layer cylindrical set-up

The external surface of the outer tube is surrounded by a flat, flexible resistive heating element of small thickness which makes it possible to ensure uniform heating thereof.

Two K type thermocouples are placed at mid-height respectively on the inner tube to measure $T_1(t)$ and on the outer tube to measure $T_2(t)$. The thermocouple wires with diameters of 0.05 mm are glued to the two tubes a few millimeters apart to obtain a mounting with separate contacts.

The inner tube is centered by two hollow discs of expanded PVC with a thickness of 8 mm, an internal diameter of 9 mm and an outer diameter of 78 mm. These two discs placed respectively at the bottom and at the top of the tubes make it possible to keep the product to be measured in the annular space between the tubes and to isolate it from the outside environment.

The experimental procedure is as follows: the annular space between the two tubes is filled with the product to be characterized and we wait until the temperatures T_1 and T_2 reach an identical stable value. The outer tube is then heated until its temperature rises to 10°C, then the heating is stopped when the temperature T_1 has reached a few degrees. The temperatures T_1 and T_2 are recorded from the start of heating and until the temperature T_1 reaches its maximum value.

5.3.2. Hypotheses and model

Modeling is based on the following hypotheses:

- the temperature of the product and of the tubes is uniform and equal to T_i at the initial time;
- the product is dry: no mass transfer;
- the temperature gradients according to Oz are negligible at mid-height of the set-up;
- the radial temperature gradient is negligible in both tubes (small body hypothesis).

By reason of cylindrical symmetry, the radial heat flux on the inner surface of the inner tube is zero and the quadrupole equation can be written as:

$$\begin{bmatrix} \theta_1(p) \\ 0 \end{bmatrix} = \begin{bmatrix} I & 0 \\ C_{1p} & I \end{bmatrix} \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} I & 0 \\ C_{2p} & I \end{bmatrix} \begin{bmatrix} \theta_2(p) \\ \Phi_0(p) \end{bmatrix}$$

where:

$$q = \sqrt{\frac{p}{a}};$$

$$A = qr_2 [K_1(qr_2) I_0(qr_1) + K_0(qr_1) I_1(qr_2)];$$

$$B = \frac{1}{2\pi\lambda L} [K_0(qr_1) I_0(qr_2) + K_0(qr_2) I_0(qr_1)];$$

$$C = 2\pi L \rho c p r_1 r_2 [K_1(qr_1) I_1(qr_2) + K_1(qr_2) I_1(qr_1)];$$

$$D = qr_1 [K_0(qr_2) I_1(qr_1) + K_1(qr_1) I_0(qr_2)];$$

$$C_1 = m_1 c_1;$$

$$C_2 = m_2 c_2;$$

$$\Phi_0(p) = \left[-\frac{\phi_0 [1 - \exp(-p t_c)]}{p} + h\theta_2(p) \right] \pi r_3 L.$$

where:

$\theta_j(p)$ is the Laplace transform of the temperature $T_j(t) - T_i$;

$\Phi_j(p)$ is the Laplace transform of the heat flow rate $\varphi_j(t)$;

α is the thermal diffusivity of the product;

ρ is the density of the product;

c is the specific heat of the product;

c_1 is the specific heat of the inner tube;

c_2 is the specific heat of the outer tube;

r_0 is the internal radius of the inner tube;

r_1 is the external radius of the inner tube;

r_2 is the internal radius of the outer tube;

r_3 is the external radius of the outer tube;

m_1 is the mass of the inner tube;

m_2 is the mass of the outer tube;

L is the length of the tube;

ϕ_0 is the heat flux on the outer tube;

t_c is the duration of heating;

h is the convective heat transfer coefficient.

We thus deduce:

$$\theta_1(p) = \frac{-\Phi_0(p)}{AC_1p + C + C_2p (BC_1p + D)} \quad [5.9]$$

$$\text{and: } \theta_2(p) = \frac{-(BC_1p + D) \Phi_0(p)}{AC_1p + C + C_2p (BC_1p + D)} \quad [5.10]$$

and the Laplace transform of the system's transfer function:

$$H(p) = \frac{\theta_1(p)}{\theta_2(p)} = \frac{1}{BC_1p + D} \quad [5.11]$$

or in real space:

$$T_1(t) = T_2(t) \otimes F(t) \quad [5.12]$$

$$\text{with: } F(t) = L^{-1} \left[\frac{1}{BC_1p + D} \right] \quad [5.13]$$

5.3.2.1. Method for estimating unknown parameters

This modeling of the system makes it possible to obtain the analytical expression of the transfer function of system $F(t)$ between the input $T_2(t)$ and the output $T_1(t)$. This transfer function depends only on λ (thermal conductivity), a (thermal diffusivity) and C_1 (heat capacity of the inner tube):

- the choice of the cylindrical geometry makes it possible to eliminate losses (that are unknown and to be estimated in other configurations) on the unheated face;

- the choice of carrying out the estimation on the transfer function makes it possible to eliminate the influence of all the parameters situated upstream of the heated surface, in particular the heat flux ϕ_0 , the heating time t_c , the heat capacity of the outer tube C_2 and the convective heat transfer coefficient h outside of this tube.

The two parameters a and ρc are estimated by minimizing the sum of the quadratic deviations between the experimental curve $T_{1exp}(t)$ and the model curve calculated by an equation of type $T_{1mod}(t) = T_{2exp}(t) \otimes F(t, \lambda, a, C_1)$ where $T_{2exp}(t)$ and C_1 are considered as data.

The transfer function $F(t)$ is calculated by calculating the inverse Laplace transform of equation [5.11] giving $H(p)$ using the De Hoog algorithm [DEH 82]. The minimization of the sum of the quadratic deviations is performed by the Levenberg–Marquardt algorithm.

This method leads to the following remarkable result and to our unique knowledge for a transient method using the entire thermogram: the only unknown parameters to be estimated are the thermal properties of the product to be characterized.

5.3.2.2. Study of reduced sensitivities

The sensitivity study was carried out on three materials with very different properties:

- heavy material: glass beads;
- medium density insulator: cork;
- a very light insulating material: expanded polystyrene beads.

The average properties used (based on data from several suppliers or measured) for each of these materials are reported in Table 5.2.

Material	$\lambda(\text{W m}^{-1} \text{K}^{-1})$	$\rho c(\text{J m}^{-3} \text{K}^{-1})$
Glass beads	0.22	1.23×10^6
Cork aggregates	0.038	1.1×10^5
Polystyrene beads	0.035	2.5×10^4

Table 5.2. Properties of the materials considered for the study of reduced sensitivities

The heating time t_c has been adjusted so as to remain less than the time from which the temperature of the inner tube begins to rise. The heat flow imposed on the outer tube was adjusted so that its temperature did not exceed 10°C .

We first place ourselves in the case where we try to estimate a and ρc . The simulated curves $T_1(t)$ and $T_2(t)$ as well as the transfer function $F(t)$ have been computed by inverse Laplace transformations applied to equations [5.9], [5.10] and [5.11]. The reduced sensitivities $a \frac{\partial T_1}{\partial a}(t)$ and $\rho c \frac{\partial T_1}{\partial \rho c}(t)$ were calculated as follows:

$$a \frac{\partial T_1}{\partial a}(t) = T_2(t) \otimes a \frac{\partial F}{\partial a}(t) \quad [5.14]$$

$$\rho c \frac{\partial T_1}{\partial p c}(t) = T_2(t) \otimes \rho c \frac{\partial F}{\partial p c}(t) \quad [5.15]$$

The set of these curves are shown for each of the three materials in Figure 5.13.

For glass beads, it can be seen that:

- the maxima of the sensitivities of T_1 to a and ρc are almost confounded and the sensitivities are almost proportional;
- the sensitivity to ρc is about 10 times lower than the sensitivity to a .

This leads us to predict that the value of ρc will be estimated with high uncertainty.

On the contrary, in the case of bulk cork and polystyrene beads, the ratio of sensitivity maxima to a and to ρc is about 2.5 for cork and 1.3 for polystyrene and also the sensitivities are not proportional. The two quantities a and ρc can therefore be estimated in this case with good precision.

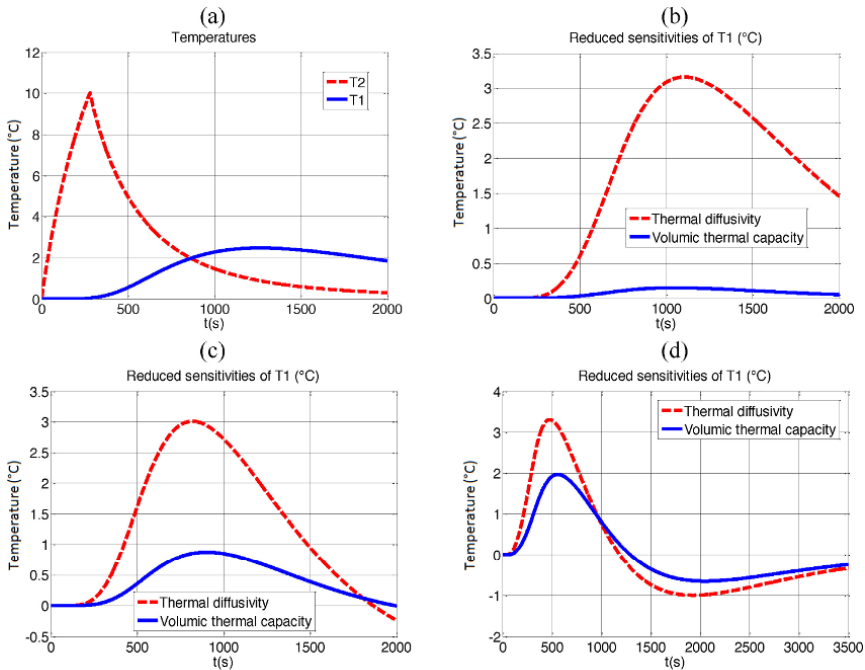


Figure 5.13. Typical temperature changes (a) and reduced sensitivities of T_1 for glass beads (b), cork aggregates (c) and polystyrene beads (d). For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

5.3.2.3. Influence of the diameter of the outer tube

The sensitivity study was carried out for the same products but now considering an outer tube with diameters of 24.5 mm/25 mm. The results obtained are shown in Figure 5.14.

It can be seen that the reduction in the diameter of the outer tube makes it possible to increase the sensitivity of T_1 to the thermal conductivity λ without this being sufficient for a material as heavy as the glass beads. This also reduces the measurement time and reduces the height necessary to avoid 2D effects in the middle of the tube.

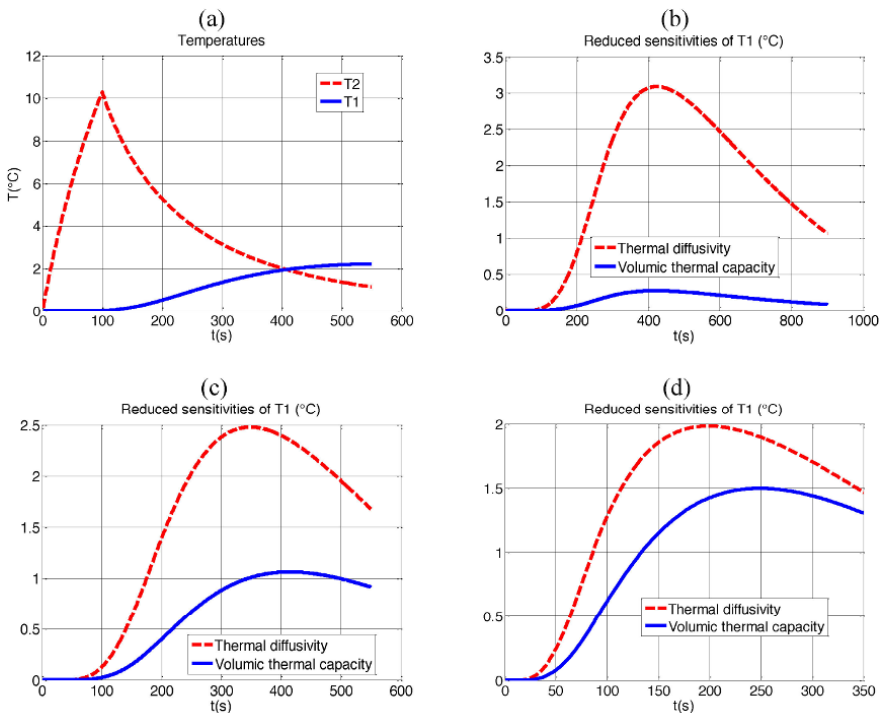


Figure 5.14. Typical temperature changes (a) and reduced sensitivities of T_1 for glass beads (b), cork aggregates (c) and polystyrene beads (d). For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

5.3.2.4. *Evaluation of the effect of an error on a given data*

The temperature T_1 not only depends on a and ρc but also on the following parameters: r_1 , r_2 and $m_1 c_1$. The influence of an error on each of the parameters was estimated in the following way [JAN 13]: a simulation was carried out with the nominal values of the parameters, parameter estimation in which the following measurement errors were successively simulated: absolute error of 0.01 mm on r_0 and on r_1 , of 0.05 mm on r_2 , and relative error of 1% on $m_1 c_1$. The estimated values for each simulation were then compared to the nominal values introduced in the simulations.

Given the different calculated deviations that can accumulate in the worst case, it is possible to estimate the accuracy of the measurement of the thermal diffusivity a with less than 2% whatever the material. Thermal conductivity can be estimated with accuracy better than 2% for insulating materials but is not measurable accurately for heavier and less insulating materials such as glass beads. These results are consistent with the findings of the reduced sensitivity study.

5.3.2.5. *Evaluation of the effect of a centering error*

The influence of an error in centering the inner tube with respect to the external tube on the estimated values of the thermal conductivity and the volumic heat capacity was also studied using 3D simulations carried out under COMSOL [JAN 13].

It has been shown that an offset of the axes as large as 0.25 mm produces an error of less than 0.3% on all the parameters except for the thermal conductivity λ and the volumic heat capacity ρc of the glass beads. An improvement consists of using two thermocouples placed at diametrically opposite points on the outer tube to measure T_2 and to retain the average of two values for the use of the results. It has been shown that this arrangement of the thermocouples makes it possible to obtain a deviation of less than 1% for all the parameters including for glass beads provided that the shift of the axes is less than 0.25 mm, which is easily realized in practice.

5.3.2.6. *Evaluation of standard deviations on estimated values*

The specific problem that arises here is the taking into account of the measurement noise on T_2 for the calculation of the standard deviation on the estimated values of a and ρc .

These two parameters are estimated by minimizing the sum of the quadratic deviations between the experimental curve $T_{1exp}(t)$ and the model curve T_{1mod}

calculated by a convolution between the experimental curve $T_{2exp}(t)$ and the transfer function, namely:

$$J(\beta) = \sum_i [T_{1exp}(t_i) - T_{1mod}(t_i, T_{2exp}, \beta)]^2$$

with: $T_1(t_i, T_{2exp}, \beta) = T_{2exp}(t_i) \otimes F(t_i, \beta)$

where: $\beta = \begin{bmatrix} a \\ \rho c \end{bmatrix}$ is the vector of the parameters to be estimated.

The function $J(\beta)$ is not “classical” for two reasons:

– the temperature T_1 depends on a parameter vector β' different from β (β' contains six parameters) leading to a calculation of the sensitivity matrix of T_1 :

$$S(\beta) = \frac{dT_1}{d\beta} = T_{2exp} \otimes \frac{dF}{d\beta} = T_{2exp} \otimes X(\beta)$$

where $X(\beta)$ is the sensitivity matrix of F ;

– the two temperatures $T_{1exp}(t)$ and $T_{2exp}(t)$ are noisy.

A complete calculation of the parametric estimation has shown that the estimation bias is of second order and that the covariance matrix is written as [MAI 13]:

$$\begin{aligned} cov(\beta) &= \sigma_T^2 [(S^T S)^{-1} + (S^T S)^{-1} S^T M_F M_F^T S (S^T S)^{-1}] = \\ &\begin{bmatrix} a_{11} & a_{12} \\ a_{21} & a_{22} \end{bmatrix} \end{aligned} \quad [5.16]$$

where: $S = M_{T_1} X$

and:

M_{T_1} is the Toeplitz matrix of T_1 ;

σ_T is the standard deviation of the measurement noise on T_1 ;

M_F is the Toeplitz matrix of F ;

X is the sensitivity matrix of F .

The standard deviations σ_a and $\sigma_{\rho c}$ on the estimated values of a and ρc are calculated by:

$$\sigma_a = \sqrt{a_{11}}$$

$$\sigma_{\rho c} = \sqrt{a_{22}}$$

The standard deviations were calculated for a recording time corresponding to the maximum value reached by the temperature of the small tube with an acquisition time step of 0.1 s and a standard deviation of the measurement noise $\sigma_T = 0.1 \text{ K}$.

It has been shown that, by choosing an acquisition time step of 0.1 s, the standard deviations due to measurement noise on the temperatures are negligible for the thermal conductivity of light materials (cork, polystyrene) and for thermal diffusivity of all materials [JAN 13]. The standard deviation on the thermal conductivity also remains less than 1% for heavier materials such as glass beads.

5.3.3. Experimental set-up

A set-up identical to that shown schematically in Figure 5.12 was made at LEMTA.

The choice of the material of the inner tube results must satisfy the following:

- it must be sufficiently conductive so that the temperature gradient in its thickness is negligible;
- it should not be too conductive to limit the longitudinal heat transfer which could invalidate the hypothesis of a purely radial heat transfer to mid-height if they were too large.

The final choice was a stainless steel with a thermal conductivity $\lambda = 16 \text{ W m}^{-1} \text{ K}^{-1}$.

The external diameter of the inner tube was chosen to be large enough (9 mm) so that the number of contact points between the grains and the tube is important even for grains of large diameters. Its thickness must also satisfy the following:

- it must be thick enough to be non-deformable;
- it should not be too thick to limit longitudinal heat transfer which could invalidate the hypothesis of a purely radial heat transfer at mid-height if they were too large.

The final choice was for a thickness of 0.5 mm.

The diameter of the outer tube was chosen to be large enough so that the distance between the two tubes is large compared to the size of the grains whose cluster properties are to be measured. Its thermal conductivity must be the best possible in order to ensure a uniform temperature over its entire surface. Its mechanical strength must be the best possible in order to avoid any deformation. All these constraints led us to choose a brass tube with an external diameter of 80 mm and a wall thickness of 1 mm.

The height of the tubes was chosen to be sufficiently large for the hypothesis of the 1D transfer at mid-height to be respected; a height of 38 cm was chosen. The validity of the 1D hypothesis for these dimensions has been validated by numerical simulations with the COMSOL[®] software.

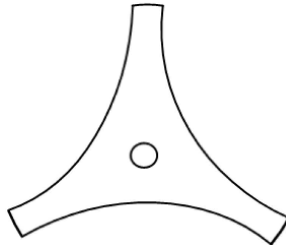


Figure 5.15. *Diagram of a centering element*

This numerical study has shown the paramount importance of the dimensions and the thermal conductivity of the two discs ensuring the centering of the inner tube with respect to the outer tube, for the respect of the 1D transfer hypothesis at mid-height. This led us to make this part in a rigid and insulating material: rigid PVC foam with thermal conductivity = $0.065 \text{ W m}^{-1} \text{ K}^{-1}$. These discs with a thickness of 8 mm were also “hollowed out” to reduce the heat transfer from the large tube to the small tube, according to the diagram in Figure 5.15.

5.3.4. Experimental practice

An experimental study was carried out at LEMTA on three products:

- glass beads;

- cork aggregates;
- polystyrene beads.

Before each experiment the products were dehydrated by evacuation for 48 h.

The specific heat values of each of these products and of the inner tube were measured using a SETARAM μ DSC3 calorimeter. The bulk densities were deduced from the weighing of a known volume of product. The results are shown in Table 5.2.

The thermal effusivity E of the glass beads was measured using an asymmetric hot plate type device described and used by Bal [BAL 11]. The measured value at 30°C is: $E_{glass} = 470.9 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$ which couples to the value of the volumic heat capacity ρc to deduce the thermal diffusivity and thermal conductivity at 30°C: $\alpha = 1.54 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$ and $\lambda = 0.185 \text{ W m}^{-1} \text{ K}^{-1}$.

Measurements were then carried out on each of the three materials using the cylindrical three-layer device. The heat flux density applied to the outer tube was modulated to maintain the temperature rise of the outer tube at approximately 10°C until the temperature of the inner tube had increased by about 2.5°C. The heating was then switched off and the recording continued until the temperature of the inner tube reached its maximum.

The results of the estimates obtained for the three materials are presented in Table 5.3. The deviations between the values measured by the three-layer method and the values measured by DSC and the centered hot plate are in agreement with the theoretical predictions: low (< 2.1%) for light materials (cork and polystyrene) and high for heavy materials (glass beads). The theoretical study predicts for the latter type of material that only the thermal diffusivity can be measured with precision. It has indeed been found that the value of the thermal diffusivity measured by the cylindrical three-layer method: $\alpha = 1.56 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$ differs only by 1.6% from the DSC measurements and the centered hot plate: $\alpha = 1.54 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$.

As an example, Figure 5.16 shows the changes in measured temperatures T_1 and T_2 as well as the changes in measured and simulated temperatures T_1 with the parameters estimated for dry cork aggregates. Estimation residues ($\times 10$), i.e. the difference between the experimental values and the measured values of T_1 , were also included. It is observed that the residues are weak and perfectly centered at approximately 0°C, which validates the 1D model.

	Cylindrical three-layer			DSC + centered hot plate		
Material	Glass	Cork	Polystyrene	Glass	Cork	Polystyrene
$a \text{ (m}^2 \text{ s}^{-1}\text{)}$	1.564×10^{-7}	3.45×10^{-7}	1.377×10^{-6}	1.54×10^{-7}	–	–
$\rho c \text{ (J m}^{-3} \text{ K}^{-1}\text{)}$	0.965×10^6	1.113×10^5	26443	1.20×10^6	1.14×10^5	26107
$\lambda \text{ (W m}^{-1} \text{ K}^{-1}\text{)}$	0.151	0.0384	0.0364	0.185	–	–

Table 5.3. *Values of the thermal properties estimated by the cylindrical three-layer and the DSC + centered hot plate methods*

A measurement was also carried out on cork with a water content of 3% in equilibrium with the ambient air. Figure 5.17 shows the evolution of the measured temperatures T_1 and T_2 as well as the evolutions of the measured and simulated temperatures T_1 with the estimated parameters, the estimating residues ($\times 10$) have also been included. It is found that the model which does not take into account the heat transfer produced by the evaporation/condensation phenomena does not make it possible to obtain a satisfactory representation of the experimental curve. The properties of a wet material can therefore only be achieved by a steady-state measurement or by a transient measurement modeled by taking into account the mass transfers. In the latter case, it is necessary to know at least the mass diffusion coefficient, which leads to a source of additional uncertainty. This applies not only to the cylindrical three-layer method but also to all transient measurement methods.

Finally, additional measurements were made on the glass beads with a device of a height reduced to 20 cm with an outer tube with diameters of 24.5/25 mm, the inner tube remaining the same. The values obtained are the following: $\lambda = 0.167 \text{ W m}^{-1} \text{ K}^{-1}$, $\rho c = 1.07 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$ and $a = 1.574 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$. The estimated value of the volumic heat capacity is slightly closer to the real value than that obtained with the large tube (10%, instead of 19.6%). The thermal diffusivity values obtained with the two devices differ by less than 1%.

The cylindrical three-layer method allows us to measure the thermal properties of powder-type materials or clusters of dry pellets. For insulating materials (therefore low density), it is possible to measure in a single experiment and with a precision better than 5% both the thermal conductivity and the volumic heat capacity. For heavy materials, it is possible to measure the thermal diffusivity, another method of measurement is then necessary to fully characterize the product, the simple method of the asymmetric centered hot plate [JAN 10] can in this case be used as a complement.

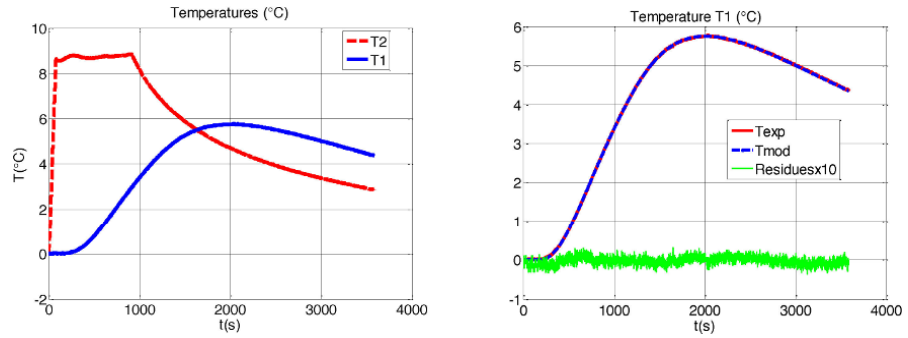


Figure 5.16. a) Experimental curves of $T_1(t)$ and $T_2(t)$; b) experimental and model curves of $T_1(t)$ and estimation residues $\times 10$ for dry cork aggregates. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

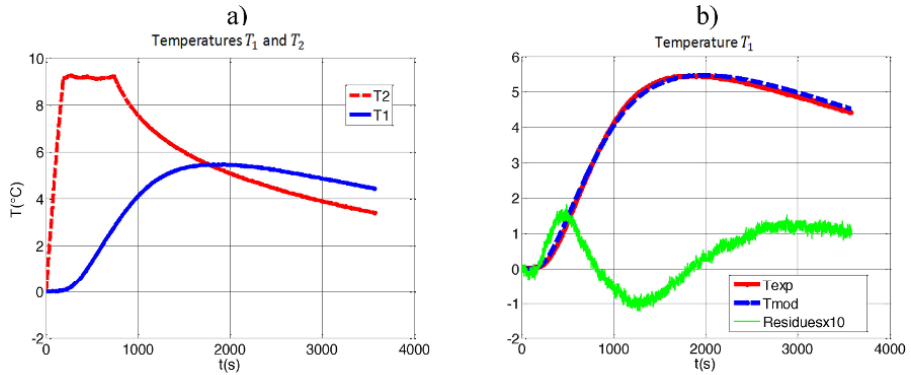


Figure 5.17. a) Experimental curves of $T_1(t)$ and $T_2(t)$; b) experimental and model curves of $T_1(t)$ and estimation residues $\times 10$ for wet cork aggregates. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

5.4. Transient fin method

5.4.1. Principle

Knowledge of the thermal properties of materials available and used in thin plates is important in several fields including that of printed circuits. The support of the printed circuits is most often made of a plastic plate with a thickness of less than 1 mm. The conductivity in the direction perpendicular to the plate is between

$\lambda_z = 0.2 \text{ W m}^{-1} \text{ K}^{-1}$ and $\lambda_z = 2 \text{ W m}^{-1} \text{ K}^{-1}$ with a factor of anisotropy $\frac{\lambda_{xy}}{\lambda_z}$ that can be up to 2.

To predict and limit the heating of the supports (and therefore of the circuits deposited therein), it is necessary to know precisely the conductivities λ_{xy} and λ_z .

The thermal conductivity λ_z can be deduced from the thermal diffusivity a_z measured by the flash method. The thermal conductivity λ_{xy} can be deduced from the thermal diffusivity a_{xy} measured by the transient fin method [HAD 92] whose principle is shown in Figure 5.18.

It is considered that the sample is initially at a uniform temperature and equal to the temperature T_a of the ambient air. A heat flow rate $\varphi_0(t)$ is applied on one of its ends, the intensity and the time variations of which are not required to be known. The temporal evolutions of the temperatures T_1 and T_2 are measured at respective distances x_1 and x_2 of the heated edge by bonded thermocouples or by an IR camera. These measurements, combined with a modeling of heat transfer, make it possible to deduce the thermal diffusivity a_{xy} .

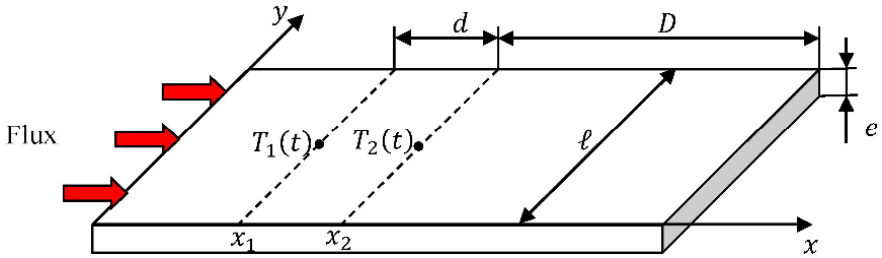


Figure 5.18. Diagram of the principle of the transient fin

5.4.2. Hypotheses and model

5.4.2.1. Model

This model is based on the following hypotheses:

- the thermal gradient in the direction of thickness e is negligible (thin system), which is verified if $Bi < 0.1$, where $Bi = \frac{he}{\lambda}$ is the Biot number;
- the plate is long enough so that it can be considered as a semi-infinite medium for the duration of the measurement;

- the plate is wide enough so that the edge effects in the direction Oy have no effect at the center of the plate;
- heat transfer by conduction in the air is negligible.

With these hypotheses, the system of equations describing the transfer is written as:

$$\frac{\partial^2 T}{\partial x^2} - \frac{h P_e}{\lambda_{xy} S} (T - T_a) = \frac{1}{a_{xy}} \frac{\partial T}{\partial t}$$

with: $T(x, t = 0) = T_a;$

$$T(x_1, t) = T_1;$$

$$T(x \rightarrow \infty, t) = T_a.$$

and where: $P_e = 2(e + \ell)$ is the perimeter;

$S = e\ell$ is area of the section associated with the heat flow rate.

Given $\bar{T} = T - T_a$ and designating $\theta(x, p)$ the Laplace transform of $\bar{T}(x, t)$, these equations become:

$$\frac{d^2 \theta}{dx^2} - \left(\frac{p}{a_{xy}} + \frac{h P_e}{\lambda_{xy} S} \right) \theta = 0$$

The general solution is written in the form: $\theta(x, p) = A \exp(-qx) + B \exp(qx)$

where: $q = \sqrt{\frac{p}{a_{xy}} + \frac{h P_e}{\lambda_{xy} S}}.$

The semi-infinite medium hypothesis leads to: $B = 0$.

The solution thus takes the form: $\theta(x, p) = A \exp(-qx)$.

In particular: $\theta_1(p) = A \exp(-qx_1)$ and $\theta_2(p) = A \exp(-qx_2)$.

Hence: $\theta_2(p) = \theta_1(p) \exp(-qd)$.

where: $d = x_2 - x_1$.

Thus θ_2 depends only on θ_1 and the following parameters: p , $\frac{d^2}{a_{xy}}$ and $\frac{h P_e d^2}{\lambda_{xy} S}$

and we can write it in real space as:

$$T_2(t) = T_1(t) \otimes F(t) \quad [5.17]$$

where: $F(t) = L^{-1}[H(p)]$ is the inverse Laplace transform of $H(p)$.

$$\text{with: } H(p) = \exp(-qd) \quad [5.18]$$

$$\text{and: } F(t) = \frac{1}{2} \frac{d}{\sqrt{\pi t^3 a_{xy}}} \exp\left(-\frac{hPe}{\rho c S} t - \frac{1}{4} \frac{d^2}{a_{xy} t}\right) \quad [5.19]$$

5.4.2.2. Method for estimating unknown parameters

This modeling of the system makes it possible to obtain the analytical expression of the transfer function of the system $F(t)$ between the input $T_2(t)$ and the output $T_1(t)$. This transfer function depends only on t , a_{xy} (thermal diffusivity) and h (convection–radiation transfer coefficient):

The two parameters a_{xy} and h are estimated by minimizing the sum of the quadratic deviations between the experimental curve $T_{2exp}(t)$ and the model curve calculated by an equation of type $T_{2mod}(t) = T_{1exp}(t) \otimes F(t, a_{xy}, h)$ where $T_{1exp}(t)$ is considered as a data point.

The function $F(t)$ is computed by equation [5.19]. The minimization of the sum of the quadratic deviations is performed by the Levenberg–Marquardt algorithm.

5.4.2.3. Study of reduced sensitivities

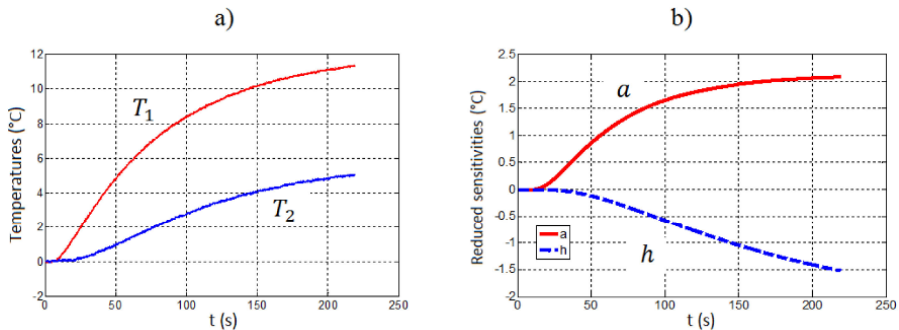


Figure 5.19. a) Temperature curves $T_1(t)$ and $T_2(t)$; b) reduced sensitivities of $T_1(t)$ to a_{xy} and h for a resin. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

It shall be noted that:

- the sensitivity to h decreases if the thickness e of the plate increases: it is therefore not necessary to work with plates that are too thin, especially if the thermal diffusivity is low;
- the sensitivity to h decreases if the value of h decreases: we will therefore work under vacuum whenever possible, which will also make it possible to limit the conductive transfer in the air neglected in the model.

5.4.3. Experimental set-up

The heat flow rate $\varphi_0(t)$ can be produced by passing a current through a resistive strip deposited on the substrate. To avoid edge effects, it is preferable to use a rather large sample (square of 10 cm side in our experiments). The values of x_1 and x_2 must be large enough so that the uncertainty about their difference d is small ($x_1 = 1$ cm and $x_2 = 2$ cm in our experiments). The distance D (see Figure 5.18) must also be sufficiently large (about 3 cm) for the semi-infinite medium hypothesis to remain valid for some time.

The measurement of the two temperatures T_1 and T_2 can be carried out either:

- by IR camera;
- by a measurement of the variation of the resistances of two thin resistive strips of which the temperature coefficient is known.

Whatever the method of measurement chosen, it should be noted that in this type of method the measurement of two quantities proportional to the temperatures (with the same coefficient of proportionality) is sufficient. For example, if the two strips are identical, it is not necessary to know their temperature coefficient. If an IR camera is used, no accurate calibration is required.

The reduced sensitivity study showed that the sensitivity to h decreases with the value of h , so it is advantageous to minimize this value. For this reason, the measuring device should be placed under vacuum whenever possible: the external transfer will be exclusively due to the radiation and therefore the transfer coefficient will be lower. Conductive heat transfer will also be avoided in the air layer adjacent to the surface which can create a thermal bridge causing a slight increase in temperature T_2 before that caused by the diffusion of heat in the plate. This effect was observed for the least diffusive materials.

5.4.4. Practice of the measurement

An example of recording of temperatures $T_{1exp}(t)$ and $T_{2exp}(t)$ as well as the experimental curves $T_{2exp}(t)$ and model $T_{2mod}(t)$ plotted on the same graph is given in Figure 5.20. The temperature measurements were carried out by an IR camera. The distance d between the measuring points is 10 mm for a value of the thermal diffusivity $a_{xy} = 8.2 \times 10^{-7} \text{ m}^2\text{s}^{-1}$ and a plate thickness of 0.8 mm. In this case, the temperature rise on the rear face is sufficient to obtain a signal-to-noise ratio allowing a good estimation of the parameters.

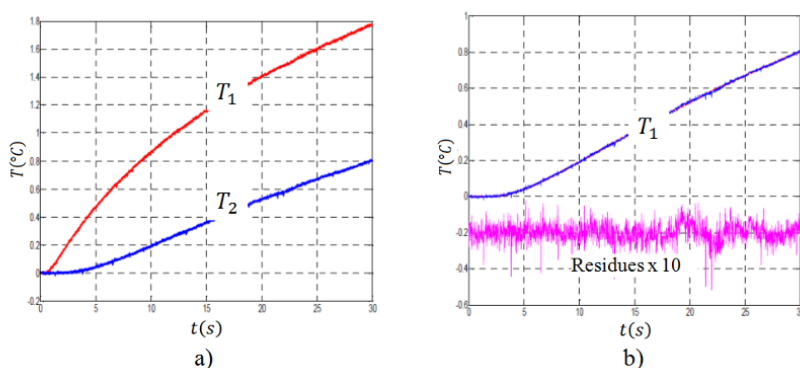


Figure 5.20. a) Experimental thermograms $T_{1exp}(t)$ and $T_{2exp}(t)$; b) experimental thermogram $T_{2exp}(t)$ and theoretical $T_{2mod}(t)$ obtained on a resin

Another series of experiments on a sample of the same thickness but of thermal diffusivity $a_{xy} = 2.2 \times 10^{-7} \text{ m}^2\text{s}^{-1}$ led to an elevation of the temperature that is too low. The distance between the measuring points has to be reduced in this case to $d = 5$ mm, which increases the relative uncertainty on its value. To maintain the value acceptable $d = 10$ mm, it would have been necessary to work under vacuum and/or increase the thickness of the plate.

NOTE.—

A series of measurements carried out under different air pressures revealed the influence of conduction in the adjacent air layer on the result of the estimation. For a material with a thermal conductivity $\lambda_{xy} = 0.48 \text{ W m}^{-1} \text{ K}^{-1}$ and thickness $e = 0.8$ mm, the pressure must be lowered to less than 10^{-2} mbar to obtain an accurate estimate of the thermal conductivity. The error obtained by performing the measurement at atmospheric pressure reaches 30%.

Choice of an Adapted Method

In this chapter, we will propose a choice of methods adapted to measurement in different types of situations which depend on:

- the range in which the thermal properties of the material to be measured lie;
- the size of the available samples;
- their moisture state: dry or wet;
- their physical state: consolidated solid, powder or cluster, liquid.

Before that, we will give some general advice for the realization of good measurements.

6.1. Measurement advice

6.1.1. *How many measurements?*

The first point concerns the number of measurements to be realized before a reliable result can be obtained. The minimum is, in our opinion, three measurements and it is essential to remove the samples from the experimental device and then to put them back in place between measurements. For example, certain measurements may be sensitive to the thermal contact resistances and the clamping of the device whose influences may vary between two experiments. It is therefore important to repeat this clamping operation to verify that it has no impact on the measurement result. Moreover, the calculated standard deviation between the results of the three measurements makes it possible to get an order of magnitude for the uncertainty due to the measurement noise.

Important reminder:

As for the uncertainty due to errors on the supposedly known parameters of the model, one way of knowing them is to work on the dimensionless expression of the model.

For example:

– for the standard 1D Flash method, the dimensioned formula gives rise to three parameters: $\frac{he}{\lambda}$, $\frac{e^2}{a}$ and $\frac{q_0}{\rho ce}$

A 1% error on e results in a 2% error on a ;

– for the semi-infinite hot plate, the dimensionless formula [4.2] gives rise to three parameters: $R_c E$, $\phi_0 R_c$ and $\frac{(mc)_s}{R_c E^2 S}$

A 1% error on ϕ_0 results in a 1% error on E .

6.1.2. Steady-state or transient mode?

A commonly accepted idea is that transient methods allow for a much faster measurement than steady-state methods. Some manufacturers of measuring devices advertise less than one second measurement durations. The reality is very different.

Indeed, all the transient regime methods are based on the same hypothesis: at the initial time, the temperature field in the sample is rigorously uniform and most often equal to the temperature of the surrounding environment. In order to place the specimen in place, it has to be held with the fingers with a surface temperature of at least 32°C, or the sample in place has already been subjected to a first measurement during which it was heated. Before carrying out a transient measurement, it will be necessary to wait until the effect of these thermal disturbances has dissipated, which amounts to waiting for the establishment of a steady state, which can take minutes or even hours.

In transient conditions, we hypothesize that the medium behaves as a homogeneous medium, which does not make it possible to characterize certain types of media such as multilayer ones for which one chooses steady-state methods.

In wet environments, heat and mass transfers occur simultaneously, making the thermal models inadequate to describe the change in the temperature field in transient mode. Only the steady-state methods allow precise measurements for light insulating materials, since the time taken to achieve equilibrium is much longer than

for a dry material because the mass diffusion coefficient is generally low. You have to be very patient to measure the thermal conductivity of a wet material!

In the steady-state methods, a single measurement is performed *a priori*, which therefore makes it possible to estimate only one parameter which is thermal resistance. Therefore, to deduce a value of thermal conductivity, thermal contact resistances must be negligible compared to the sample's thermal resistance. Therefore, the steady-state methods do not make it possible to accurately measure high thermal conductivities.

6.1.3. What if the material is wet?

If the material is wet, the only reliable measurement methods are steady-state measurements. Experimental studies carried out on wet insulators using the hot plate and three-layer methods have shown that mass transfer, although slower than heat transfer, alters the temperature evolution (relative to that obtained on a dry medium) from the start of the heating and leads to an incorrect estimation of the values of the thermal properties in this case. For much heavier materials containing less water, a hot plate-type method may be acceptable. The criterion for determining whether a transient method is appropriate or not is the study of residues: if they are signed (not flat) as in Figure 5.17(b), then the obtained result is not reliable.

It is therefore preferable to measure the thermal conductivity with a steady-state method of the centered hot plate type with the entire device placed in an enclosure in which both the temperature and the relative humidity of the air can be adjusted. The time taken to reach equilibrium is much longer than with a dry material because of the low value of the mass diffusivity.

The specific heat c_{dry} of the dry material is measured by differential scanning calorimetry. If we characterize the moisture content of a material by its dry basis water content X ($\text{kg}_{\text{water}}.\text{kg}_{\text{dry}}^{-1}$) defined by:

$$X = \frac{m_{\text{water}}}{m_{\text{dry}}} \quad [6.1]$$

where

m_{water} is the mass of water contained in a volume V of wet solid;

m_{dry} is the mass of dry matter contained in the same volume.

We can deduce the value of its specific heat c by a simple additivity law [BAL 12]:

$$c = \frac{c_{dry} + Xc_{water}}{1 + X} \quad [6.2]$$

NOTE.—

On the contrary, the thermal conductivity of a wet medium cannot be calculated by an additivity law. It has been shown experimentally that a small increase in water content can sometimes cause a significant increase in thermal conductivity. Water can in fact be placed at the level of contacts between grains or fibers and thus greatly reduce the thermal resistance of a material [BAL 13].

6.1.4. What if the material is semi-transparent?

In some cases, when it is not perfectly opaque, the material can transmit radiation, and this radiation flux will be added to the conduction flux and will disturb the measurement since the thermal conductivity is a characteristic of the conduction:

$$\phi_c = -\lambda \text{ grad}(T)$$

where ϕ_c is the conduction flux and not the total flux (conduction + radiation).

A material that is not completely opaque is said to be “semi-transparent”. There are three types of semi-transparent media [DEG 02, MAI 00]:

- purely absorbing medium;
- purely scattering medium;
- medium that is both absorbing and scattering (participating medium).

To be purely scattering, a medium must have very little solid material (low density), as is the case for light insulators (glass wool, aerogel, etc.). Conversely, mass materials such as electrical insulators and semiconductors are purely absorbing (PVC, glass, silicon, etc.).

In practice, to detect a semi-transparency problem, it is necessary to carry out two measurements at different thicknesses, if the obtained conductivities are different, this is certainly a semi-transparency problem. In transient experiments, signed estimation residues may also be a sign of semi-transparency.

In a steady state, on a single measurement, it is not possible to separate the radiation contribution from the conduction one; the measurement will give an apparent conductivity taking into account the two contributions. In transient conditions, in some cases, it is possible to separate the contributions, the conductive transfer and the radiative transfer behaving in different ways in transient mode.

6.1.4.1. Purely scattering media

In this case there is no coupling between the conduction and the radiation fluxes. Figure 6.1 describes the steady state (a) and transient (b) thermal models.

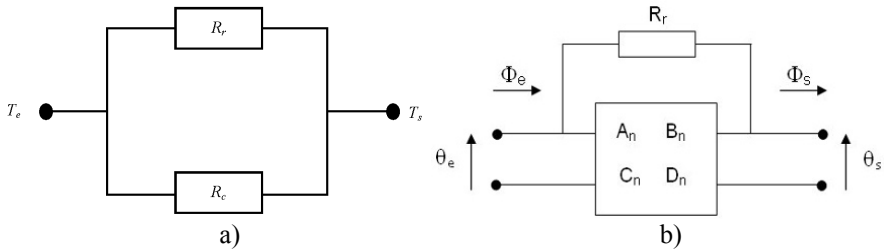


Figure 6.1. *Electrical equivalent diagrams for purely scattering media: a) steady-state mode; b) transient mode*

With: $R_c = \frac{e}{\lambda_c S}$; $R_r = \frac{e}{\lambda_r S}$

and:
$$\lambda_r = \frac{4n^2\sigma T^3 e}{\frac{1}{\varepsilon_1} + \frac{1}{\varepsilon_2} - 1 + K\beta e} \quad [6.3]$$

where:

n is the medium's optical index (for light porous media $n = 1$);

σ is Stefan's constant;

e is the medium's thickness;

β is the extinction coefficient (βe is the optical thickness of the medium);

$\varepsilon_1, \varepsilon_2$ is the emission coefficients of the sample's limit;

K is the function of βe which varies from 3/4 (Deissler's approximation for optically thick media) to 1 ("two fluxes" approximation for optically thin media).

In a steady-state experiment, the radiation conductivity λ_r is added to the phonic conductivity λ_c , i.e.:

$$\lambda = \lambda_c + \lambda_r$$

Note: the radiation conductivity increases with the thickness of the sample.

6.1.4.2. *Purely absorbing media*

Two cases can be modeled simply, optically thick media ($\beta e > 10$) and optically thin media ($\beta e < 0.1$):

– Optically thick media:

- the media follow the Rosseland law, i.e. the radiation transfer behaves like the conduction transfer (diffusion mechanism), with a radiative conduction λ_r which is added to the phonic conductivity λ_c , namely: $\lambda = \lambda_c + \lambda_r$

where
$$\lambda_r = \frac{16n^2\sigma T^3}{3\beta} \quad [6.4]$$

– Optically thin media:

- the heat transfers are in parallel as for the purely scattering media, and the radiation conductivity is calculated by equation [6.3] with $K = 1$.

– In the general case, conduction and radiation heat transfers are coupled and modeling is more complex [DEG 02, MAI 00].

In practice, as we have already pointed out:

– in steady-state measurement techniques, conduction and radiation contributions cannot be separated in a single experiment. It is necessary to carry out several measurements with different thicknesses and extrapolate for $e = 0$;

– in transient mode, the radiation can be taken into account initially by placing a resistance in parallel on the conductive quadrupole (Figure 6.1(b)), a resistance that will be identified at the same time as the thermal diffusivity and heat losses.

In some cases, the problem cannot be simplified and a system of coupled equations must be written and solved [SCH 12, NGU 08, MAI 00, REM 06].

6.2. Choice of method

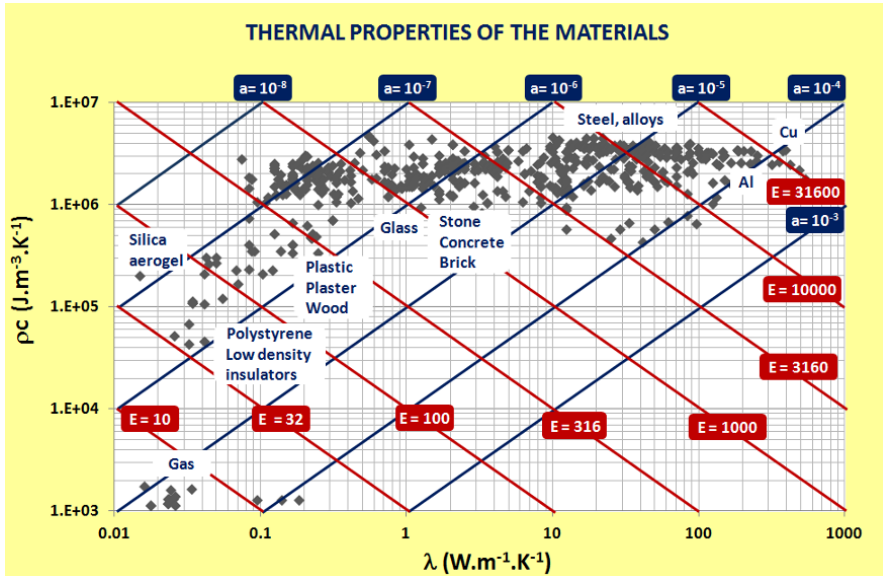


Figure 6.2. Representation of the thermal properties of materials [EDW 69]. For a color version of the figure, see www.iste.co.uk/jannot/thermal.zip

Representative points of a large number of materials in a system of axes (thermal conductivity λ vs. volumic heat capacity ρc) have been shown in Figure 6.2. There is a very wide variety of thermal properties: λ varying between 0.015 and 400 $\text{W m}^{-1} \text{K}^{-1}$ and ρc varying between 10^3 and $4 \times 10^6 \text{ J m}^{-3} \text{K}^{-1}$, so that there is no method for accurately measuring the thermal properties of all materials. The choice of measurement method depends on the range of thermal properties to be measured: we do not measure the thermal conductivity of a superinsulator ($\lambda < 0.026 \text{ W m}^{-1} \text{K}^{-1}$) with the same method used in the case of a metal ($\lambda > 10 \text{ W m}^{-1} \text{K}^{-1}$).

6.2.1. Consolidated solid

Taking into account the characteristics of each of the methods detailed in the preceding chapters, we have made recommendations concerning the thermal

characterization methods adapted to the different thermal conductivity ranges of solids. Table 6.1 summarizes the results.

Whenever possible, several different methods will be used to measure the same thermal property: this is often where the problems begin, but it is often the only way to be confident in the accuracy of its measurement. For example, to measure the thermal properties of a plastic, the following measurements can be carried out:

- hot plate on semi-infinite medium to estimate E ;
- hot wire to estimate λ ;
- differential scanning calorimetry to estimate c and separate measurement of ρ .

The combination of the first two measurements makes it possible to calculate $\rho c = \frac{E^2}{\lambda}$, a value that will be compared with that measured by differential scanning calorimetry. The difference between the two values will also indicate the precision that can be given to the measurement. A deviation of more than 5% should lead to questions about the measurements carried out and to an analysis of any measurement errors or biases whose possible causes have been analyzed in Chapter 2. A case study illustrating this approach can be found in Appendix 10.

Conductivity range (W.m ⁻¹ .K ⁻¹)	Recommended methods	Measured quantities	Notes
$\lambda > 5$	Flash 1D	a	
	Flash 3D	a	Suitable for anisotropic materials
	Differential scanning calorimetry	c	
	Hot wire	λ	Semi-infinite medium condition to be verified
$5 > \lambda > 0.5$	Centered hot plate isolated rear face	$\lambda, \rho c$	1D transfer condition to be verified
	Flash 1D	a	
	Flash 3D	a	Suitable for anisotropic materials
	Differential calorimetry	c	
	Hot wire	λ	Semi-infinite medium condition to be verified
	Hot disc	λ, a	Weak precision on the value of a

$0.5 > \lambda > 0.05$	Centered hot plate constant T_{rear}	λ	
	Permanent hot strip	λ	Suitable for anisotropic materials
	Differential calorimetry	c	
	Guarded hot plate	λ	Reference method
	Flash 1D	a	
	Flash 3D	a	Suitable for anisotropic materials
	Hot wire	λ	Semi-infinite medium condition to be verified
	Hot disc	λ, a	Weak precision on the value of a
$\lambda < 0.05$	Differential calorimetry	c	
	Guarded hot plate	λ	Reference method
	Centered hot plate constant T_{rear}	E, λ	
	Permanent hot strip	λ	Suitable for anisotropic materials
	Three-layer	λ, a	For light insulators of small dimensions
	Hot wire	λ	Semi-infinite medium condition to be verified
	Hot disc	λ, a	Weak precision on the value of a

Table 6.1. *Recommended methods for measuring the thermal properties of a solid (as a function of conductivity)*

6.2.2. Liquids

With regard to the measurement of the thermal properties of liquids, the most reliable methods seem to be the following:

- differential scanning calorimetry to estimate c and separate measurement of ρ ;
- hot tube method for estimating λ .

The hot wire method is more difficult to use because it is difficult to know when convection occurs. Moreover, it must be ensured that the influence of the radius and the mass of the wire was already negligible before this moment in order to make it possible to measure the unbiased thermal conductivity.

6.2.3. Powders

The cylindrical three-layer method makes it possible to measure all the thermal properties of “light” bulk materials and the thermal diffusivity of heavier bulk materials. It can only be used on dry materials.

For heavy and dry materials, the measurement of thermal diffusivity may be completed:

- by measuring the thermal effusivity using the asymmetric hot plate method;
- by measuring specific heat by calorimetry (DSC).

For wet materials, the hot tube method in steady state will be used to measure the thermal conductivity. The specific heat of the dry material will also be measured by calorimetry and the volumic heat capacity ρc of the wet material will be deduced by an additivity law. All the thermal properties can then be deduced as a function of the water content.

6.2.4. Thin films

To measure the thermal conductivity of thin films, the hot strip method is used in periodic regime (3ω) or in transient regime.

Analogies Between Different Transfers

In the following, we will show the analogies that exist between certain types of transfers, namely:

- heat transfer;
- mass transfer;
- flow of a fluid in a porous medium.

Of all these fields, the thermal field is undoubtedly the one which has generated the widest variety of characterization methods; the presentation that has been made in this book is not exhaustive. The analogy between the equations governing these different transfers and the physical quantities that characterize them can make it possible to transpose certain thermal characterization methods to other fields, as we will see in an example.

7.1. Diffusion of heat by conduction

As seen in the first chapter, heat transfer by conduction is governed by Fourier's law:

$$\varphi = -\lambda S \frac{\partial T}{\partial x} \quad [7.1]$$

where λ is the thermal conductivity ($\text{W m}^{-1} \text{K}^{-1}$);

S is the flux cross-section (m^2).

The temperature within a conductive medium in transient state verifies the heat equation:

$$a \nabla^2 T = \frac{\partial T}{\partial t} \quad [7.2]$$

The ratio $a = \frac{\lambda}{\rho c}$ is called the thermal diffusivity.

Initial and boundary conditions can take one of the following forms:

System boundary conditions:

– If the resistance to heat transfer at the surface is negligible:

$$T_s = T_\infty$$

– In the opposite case:

$$-\lambda \left(\frac{\partial T}{\partial x} \right)_s = h (T_s - T_\infty)$$

where: h is the convection heat transfer coefficient at the interface ($\text{W m}^{-2} \text{K}^{-1}$);

T_∞ is the temperature of the fluid in contact with the solid ($^\circ\text{C}$).

– If the medium is semi-infinite:

$$\lim_{x \rightarrow \infty} T(x, t) = T(x, t = 0)$$

– If the medium is a wall of thickness e with the same transfer on both sides, the symmetry imposes:

$$\left(\frac{\partial T}{\partial x} \right)_{x=e/2} = 0$$

7.2. Diffusion of water vapor

Let us consider by way of example the unidirectional transfer of vapor inside a porous medium as represented in Figure 7.1.

The mass transfer is governed by Fick's first law which is written as:

$$\varphi_m = -\rho_{dry} D S \frac{\partial X}{\partial x} \quad [7.3]$$

where: φ_m is the mass flow rate (kg s^{-1});

ρ_{dry} is the density of dry solid (kg m^{-3});

D is the mass diffusion coefficient ($\text{m}^2 \text{s}^{-1}$);

X is the dry basis water content ($\text{kg}_{\text{water}} \text{kg}_{\text{dm}}^{-1}$).

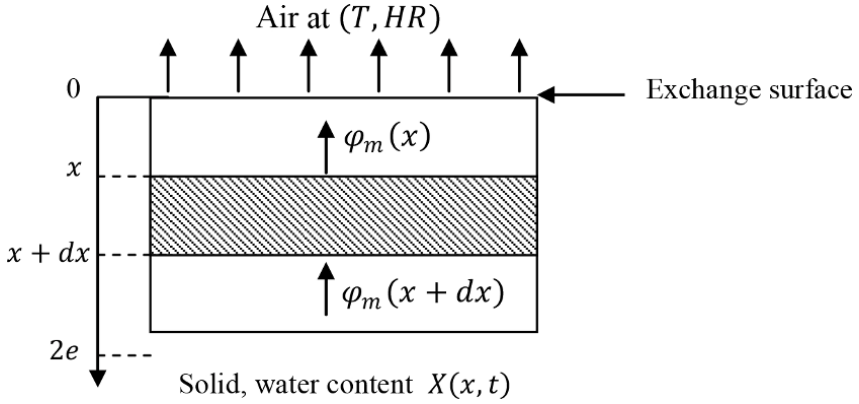


Figure 7.1. Schematic diagram of water vapor transfer

Let us carry out a mass balance between t and $t + dt$ on the volume element between x and $x + dx$:

$$[\varphi_m(x) - \varphi_m(x + dx)]dt = \rho_{dry} S dx dX$$

$$\text{or} \quad \rho_{dry} D S \left(\frac{\partial X}{\partial x} \right)_x - \rho_{dry} D S \left(\frac{\partial X}{\partial x} \right)_{x+dx} = \rho_{dry} S dx dX$$

Hence Fick's second law:

$$\frac{\partial}{\partial x} \left(D \frac{\partial X}{\partial x} \right) = \frac{\partial X}{\partial t} \quad [7.4]$$

This equation is easily generalizable to a three-dimensional transfer and, in the case where the diffusion coefficient D does not depend on X , it is written as:

$$D \nabla^2 X = \frac{\partial X}{\partial t} \quad [7.5]$$

System boundary conditions:

- If the resistance to mass transfer to the surface is negligible:

$$X_s = X_\infty$$

- In the opposite case:

$$-\rho_{dry} D \left(\frac{\partial X}{\partial x} \right)_s = h_m (X_s - X_\infty)$$

where: h_m is the mass transfer coefficient at the interface (kg m^{-2});

X_∞ is the dry basis water content of the solid in equilibrium with air at (T, HR) .

- If the surface is perfectly airtight:

$$\left(\frac{\partial X}{\partial x} \right)_s = 0$$

- If the medium is semi-infinite:

$$\lim_{x \rightarrow \infty} X(x, t) = X(x, t = 0)$$

7.3. Flow of a gas in a porous medium

Let us consider by way of example the unidirectional (1D) flow of a gas in a porous medium as represented in Figure 7.2.

The mass transfer is governed by Darcy's law which is written as:

$$\varphi_m = -\frac{k S}{v} \frac{\partial P}{\partial x} \quad [7.6]$$

where: φ_m is the mass flow rate (kg s^{-1});

k is the permeability of the solid (m^2);

v is the kinematic viscosity of the gas (Pa s);

P is the pressure (Pa).

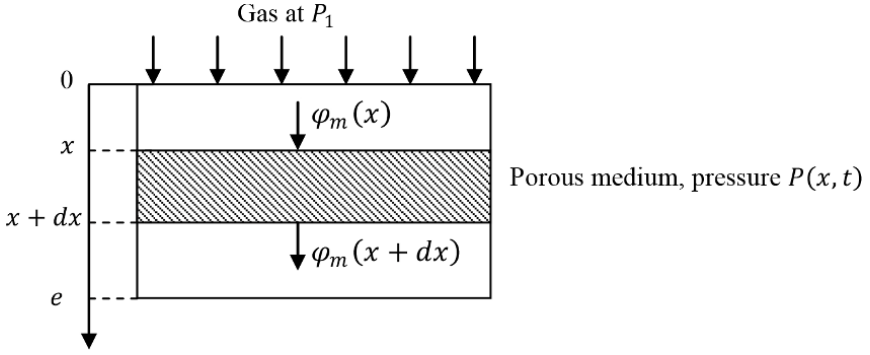


Figure 7.2. Diagram of the flow of a gas in a porous medium

Let us carry out a mass balance between t and $t + dt$ on the volume element between x and $x + dx$:

$$[\varphi_m(x) - \varphi_m(x + dx)] dt = \varepsilon S dx d\rho$$

where: ε is the solid porosity.

$$\text{Hence } -\frac{k}{\nu} \left(\frac{\partial P}{\partial x} \right)_x + \frac{k}{\nu} \left(\frac{\partial P}{\partial x} \right)_{x+dx} = \varepsilon dx \frac{\partial \rho}{\partial t}$$

$$\text{By using the law of perfect gases: } P = \rho \frac{R}{M} T$$

$$\text{we obtain: } \frac{\partial \rho}{\partial t} = \frac{M}{RT} \frac{\partial P}{\partial t}$$

and knowing that: $\nu = \frac{\mu}{\rho}$, we deduce:

$$P \nabla^2 P = \frac{\varepsilon \mu}{k} \frac{\partial P}{\partial t} \quad [7.7]$$

Two types of boundary conditions can be considered:

– In the case of contact of a surface with a gaseous volume, the resistance to mass transfer in a gas is negligible in comparison with that in a porous medium and

the pressure at the surface is equal to that of the surrounding medium: $P_s(t) = P_{ext}$. A gaseous volume can therefore be considered as a pure capacity.

- The surface can be sealed and in this case: $\left(\frac{\partial P}{\partial x}\right)_s = 0$.

If P_0 is the uniform pressure in a porous medium at time $t = 0$ and if we take a time interval in which $P(t)$ has varied slightly (a few %), we can write equation [7.7] in the following simplified form:

$$\nabla^2 P = \frac{\varepsilon \mu}{k P_0} \frac{\partial P}{\partial t} \quad [7.8]$$

7.4. Analogy between the different transfers

The equations governing the transfers of heat, water vapor and air have strong similarities highlighted in Table 7.1.

The evolution of the temperature, humidity and pressure of a gas inside a porous medium results from the various transfers that occur through its walls. The physical data characteristics of the wall that must be known to calculate the different flows through it are:

- thermal conductivity λ ($\text{W m}^{-2} \text{K}^{-1}$);
- thermal diffusivity a ($\text{m}^2 \text{s}^{-1}$);
- convective heat transfer coefficient h ($\text{W m}^{-2} \text{K}^{-1}$);
- diffusivity to water vapor D ($\text{m}^2 \text{s}^{-1}$);
- equilibrium water content X_{eq} ($\text{kg}_{\text{water}} \text{kg}_{\text{dm}}^{-1}$);
- density of dry material ρ_{dry} (kg m^{-3});
- mass transfer coefficient h_m (kg m^{-2});
- air permeability k (m^2);
- porosity ε .

Table 7.1 summarizes the equations governing these different modes of transfer and the resulting analogies in terms of physical properties.

Heat	Water vapor	Gas
Fourier's law $\varphi = -\lambda S \frac{\partial T}{\partial x}$	Fick's first law $\varphi_m = -\rho_{sec} D S \frac{\partial X}{\partial x}$	Darcy's law $\varphi_m = -\frac{k S}{v} \frac{\partial P}{\partial x}$
Heat equation $a \nabla^2 T = \frac{\partial T}{\partial t}$	Fick's second law $D \nabla^2 X = \frac{\partial X}{\partial t}$	If weak variation of P $\nabla^2 P = \frac{\varepsilon \mu}{k P_0} \frac{\partial P}{\partial t}$
Thermal conductivity λ	Mass conductivity $\rho_{sec} D$	Mass conductivity $\frac{k}{v}$
Thermal diffusivity a	Mass diffusivity D	Mass diffusivity $\frac{k P_0}{\varepsilon \mu}$
Thermal effusivity $E = \sqrt{\lambda \rho c} = \frac{\lambda}{\sqrt{a}}$	Mass effusivity $E_m = \rho_{dry} \sqrt{D}$	Mass effusivity $E_m = \sqrt{\frac{k \varepsilon \rho}{v P_0}}$
Heat capacity ρc	Mass capacity ρ_{sec}	Mass capacity $\frac{\varepsilon \rho}{P_0}$
Newton's law $\varphi = h (T_s - T_\infty)$	$\varphi_m = h_m (X_s - X_\infty)$	-

Table 7.1. Analogies between the different modes of transfers

7.5. Example of adaptation of a thermal method to another domain

From the data grouped in Table 7.1, it is possible to transpose a method designed to measure thermal properties to another transport phenomenon. However, it is important to take into account the differences between the studied transport modes from a practical point of view:

- it is possible to measure a temperature at a point (or even a temperature field with an IR camera) whereas we can only measure the mean value of the water content in a solid (by weighing) and the value of the pressure in a volume;

- thermal losses at the limits of a solid can never be neglected, whereas the losses of water vapor or fluid can be strictly zero (possibility of achieving a perfect fluid tightness).

From all these considerations, the concept of a thermal characterization device, the three-layer method [JAN 09], has been transposed to design a new device characterizing the properties related to the flow of a gas in a solid (porosity and permeability): the step decay method [LAS 11].

Figure 7.3 recalls the principle of the three-layer method: the sample to be characterized, initially at thermal equilibrium with its environment, is inserted between two brass discs (pure capacities). One of these capacities is excited thermally and the evolution of the temperatures T_1 and T_2 of the two plates is simultaneously recorded. The transfer function $F(\lambda, \rho c)$ between the two plates is modeled and the values of the parameters which minimize the sum of the quadratic deviations between T_{2exp} and $T_{2mod} = T_{1exp} \otimes F_{mod}(\lambda, \rho c)$ are estimated.

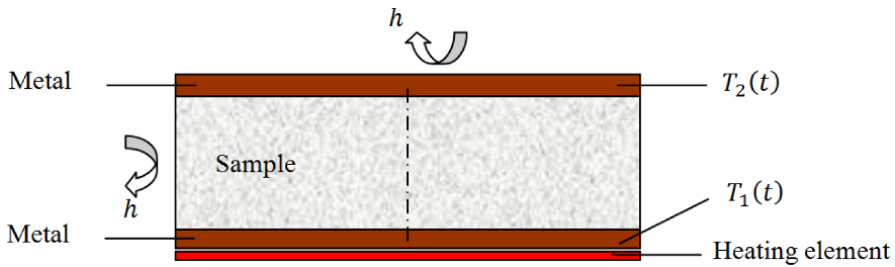


Figure 7.3. Schematic diagram of the three-layer method

We designed the step decay method from the analogies highlighted in Table 7.1:

- Potential: T or P .
- Conductivity: λ or $\frac{k}{v}$.
- Capacity: ρc or $\frac{\varepsilon \rho}{P_0}$.
- Pure capacity: thin conductive plate or volume of air.

Its schematic diagram is given in Figure 7.4.

The cylindrical sample is surrounded by a silicone sleeve on which a high gas pressure is applied to ensure the sealing on the lateral face. At the initial time, volumes V_0 and V_1 are at pressure P_{0i} . The valves V_e and V_c are closed and the pressure in V_0 is set to a value P_0 greater than P_{0i} . The valve V_e is then opened and the pressure changes of $P_0(t)$ and $P_1(t)$ are recorded.

As in the three-layer method, the transfer function $F(k, \varepsilon)$ between the two pressures P_0 and P_1 is modeled and the values of the parameters are estimated which make it possible to minimize the sum of the quadratic deviations between P_{1exp} and $P_{1mod} = P_{0exp} \otimes F_{mod}(k, \varepsilon)$.

In this application, the permeability depends on the pressure according to the Klinkenberg law:

$$k = k_l \left(1 + \frac{b}{p} \right) \quad [7.9]$$

therefore the calculation of P_{1mod} is carried out numerically by a 1D finite differences method and several pressure steps are successively applied in order to obtain a greater sensitivity to the parameter b . Figure 7.5 shows an example of experimental pressures P_0 and P_1 .

The approach adopted for the development of this set-up could be applied to other thermal characterization methods.

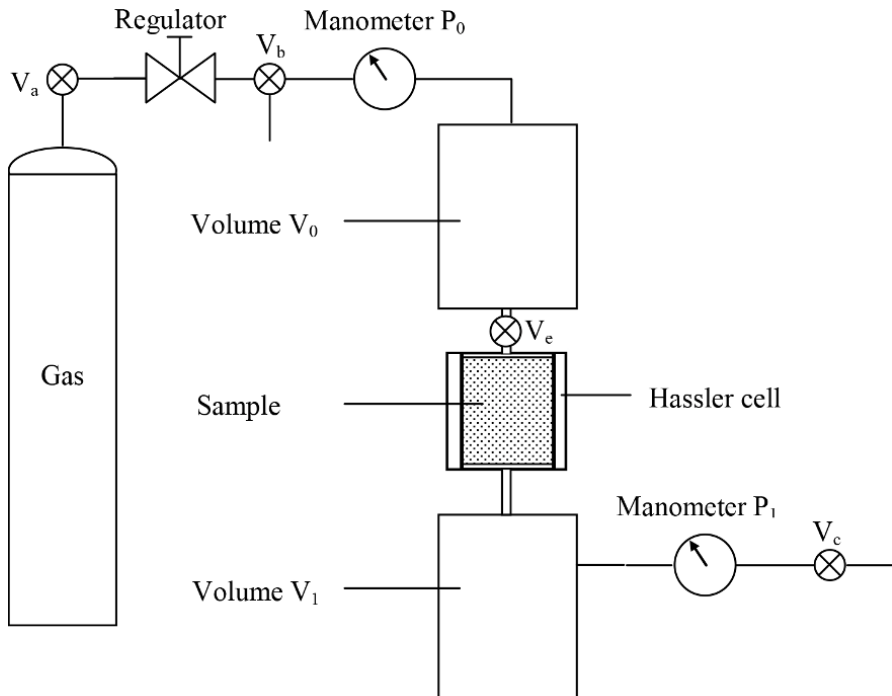


Figure 7.4. Schematic diagram of the step decay set-up

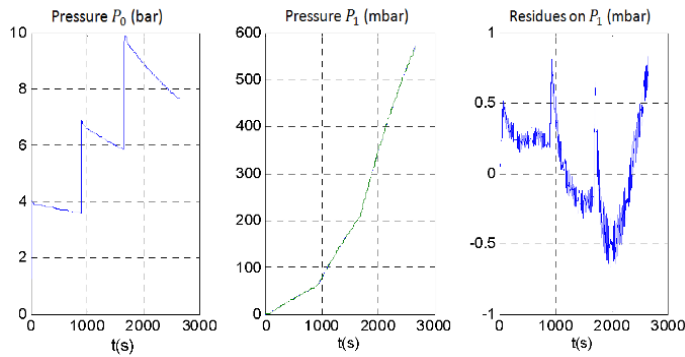


Figure 7.5. Experimental and estimation results on a wood sample using the step decay method. a) Evolution of P_0 (three pulses); b) evolution of P_1 measured and resulting from the estimation; c) residues on $P_1(t)$ after estimation

Appendices

Appendix 1

Physical Properties of Some Materials

	ρ kg m ⁻³	c J kg ⁻¹ K ⁻¹	λ W m ⁻¹ K ⁻¹
Metals and alloys			
Carbon steel	7833	465	54
Stainless steel 15% Cr, 10% Ni	7864	460	20
Stainless steel 18% Cr, 8% Ni	7816	460	16.3
Stainless steel 25% Cr, 20% Ni	7864	460	13
Alumine			29
Aluminum	2707	896	204
Silver	10525	234	407
Bronze 75% Cu, 25% Sn	8800	377	188
Bronze 92% Cu, 8% Al	7900	377	71
Carbon graphite	2250	707	147
Silicon carbide			13
Chromium	7150	450	94
Constantan 60% Cu, 40% Ni	8922	410	22.7
Copper	8954	383	386
Cupronickel 70% Cu, 30% Ni	8900	377	29.3
Duralumin	2787	883	164
Tin	7304	226	64
Iron	7870	452	73
Cast iron	7849	460	59
Brass 70% Cu, 30% Zn	8522	385	111
Magnesium	1740	1004	151
Gold	19300	128	312
Platinum	21400	140	69

	ρ kg m ⁻³	c J kg ⁻¹ K ⁻¹	λ W m ⁻¹ K ⁻¹
Metals and alloys			
Lead	11373	130	35
Liquid sodium	930	1381	84.5
Titanium	4500	523	20.9
Tungsten	19350	134	163
Zinc	7144	384	112
Diverse materials			
Asbestos	575	1046	0.15
Asphalt	2115	920	0.062
Rubber (natural)	1150		0.28
Rubber (vulcanized)	1100	2010	0.13
Cardboard	86	2030	0.048
Leather	998		0.159
Ice	920	2040	1.88
Plexiglass	1190	1465	0.19
Porcelain	2400	1088	1.035
Polyethylene	929	1830	0.46
PVC	1459	930	0.19
Sand	1515	800	0.2–1.0
Teflon	2170	1004	0.25
Earth, dry	1900	2000	2
Earth, wet	1500	1900	1
Glass	2300	837	1.05
Pyrex glass	2220	728	1.13

	ρ kg m ⁻³	c J kg ⁻¹ K ⁻¹	λ W m ⁻¹ K ⁻¹
Building materials			
Slate	2400	879	2.2
Basalt	2850	881	1.6
Cavernous concrete	1900	879	1.4
Solid concrete	2300	878	1.75
Bitumen (cardboard)	1050	1305	0.23
Low density wood	525	1250 (dry)	0.12
Medium density wood	675	1250 (dry)	0.15
High density wood	800	1250 (dry)	0.23
Clay brick	1800	878	1.15
Hard limestone	2450	882	2.4
Soft limestone	1650	879	1
Floor tile	2400	875	2.4
Okoumé plywood	400	1600	0.12
Pine plywood	500	1600	0.15
Granite	2600	881	3
Gravel (bulk)	1800	889	0.7
Sandstone	2500	880	2.6
Washed sand	2350	881	1.1
Marble	2700	881	2.5
Plaster	1440	840	0.48
Schist	2400	879	2.2

	ρ kg m ⁻³	c J kg ⁻¹ K ⁻¹	λ W m ⁻¹ K ⁻¹
Insulating materials			
Balsa	140		0.054
Cotton	80	1300	0.06
Kapok			0.035
Rock wool	20	880	0.047
	55	880	0.038
	135	880	0.041
Glass wool	8	875	0.051
	10	880	0.045
	15	880	0.041
	40	880	0.035
Expanded cork	120	2100	0.044
Carpet	200	1300	0.06
Polyurethane (foam)	32	1300	0.03
	50	1360	0.035
	85	1300	0.045
PVC (rigid foam)	30	1300	0.031
	40	1300	0.041
Expanded polystyrene	12	1300	0.047
	14	1300	0.043
	18	1300	0.041
Styrofoam	30		0.032

Appendix 2

Physical Properties of Air and Water

Properties of saturated water							Properties of air at 1 atm						
<i>T</i>	ρ	<i>c</i>	λ	$10^6 \cdot \mu$	$10^7 \cdot \alpha$	<i>Pr</i>	<i>T</i>	ρ	<i>c</i>	λ	$10^5 \cdot \mu$	$10^7 \cdot \alpha$	<i>Pr</i>
°C	kg m ⁻³	J kg ⁻¹ K ⁻¹	W m ⁻¹ K ⁻¹	Pa s ⁻¹	m ² s ⁻¹		°C	kg m ⁻³	J kg ⁻¹ K ⁻¹	W m ⁻¹ K ⁻¹	Pa s ⁻¹	m ² s ⁻¹	
0	1002	4218	0.552	17.90	1.31	13.06	0	1.292	1006	0.0242	1.72	1.86	0.72
20	1001	4182	0.597	10.10	1.43	7.02	20	1.204	1006	0.0257	1.81	2.12	0.71
40	995	4178	0.628	6.55	1.51	4.34	40	1.127	1007	0.0272	1.90	2.40	0.70
60	985	4184	0.651	4.71	1.55	3.02	60	1.059	1008	0.0287	1.99	2.69	0.70
80	974	4196	0.668	3.55	1.64	2.22	80	0.999	1010	0.0302	2.09	3.00	0.70
100	960	4216	0.680	2.82	1.68	1.74	100	0.946	1012	0.0318	2.18	3.32	0.69
120	945	4250	0.685	2.33	1.71	1.45	120	0.898	1014	0.0333	2.27	3.66	0.69
140	928	4283	0.684	1.99	1.72	1.24	140	0.854	1016	0.0345	2.34	3.98	0.69
160	910	4342	0.680	1.73	1.73	1.10	160	0.815	1019	0.0359	2.42	4.32	0.69
180	889	4417	0.675	1.54	1.72	1.00	180	0.779	1022	0.0372	2.50	4.67	0.69
200	867	4505	0.665	1.39	1.71	0.94	200	0.746	1025	0.0386	2.57	5.05	0.68
220	842	4610	0.652	1.26	1.68	0.89	220	0.700	1028	0.0399	2.64	5.43	0.68
240	816	4756	0.635	1.17	1.64	0.88	240	0.688	1032	0.0412	2.72	5.80	0.68
260	786	4949	0.611	1.08	1.58	0.87	260	0.662	1036	0.0425	2.79	6.20	0.68
280	753	5208	0.580	1.02	1.48	0.91	280	0.638	1040	0.0437	2.86	6.59	0.68
300	714	5728	0.540	0.96	1.32	1.02	300	0.616	1045	0.0450	2.93	6.99	0.68

Correlations between 0 and 100 °C (T temperature in °C)**For air**

$$\rho = \frac{353}{T+273} \quad \text{kg m}^{-3}$$

$$c_p = 1008 \quad \text{J kg}^{-1} \text{ K}^{-1}$$

$$\lambda = 7.57 \times 10^{-5} T + 0.0242 \quad \text{W m}^{-1} \text{ K}^{-1}$$

$$\mu = 10^{-5}(0.0046 T + 1.7176) \quad \text{kg m}^{-1} \text{ s}^{-1}$$

$$a = 10^{-5}(0.0146 T + 1.8343) \quad \text{m}^2 \text{ s}^{-1}$$

$$Pr = -2.54 \times 10^{-4} T + 0.7147$$

$$\beta \approx \frac{1}{T+273} \quad \text{K}^{-1}$$

For water

$$\rho = -0.00380 T^2 - 0.0505 T + 1002.6 \quad \text{kg m}^{-3}$$

$$c_p = 4180 \quad \text{J kg}^{-1} \text{ K}^{-1}$$

$$\lambda = -9.87 \times 10^{-6} T^2 + 2.238 \times 10^{-3} T + 0.5536 \quad \text{W m}^{-1} \text{ K}^{-1}$$

$$\mu = 10^{-4} \frac{0.0003354 T^2 - 0.07377 T + 17.9}{8.765 \cdot 10^{-5} T^2 + 0.03032 T + 1} \quad \text{kg m}^{-1} \text{ s}^{-1}$$

$$a = 10^{-7}(-0.00360 T + 1.340) \quad \text{m}^2 \text{ s}^{-1}$$

$$Pr = \frac{-0.0037 T^2 + 1.387 T + 13.06}{0.005297 T^2 + 0.1241 T + 1}$$

$$\frac{g\beta\rho^2c}{\lambda\mu} = (0.0105 T^2 + 0.477 T - 0.0363) \times 10^9 \quad \text{K}^{-1} \text{ m}^{-3}$$

$$\log_{10}[p_{\text{sat}}(T + 273)] = 20.3182 - \frac{2795}{T+273}$$

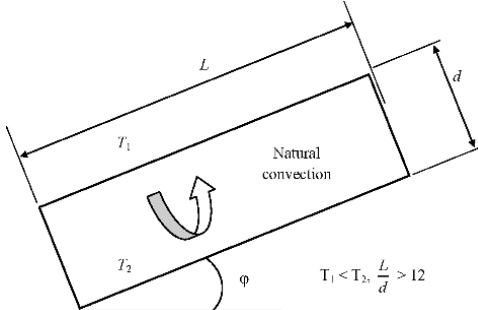
$$-3.868 \log_{10}(T + 273) \quad \text{mmHg} \quad -50^\circ\text{C} < T < 200^\circ\text{C}$$

$$L_v = 2495 - 2.346 T \quad \text{kJ kg}^{-1} \quad 0^\circ\text{C} < T < 100^\circ\text{C}$$

Appendix 3

Transfer Coefficients in Natural Convection

<i>Correlations valid for all fluids: $Nu = C (GrPr)^m$ (according to [HOL 90])</i>			
Geometry	$GrPr$	C	m
Vertical cylinders and plates	10^4-10^9	0.59	1/4
	10^9-10^{13}	0.1	1/3
Horizontal cylinders	$10^{-10}-10^{-2}$	0.675	0.058
	$10^{-2}-10^2$	1.02	0.148
	10^2-10^4	0.850	0.188
	10^4-10^7	0.480	0.25
	10^7-10^{12}	0.125	0.33
Upper face of a hot plate or lower face of a cold plate	$2 \times 10^4-8 \times 10^6$	0.54	0.25
	$8 \times 10^6-10^{11}$	0.15	0.33
Lower face of a hot plate or upper face of a cold plate	10^5-10^{11}	0.27	0.25

Inclined rectangular closed cell  $T_1 < T_2, \frac{L}{d} > 12$	$Nu = 1 + 1.44 \left[1 - \frac{1708}{GrPr \cos(\varphi)} \right]^* \left\{ 1 - \frac{1708[\sin(1.8\varphi)^{1.6}]}{GrPr \cos(\varphi)} \right\} + \left[\frac{GrPr \cos(\varphi)}{5830} \right]^{1/3} - 1 \Bigg]^*$ If: $0 < \varphi < \tan^{-1}(4800 Pr)$ The quantities * are taken equal to 0 if the result of their calculation leads to a negative number [HOL 75]	
<i>Simplified equations for air at atmospheric pressure and at 20°C</i>		
Geometry	Laminar $10^4 < GrPr < 10^9$	Turbulent $GrPr > 10^9$
Vertical cylinders and plates	$h = 1.54 \left(\frac{\Delta T}{L} \right)^{1/4}$	$h = 1.22(\Delta T)^{1/3}$
Horizontal cylinder	$h = 1.26 \left(\frac{\Delta T}{D} \right)^{1/4}$	$h = 1.52(\Delta T)^{1/3}$
The upper face of a hot plate or the lower face of a cold plate	$h = 1.41 \left(\frac{\Delta T}{L} \right)^{1/4}$	$h = 1.83(\Delta T)^{1/3}$
The lower face of a hot plate or the upper face of a cold plate	$h = 0.71 \left(\frac{\Delta T}{L} \right)^{1/4}$	$h = 0.71 \left(\frac{\Delta T}{L} \right)^{1/4}$

Appendix 4

Main Integral Transformations: Laplace, Fourier and Hankel

A4.1. Laplace transform

A4.1.1. Definition

$L[T(t)] = \theta(p) = \int_0^{\infty} \exp(-pt) T(t) dt$ and $L^{-1}[\theta(p)] = T(t)$ (Inverse transform)

A4.1.2. Properties

Linearity: $L[a_1 T_1(t) + a_2 T_2(t)] = a_1 L[T_1(t)] + a_2 L[T_2(t)]$, same for L^{-1}

Translation: $L[\exp(at)T(t)] = \theta(p - a)$; $L^{-1}[\theta(p - a)] = \exp(at)T(t)$

$L^{-1}[\exp(-ap)\theta(p)] = T(t - a)$ if $t > a$

$L^{-1}[\exp(-ap)\theta(p)] = 0$ if $t \leq a$

Change of scale: $L[T(at)] = \frac{1}{a} \theta\left(\frac{p}{a}\right)$; $L^{-1}[\theta(ap)] = \frac{1}{a} T\left(\frac{t}{a}\right)$

Derivation: $L[T'(t)] = p\theta(p) - T(0)$; $L^{-1}[\theta^{(n)}(p)] = (-1)^n t^n T(t)$

$L[T''(t)] = p^2 \theta(p) - pT(0) - T'(0)$

Integration: $L\left[\int_0^t T(u) du\right] = \frac{\theta(p)}{p}$; $L^{-1}\left[\int_p^{\infty} \theta(u) du\right] = \frac{T(t)}{t}$

Multiplication by t^n : $L[t^n T(t)] = (-1)^n \theta^{(n)}(p)$; $L^{-1}[p\theta(p)] = T'(t) - T(0)\delta(t)$

Division by t : $L\left[\frac{T(t)}{t}\right] = \int_p^\infty \theta(u)du$; $L^{-1}\left[\frac{\theta(p)}{p}\right] = \int_0^t T(u)du$

Periodic functions (Period P): $L[T(t)] = \frac{\int_0^P \exp(-pt)T(t)dt}{1-\exp(-pP)}$

A4.2. Complex Fourier transform

A4.2.1. Definition

$$F[T(x)] = \theta(\omega) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} e^{-i\omega x} T(x) dx$$

$$T(x) = F^{-1}[\theta(\omega)] = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} e^{i\omega x} \theta(\omega) d\omega$$

A4.2.2. Properties

$$F\left[\frac{\partial T}{\partial x}\right] = i\omega\theta(\omega)$$

$$F\left[\frac{\partial^2 T}{\partial x^2}\right] = -\omega^2\theta(\omega)$$

A4.3. Fourier transform in sine and cosine

A4.3.1. Definitions

Sine

$$F_s[T(x)] = \theta_s(\omega) = \sqrt{\frac{2}{\pi}} \int_0^\infty T(x) \sin(\omega x) dx$$

$$T(x) = F_s^{-1}[\theta_s(\omega)] = \sqrt{\frac{2}{\pi}} \int_0^\infty \theta_s(\omega) \sin(\omega x) d\omega$$

Cosine

$$F_c[T(x)] = \theta_c(\omega) = \sqrt{\frac{2}{\pi}} \int_0^\infty T(x) \cos(\omega x) dx$$

$$T(x) = F_c^{-1}[\theta_c(\omega)] = \sqrt{\frac{2}{\pi}} \int_0^\infty \theta_c(\omega) \cos(\omega x) d\omega$$

A4.3.2. Properties

$$F_s \left[\frac{\partial T}{\partial x} \right] = -\omega \theta_c(\omega); F_c \left[\frac{\partial T}{\partial x} \right] = -\omega \theta_s(\omega) - \sqrt{\frac{2}{\pi}} T(0)$$

$$F_s \left[\frac{\partial^2 T}{\partial x^2} \right] = -\omega^2 \theta_s(\omega) + \omega \sqrt{\frac{2}{\pi}} T(0); F_c \left[\frac{\partial^2 T}{\partial x^2} \right] = -\omega^2 \theta_c(\omega) - \sqrt{\frac{2}{\pi}} \left(\frac{\partial T}{\partial x} \right)_{x=0}$$

A4.4. Finite Fourier transform in sine and cosine

A4.4.1. Definitions

If the temperature $T(x)$ is defined only on the interval $[0, L]$, we can use a finite Fourier transformation in sine or cosine:

$$F_s[T(x)] = \theta_s(n) = \int_0^L T(x) \sin\left(\frac{n\pi x}{L}\right) dx \quad \text{or} \quad F_c[T(x)] = \theta_c(n) = \int_0^L T(x) \cos\left(\frac{n\pi x}{L}\right) dx$$

$$T(x) = F_s^{-1}[\theta_s(n)] = \frac{2}{L} \sum_{n=1}^{\infty} \theta_s(n) \sin\left(\frac{n\pi x}{L}\right)$$

$$\text{or: } T(x) = F_c^{-1}[\theta_c(n)] = \frac{1}{L} \theta_c(0) + \frac{2}{L} \sum_{n=1}^{\infty} \theta_c(n) \cos\left(\frac{n\pi x}{L}\right)$$

A4.4.2. Properties

$$F_s \left[\frac{\partial^2 T}{\partial x^2} \right] = \frac{n\pi}{L} [T(0) - (-1)^n T(L)] - \frac{n^2 \pi^2}{L^2} \theta_s(n)$$

$$F_c \left[\frac{\partial^2 T}{\partial x^2} \right] = (-1)^n \left(\frac{\partial T}{\partial x} \right)_{x=L} - \left(\frac{\partial T}{\partial x} \right)_{x=0} - \frac{n^2 \pi^2}{L^2} \theta_c(n)$$

A4.5. Hankel transform of order ν

A4.5.1. Definition

For $\nu > -\frac{1}{2}$:

$$H_\nu[T(r)] = \theta_\nu(\sigma) = \int_0^\infty r J_\nu(\sigma r) T(r) dr; T(r) = H_\nu^{-1}[\theta_\nu(\sigma)] = \int_0^\infty \sigma J_\nu(\sigma r) \theta_\nu(\sigma) d\sigma$$

A4.5.2. Properties

$$H_\nu \left[\frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial T}{\partial r} \right) - \frac{\nu^2 T}{r^2} \right] = -\sigma^2 \theta_\nu(\sigma);$$

at order 0: $H_0[T(r) = T_i] = T_i \int_0^\infty r J_0(\sigma r) dr = T_i \frac{r}{\sigma} J_1(\sigma r)$

Appendix 5

Inverse Laplace Transformation

A5.1. Analytical method

The Laplace transform $\theta(p)$ of the function $T(t)$ is given by:

$$L[T(t)] = \theta(p) = \int_0^{\infty} \exp(-pt) T(t) dt.$$

There is no general analytical formula for calculating $T(t)$ knowing $\theta(p)$. However, we know the exact expression of $T(t)$ for some particular functions $\theta(p)$, examples of which can be found on the following page (see [SPI 90] for more complete tables). The use of these tables associated with the particular properties of the inverse Laplace transformation recalled in Appendix A2.2 can solve a number of cases. We will always try to decompose a complex function in the sum, product, and series of simple functions that are more easily invertible.

A5.2. Numerical methods

For those cases where an analytical solution cannot be found, one of the following two numerical methods can be used:

A5.2.1. Stehfest method

The inverse transform of the function $\theta(p)$ can be calculated by [STE 70]:

$$T(t) = \frac{\ln(2)}{t} \sum_{j=1}^N V_j \theta \left[\frac{j \ln(2)}{t} \right]$$

$N = 20$ (double precision):

$$V_1 = -5.511463844797178 \times 10^{-6} \quad V_2 = 1.523864638447972 \times 10^{-1}$$

$$V_3 = -1.174654761904762 \times 10^2 \quad V_4 = 1.734244933862434 \times 10^4$$

$$V_5 = -9.228069289021164 \times 10^5 \quad V_6 = 2.377408778710318 \times 10^7$$

$$V_7 = -3.494211661953704 \times 10^8 \quad V_8 = 3.241369852231879 \times 10^9$$

$$V_9 = -2.027694830723779 \times 10^{10} \quad V_{10} = 8.946482982379724 \times 10^{10}$$

$$V_{11} = -2.870209211471027 \times 10^{11} \quad V_{12} = 6.829920102815115 \times 10^{11}$$

$$V_{13} = -1.219082330054374 \times 10^{12} \quad V_{14} = 1.637573800842013 \times 10^{12}$$

$$V_{15} = -1.647177486836117 \times 10^{12} \quad V_{16} = 1.221924554444226 \times 10^{12}$$

$$V_{17} = -6.488065588175326 \times 10^{11} \quad V_{18} = 2.333166532137059 \times 10^{11}$$

$$V_{19} = -5.091380070546738 \times 10^{10} \quad V_{20} = 5.091380070546738 \times 10^9$$

$N = 10$ (single precision):

$$V_1 = \frac{1}{12}, \quad V_2 = -\frac{385}{12}, \quad V_3 = 1279, \quad V_4 = -\frac{46871}{3}, \quad V_5 = \frac{505465}{6},$$

$$V_6 = -\frac{473915}{2}, \quad V_7 = \frac{1127735}{3}, \quad V_8 = -\frac{1020215}{3}, \quad V_9 = \frac{328125}{2}, \quad V_{10} = -\frac{65625}{2}$$

A5.2.2. Fourier method

$$T(t) = \frac{\exp(ct)}{t_{\max}} \left(\frac{\theta(c)}{2} + \sum_{k=1}^{\infty} \{ \operatorname{Re}[\theta(c + j\omega_k)] \cos(\omega_k t) - \operatorname{Im}[\theta(c + j\omega_k)] \sin(\omega_k t) \} \right)$$

where: $\omega_k = \frac{k\pi}{t_{\max}}$

The infinite sum is in practice calculated for a finite number N of terms; we will generally take $N > 100$. This method requires choosing two parameters: c and $t_{\max}$. We must ensure *a posteriori* that: $\exp(-2ct_{\max})(2t_{\max}) \approx 0$.

A5.2.3. Choice of a method and verification of results

The Stehfest method is simple to implement because it does not require us to choose certain parameters. The Fourier method can lead to a better result in the case of inversion of certain functions such as, for example, periodic functions [MAI 00]. It is also possible to directly use the Matlab subprogram “Invlap” based on the De Hoog algorithm [DEH 82].

The study of the behavior of the function $\theta(p)$ at long times ($t \rightarrow \infty$ or $p \rightarrow 0$) and at short times ($t \rightarrow 0$ or $p \rightarrow \infty$) can lead to approximate formulas of $\theta(p)$ for which we can then find the inverse Laplace transform analytically. The comparison of these analytical solutions with the results of the numerical inversion gives an indication of the accuracy of the numerical inversion.

(According to Spiegel, [SPI 90])

$$q = \sqrt{\frac{p}{a}}$$

$\theta(p) = L [T(t)]$	$T(t)$	$\theta(p) = L [T(t)]$	$T(t)$
$\frac{1}{p}$	1	$\frac{\ln(p)}{p}$	$-\ln(t) - \gamma; \gamma = 0.57721$
1	$\delta(t)$ Dirac	$\frac{1}{\sqrt{p}}$	$\frac{1}{\sqrt{\pi t}}$
$\frac{1}{p + \beta}$	$e^{-\beta t}$	$\frac{1}{p\sqrt{p}}$	$\frac{2}{\sqrt{\pi}}\sqrt{t}$
$\frac{\omega^2}{p^2 + \omega^2}$	$\sin(\omega t)$	$\frac{\omega}{p^2 - \omega^2}$	$\sinh(\omega t)$
$\frac{p}{p^2 + \omega^2}$	$\cos(\omega t)$	$\frac{p}{p^2 - \omega^2}$	$\cosh(\omega t)$
$\frac{b}{p(b + \sqrt{p})}$	$1 - \exp(b^2 t) \operatorname{erfc}(b\sqrt{t})$	$\frac{1}{p^n} \quad n = 2, 3, \dots$	$\frac{t^{n-1}}{(n-1)!}$

$\theta(p) = L [T(t)]$	$T(t)$
e^{-qx}	$\frac{x}{2\sqrt{\pi at^3}} \exp\left(-\frac{x^2}{4at}\right)$
$\frac{e^{-qx}}{q}$	$\sqrt{\frac{a}{\pi t}} \exp\left(-\frac{x^2}{4at}\right)$
$\frac{e^{-qx}}{p}$	$\operatorname{erfc}\left(\frac{x}{2\sqrt{at}}\right)$
$\frac{e^{-qx}}{pq}$	$2\sqrt{\frac{a}{\pi t}} \exp\left(-\frac{x^2}{4at}\right) - x \operatorname{erfc}\left(\frac{x}{2\sqrt{at}}\right)$
$\frac{e^{-qx}}{p^2}$	$\left(t + \frac{x^2}{2a}\right) \operatorname{erfc}\left(\frac{x}{2\sqrt{at}}\right) - x \sqrt{\frac{t}{\pi a}} \exp\left(-\frac{x^2}{4at}\right)$
$\frac{e^{-qx}}{q+h}$	$\sqrt{\frac{a}{\pi t}} \exp\left(-\frac{x^2}{4at}\right) - ha \exp(hx + ath^2) \operatorname{erfc}\left(\frac{x}{2\sqrt{at}} + h\sqrt{at}\right)$
$\frac{e^{-qx}}{q(q+h)}$	$a \exp(hx + ath^2) \operatorname{erfc}\left(\frac{x}{2\sqrt{at}} + h\sqrt{at}\right)$
$\frac{e^{-qx}}{p(q+h)}$	$\frac{1}{h} \operatorname{erfc}\left(\frac{x}{2\sqrt{at}}\right) - \frac{1}{h} \exp(hx + ath^2) \operatorname{erfc}\left(\frac{x}{2\sqrt{at}} + h\sqrt{at}\right)$
$\frac{e^{-qx}}{pq(q+h)}$	$\frac{2}{h} \sqrt{\frac{a}{\pi t}} \exp\left(-\frac{x^2}{4at}\right) - \frac{1+hx}{h^2} \operatorname{erfc}\left(\frac{x}{2\sqrt{at}}\right) + \frac{1}{h^2} \exp(hx + ath^2) \operatorname{erfc}\left(\frac{x}{2\sqrt{at}} + h\sqrt{at}\right)$

Appendix 6

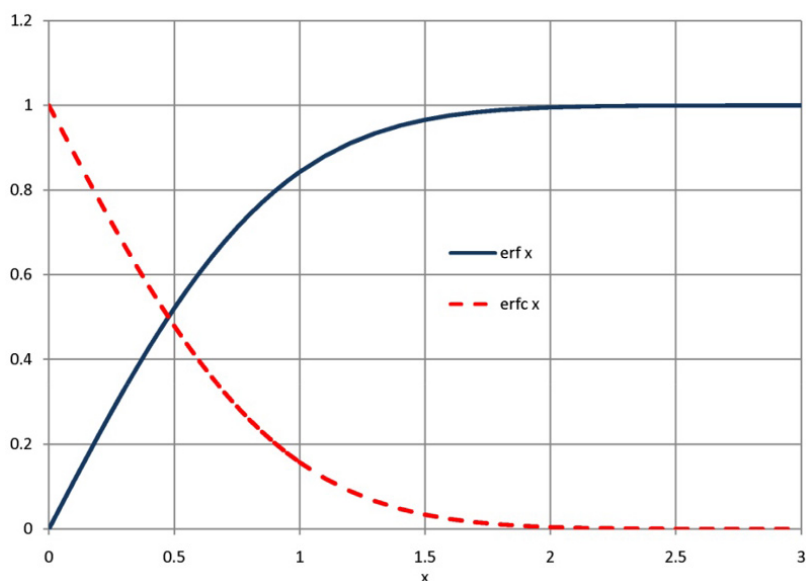
Value of the Function ERF

Properties:

$$\operatorname{erf}(u) = \frac{2}{\sqrt{\pi}} \int_0^u \exp(-t^2) dt ; \operatorname{erfc}(u) = 1 - \operatorname{erf}(u);$$

$$\operatorname{ierfc}(u) = \frac{1}{\sqrt{\pi}} \exp(-u^2) - u[1 - \operatorname{erf}(u)]; \quad \frac{d}{dx} [\operatorname{erf}(x)] = \frac{2}{\sqrt{\pi}} \exp(-x^2)$$

$$\text{For } x \rightarrow \infty : \operatorname{erfc}(x) = \frac{\exp(-x^2)}{\sqrt{\pi}} \left[\frac{1}{x} - \frac{1}{2x^3} + \frac{1 \times 3}{2^2 x^5} - \frac{1 \times 3 \times 5}{2^3 x^7} + \dots \right]$$



x	$erf(x)$	$erfc(x)$	$ierfc(x)$	x	$erf(x)$	$erfc(x)$	$ierfc(x)$
0	0.000000	1.000000	0.56419	1.1	0.880205	0.11980	0.03647
0.05	0.056372	0.943628	0.5156	1.2	0.910314	0.08969	0.02605
0.1	0.112463	0.887537	0.46982	1.3	0.934008	0.06599	0.01831
0.15	0.167996	0.832004	0.42684	1.4	0.952285	0.04772	0.01267
0.2	0.222703	0.777297	0.38661	1.5	0.966105	0.03390	0.00862
0.25	0.276326	0.723674	0.34909	1.6	0.976378	0.02362	0.00577
0.3	0.328627	0.671373	0.31422	1.7	0.983790	0.01621	0.0038
0.35	0.379382	0.620618	0.28193	1.8	0.989091	0.01091	0.00246
0.4	0.428392	0.571608	0.25213	1.9	0.992790	0.00721	0.00156
0.45	0.475482	0.524518	0.22473	2	0.995322	0.00468	0.00098
0.5	0.520500	0.479500	0.19964	2.1	0.997021	0.00298	0.0006
0.55	0.563323	0.436677	0.17675	2.2	0.998137	0.00186	0.00036
0.6	0.603856	0.396144	0.15594	2.3	0.998857	0.00114	0.00022
0.65	0.642029	0.357971	0.13709	2.4	0.999311	0.00069	0.00013
0.7	0.677801	0.322199	0.1201	2.5	0.999593	0.00041	0.0000
0.75	0.711156	0.288844	0.10483	2.6	0.999764	0.00024	0.00004
0.8	0.742101	0.257899	0.09117	2.7	0.999866	0.00013	0.00002
0.85	0.770668	0.229332	0.079	2.8	0.999925	0.00008	0.00001
0.9	0.796908	0.203092	0.0682	2.9	0.999959	0.00004	0.00000
0.95	0.820891	0.179109	0.05866	3	0.999978	0.00002	0.00000
1	0.842701	0.157299	0.05025				

Appendix 7

Quadrupole Matrices for Different Configurations

$$q = \sqrt{\frac{p}{a}}; I_0, I_1, K_0, K_1: \text{Bessel's functions, see Appendix 8}$$

A7.1. Quadrupole associated with unidirectional transfer in a medium without energy generation [MAI 00]

A7.1.1. Medium of finite thickness

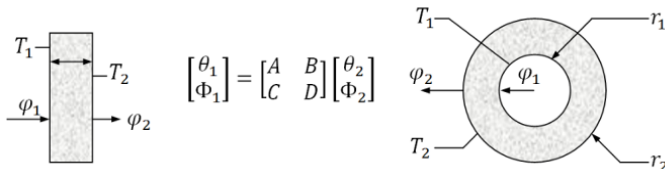


	Plate of thickness e	Hollow cylinder of radii r_1 and r_2	Hollow sphere of radii r_1 and r_2
A	$\cosh(qe)$	$qr_2[I_0(qr_1)K_1(qr_2) + I_1(qr_2)K_0(qr_1)]$	$\frac{r_2}{r_1} \cosh(s) - \frac{\sinh(s)}{qr_1}$
B	$\frac{\sinh(qe)}{\lambda q S}$	$\frac{1}{2\pi\lambda L} [I_0(qr_2)K_0(qr_1) - I_0(qr_1)K_0(qr_2)]$	$\frac{\sinh(s)}{4\pi\lambda qr_1 r_2}$
C	$\lambda q S \sinh(qe)$	$2\pi\lambda L q r_1 q r_2 \left[\begin{matrix} I_1(qr_2)K_1(qr_1) - \\ I_1(qr_1)K_1(qr_2) \end{matrix} \right]$	$4\pi\lambda r_2 \left[\begin{matrix} \left(1 - \frac{r_1}{r_2}\right) \cosh(s) + \\ \left(qr_1 - \frac{1}{qr_2}\right) \sinh(s) \end{matrix} \right]$
D	$\cosh(qe)$	$qr_1[I_0(qr_2)K_1(qr_1) + I_1(qr_1)K_0(qr_2)]$	$\frac{r_1}{r_2} \cosh(s) - \frac{\sinh(s)}{qr_2}$

Where: $s = q (r_2 - r_1)$.

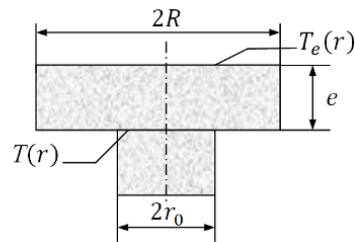
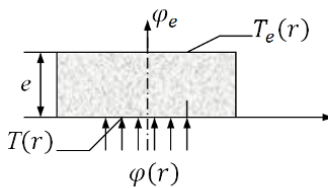
A7.1.2. Semi-infinite medium

The Laplace transform Φ of the heat flow rate φ is written as: $\Phi = \frac{\theta}{Z}$ with:

	Semi-infinite wall	Semi-infinite cylinder with inner radius r_1	Semi-infinite sphere with inner radius r_1
Z	$\frac{1}{ES\sqrt{p}}$	$\frac{1}{2\pi\lambda L} \frac{K_0(qr_1)}{qr_1 K_1(qr_1)}$	$\frac{1}{4\pi\lambda r_1(1 + qr_1)}$

where $E = \sqrt{\lambda\rho c}$ is the thermal effusivity.

A7.2. Quadrupole associated with constriction resistance [MAI 00]



Transfer of a flux $\varphi(r)$ to the surface of a cylinder of radius R and of thickness e , with $\varphi(r) = 0$ if $r > r_0$

	infinite R and e		finite R and e	
	$\Phi = \frac{\bar{\theta}_{0-r_0}}{R_c}$		$[\theta] = \begin{bmatrix} 1 & R_c \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta_e \\ \Phi_e \end{bmatrix}$	
	$T(r) = T_0$ for $r < r_0$	$\varphi(r) = \varphi_0$ for $r < r_0$	$T_e(r) = T_{e0}$	$\varphi_e(r) = \varphi_{e0}$
R_c	$\frac{1}{4\lambda r_0 \left(1 + \frac{\pi}{4} q r_0\right)}$	$\frac{8}{3\pi^2 \lambda r_0 \left(1 + \frac{8}{3\pi} q r_0\right)}$	$\sum_{n=1}^{\infty} F_n \tanh(\gamma_n e)$	$\sum_{n=1}^{\infty} \frac{F_n}{\tanh(\gamma_n e)}$

With $A = D = \cosh(qe)$; $B = \frac{\sinh(qe)}{\lambda q \pi R^2}$; $C = \lambda q \pi R^2 \sinh(qe)$

$$F_n = \frac{4J_1^2(\alpha_n r_0)}{\lambda \pi R^2 r_0^2 \alpha_n^2 \gamma_n J_0^2(\alpha_n r_0)} \text{ with } \alpha_n \text{ solution of } J_1(\alpha_n R) = 0 \text{ and: } \gamma_n = \alpha_n^2 + \frac{p}{a}$$

$$\text{If: } \frac{e}{R} > 1, \text{ then: } \tanh(\gamma_n e) = 1$$

A7.3. Quadrupole associated with unidirectional transfer in a medium with energy generation

Inlet temperature = average temperature of the heating element

Inlet flux = total power injected into the heating element

$$\begin{bmatrix} \bar{\theta} \\ \Phi_0 \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta_s \\ \Phi_s \end{bmatrix}$$

	Plate of thickness e	Full cylinder of radius r and height L	Full sphere of radius r
A	1	1	1
B	$\frac{1}{\lambda q S \tanh(qe)} - \frac{1}{\rho c S e p}$	$\frac{1}{2\pi\lambda L q r} \frac{I_0(qr)}{I_1(qr)} - \frac{1}{\rho c \pi r^2 L p}$	$\frac{1}{4\pi\lambda r [qr \cosh(qr) - 1]} - \frac{3}{4\rho c \pi r^3 p}$
C	$\rho c S e p$	$\rho c \pi r^2 L p$	$\frac{4}{3} \rho c \pi r^3 p$
D	$\frac{qe}{\tanh(qe)}$	$\frac{qr I_0(qr)}{2 I_1(qr)}$	$\frac{(qr)^2}{3[qr \cosh(qr) - 1]}$

Appendix 8

Bessel Equations and Functions

A8.1. Bessel's special equations and their solutions

$$y'' + \frac{y'}{x} + m^2 y = 0 \quad \Rightarrow \quad y = k_1 J_0(mx) + k_2 Y_0(mx)$$

$$x^2 y'' + xy' + (x^2 - n^2)y = 0 \quad \Rightarrow \quad y = k_1 J_n(x) + k_2 Y_n(x) \quad (n \text{ integer})$$

$$y'' + \frac{y'}{x} - m^2 y = 0 \quad \Rightarrow \quad y = k_1 I_0(mx) + k_2 K_0(mx)$$

$$x^2 y'' + xy' - (x^2 - n^2)y = 0 \quad \Rightarrow \quad y = k_1 I_n(x) + k_2 K_n(x) \quad (n \text{ integer})$$

J_n is the non-modified Bessel function of the first kind of order n ;

I_n is the modified Bessel function of the first kind of order n ;

Y_n is the non-modified Bessel function of the second kind of order n ;

K_n is the modified Bessel function of the second kind of order n .

A8.2. Main properties of Bessel functions

Recurrence

$$J_{n+1}(u) = -J_{n-1}(u) + \frac{2n}{u} J_n(u) \quad Y_{n+1}(u) = -Y_{n-1}(u) + \frac{2n}{u} Y_n(u)$$

$$I_{n+1}(u) = I_{n-1}(u) - \frac{2n}{u} I_n(u) \quad K_{n+1}(u) = K_{n-1}(u) - \frac{2n}{u} K_n(u)$$

Derivative

$$\frac{dJ_0(u)}{du} = -J_1(u) ; \frac{dI_0(u)}{du} = I_1(u) ; \frac{dK_0(u)}{du} = -K_1(u) ; \frac{dY_0(u)}{du} = -Y_1(u) ;$$

$$\frac{d[uJ_1(u)]}{du} = uJ_0(u)$$

A8.3. Limits of Bessel functions of order 0 and 1

$$\text{If } u \rightarrow 0 : \quad J_0(u) \rightarrow 1 \quad J_1(u) \rightarrow 0 \quad Y_0(u) \rightarrow -\infty \quad Y_1(u) \rightarrow -\infty$$

$$I_0(u) \rightarrow 1 \quad I_1(u) \rightarrow 0 \quad K_0(u) \rightarrow +\infty \quad K_1(u) \rightarrow +\infty$$

$$\text{If } u \rightarrow \infty : \quad J_0(u) \rightarrow 0 \quad J_1(u) \rightarrow 0 \quad Y_0(u) \rightarrow 0 \quad Y_1(u) \rightarrow 0$$

$$I_0(u) \rightarrow +\infty \quad I_1(u) \rightarrow +\infty \quad K_0(u) \rightarrow 0 \quad K_1(u) \rightarrow 0$$

A8.4. Asymptotic behavior of Bessel functions of order 0 and 1

If $u \rightarrow 0$:

$$J_0(u) \rightarrow 1 \quad J_1(u) \rightarrow \frac{u}{2} \quad Y_0(u) \rightarrow \left(\frac{2}{\pi}\right) \ln(u) \quad Y_1(u) \rightarrow \frac{2}{\pi u}$$

$$I_0(u) \rightarrow 1 \quad I_1(u) \rightarrow \frac{u}{2} \quad K_0(u) \rightarrow -\ln(u) \quad K_1(u) \rightarrow \frac{1}{u}$$

If $u \rightarrow \infty$:

$$J_0(u) \rightarrow \sqrt{\frac{2}{\pi u}} \cos\left(u - \frac{\pi}{4}\right) \quad J_1(u) \rightarrow \sqrt{\frac{2}{\pi u}} \cos\left(\frac{u}{2} - \frac{\pi}{4}\right) \quad Y_0(u) \rightarrow \sqrt{\frac{2}{\pi u}} \sin\left(u - \frac{\pi}{4}\right)$$

$$Y_1(u) \rightarrow \sqrt{\frac{2}{\pi u}} \sin\left(\frac{u}{2} - \frac{\pi}{4}\right) \quad I_0(u), I_1(u) \rightarrow \sqrt{\frac{2}{\pi u}} \exp(u) \quad K_0(u), K_1(u) \rightarrow \sqrt{\frac{\pi}{2u}} \exp(-u)$$

A8.5. Integration

$$\int_0^R r [J_0(\alpha r)]^2 dr = \frac{R^2}{2} \{[J_0(\alpha R)]^2 + [J_1(\alpha R)]^2\}$$

Appendix 9

Influence of Radiation on Temperature Measurement

The two examples discussed below are intended to show that measuring the temperature is not always simple to realize in an accurate manner and that it may be affected by a large error if certain precautions are not taken. They show the parameters which influence the result of the temperature measurement.

A9.1. Measuring the temperature of a gas in a hot pipe

A thermometer with a diameter d_t possessing an emissivity $\varepsilon_t = 0.8$ is used to measure the temperature of a transparent gas flowing in a large pipe whose walls are at 250°C . The actual gas temperature is 500°C and the convective exchange coefficient is $h_c = 122 \text{ W m}^{-2}\text{K}^{-1}$.

To calculate the temperature T_t indicated by the thermometer, we write the heat balance of the thermometer: $h_c(T_g - T_t) = \sigma\varepsilon_t(T_t^4 - T_p^4)$.

With $\varepsilon_t = 0.8$; $T_g = 500^\circ\text{C}$; $T_p = 250^\circ\text{C}$ and $h_c = 122 \text{ W m}^{-2}\text{K}^{-1}$.

We find: $T_t = 434.5^\circ\text{C}$ which is an error of 65.5°C .

In order to reduce the measurement error, we can think of covering the thermometer with aluminum foil of emissivity 0.1. With $T_g = 500^\circ\text{C}$; $T_p = 250^\circ\text{C}$; $h_c = 122 \text{ W m}^{-2}\text{K}^{-1}$, we find: $T_t = 487.9^\circ\text{C}$, the error is only (or still!) of 12.1°C .

In order to further improve the accuracy of the measurement, the thermometer is protected against radiation by a thin cylindrical screen with a diameter of $d_e = 4d_t$ and an emission factor $\varepsilon_e = 0.3$. We admit that T_p , T_g , ε_t and h_c retain the same numerical values and that h_e (convective transfer coefficient on each side of the screen) is equal to $114 \text{ W m}^{-2} \text{ K}^{-1}$.

The heat balances are written as [JAN 16]:

$$\text{For the thermometer: } h_c S_t (T_g - T_t) + \frac{\sigma S_t (T_e^4 - T_t^4)}{\frac{1}{\varepsilon_t} + \frac{S_t}{S_e} \left(\frac{1}{\varepsilon_e} - 1 \right)} = 0$$

$$\text{For the screen: } 2h_e S_e (T_g - T_e) + \frac{\sigma S_t (T_t^4 - T_e^4)}{\frac{1}{\varepsilon_t} + \frac{S_t}{S_e} \left(\frac{1}{\varepsilon_e} - 1 \right)} + \sigma \varepsilon_e S_e (T_p^4 - T_e^4) = 0$$

The numerical resolution of the system of equations leads to:

$$T_t = 494.8^\circ\text{C} \text{ and } T_e = 483.1^\circ\text{C} \text{ or a measurement error of } 5.2^\circ\text{C}.$$

It should be noted that it is advantageous to use a thermometer that is as thin as possible and to have the highest possible air velocity to increase the value of h_c . It is also advantageous to have the lowest possible emissivity ε_t for the thermometer.

A9.2. Measurement of outside air temperature

A cylindrical thermometer with diameter $D = 5 \text{ mm}$ is used to measure the temperature of the outside air. The user places it outdoors vertically without any protection from solar radiation. The actual air temperature at midday was measured at 25°C at a nearby meteorological station.

The absorption factor of the thermometer for $0 < \lambda < 3 \mu\text{m}$ is $\alpha_0 = 0.8$ and its absorption factor for $\lambda > 3 \mu\text{m}$ is $\alpha_1 = 0.8$.

The radiation emitted by the atmosphere and the night sky towards the surface of the Earth can be calculated by the following equation:

$$\phi_{sky} = \sigma T_c^4 \text{ with } T_c = T_a - 12 \text{ K}$$

where T_a is the atmospheric air temperature (K).

The ground is assimilated to a black body at the temperature T_a .

The convective coefficient around the thermometer is taken to be equal to: $h = 15 \text{ W m}^{-2} \text{ K}^{-1}$.

The height of the Sun in the sky at midday is $H = 45^\circ$.

The solar radiation on a horizontal surface is $G^* = 700 \text{ W m}^{-2}$.

The solar flux density received by the vertical thermometer is identical to the flux density G^* received on a horizontal plate since the height of the Sun is 45° . The apparent surface of the thermometer seen by the Sun is: $D \times \text{height}$. Radiation of the atmosphere is assumed to be diffuse (non-directional).

By symmetry, the geometric shape factor of the thermometer with respect to the ground is identical to its shape factor with respect to the sky and is equal to 0.5. It can therefore be considered that the thermometer exchanges by radiation with a body at an average temperature $T_r = 0.5 (T_a + T_{sky})$.

The heat balance of the thermometer is written as (per unit of height):

$$\alpha_0 G^* D = \sigma \alpha_1 (T^4 - T_r^4) \pi D + h(T - T_a) \pi D$$

We can assume: $\sigma \alpha_1 (T^4 - T_r^4) = h_r (T - T_r)$ with $h_r = \sigma \alpha_1 (T^2 + T_r^2) (T + T_r) \approx 4 \sigma \alpha_1 T_r^3$.

The balance becomes: $\alpha_0 G^* = h_r (T - T_r) \pi + h(T - T_a) \pi$

$$\text{We thus deduce: } T = \frac{\frac{\alpha_0 G^*}{\pi} + h_r T_r + h T_a}{h + h_r}$$

With $\alpha_0 = \alpha_1 = 0.8$, we obtain: $h_r = 4.80 \text{ W m}^{-2} \text{ K}^{-1}$ and $T = 31.1^\circ \text{C}$ or a measurement error of 6.1°C .

To reduce the measurement error, we can cover the thermometer with aluminum foil whose absorption coefficients are: $\alpha_0 = \alpha_1 = 0.1$, we obtain: $h_r = 0.60 \text{ W m}^{-2} \text{ K}^{-1}$ and $T = 26.0^\circ \text{C}$ which reduces the measurement error to 1.0°C .

It is also possible to envisage moving the air to increase the convection coefficient which is calculated by the equation [JAN 16]: $Nu = C Re^n Pr^{1/3}$.

With an air velocity $u = 2 \text{ m s}^{-1}$, we obtain:

$$Re = \frac{\rho u D}{\mu} = \frac{1.185 \times 2 \times 0.005}{1.83 \times 10^{-5}} = 6475$$

Thus $C = 0.93$ and $n = 0.618$.

$$\text{We find: } Nu = 39.0 \text{ and } h = \frac{\lambda Nu}{D} = \frac{0.026 \times 39.0}{0.005} = 202.9 \text{ W m}^{-1} \text{ K}^{-1}.$$

We can deduce for the two previous cases: $T = 25.6^\circ\text{C}$ and $T = 25.1^\circ\text{C}$.

The coefficient h would be higher and therefore the measurement would be more accurate if a thermometer of smaller diameter of the thermocouple type was used.

Appendix 10

Case Study

A10.1. Measurement of the thermal properties of a cosmetic product

An industrial enterprise wanted to know the thermal properties of a cosmetic product of pasty constitution. It was therefore necessary to choose methods of measurement adapted to the specificity of the material.

Using a differential calorimeter, we first performed a measurement of the specific heat, which is advisable for any study if we have this type of equipment.

Next, we must measure another quantity which is thermal conductivity, diffusivity or effusivity. Given the “sticky” nature of the material, we avoided the hot wire and hot strip methods, the flash method also seems difficult to implement practically with this type of material. We therefore opt *a priori* for an asymmetric centered hot plate method with realization of a thin-edged frame to contain the paste under a controlled thickness. We will perform a steady-state measurement to estimate thermal conductivity and then a confirmation measurement in transient regime to estimate thermal effusivity.

A10.1.1. Specific heat

The specific heat c ($\text{J kg}^{-1} \text{K}^{-1}$) of the samples was measured by a Setaram calorimeter μdSc3 .

A preliminary verification of the calibration of the device was carried out with sapphire, the specific heat of which was measured between 20°C and 90°C and compared with the reference values (see Figure A10.1). The maximum deviation observed is 0.6%.

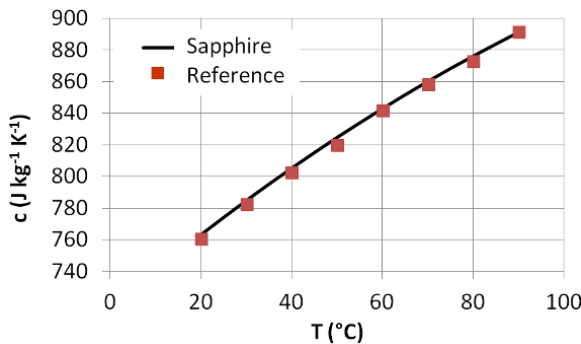


Figure A10.1. Measured and reference values of the specific heat of sapphire

The results obtained for the cosmetic product sample are given in Table A10.1. The accuracy of the measurement is estimated at 3%.

<i>T</i> (°C)	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C
<i>c</i> (J kg ⁻¹ K ⁻¹)	1808	1964	2068	2075	2038	2001	1931	1868

Table A10.1. Values of the specific heat *c* (J kg⁻¹ K⁻¹) measured by the Setaram calorimeter µdSc3

The density was measured by weighing a container of known volume, empty and then filled with water, and finally with the pasty product. The value thus obtained is:

$$\rho = 1070 \text{ kg m}^{-3}$$

A10.1.2. Thermal conductivity

The thermal conductivity was measured in steady state by the asymmetric centered hot plate method [JAN 17, DAM 16, JAN 10]. The device used, which has been placed in a climatic chamber maintained at a temperature *T_a*, is shown schematically in Figures A10.2 and A10.3. The section of the samples and of the heating element is 0.102 × 0.102 m².

Thermal conductivity was calculated as: $\lambda = \frac{e}{T-T_1} \left[\frac{R_{el}}{S} I^2 - \frac{\lambda_0}{e_0} (T - T_0) \right]$

where:

e is the thickness of the PVC frame (and thus of the pasty product);

$\frac{R_{el}}{s}$ is the ratio of the electrical resistance to the section, calibrated with a material of known properties (see [JAN 17]);

I is the intensity;

λ_0 is the (known) thermal conductivity of extruded polystyrene;

e_0 is the thickness of extruded polystyrene.

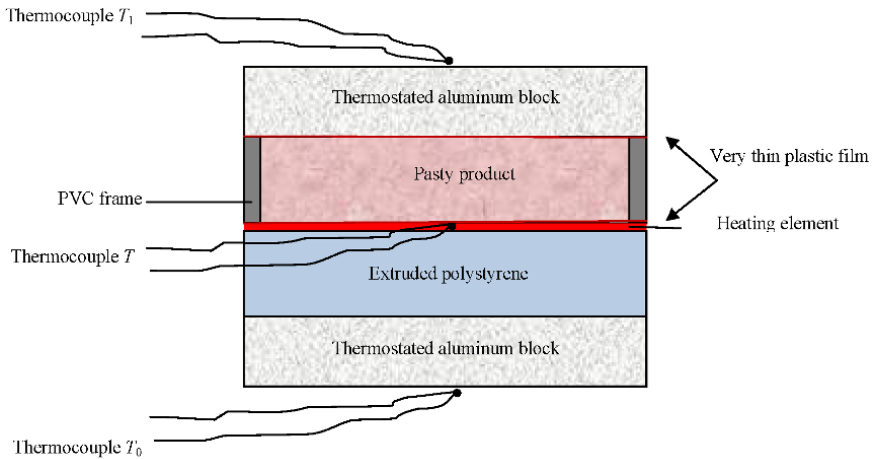


Figure A10.2. Schematic diagram of the experimental device

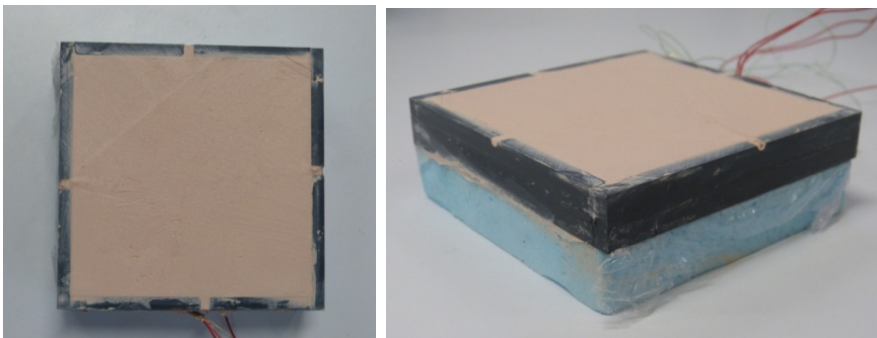


Figure A10.3. View of the device used for holding the pasty product

We first performed a 3D digital simulation under COMSOL of the device to check that the temperature at the center of the heating element was not affected by the edge effects due to the presence of the PVC frame and by convection and radiation heat transfer on the side walls.

According to the established results [JAN 17], in order to carry out a measurement at an average temperature T_0 we have imposed the following temperature values: $T = T_m + 5^\circ\text{C}$; $T_0 = T_1 = T_m - 5^\circ\text{C}$ and $T_a = T_m + 3^\circ\text{C}$.

The experimental results are reported in Table A10.2. The thermal conductivity is constant over the temperature range $[20^\circ\text{C}; 50^\circ\text{C}]$ and has the value:

$$\lambda = 0.206 \text{ W m}^{-1} \text{ K}^{-1}.$$

T	T_0	T_1	e_0	e	λ_0	I	T_m	λ
$^\circ\text{C}$			m	m	$\text{W m}^{-1} \text{ K}^{-1}$	A	$^\circ\text{C}$	$\text{W m}^{-1} \text{ K}^{-1}$
25.02	14.88	15.25	0.01875	0.0156	0.0278	0.19	20.0	0.206
40.3	29.6	29.91	0.01875	0.0156	0.0278	0.195	35.0	0.206
55.67	44.9	45.14	0.01875	0.0156	0.0278	0.195	50.3	0.205

Table A10.2. Measured values of the thermal conductivity

A10.1.3. Thermal effusivity

To confirm the results of the first two devices, we used the device shown in Figure A10.2 for an evaluation of the results of the transient hot plate. For this purpose, we have fixed the intensity passing through the resistance to a value which allows a temperature rise of the order of 10°C to be obtained in 180 s and we have recorded the change in temperature $T(t)$ from the start of heating with a time step of 0.1 s. It has been shown previously that, after a certain time (depending on the thermal capacity C_h of the heating element) and during the time when the semi-infinite medium hypothesis is valid, the curve $T(t) = f(\sqrt{t})$ can be assimilated to a straight line with a slope α . The thermal effusivity E can then be estimated by equation (4.2.11): $E = \frac{2\phi_0}{\alpha\sqrt{\pi}} - E_i$

where E_i is the thermal effusivity of the extruded polystyrene in our case and ϕ_0 the heat flux produced by the Joule effect in the heating element.

Preliminary measurements of specific heat by DSC, density and thermal conductivity by the centered hot plate method allowed us to calculate the effusivity value of extruded polystyrene at 20°C:

$$E_i = \sqrt{0.0302 \times 39.3 \times 1212} = 37.9 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$$

The experimental curves $T = f(t)$ and $T = f(\sqrt{t})$ obtained with the product to be characterized are given in Figure A10.4.

A first estimate was made by calculating the slope of the curve $T = f(\sqrt{t})$ over the entire recording interval, i.e. [0; 290 s]. We have thus obtained the value $E = 624.3 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$, the estimation residues, i.e. the difference $T_{exp}(t) - T_{mod}(t)$ are shown in Figure A10.5(a). It is found that they are neither flat nor centered on the zero value over the entire estimation interval, but a horizontal plateau is observed between 50 s and 150 s. The estimate is then resumed by reducing the interval to [50 s; 150 s] for calculating the slope of the curve $T = f(\sqrt{t})$. We then obtain $E = 625.0 \text{ J m}^{-2} \text{ K}^{-1} \text{ s}^{-1/2}$ and flat residues centered on zero over the chosen estimation interval, shown in Figure A10.5(b). It will be noted, however, that in this particular case the first value was very close to the final result, which is not always the case.

The knowledge of the effusivity, the density and the thermal conductivity makes it possible to estimate the specific heat by: $c = \frac{E^2}{\rho\lambda}$ which allows us to obtain:

$$c = \frac{625^2}{1070 \times 39.3} = 1772 \text{ J kg}^{-1} \text{ K}^{-1}$$

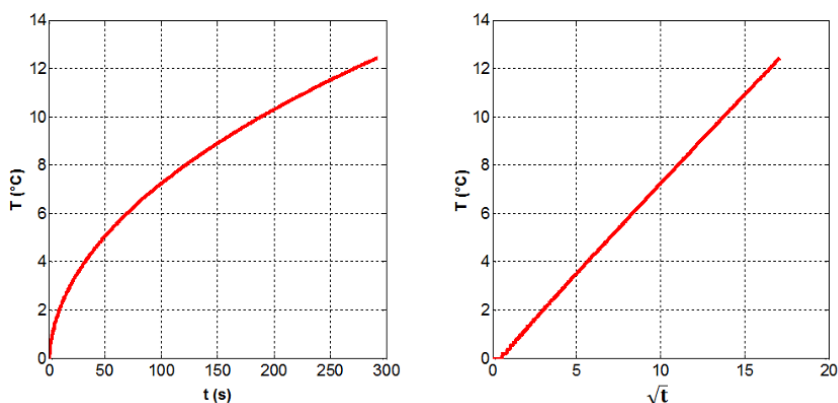


Figure A10.4. Examples of experimental curves $T = f(t)$ and $T = f(\sqrt{t})$

This value should be compared with that measured by DSC, namely: $c = 1808 \text{ J kg}^{-1} \text{ K}^{-1}$. The difference is only 2%. The last two measurements carried out with a very simple device of the asymmetrical hot plate type could thus alone make it possible to measure the thermal properties of this product sufficiently accurately.

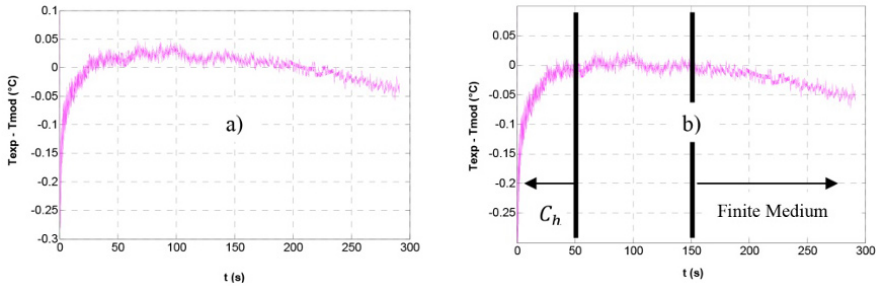


Figure A10.5. Estimation residues realized on a) [0; 290 s] and b) [50; 150 s]

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