



Thermal conductivity measurement of insulating materials with a three layers device

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ABSTRACT

This paper presents a new method dedicated to thermal conductivity measurement of low-density insulating materials. The three layers experimental device (brass/sample/brass) and the principle of the measurement based on a pulsed method are presented. The three-dimensional modelling of the system is used for a sensitivity analysis. The estimation method is described and applied to experimental measurements carried out at atmospheric pressure and under vacuum. We conclude that the thermal conductivity is estimated with a precision better than 5% and that the precision of the thermal diffusivity estimation depends on the density of the material.

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1. Introduction

The existing methods are not suited to thermal conductivity measurements of low density insulating materials. The contact transient methods using plane or linear heating element: hot disk [1–2], hot wire [3], hot plate [4], hot strip [5–6] cannot measure precisely thermal conductivity of low density insulating materials for the following reasons:

- The thermal capacity and the thermal resistance of the heating element (often heterogeneous and made of a metal wire inserted between two plastic films) is not known with precision and is often taken into account by a simplified model.
- The sensitivity of the measured temperature to the thermal capacity of the heating element is very high if the thermal capacity of the insulating material is low (case of a low density material)
- The thermal conductivity of the heating element is higher than the conductivity of the insulating material. The longitudinal heat transfer (parallel to the contact surface between the heat source and the sample) in the heating element that is not taken into account in the models may be and lead to estimation errors.

The Flash method [7] is difficult to use for the following reasons:

- The insulating materials are often semi-transparent to the radiations of the Flash lamp,
- It is very difficult to measure precisely a surface temperature on a low density material,
- The heat transfer on the heated face is often very different of the heat transfer on the other faces (very important temperature differences).

To avoid the first two disadvantages, the sample may be inserted between two heat conducting plates; a device based on this principle has already been used for the liquids [8]. For very low-density materials, one can show that the sensitivity of the unheated face temperature to the thermal conductivity is highly correlated to the sensitivity to the convective losses and that the sensitivity to the thermal diffusivity is low.

The aim of this work was to develop a new method suited to the thermal conductivity (and eventually diffusivity) measurement of low- and very-low-density insulating materials.

2. Principle and experimental device

The experimental device includes a cylindrical sample ($R = 2$ cm, $e = 5$ – 10 mm) of the material to be characterized inserted between two brass discs with a thickness, $e_b = 0.4$ mm and the same radius (cf. Fig. 1). Two type K thermocouple with wire diameter 0.05 mm are welded on the external face of each brass disc by the technique of the separated contact (with a distance of 5 mm between the two wires). The lower disc is in direct contact with a plane circular heating element having the same diameter

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Nomenclature

a	thermal diffusivity ($\text{m}^2 \text{s}^{-1}$)	θ	Laplace transform of the temperature
c	specific heat ($\text{J kg}^{-1} \text{ }^\circ\text{C}^{-1}$)	φ_0	heat flux density (W m^{-2})
D	diameter (m)	Φ_0	Laplace transform of the heat flux density
e	thickness (m)	λ	thermal conductivity ($\text{W m}^{-1} \text{ }^\circ\text{C}^{-1}$)
h	convection heat transfer coefficient ($\text{W m}^{-2} \text{ }^\circ\text{C}^{-1}$)	ρ	density (kg m^{-3})
H	transfer function		
p	Laplace parameter		
R_c	thermal contact resistance ($^\circ\text{C W}^{-1}$)		
S	area (m^2)		
t	time (s)		
t_{max}	time at which maximum temperature is reached (s)		
t_{ub}	upper bound of the time estimation interval (s)		
T	temperature ($^\circ\text{C}$)		
ε	porosity		
		Subscripts	
		air	air
		b	brass
		1	heated face
		2	unheated face
		3	lateral faces
		s	solid matrix of the porous medium
		v	vacuum

and set on an insulating material. A pressure is applied on the unheated brass disc by four PVC (chosen for its low thermal conductivity) tips with a very low contact surface area. The upper surface of the unheated brass disc exchanges with the ambient air by natural convection and radiation.

A heat flux is applied during a few seconds to the heating element and the temperatures, $T_{b1}(t)$ and $T_{b2}(t)$ of the brass discs are recorded. A three-dimensional model associated to an inverse method is then used to estimate the thermal conductivity and the thermal diffusivity of the insulating material inserted between the two brass discs.

The heat flux is produced by a plane heating element during a few seconds instead of being produced by a flash lamp (device initially tested) during a few milliseconds for the following reasons:

- The temperature increasing of the heated face must not be too fast to be compatible with the thermocouple response time.
- A uniform pressure may be easily applied on the brass discs through the plane heating element.
- A light part of the flash may reach the lateral surfaces by reflection, this disadvantage is avoided with a plane heating element.

3. Model and estimation method

3.1. Assumptions

- The temperatures, T_{b1} and T_{b2} in the brass discs are uniform and

- The thermal contact resistances (typically $10^{-4} \text{ m}^2 \text{ K W}^{-1}$) between the sample and the brass discs are negligible in comparison with the thermal resistance of the sample (greater than $5 \times 10^{-2} \text{ m}^2 \text{ K W}^{-1}$ for the tested samples).

As a first step, the following case is considered: a unique sample with heat transfer by convection with the ambient air on all its faces receives a direct and short heating on one face (no brass discs as represented in Fig. 2).

Setting $\bar{T}(r, z, t) = T(r, z, t) - T_e$

The equation of heat becomes:

$$\frac{\partial^2 \bar{T}(r, z, t)}{\partial r^2} + \frac{1}{r} \frac{\partial \bar{T}(r, z, t)}{\partial r} + \frac{\partial^2 \bar{T}(r, z, t)}{\partial z^2} = \frac{1}{a} \frac{\partial \bar{T}(r, z, t)}{\partial t} \quad (1)$$

Initial and boundary conditions may be written as:

$$z = 0 \rightarrow \lambda \frac{\partial \bar{T}(r, 0, t)}{\partial z} = h_1 \bar{T}(r, 0, t) - \phi_0(t) \quad (2)$$

$$z = e \rightarrow -\lambda \frac{\partial \bar{T}(r, e, t)}{\partial z} = h_2 \bar{T}(r, e, t) \quad (3)$$

$$r = 0 \rightarrow \frac{\partial \bar{T}(0, z, t)}{\partial r} = 0 \quad (4)$$

$$r = R \rightarrow -\lambda \frac{\partial \bar{T}(R, z, t)}{\partial r} = h_3 \bar{T}(R, z, t) \quad (5)$$

$$t = 0 \rightarrow \bar{T}(r, z, 0) = 0 \quad (6)$$

The Laplace transform applied to relation (1) with $L[\bar{T}(r, z, t)] = \theta(r, z, p)$ leads to:

$$\frac{\partial^2 \theta(r, z, p)}{\partial r^2} + \frac{1}{r} \frac{\partial \theta(r, z, p)}{\partial r} + \frac{\partial^2 \theta(r, z, p)}{\partial z^2} = \frac{p}{a} \theta(r, z, p) \quad (7)$$

The Laplace transforms of the boundary conditions are:

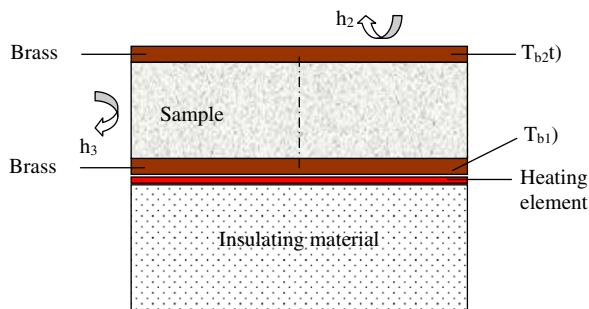


Fig. 1. Experimental device.

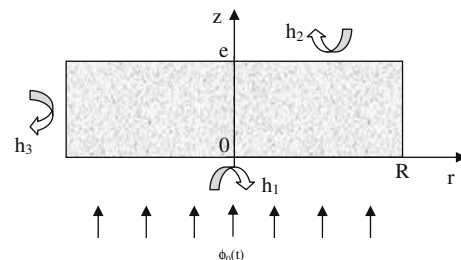


Fig. 2. Experiment schema for a unique sample.

$$\lambda \frac{\partial \theta(r, 0, p)}{\partial z} = h_1 \theta(r, 0, p) - \Phi_0(p) \tag{8}$$

$$-\lambda \frac{\partial \theta(r, e, p)}{\partial z} = h_2 \theta(r, e, p) \tag{9}$$

$$\frac{\partial \theta(0, z, p)}{\partial r} = 0 \tag{10}$$

$$-\lambda \frac{\partial \theta(R, z, p)}{\partial r} = h_3 \theta(R, z, p) \tag{11}$$

$$\theta(r, z, 0) = 0 \tag{12}$$

Setting: $\theta(r, z, p) = R(r, p)Z(z, p)$

One obtains : $\theta(r, z, p) = \sum_{n=1}^{\infty} A_n J_0(\alpha_n r) \{ \beta_n \text{ch}[\gamma_n(e-z)] + H_2 \text{sh}[\gamma_n(e-z)] \}$ (13)

Where : $A_n = \frac{2\Phi_0(p) \frac{e}{\lambda}}{\omega_n \left(1 + \frac{\omega_n^2}{H_3^2} \right) J_1(\omega_n) \left[(\beta_n^2 + H_2 H_1) \text{sh}(\beta_n) + \beta_n (H_2 + H_1) \text{ch}(\beta_n) \right]}$ (14)

ω_n is solution of : $\omega J_1(\omega) = H_3 J_0(\omega)$ (15)

$\beta_n = \sqrt{\frac{pe^2}{a} + \left(\frac{e}{R}\right)^2 \omega_n^2}$ (16)

$H_1 = \frac{eh_1}{\lambda}; H_2 = \frac{eh_2}{\lambda}; H_3 = \frac{h_3 R}{\lambda}$ (17)

The mean temperatures for $z = 0$ and e may be calculated by integration of relation (13) between $r = 0$ and R :

$$\theta_{\text{moy}}(0, p) = \frac{\sum_{n=1}^{\infty} \frac{4\Phi_0(p) \frac{e}{\lambda} [\beta_n \text{ch}(\beta_n) + H_2 \text{sh}(\beta_n)]}{\omega_n^2 \left(1 + \frac{\omega_n^2}{H_3^2} \right) [(\beta_n^2 + H_2 H_1) \text{sh}(\beta_n) + \beta_n (H_2 + H_1) \text{ch}(\beta_n)]}}{\tag{18}}$$

$$\theta_{\text{moy}}(e, p) = \frac{\sum_{n=1}^{\infty} \frac{4\Phi_0(p) \frac{e}{\lambda} \beta_n}{\omega_n^2 \left(1 + \frac{\omega_n^2}{H_3^2} \right) [(\beta_n^2 + H_2 H_1) \text{sh}(\beta_n) + \beta_n (H_2 + H_1) \text{ch}(\beta_n)]}}{\tag{19}}$$

The next step is the study of the three layers device represented in Fig. 3.

The relations (1), (4), (5), and (6) remain valid in this case.

The local thermal balance at radius, r , on the heated brass disc (supposed at uniform temperature, $T_{b1}(t)$) is:

$$\pi R^2 e_1 \rho_b c_b \frac{\partial T_{b1}}{\partial t} = \phi_0(t) \pi R^2 - h_1 \pi R^2 T_{b1} - h_3 2\pi R e_1 T_{b1} + \int_0^R 2\pi r dr \lambda_s e \frac{\partial T(r, 0)}{\partial z} \tag{20}$$

The integration of this local balance between, $r = 0$ and $r = R$ with a Laplace transformation leads to:

$$-\lambda \frac{\partial \theta_{\text{moy}}(e, p)}{\partial z} = \Phi_0(p) - \left[\rho_b c_b e_1 p + \left(h_1 + \frac{2h_3 e_1}{R} \right) \right] \theta_{b1} \tag{21}$$

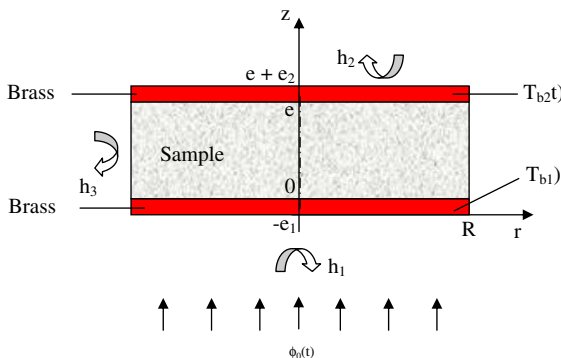


Fig. 3. Experimental schema of a three layers device.

This equation is similar to the relation (8) considering the mean temperature for a given value of z instead of the local temperature at (r, z) and replacing h_1 by a corrected

coefficient : $h_{1c} = \rho_b c_b e_1 p + \left(h_1 + \frac{2h_3 e_1}{R} \right)$ (22)

The thermal balance of the unheated disc leads similarly to a corrected coefficient:

$$h_{2c} = \rho_b c_b e_2 p + \left(h_2 + \frac{2h_3 e_2}{R} \right) \tag{23}$$

By considering the mean temperature at z instead of the local temperature at (r, z) , this boundary conditions are the same as for a unique sample. So that for the three layers system brass/sample/brass, the expressions (18) and (19) of the mean temperatures, respectively, at $z = 0$ and e for a unique sample remains valid if h_1 is replaced by h_{1c} and h_2 by h_{2c} in relation (17).

The transfer function $H(p)$ of the system may be written as:

$$H(p) = \frac{\theta(e, p)}{\theta(0, p)} \tag{24}$$

and : $T_{b2}(t) = T_{b1}(t) \otimes L^{-1}[H(p)]$ (25)

These two relations are true whatever the boundary condition on the heated brass disc is, particularly if this disc exchange heat by conduction with an insulating material (as described in Fig. 1) rather than by convection with air.

The principle of the method is to estimate the transfer function $H(p)$ by estimating the values of the three parameters: a , λ and $h = h_2 = h_3$ that minimize the sum of the quadratic errors between the experimental values of $T_{b2}(t)$ and those calculated by relation (25) with experimental values of $T_{b1}(t)$. The number of parameters to be estimated is the same that in the classical flash method (heat flux density, ϕ ; thermal diffusivity, a and convection heat transfer coefficient, h). The minimization is realized by using the Levenberg–Marquart method.

One of the advantages of the method is that it is not sensitive to the heat transfer on the heated face; in the case of a convective heat transfer with important variations of temperature on this face, the hypothesis of a constant convection coefficient being the same on the heated and on the unheated face is not totally true. It will be shown that this model approximation can lead to errors in the estimated parameters particularly in the case of a measurement realized on an insulating material where the heated face temperature may reach several decades degrees and where the resistance to the external transfer (convection) is of the same order of magnitude that the resistance to the internal transfer (conduction).

Compared to the limits (described in introduction) of the classical contact methods and of the Flash method for the thermal conductivity measurement of low density material, the proposed method has the following advantages:

- The temperature measurements are more precise since they are done on a heavy conductive material (brass),
- The thermal capacity of the two discs (homogeneous) is known precisely and taken into account, and
- The longitudinal heat transfers in the discs are taken into account without approximation in the model (boundary condition of uniform temperature).

4. Sensitivity analysis

The reduced sensitivities of the transfer function $H(p)$ have been calculated and represented in Fig. 4 for a three layers system with the following characteristics:

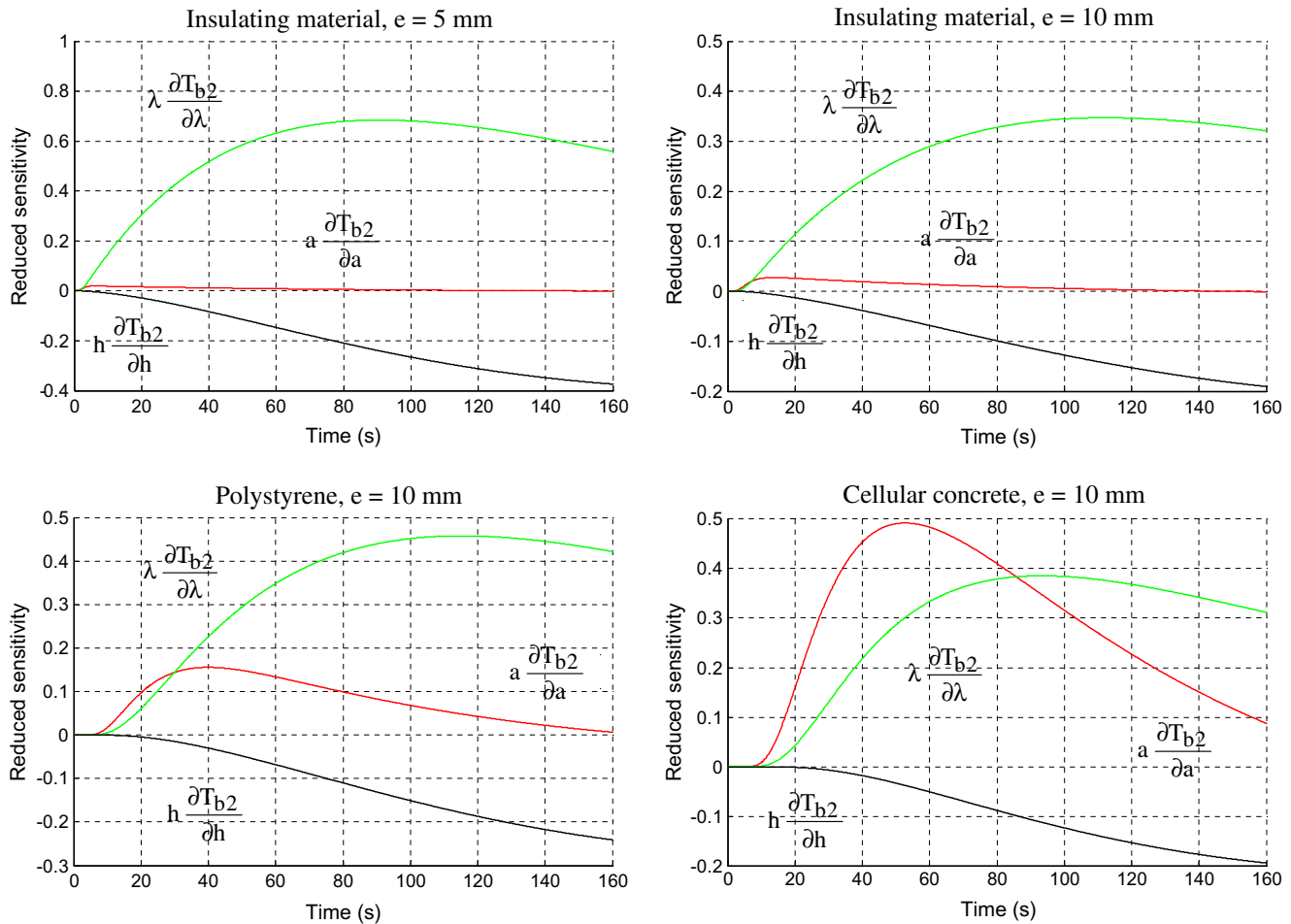


Fig. 4. Reduced sensitivities of the transfer function $H(p)$ calculated by relation (25).

- Brass discs: $e_b = 0.4$ mm, $D = 40$ mm, $\lambda = 100.4$ W m⁻¹ K⁻¹, $a = 3.14 \times 10^{-5}$ m² s⁻¹;
- Samples:
 1. *Insulating material*: $\lambda = 0.02$ W m⁻¹ K⁻¹, $a = 4 \times 10^{-6}$ m² s⁻¹, $e = 5$ mm, $e = 10$ mm;
 2. *Polystyrene*: $\lambda = 0.035$ W m⁻¹ K⁻¹, $a = 8 \times 10^{-7}$ m² s⁻¹, $e = 10$ mm; and
 3. *Cellular concrete*: $\lambda = 0.15$ W m⁻¹ K⁻¹, $a = 5 \times 10^{-7}$ m² s⁻¹, $e = 10$ mm.

It can be noticed that:

- For a given material, if the sample thickness decreases then the sensitivity to the thermal diffusivity increases and the sensitivity to the thermal conductivity decreases.
- For a given sample thickness, if the thermal capacity of the sample increases then the sensitivity to the thermal diffusivity increases.
- The sensitivity to the thermal diffusivity is quite low for very low density materials so that the thermal diffusivity will not be measurable with this method. Nevertheless, this sensitivity is high enough to estimate the thermal diffusivity of insulating material with higher density such as polystyrene or cellular concrete.
- The sensitivity to the thermal conductivity is high in each case and is not correlated with the sensitivities to the convection coefficient and to the thermal diffusivity. The thermal conductivity is thus estimable by this method for all type of insulating materials

5. Results

5.1. Estimation from numerical simulations

The temperatures in a three layers device have been simulated with COMSOL. The following data has been considered:

- *Sample*: diameter = 35 mm, thickness = 5.6 mm, thermal properties: $\lambda = 0.02$ W m⁻¹ K⁻¹, $\rho c = 5000$ J m⁻³ K⁻¹, $a = 4 \times 10^{-6}$ m² s⁻¹ and
- *Metallic discs (copper)*: diameter = 35 mm, thickness = 0.4 mm, thermal properties: $\lambda = 397.5$ W m⁻¹ K⁻¹, $\rho = 8940$ kg m⁻³, $C_p = 384.9$ J kg⁻¹ K⁻¹.

First, the simulation of the temperatures has shown that the hypothesis of uniform temperature in the two metallic discs is valid.

Then, the simulated temperatures $T_{b1}(t)$ and $T_{b2}(t)$ have been considered as experimental data and an estimation parameter has been applied according to the two following methods:

1. The unheated face temperature, $T_{b2}(t)$ is the only experimental data considered as in the classical flash method. An inversion method is used to estimate the heat flux density, φ_0 ; the thermal diffusivity, a ; the thermal conductivity, λ and the convection coefficient h that minimize the sum of the quadratic errors between the experimental curve and the simulated curve $T_{b2}(t)$ calculated by relation (19) with the corrected convection coefficients given by relations (22) and (23).

2. The temperature $T_{b1}(t)$ of the heated face is considered as input experimental data of the system both with the temperature $T_{b2}(t)$. An inversion method is used to estimate the thermal diffusivity, a ; the thermal conductivity, λ and the convection coefficient h that minimize the sum of the quadratic errors between the experimental curve and the simulated curve $T_{b2}(t)$ calculated by relation (25) of the unheated face temperature.

The results of these two estimations are reported in Table 1, where t_{up} is the upper bound of the estimation time interval.

It can be noticed that:

- The method 1, based on the 3D model and considering that the only known temperature is $T_{b2}(t)$, does not lead to a precise estimation of λ if the convection coefficient h_1 on the heated face is different of the coefficients, h_2 and h_3 , on the other faces.
- The method 2 gives in all cases a precise estimation of the thermal conductivity, even when a random noise with an amplitude of 0.01 °C has been added to the temperatures simulated with COMSOL. The estimation of the thermal diffusivity becomes less precise if the measurement noise increases.
- A simulation based on method 2 with $h_2 = 5 \text{ W m}^{-2} \text{ K}^{-1}$ and $h_1 = h_3 = 10 \text{ W m}^{-2} \text{ K}^{-1}$ leads to an error of 3.5% on the estimated value of λ and of 2% on the estimated value of a . An extreme case has been considered ($h_3 = 2h_2$) so that the errors with experimental data will be less important.

The effect of an error on the supposed “known” value of the thermal capacity, ρc_{mp} of the two metallic plates has also been investigated. It was found that an error of $x\%$ on ρc_{mp} leads to the same error on the estimated value of λ but has no significant influence on the estimated value of a , for $x < 5\%$. It has been verified that an error of 5% on the thermal conductivity of the metallic plates has no influence on the estimated values.

5.2. Estimation from experimental results

Experimental measurements with the three layers method have been realized for 2 materials: a rigid foam of 10.0 mm thickness and a cellular concrete of 8.1 mm thickness. The sample's diameter was 40 mm.

The Tiny hot plate method [9] has also been used to estimate the thermal conductivities of these two materials and the hot plate method [4] has been used to estimate the thermal effusivity of the cellular concrete.

A series of three tests has been realized on each sample. Fig. 5 represents an example of experimental curves obtained with the rigid foam. Fig. 6 represents the experimental and simulated curves calculated with the values obtained by the inversion

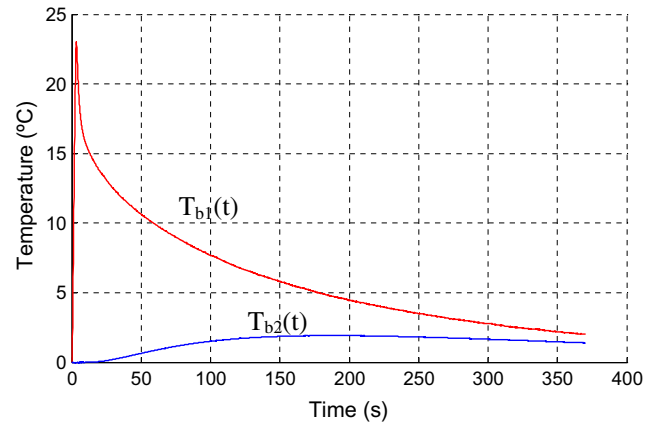


Fig. 5. Experimental curves $T_{b1}(t)$ and $T_{b2}(t)$ for a rigid foam.

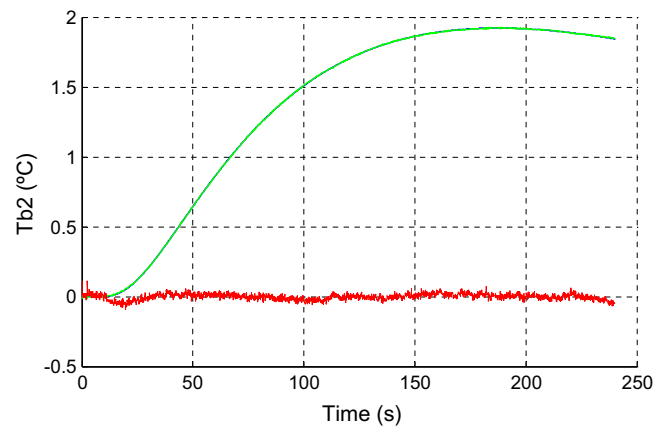


Fig. 6. Experimental and simulated curves $T_{b2}(t)$ for a rigid foam and residues, $10 \times$.

Table 2

Estimated values of a , λ and h as a function of the upper bond t_{ub} of the time estimation interval for a test on a rigid foam ($t_{max} = 185 \text{ s}$).

$t_{ub} \text{ (s)}$	$a \text{ (m}^2 \text{ s}^{-1}\text{)}$	$\lambda \text{ (W m}^{-1} \text{ K}^{-1}\text{)}$	$\rho c \text{ (J m}^{-3} \text{ K}^{-1}\text{)}$	$h \text{ (W m}^{-2} \text{ K}^{-1}\text{)}$
80	4.67×10^{-7}	0.0393	8.40×10^4	4.15
100	4.59×10^{-7}	0.0410	8.92×10^4	4.92
120	4.55×10^{-7}	0.0420	9.21×10^4	5.29
150	4.52×10^{-7}	0.0425	9.38×10^4	5.47
160	4.52×10^{-7}	0.0425	9.38×10^4	5.47
180	4.52×10^{-7}	0.0425	9.37×10^4	5.46
240	4.56×10^{-7}	0.0417	9.14×10^4	5.30
300	4.68×10^{-7}	0.0405	8.65×10^4	5.03
360	4.67×10^{-7}	0.0405	8.64×10^4	5.02

Table 1

Results of the estimations realized by two different methods from temperatures simulated with COMSOL.

Estimation method	$t_s = 100 \text{ s}$			$t_s = 200 \text{ s}$			$t_s = 300 \text{ s}$		
	$10^6 a$	$10^3 \lambda$	h	$10^6 a$	$10^3 \lambda$	h	$10^6 a$	$10^3 \lambda$	h
$h_1 = h_2 = h_3 = 10 \text{ W m}^{-2} \text{ K}^{-1}$									
1	4.18	17.7	11.0	4.19	16.4	11.3	4.2	16.5	11.3
2	4.01	19.8	11.1	3.95	19.8	11.1	3.8	19.9	11.2
$h_1 = 15 \text{ W m}^{-2} \text{ K}^{-1}; h_2 = h_3 = 5 \text{ W m}^{-2} \text{ K}^{-1}$									
1	4.12	33.8	7.4	4.11	33.8	7.4	4.1	34.0	7.5
2	3.95	19.9	5.4	3.93	19.9	5.4	3.9	19.9	5.5
$h_1 = 15 \text{ W m}^{-2} \text{ K}^{-1}; h_2 = h_3 = 5 \text{ W m}^{-2} \text{ K}^{-1}; dT_{b1} = dT_{b2} = 0.01 \text{ }^\circ\text{C}$									
1	4.16	27.0	8.4	4.22	31.7	7.7	4.1	33.7	7.5
2	3.88	20.1	5.3	3.85	20.5	5.6	3.8	20.6	5.7
$h_1 = h_3 = 10 \text{ W m}^{-2} \text{ K}^{-1}; h_2 = 5 \text{ W m}^{-2} \text{ K}^{-1}$									
1	3.92	19.4	6.2	19.3	16.4	6.1	4.01	19.3	6.1

Table 3
Estimated values of a , λ and h for the three layers method.

Material	Test number	a ($\text{m}^2 \text{s}^{-1}$)	λ ($\text{W m}^{-1} \text{K}^{-1}$)	ρc ($\text{J m}^{-3} \text{K}^{-1}$)	h ($\text{W m}^{-2} \text{K}^{-1}$)
Rigid foam	1	4.54×10^{-7}	0.0420	9.25×10^4	5.38
	2	4.52×10^{-7}	0.0425	9.37×10^4	5.46
	3	4.56×10^{-7}	0.0423	9.26×10^4	5.33
	Mean	4.54×10^{-7}	0.0423	9.29×10^4	5.39
	Standard deviation (%)	0.36	0.49	0.58	0.99
Cellular concrete	1	2.54×10^{-7}	0.156	6.11×10^5	7.26
	2	2.58×10^{-7}	0.149	5.76×10^5	6.99
	3	2.54×10^{-7}	0.155	6.11×10^5	7.24
	Mean	2.55×10^{-7}	0.153	5.99×10^5	7.16
	Standard deviation (%)	0.90	2.47	3.37	2.10

Table 4
Estimated values of a , λ and ρc of a rigid foam at atmospheric pressure and under 10^{-2} mbar.

	a ($\text{m}^2 \text{s}^{-1}$)	λ ($\text{W m}^{-1} \text{K}^{-1}$)	ρc ($\text{J m}^{-3} \text{K}^{-1}$)
$P = 1$ bar	4.54×10^{-7}	0.0423	9.29×10^4
$P = 10^{-2}$ mbar	2.12×10^{-7}	0.0183	8.75×10^4

method. The residues (multiplied by 10) that are also represented show that the modelled curve is very close to the experimental one.

Table 2 gives the estimated values of λ , a and h as a function of the upper bound t_{ub} of the estimation time interval for a test with the rigid foam. One can notice a very low variation of the estimated values, the estimation for this material has further been realized between $t=0$ and the time, t_{max} where the temperature, T_{b2} reaches its maximum value. All the experimental results are presented in Table 3 showing that the reproducibility of the results is quite good.

The difference between the thermal conductivity values estimated by the tiny hot plate ($\lambda = 0.0405 \text{ W m}^{-1} \text{K}^{-1}$ for the rigid foam and $\lambda = 0.165 \text{ W m}^{-1} \text{K}^{-1}$ for the cellular concrete) and by the three layers method is lower than 5%.

For the cellular foam, the thermal diffusivity may be calculated from the thermal effusivity measured by the hot plate ($E = 325 \text{ J m}^{-2} \text{K}^{-1} \text{s}^{-1/2}$) and from the thermal conductivity measured by the Tiny hot plate method ($\lambda = 0.165 \text{ W m}^{-1} \text{K}^{-1}$), the result is $a = 2.56 \times 10^{-7} \text{ m}^2 \text{s}^{-1}$. This difference between this value and the value measured by the three layers method is lower than 1%.

A last measurement series of three tests has been done with the rigid foam sample under a pressure of 10^{-2} mbar. Table 4 reports the thermal properties estimated under the atmospheric pressure and under a pressure of 10^{-2} mbar.

If the radiation heat transfer is negligible (low emissivity of the brass disc surface and important diffusion inside the material), the hypothesis of parallel thermal resistance generally considered in high porosity porous media leads to an equivalent thermal conductivity [10]:

$$\lambda = \varepsilon \lambda_{\text{air}} + (1 - \varepsilon) \lambda_s \quad (26)$$

The values of λ and λ_s have been estimated from two experimental sets of curves $T_1(t)$ and $T_2(t)$ without any assumption on the porosity value.

The porosity may thus be estimated by: $\varepsilon = \frac{\lambda - (1 - \varepsilon) \lambda_s}{\lambda_{\text{air}}} = 0.92$

Considering the expression of the thermal capacity of the porous medium:

$$\rho c = \rho_s (1 - \varepsilon) c_s + \rho_{\text{air}} \varepsilon c_{\text{air}} \quad (27)$$

The thermal capacity of the rigid foam under vacuum may be estimated as:

$$\begin{aligned} \rho_v c_v &= \rho c - \rho_{\text{air}} \varepsilon c_{\text{air}} = 92,900 - 1.2 \times 0.92 \times 1006 \\ &= 91,789 \text{ J m}^{-3} \text{K}^{-1} \end{aligned}$$

The difference between this predicted value and the measured value $87,500 \text{ J m}^{-3} \text{K}^{-1}$ given in Table 4 is lower than 5% and validates our measurement under low pressure.

Furthermore, the thermal conductivity of the solid material constituting the matrix may be evaluated by: $\lambda_s = \frac{\lambda - \varepsilon \lambda_{\text{air}}}{1 - \varepsilon} = 0.231 \text{ W m}^{-1} \text{K}^{-1}$

And its thermal capacity $\rho_s c_s$ by:

$$\rho_s c_s = \frac{\rho c - \varepsilon \rho_{\text{air}} c_{\text{air}}}{1 - \varepsilon} = 1.15 \times 10^6 \text{ J m}^{-3} \text{K}^{-1}$$

These two values are compatible with known values although they are not estimated with precision since the porosity has been estimated from thermal conductivity measurement and considering the assumption of parallel thermal resistances in the porous medium. Nevertheless, a specific and more precise measurement of the porosity (carried out with a picnometer for example) may lead to precise estimations of the thermal conductivity and of the thermal capacity of the solid matrix of the tested porous medium.

6. Conclusions

The proposed method is based on a three layers system which input and output temperatures are measured after applying a short heat flux on one layer. The 3D transfer function of the system is estimated by applying an inverse method to a convolution product. This method leads to a satisfying precision for the estimation of the thermal conductivity (<5%) of the low to very low conductivity materials ($\lambda < 0.15 \text{ W m}^{-1} \text{K}^{-1}$). It also gives an estimation of the thermal diffusivity of the material having a thermal capacity greater than $4 \times 10^4 \text{ W m}^{-3}$. The measurement may be done at atmospheric pressure or under vacuum. The thermal diffusivity of the super-insulating materials with very low density seems difficult to measure with this method.

References

- [1] S.E. Gustafsson, Transient plane source techniques for thermal conductivity and thermal diffusivity measurements of solid materials, Rev. Sci. Instrum. 62 (3) (1991) 797–804.
- [2] Y. He, Rapid thermal conductivity measurement with a hot disk sensor. Part 1. Theoretical considerations, Thermochim. Acta 436 (2005) 122–129.
- [3] Y. Nagazaka, A. Nagashima, Simultaneous measurement of the thermal conductivity and the thermal diffusivity of liquids by the transient hot-wire method, Rev. Sci. Instrum. 52 (2) (1981) 229–232.
- [4] X. Zhang, A. et Degiovanni, Mesure de l'effusivité thermique de matériaux solides et homogènes par une méthode de «sonde» plane, J. Phys. III 6 (1993) 1243–1265.

- [5] U. Hammerschmidt, A new pulse hot strip sensor for measuring thermal conductivity and thermal diffusivity of solids, *Int. J. Thermophys.* 24 (3) (2003) 675–682.
- [6] Y. Jannot, P. Meukam, Simplified estimation method for the determination of thermal effusivity and thermal conductivity with a low cost hot strip, *Measure. Sci. Technol.* 15 (2004) 1932–1938.
- [7] A. Degiovanni, A. Laurent, Une nouvelle technique d'identification de la diffusivité thermique pour la méthode flash, *Rev. Phys. Appl.* 21 (1986) 229–237.
- [8] B. Rémy, A. Degiovanni, Parameters estimation and measurement of thermophysical properties of liquids, *Int. J. Heat Mass Transfer* 48 (2005) 4103–4120.
- [9] J.C. Batsale, A. Degiovanni, Mesure de résistance thermique de plaques minces à l'aide d'une mini-plaque chaude, *Rev. Gen. Therm.* 390–391 (1994) 387–391.
- [10] S. Bories, M. Prat, Transferts de chaleur dans les milieux poreux, *Tech. Ing.* B8 (250) (1995).