Transient hot plate method with two temperature measurements for thermal characterization of metals

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Abstract
This paper presents a study of the transient hot plate method with
simultaneous measurements of front (heated) and rear face
temperatures. In contrast to the classical device, a single sample of
the material to be thermally characterized is set in contact with a planar
heating element and inserted between two pieces of insulating material.
The purpose was to simultaneously estimate thermal effusivity and
conductivity of metals in a limited time t_2 (<90 s) using a low-cost
device. Heat transfer has been modelled with a quadrupole formalism to
simulate the front and rear face temperatures $T_0(t)$ and $T_2(t)$. Simulation
is used to fix the minimal thickness of the sample so that the front face
temperature remains independent of thermal conductivity during a time
$t_1 > 30$ s. The thermal effusivity is estimated between 0 and t_1 by
minimization of the quadratic errors between the experimental curve and
the simulated curve $T_0(t)$. The thermal conductivity is estimated between
0 and t_2 by minimization of the quadratic errors between the experimental
curve and the simulated curve $T_2(t)$. To validate the model and the
estimation process, experimental tests were realized on four samples of
metals with conductivities varying from 6 to 140 W m^{-1} °C^{-1} and
having typical area 44.5 × 44.5 mm^2 and thickness varying from 16.7 to
80 mm.

Keywords: transient hot plate, rear face, front face, thermal quadrupoles,
effusivity, conductivity, parameter estimation

Nomenclature

a	thermal diffusivity (m^2 s^{-1})	R_c	thermal contact resistance (°C W^{-1})
c	specific heat of the heating element (J kg^{-1} °C^{-1})	S	heating element area (m^2)
e	sample thickness (m)	T_0	temperature increase of the front (heated) face (°C)
E	thermal effusivity of the material (J m^{-2} °C^{-1} s$^{-1/2}$)	T_2	temperature increase of the rear face (°C)
Ei	thermal effusivity of the insulating material (J m^{-2} °C^{-1} s$^{-1/2}$)	V_j	coefficients of the Stehfest algorithm
h	convective heat transfer coefficient (W m^{-2} °C^{-1})	θ_0	Laplace transform of T_0
m	mass of the heating element (kg)	θ_2	Laplace transform of T_2
p	Laplace parameter	φ_0	heat flux dissipated in the heating element (W)
p_e	sample perimeter (m)	Φ	Laplace transform of the heat flux
		λ	thermal conductivity (W m^{-1} °C^{-1})

1. Introduction

The aim of this study was to develop a relatively short-time method (< 90 s) using a low-cost probe to estimate simultaneously thermal effusivity and thermal conductivity from a single temperature recording. The thermal effusivity is defined as

$$E = \sqrt{\lambda \rho c},$$

where E is the thermal effusivity (J m^{-2} °C^{-1} s$^{-1/2}$), λ is the thermal conductivity (W m^{-1} °C^{-1}), ρ is the density (kg m^{-3}) and c is the specific heat (J kg^{-1} °C^{-1}). This method is dedicated to high conductivity materials (> 6 W m^{-1} °C^{-1}) for which the semi-infinite medium hypothesis is true only for low values of the ratio of the measuring time to the sample dimensions. All the methods based on the hypothesis of a semi-infinite medium for the sample are thus limited to this type of material (hot plate [1], hot wire [2, 3], hot strip [4–7] or hot disc [8]) since they need to work with very large dimension samples or have too short an estimation time to reach an acceptable precision. Nevertheless, some devices allowing the simultaneous estimation of thermal effusivity and conductivity of thermally finite samples have been achieved; they are all based on the processing of transient measurement of the temperature of a heating probe set between two samples of the material to be characterized.

The main devices are as follows.

(1) The hot strip method with imposed temperature on the unheated faces as studied by Ladevie [9]. Its main disadvantages are the experimentation duration (> 5 mn), complex data processing and the need to estimate the convective heat losses on the sample lateral faces. Moreover, this method is dedicated to low conductivity materials.

(2) The DPS method (dynamic plane source) introduced by Malinarič [10]. In this method, the probe mass is neglected and the parameter estimation interval is chosen so that the influence of the thermal contact resistance is minimized. This choice remains an uncertainty factor difficult to estimate.

(3) The periodic method developed by Boudenne *et al* [11] in which a sample inserted between two metallic masses is submitted to a periodical heat flux. This method is relatively complex to carry out (more complex heat flux to be generated, partial vacuum enclosure, thermally conductive grease to be put on the sample faces to minimize thermal contact resistances).

The proposed device represented in figure 1 is based on the use of a simple heating element inserted between a plane face sample of the material to be characterized and a sample of an insulating material (polystyrene) of perfectly known thermal effusivity. The heating element and the samples have the same area so that the heat transfer may be considered as unidirectional as long as the convective heat losses are negligible. The initial temperature distribution in the sample and in the heating element is uniform. A first thermocouple is set on the heating element face in contact with the insulating material (front face). A second thermocouple is fixed on the unheated face of the sample in contact with the insulating

material (rear face). The sample thickness is chosen so that the hypothesis of a semi-infinite medium for the sample remains true for at least 30 s. The principle of the method is to use the front face temperature $T_0(t)$ recording at the beginning (when $T_0(t)$ is insensitive to the thermal conductivity λ and when convective heat losses may be neglected) to estimate the sample thermal effusivity E, the thermal capacity mc of the heating element and the thermal contact resistances R_{c1} and R_{c2} on the two faces of the heating element. A complete modelling of heat transfer in the sample to be characterized and in the insulating samples associated with a parameter estimation method will then be used to estimate the thermal conductivity λ of the sample by processing the rear face temperature $T_2(t)$ recording. The time estimation interval is 0–90 s when the convective heat losses are negligible.

The main advantages of this method are the very low cost of the probe, a simple to use device and an estimation method based on a complete simulation model of the front face temperature $T_0(t)$ and of the rear face temperature $T_2(t)$. This method does not need either the estimation of the lateral face heat losses or the choice of a restricted time interval for the parameter estimation.

2. Modelling

Using the quadrupole formalism presented by Maillet *et al* [12], neglecting the convection lateral heat losses and considering the heating element as a thin system (no thermal gradient in its thickness), the following matrix relations can be written:

$$\begin{bmatrix} \theta_0 \\ \Phi_{01} \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ mcp & 1 \end{bmatrix} \begin{bmatrix} 1 & R_{c1} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} 1 & R_{c2} \\ 0 & 1 \end{bmatrix}$$
$$\times \begin{bmatrix} & \theta_3 \\ E_i & S\sqrt{p}\theta_3 \end{bmatrix}$$

$$\begin{bmatrix} \theta_0 \\ \Phi_{04} \end{bmatrix} = \begin{bmatrix} 1 & R_{c3} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} & \theta_4 \\ E_i & S\sqrt{p}\theta_4 \end{bmatrix}$$

$$\frac{\varphi_0}{p} = \Phi_{01} + \Phi_{04}$$

where

$$\begin{bmatrix} A & B \\ C & D \end{bmatrix} = \begin{bmatrix} \text{ch}(qe) & \frac{1}{\lambda q S}\text{sh}(qe) \\ \lambda q S\,\text{ch}(qe) & \text{ch}(qe) \end{bmatrix} \quad \text{with} \quad q = \sqrt{\frac{p}{a}}$$

and

- θ_0 is the Laplace transform of the difference $T_0(t) - T_0$ $(t = 0)$,
- θ_3 is the Laplace transform of the difference $T_3(t) - T_3$ $(t = 0)$,
- R_{c1} is the contact resistance at the interface sample/heating element (°C W^{-1}),
- R_{c2} is the contact resistance at the interface sample/insulating material (°C W^{-1}),
- R_{c3} is the contact resistance at the interface heating element/insulating material (°C W^{-1}),
- m is the mass of the heating element (kg),
- c is the specific heat of the heating element (J kg^{-1} °C^{-1}),
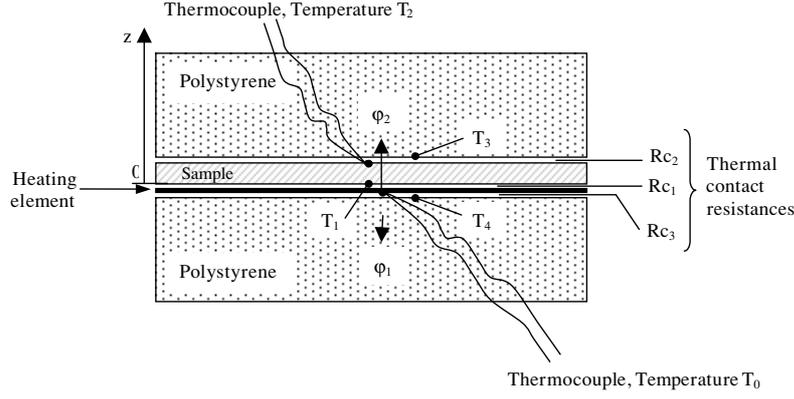- E is the thermal effusivity of the material to be characterized (J m^{-2} °C^{-1} s$^{-1/2}$),

Figure 1. Experimental device.

- E_i is the thermal effusivity of the insulating material ($J\,m^{-2}\,{}^{\circ}C^{-1}\,s^{-1/2}$),
- a is the thermal diffusivity of the material to be characterized ($m^2\,s^{-1}$),
- p is the Laplace parameter,
- S is the heating element area (m^2),
- φ_0 is the heat flux produced in the heating element (W).

The first matrix relation may also be written as

$$\begin{bmatrix} \theta_0 \\ \Phi_{01} \end{bmatrix} = \begin{bmatrix} A_{03} & B_{03} \\ C_{03} & D_{03} \end{bmatrix} \begin{bmatrix} \theta_3 \\ E_i S \sqrt{p}\,\theta_3 \end{bmatrix}$$

where

$$\begin{bmatrix} A_{03} & B_{03} \\ C_{03} & D_{03} \end{bmatrix} = \left[\begin{array}{c} (A + R_{c1}C) \\ mcpA + (1 + mcpR_{c1})C \end{array} \right.$$

$$\left. \begin{array}{c} (A + R_{c1}C)R_{c2} + (B + R_{c1}D) \\ [mcpA + (1 + mcpR_{c1})C]R_{c2} + mcpB + (1 + mcpR_{c1})D \end{array} \right].$$

It can be deduced that

$$\theta_0(p) = \frac{\frac{\varphi_0}{p}}{\frac{C_{03} + D_{03}E_i S\sqrt{p}}{A_{03} + B_{03}E_i S\sqrt{p}} + \frac{E_i S\sqrt{p}}{1 + R_{c3}E_i S\sqrt{p}}}. \tag{1}$$

The thermal balance between the heating element and the rear face of the sample at temperature $T_2(t)$ can also be written as

$$\begin{bmatrix} \theta_0 \\ \Phi_{01} \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ mcp & 1 \end{bmatrix} \begin{bmatrix} 1 & R_{c1} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta_2 \\ \Phi_2 \end{bmatrix}$$

$$= \begin{bmatrix} A_{02} & B_{02} \\ C_{02} & D_{02} \end{bmatrix} \begin{bmatrix} \theta_2 \\ \Phi_2 \end{bmatrix}$$

where

$$\begin{bmatrix} A_{02} & B_{02} \\ C_{02} & D_{02} \end{bmatrix}$$

$$= \left[\begin{array}{cc} A + R_{c1}C & B + R_{c1}D \\ mcpA + (1 + mcpR_{c1})C & mcpB + (1 + mcpR_{c1})D \end{array} \right].$$

By combining the previous relation with the following one:

$$\begin{bmatrix} \theta_0 \\ \Phi_{01} \end{bmatrix} = \begin{bmatrix} A_{03} & B_{03} \\ C_{03} & D_{03} \end{bmatrix} \begin{bmatrix} \theta_3 \\ E_i S \sqrt{p}\,\theta_3 \end{bmatrix}$$

the Laplace transform of the rear face temperature can be deduced as

$$\theta_2(p) = \left[D_{02} - B_{02} \frac{C_{03} + D_{03}E_i S\sqrt{p}}{A_{03} + B_{03}E_i S\sqrt{p}} \right] \theta_0(p). \tag{2}$$

Applying the inverse Laplace transform by the Stehfest method [13], the temperatures $T_0(t)$ and $T_2(t)$ may finally be calculated by

$$T_i(t) = \frac{\ln(2)}{t} \sum_{j=1}^{n_L} V_j \theta_i \left(\frac{j \ln(2)}{t} \right)$$

where $V_1 = 0.083\,3333$, $V_2 = -32.0833$, $V_3 = 1279$, $V_4 = -15\,623.667$, $V_5 = 84\,244.167$, $V_6 = -36\,957.5$, $V_7 = 375\,911.667$, $V_8 = -340\,071.667$, $V_9 = 164\,062.5$ and $V_{10} = -32\,812.5$.

The lateral convective losses can be taken into account in a simple way with the hypothesis that the temperature is uniform in any plane parallel to the front face. In this case studied by Ladevie [9], all the previous relations remain valid when replacing the Laplace parameter p by

$$p + \frac{hap_e}{\lambda S}$$

where p_e is the perimeter of the sample lateral area (m), a is the sample diffusivity ($m^2\,s^{-1}$), h is the convective heat transfer coefficient ($W\,{}^{\circ}C^{-1}\,m^{-2}$), λ is the sample thermal conductivity ($W\,{}^{\circ}C^{-1}\,m^{-1}$) and S is the heating element area (m^2).

Actually, the temperature of the sample lateral faces is slightly lower than the average temperature and than the centre temperature in the same plane, so that the lateral heat losses are overestimated in the simplified expression proposed by Ladevie [9].

3. Sensitivity analysis

Using relations (1) and (2) of the complete model and the Stehfest method, the reduced sensitivities of the two temperatures $T_0(t)$ and $T_2(t)$ to the parameters E, λ, R_{c1} and mc have been calculated numerically. The reduced sensitivity of the temperature T to a parameter X is defined as $X\frac{\partial T}{\partial X}$. The sample thickness has been chosen so that the sensitivity of $T_0(t)$ to λ is negligible between 0 and 30 s to allow an accurate estimation of the effusivity E. It has been checked that the sensitivities of $T_0(t)$ and $T_2(t)$ to the thermal contact resistances R_{c1} and R_{c3} with the insulating material are totally negligible. Their values have been fixed to $10^{-10}\,{}^{\circ}C\,W^{-1}$ because higher values for R_{c2} lead to numerical instabilities on $T_2(t)$ for short time values. The theoretical results obtained for pyroceram
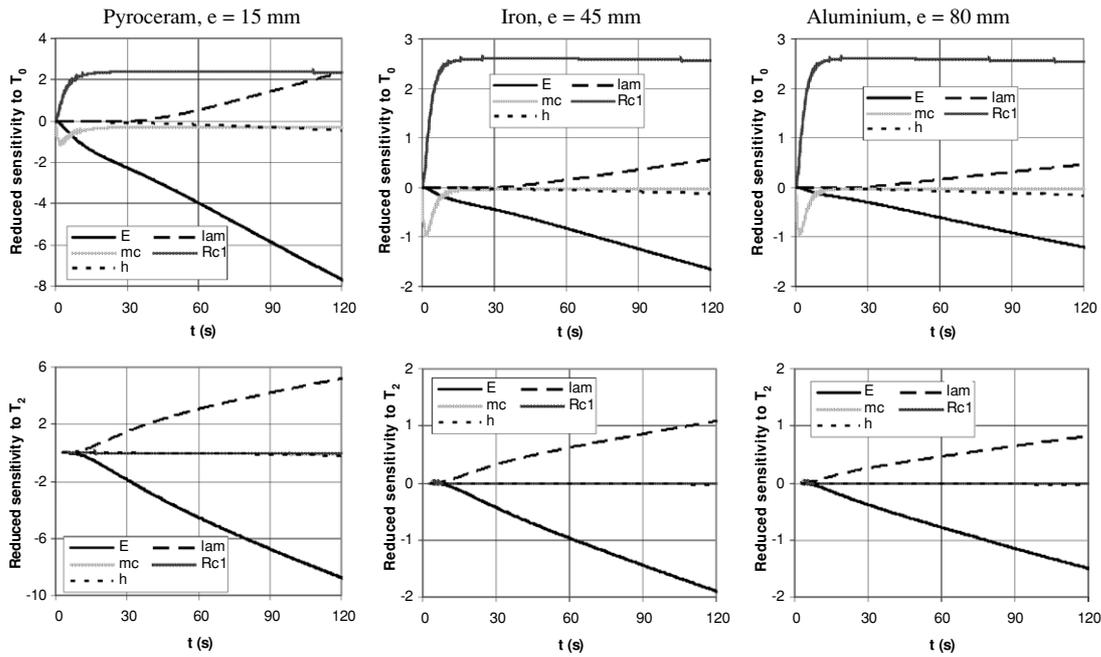
Figure 2. Reduced sensitivity of $T_0(t)$ and $T_2(t)$ to the parameters E, mc, R_{c1} and λ.

($\lambda = 3.98$ W m^{-1} °C^{-1}, $E = 2892$ J m^{-2} °C^{-1} s$^{-1/2}$, $a = 1.89 \times 10^{-6}$ m^2 s^{-1}), for iron ($\lambda = 72.7$ W m^{-1} °C^{-1}, $E = 115\,992$ J m^{-2} °C^{-1} s$^{-1/2}$, $a = 2.07 \times 10^{-5}$ m^2 s^{-1}) and for aluminium ($\lambda = 204$ W m^{-1} °C^{-1}, $E = 15\,567$ J m^{-2} °C^{-1} s$^{-1/2}$, $a = 8.418 \times 10^{-5}$ m^2 s^{-1}) are plotted as an example in figure 2; the physical properties used in the calculation are extracted from [14]. Calculations have been done with the following values: $S = 24.5$ cm^2, $mc = 2.0$ J °C^{-1} and $S\,R_{c1} = 0.002$ °C m^2 W^{-1}.

One can note that the sensitivity of T_0 to the thermal capacity mc of the heating element is constant only after 15 s so that a model neglecting the heating element inertia (with $mc = 2.0$ J °C^{-1}) could not process the first 15 s of the temperature $T_0(t)$ recording. This emphasizes the interest of using a complete model. The temperature $T_2(t)$ is not sensitive to the parameter mc.

The sensitivity of T_0 to the thermal contact resistance R_{c1} between the heating element and the sample becomes constant only after 15 s as for mc.

The sensitivity of T_0 to h (overestimated in our estimation as pointed out previously and calculated with $h = 10$ W m^{-2} °C^{-1}) is negligible for times lower than 30 s.

The sensitivity of T_0 to λ is also negligible between 0 and 30 s. The sensitivity of T_0 to E is increasing and uncorrelated with the sensitivities to mc and R_{c1} between 0 and 30 s; this will allow a precise estimation of E on this time interval. One could think of using the temperature $T_0(t)$ between 30 and 90 s to estimate λ but the sensitivities of T_0 to the parameters λ and h are correlated on this time interval preventing any separate estimation. The estimation of λ becomes possible when the transverse dimensions of the samples are great compared to its thickness. In this case, the temperature at the centre of the heating element is independent of the lateral heat losses over a sufficiently long time for a correct estimation of λ.

The temperature T_2 is only sensitive to the parameter E and between 0 and 90 s; a low sensitivity to the parameter h

(with $h = 10$ W m^{-2} °C^{-1}) appears after 90 s for pyroceram. After having estimated E between 0 and 30 s by a hot plate type model applied to $T_0(t)$, it will thus be possible to estimate λ with a good precision using a complete model for $T_2(t)$ between 0 and 90 s.

It can be noted that the considered thickness is the minimum acceptable value for each material. The parameter estimation is still possible for any greater value of the thickness but it will require a time estimation longer than 90 s to keep the temperature $T_2(t)$ sufficiently increasing.

4. Parameter estimation method

Initial values are given to the parameters E, mc and R_{c1}, and the values of R_{c2} and R_{c3} are fixed equal to 10^{-10} °C W^{-1}. The theoretical curve $T_0(t)$ is calculated using the Stehfest method to relation (1). The Newton method integrated in the solver function of a common spreadsheet is used to estimate the values of the parameters E, mc and R_{c1} minimizing the sum of the quadratic error between the theoretical and experimental curves $T_0(t)$ between 0 and 30 s. These values are then considered as known data.

An initial value is given to the thermal conductivity λ. The theoretical curve $T_2(t)$ is calculated by applying the Stehfest method to relation (2). As previously done, the Newton method integrated in the solver function of a common spreadsheet is used to estimate the value of the parameter λ minimizing the sum of the quadratic errors between the theoretical and experimental curves $T_2(t)$ between 0 and typically 90 s.

5. Experimental results and discussion

The heating element used was a Minco HK 5592 in kapton, with an electrical resistance of 53.9 Ω and an area of 44.5×44.5 mm^2.

Table 1. Mean experimental values obtained with the hot plate device with two temperature measurements, with the hot disc and with the differential scanning calorimeter.

			Titanium alloy TA6V	Steel Z35CDV5	Steel A60	Aluminium alloy AU4G
Hot plate	E	(J m^{-2} °C^{-1} s$^{-1/2}$)	4020 (0.8)	9565 (3.1)	13 771 (2.0)	18 073 (1.8)
	λ	(W m^{-1} °C^{-1})	6.61 (1.1)	25.6 (5.0)	52.6 (2.9)	138.5 (2.4)
	a	(m^2 s^{-1})	2.70×10^{-6} (0.9)	7.16×10^{-6} (5.7)	1.45×10^{-5} (4.0)	5.88×10^{-5} (2.2)
	ρc	(J m^{-3} °C^{-1})	2.44×10^{6} (0.8)	3.62×10^{6} (3.1)	3.58×10^{6} (2.9)	2.36×10^{6} (1.9)
Hot disc	λ	(W m^{-1} °C^{-1})	6.60	27.3	55.5	155.3
	a	(m^2 s^{-1})	2.83×10^{-6}	7.28×10^{-6}	1.55×10^{-5}	5.80×10^{-6}
DSC + volumeter	ρc	(J m^{-3} °C^{-1})	2.32×10^{6}	3.35×10^{6}	3.54×10^{6}	2.20×10^{6}
Deviation hot plate/	λ	(%)	0.2	6.3	5.2	10.8
hot disc	a	(%)	4.6	1.6	6.5	1.4
Deviation hot plate/DSC	ρc	(%)	5.4	5.3	1.0	7.4

Note. The values within parentheses denote standard deviation (%).

Temperatures have been measured with type T thermocouples made of wires 0.003 mm in diameter. The measurement of the rear face temperature T_2 has been realized with separated contacts since the tested samples were electrical conductors. The front face temperature T_0 has been measured by a thermocouple fixed on the face of the heating element in contact with the thermal insulator to limit the thermal contact resistance R_{c1} between the heating element and the sample. The cold junction of the thermocouples was inserted into isothermal metallic pieces and the electrical tensions were recorded with an Almemo 2290-5 apparatus. The acquisition time was 0.1 s and the resolution was 1 μV corresponding to 0.025 °C for a type T thermocouple.

The tested samples were the following:

- Titanium alloy TA6V with dimensions 44.5 × 44.5 × 16.7 mm^3.
- Steel Z35CDV5 with dimensions 44.5 × 44.5 × 28 mm^3.
- Steel A60 with dimensions 44.5 × 44.5 × 50 mm^3.
- Aluminium alloy AU4G with dimensions 44.5 × 44.5 × 80 mm^3.

Five measurements have been realized on each sample with the described device. The thermal diffusivity and conductivity have also been measured on the same samples with the 'hot disc thermal constant analyser' apparatus. The thermal capacity c has been measured with the differential scanning calorimeter 'Setaram TG-DSC111' and the density ρ has been measured with the mercury volumemeter described by Talla *et al* [15].

All the results are reported in table 1; the results for the hot plate method are the average of five measurements. One can note a good fit between the values measured by our hot plate method and those measured with the hot disc: the mean deviation is 5.6% for the thermal conductivity λ and 3.5% for the thermal diffusivity a. A good fit may also be noted between the values of the product ρc measured by our hot plate method and those measured with the DSC + volumemeter: the mean deviation is 4.8%.

As an example, figure 3 presents the experimental and theoretical curves $T_0(t)$ and $T_2(t)$ obtained with titanium alloy TA6V and with aluminium alloy AU4G. The fitting between the curves is quite satisfying.
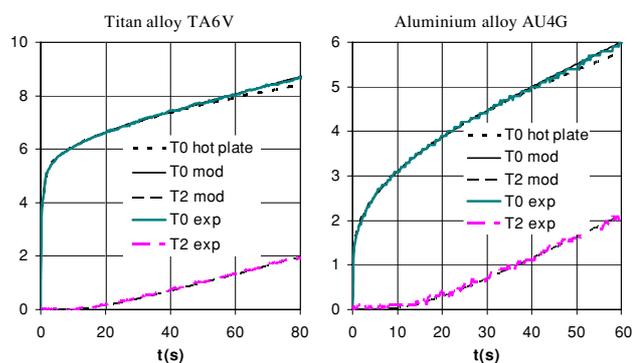

Figure 3. Experimental and theoretical curves $T_0(t)$ and $T_2(t)$ (°C) obtained with titanium and aluminium alloys.

(This figure is in colour only in the electronic version)

6. Conclusion

This study has shown that it is possible to estimate simultaneously with an acceptable precision the thermal conductivity and effusivity of high conductivity materials (>6 W m^{-1} °C^{-1}) with a simple low-cost device: a transient hot plate applied to a single sample with recording of the front and rear face temperatures measured with thermocouples. The specificity of the method is based on the use of all the recorded temperature measurements and on the simplicity of the parameter estimation method: the estimation is realized using the solver function of a very common spreadsheet. The method has been tested on four metallic alloys with thermal conductivities varying from 6 to 140 W m^{-1} °C^{-1} with quite satisfying results since the average deviation from measurements realized with a hot disc or a scanning differential calorimeter was around 5%.

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