Thermal diffusivity identification by 2^{nd} derivative analysis of transient temperature profile

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Abstract This paper deals with a new method to identify the thermal diffusivity of isotropic materials. Contrary to the flash method, a continuous and constant heating is applied on the rear surface of the sample. An analytical solution of the 1-D transient equation is developed based on Green's function and gives the temperature profile on the opposite surface at the first moments. Regarding the 2^{nd} derivative of this equation, we found that it reaches a maximum value at a characteristic time $t_{c2^{nd}}$ which is proportional to the thermal diffusivity α and the sample thickness e_s . Experiments are performed on a well-known material, ARMCO Iron, to check the feasibility and the accuracy of the identification with noisy temperature measurements. The method is also applied to insulation materials. Uncertainties on the identified values are discussed and rules are given to

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minimize these uncertainties. The results show that this method can be a real alternative or complementary to the flash method.

Keywords Thermal diffusivity · Characteristic time · 2^{nd} derivative · Green Function

1 Methods for the thermal diffusivity identification

The thermal diffusivity defines the ability of a material to transfer the heat in transient conditions. This parameter depends on the thermal conductivity λ and on the ability of the material to store the heat, represented by the product of the specific heat c_p by the material density ρ . In some applications, the knowledge of the thermal diffusivity is essential to solve local heating problems. For example, increasing the thermal diffusivity of thin layer thickness on electronic components can help to decrease local heating and reduce the maximum temperature reached.

Methods have been proposed over the last decades to identify this value. The photoacoustic technique is based upon the measurement of a photoacoustic signal as a function of the modulation frequency in the region where the thermal diffusion length equals to the sample thickness [1]. The Thermal Wave Interferometry also uses a periodical heating of a sample, typically by a modulated laser beam. This technique is particularly adapted for coatings and thin slabs. The thermal waves are partially reflected at the materials interface and the signal analysis allows identifying the thermal thickness [2]. Complementary photothermal techniques have been recently investigated [3] combining photopyroelectric calorimetry and infrared lock-in IR thermography. These allow identifying simultaneously the thermal effusivity and the thermal diffusivity of porous and semi-transparent solids.

Nowadays, the flash method is the most commonly used technique to identify the thermal diffusivity of isotropic solids but is also suitable to determine specific heat for metals [4]. Most of commercial equipments are based on this method and the standards recommend its application to measure the thermal diffusivity [5][6]. This consists of imposing a very short heat flux on a sample surface and recording the temporal evolution of the temperature on the rear surface. This signal is easily usable compared to the temperature evolution on the front face which presents singularities on shorter times. Parker has suggested to identify the thermal diffusivity from a characteristic time $t_{1/2}$ [7]. This is the time to reach the half of the temperature variation T_{lim} and is linked to the thermal diffusivity α and to the sample thickness e_s by the equation (Eq.1).

$$\alpha = \frac{0.139 * e_s^2}{t_{1/2}} \quad (1)$$

This characteristic time $t_{1/2}$ is suitable because the sensibility function $\frac{\partial T}{\partial \alpha}$ reaches its maximum for a time very close to this value. This means that an error on the temperature value has less influence on the α estimated value and then the noise on the thermogram does not affect significantly the response. As various materials are sensitive to oxygen and moisture in ambient conditions, Pohlmann [8] recently develops an apparatus to perform the flash method in inert atmosphere. This also reduces the convective heat losses during measurements.

In practice some experimental problems induce a bias in the parameter identification. The duration of the impulsion can be very short with laser beam but not enough regarding the model boundary condition. Degiovanni has suggested

corrections [9], especially in growing the sample thickness at the cost of the signal-noise ratio. Using a thermally thick sample allows also reducing the influence of the non-uniformity of the flash spot [10]. Measuring a mean temperature on the entire rear surface can also help to bypass the non-uniformity problem. One limitation in the flash method is the necessity to use a high heat flux to get a usable thermal response on the rear surface because of the short duration. This induces high thermal gradient on the front surface and then a high temperature in the first moments. The temperature gradient can cause variations on thermal properties along the sample thickness. Their influence have been studied by Soilili [11] and he shows that a model with constant thermal properties corresponding to the limit temperature T_{lim} . To take into account these experimental uncertainties, the quadripole method [12] has been applied by several author who identifies α by parameter estimation methods [13][14][15].

Here we develop a method based on a constant heat flux heating on the front face of a sample and the analysis of the 2^{nd} order time derivative of the temperature profile on the rear surface. The resolution of the 1D transient heat equation by Green's function leads to a new characteristic time t_{c2} which is also proportional to the sample thickness and the thermal diffusivity. Experiments are performed on a calibrated material, ARMCO Iron, to demonstrate the feasibility to identify α from a thermogram and the accuracy of this new method. Then the method is applied to a ceramic and resins to illustrate that it is suitable for insulation materials. Using a constant heat flux allows getting a lower thermal gradient compared to the flash method. Some rules are given to choose the appropriate sample thickness and heat flux intensity regarding the studied material.

2 A method based on the 2nd derivative temperature profile

2.1 Analytical solution and derivatives at first instants

Fig. 1 Schematic representation of the 1D heat transfer configuration

In this method, one side of a flat plate is heating continuously and uniformly with a constant heat flux density φ_0 as illustrated on the figure Fig.1. The back surface is considered cooled down by convective and radiative transfers. Regarding the boundary condition on the heated surface, the heat losses are considered negligible compared to the heat flux imposed. The heat flux density experimentally imposed by the LASER is higher than 10^8mW.m^{-2} . On the heated surface, the maximum temperature elevation is lower than 50K at the end of a test. The convective coefficient can be estimated in natural convection by the equation $h_{conv} = 1320 * (\Delta T / \varnothing_{sample})^{0.25} \text{mW.m}^{-2}.\text{K}^{-1}$ [16] and is then lower than $12 * 10^3 \text{mW.m}^{-2}.\text{K}^{-1}$. For the same temperature elevation of 50K, the equivalent radiative coefficient h_{rad} is lower than $8 * 10^3 \text{mW.m}^{-2}.\text{K}^{-1}$ if we consider an emissivity $\epsilon=1$, a background temperature of 293K and a surface temperature of 343K. This seems that

the total heat loss on the heated surface is lower than 10^6W.m^{-2} and are then 100 times lower compared to the heating intensity. On the lateral face, the heat losses are lower because the maximum temperature elevation is lower compared to the heat surface. Then the plate's temperature field is assumed unidirectional in the following model during the heating period.

Before heating, the whole sample is at ambient temperature T_∞ and no convective and radiative exchanges exist. The temperature difference $\theta(x,t)$ defines the gap between the sample temperature $T(x,t)$ and the ambient temperature T_∞ . The heat equation, the boundary conditions and the initial condition can be expressed by the system (Eq.2). On the heated surface ($x=e_s$), $\varphi(t)$ is equal to 0 for $t < 0$ and takes a constant value φ_0 for $t \geq 0$. The convective and radiative transfer are considered by an equivalent coefficient h which is assumed to be constant with the sample temperature.

$$\left\{ \begin{array}{l} \frac{1}{a} \frac{\partial \theta(x,t)}{\partial t} - \frac{\partial^2 \theta(x,t)}{\partial x^2} = 0 \\ \theta(x,0) = 0 \\ \frac{\partial \theta}{\partial x}(x,t)|_{x=e} = \frac{\varphi(t)}{\lambda} \\ \frac{\partial \theta}{\partial x}(x,t)|_{x=0} = \frac{h}{\lambda} * \theta(0,t) \end{array} \right. \quad (2)$$

The system (Eq.2) can be expressed using the Green's function theory giving the system (Eq.3)(a) and Laplace transforms are applied (Eq.3)(b) to solve it easily. In the equation (Eq.3)(b), the parameter q is defined according to the Laplace Transporm parameter p and the thermal diffusivity α ($q = \sqrt{p/\alpha}$).

$$(a) \left\{ \begin{array}{l} \frac{1}{a} \frac{\partial G(x, \xi, t)}{\partial t} - \frac{\partial^2 G(x, \xi, t)}{\partial x^2} = \delta(x - \xi) \delta(t) \\ G(x, \xi, 0) = 0 \quad \text{if } t < 0 \\ \frac{\partial G}{\partial x}(x, \xi, t)|_{x=e} = 0 \\ \frac{\partial \theta}{\partial x}(x, t)|_{x=0} = \frac{h}{\lambda} * G(0, \xi, t) \end{array} \right. \rightarrow (b) \left\{ \begin{array}{l} q^2 \bar{G} - \frac{\partial^2 \bar{G}}{\partial x^2} = \delta(x - \xi) \\ \bar{G}(x, \xi, 0) = 0 \\ \frac{\partial \bar{G}}{\partial x}|_{x=e} = 0 \\ \frac{\partial \bar{G}}{\partial x}|_{x=0} = \frac{h}{\lambda} * \bar{G}(0, \xi, p) \end{array} \right. \quad (3)$$

The solution $\bar{G}$ of the equation (Eq.3)(b) is expressed as a sum of 2 functions as denoted by the equation (Eq.4). Then the 1^{rst} and the 2^{nde} derivative of $\bar{G}$ are given by (Eq.5) considering that $\frac{x - \xi}{|x - \xi|} = 2E(x - \xi) - 1$ and the property of the Heaviside function ($\frac{\partial}{\partial x}(E(x - \xi)) = \delta(x - \xi)$).

$$\bar{G}(x, \xi, p) = F(p)e^{-q|x-\xi|} + D(x, \xi, p) \quad (4)$$

$$\left\{ \begin{array}{l} \frac{\partial \bar{G}(x, \xi, p)}{\partial x} = -q \frac{x - \xi}{|x - \xi|} F(p)e^{-q|x-\xi|} + \frac{\partial D}{\partial x} \\ \frac{\partial^2 \bar{G}(x, \xi, p)}{\partial x^2} = q^2 F(p)e^{-q|x-\xi|} - 2q\delta(x - \xi)F(p)e^{-q|x-\xi|} + \frac{\partial^2 D}{\partial x^2} \end{array} \right. \quad (5)$$

This leads to the following equations :

$$\left\{ \begin{array}{l} F(p) = \frac{1}{2q} \\ q^2 D(x, \xi, p) - \frac{\partial^2 D(x, \xi, p)}{\partial x^2} = 0 \end{array} \right. \quad (6)$$

The differential equation based on D has a general solution expressed in (Eq.7) inducing a general solution for the Green function $\bar{G}(x, \xi, p)$.

$$\left\{ \begin{array}{l} D(x, \xi, p) = A(\xi, p)e^{-qx} + B(\xi, p)e^{qx} \\ \bar{G}(x, \xi, p) = \frac{1}{2q}e^{-q|x-\xi|} + A(\xi, p)e^{-qx} + B(\xi, p)e^{qx} \end{array} \right. \quad (7)$$

Then the boundary conditions terms can be expressed according to A and B as denoted in (Eq.8). The deduced terms A and B in can be obtained (Eq.9).

$$\begin{cases} \frac{e^{-q\xi}}{2q}(q-H) = (H+q)A(\xi, p) + (H-q)B(\xi, p) \\ \frac{e^{-q(e_s-\xi)}}{2q} = -A(\xi, p)e^{-qe_s} + B(\xi, p)e^{qe_s} \end{cases} \quad (8)$$

$$\begin{cases} A(\xi, p) = \frac{-(H-q) \left[\frac{e^{-q(e_s-\xi)}}{2} + \frac{e^{-q(\xi-e_s)}}{2} \right]}{q[(H+q)e^{qe_s} + (H-q)e^{-qe_s}]} \\ B(\xi, p) = \frac{-(H-q) \frac{e^{-q(e_s+\xi)}}{2} + (H+q) \frac{e^{-q(e_s-\xi)}}{2}}{q[(H+q)e^{qe_s} + (H-q)e^{-qe_s}]} \end{cases} \quad (9)$$

The Green function can then be expressed regarding p, α and the sample thickness (Eq.10) and its value on the free surface ($x=0$) becomes (Eq.11).

$$\bar{G}(x, \xi, p) = \left[\frac{(H-q) * \left(e^{-q(x-\xi)} - e^{q(\xi-x)} - e^{-q(\xi-x)} - e^{q(2e_w-x-\xi)} \right) + (H+q) * \left(e^{q(x+\xi)} + e^{-q(x-\xi-2e_w)} \right)}{2q((H-q) + (H+q) * e^{2qe_w})} \right] \quad (10)$$

$$\bar{G}(0, 0, p) = \frac{e^{-2qe_w} + 1}{(H-q) * e^{-2qe_w} + (H+q)} \quad (11)$$

The Laplace transform of the temperature difference θ on the non-heated surface is then deduced in (Eq.12). Considering that p is very large, this form behaves like $\theta^*(0, p)$ given in (Eq.13)

$$\bar{\theta}(0, p) = 2 \frac{\varphi_0}{\lambda p} \left[\frac{1}{(H-q) * e^{-qe_w} + (H+q) * e^{qe_w}} \right] \quad (12)$$

$$\bar{\theta}(0, p \rightarrow \infty) : \bar{\theta}^*(0, p) = 2 \frac{\varphi_0}{\lambda} \frac{e^{-e_w q}}{pq} \quad (13)$$

According to a theorem cited in [17], for time close to 0, the inverse Laplace transform of the function $\theta(0, p)$ can be assumed equal to the inverse Laplace transform of $\theta^*(0, p)$. The approached temperature function $\theta^*(0, t)$ is given in the equation system (Eq.14). The first and the second derivative with time are also

given.

$$\begin{cases} \theta^*(0, t) = 2 \frac{\phi_0}{\lambda} \left[2 \sqrt{\frac{\alpha t}{\pi}} e^{\frac{-e_w^2}{4\alpha t}} - e_w * \operatorname{erfc} \left(\frac{e_w}{2\sqrt{\alpha t}} \right) \right] \\ \frac{d\theta^*}{dt}(0, t) = 2 * \frac{\phi_0}{\lambda} * \sqrt{\frac{a}{\pi t}} * \exp\left(\frac{-e_s^2}{4at}\right) \\ \frac{d^2\theta^*}{dt^2}(0, t) = \frac{\phi_0}{\lambda} * \sqrt{\frac{a}{\pi t^3}} * \exp\left(\frac{-e_s^2}{4at}\right) * \left(\frac{e_s^2}{2at} - 1\right) \end{cases} \quad (14)$$

2.2 Analysis of the theoretical solution

Let us define the characteristic time t_c (Eq.15).

$$t_c = \frac{e_s^2}{2 * \alpha} \quad (15)$$

The approximated temperature variation θ^* can be expressed regarding t_c . Besides θ^* can be also adimensioned by dividing the expression by $\frac{\phi_0 * e_s}{\lambda}$ which is homogeneous to a temperature. The figure Fig.2 represents the temporal evolution of the adimensioned temperature θ^* regarding an adimensioned time β ($t = \beta * t_c$).

Fig. 2 Theoretical temporal evolution of θ on the rear surface

The real temperature θ on the non-heated surface is also plotted. The second one is obtained numerically by 1-D finite difference model. The temperature and the time are also adimensioned for a comparison. A good agreement between the temporal evolution are found in the shortest time between numerical and analytical model. For $\beta > 1.5$, the analytical model begins to diverge to the numerical solution and the curves slopes are clearly different for larger time values ($\beta > 2$). This is the consequence of the assumption made to do the inverse Laplace transform of the equation (Eq.13).

Looking at the 1st and 2nd temperature derivatives, the analytical solution offers two interesting characteristic times. The derivatives of the analytic and numerical solutions are also adimensioned and plotted on the figure Fig.3 for β in the range 0-2.

Fig. 3 Theoretical temporal evolution of 1st and 2nd derivative of θ with time

The θ curves divergence is highlighted by the 1st derivative. The derivative of the numerical solution tends to an asymptotic curve for $\beta > 2$. Regarding the ana-

lytical solution, the 1^{rst} derivative reaches its maximum at $\beta = 1$. In other words, the characteristic time t_c defines the time at which the slope of θ^* is maximum.

A second characteristic time can be defined regarding the second derivative of θ and θ^* . Graphically, it can be observed a maximum value close to $\beta = 0.18$. Contrary to the 1^{rst} derivative, the maximum of the two curves are reached for the same β value. This means that the 2^{nde} derivative of θ^* and of a temperature measurement will reach a maximum at the same time. Regarding the 3rd derivative of θ^* with time, the expression (Eq.16) is obtained and the polynomial term allows finding the exact value of β when $\frac{\partial^2 \theta^*}{\partial t^2}$ is maximum.

$$\frac{d^3 \theta^*}{dt^3}(0, t) = \frac{\phi_0}{\lambda} * \sqrt{\frac{a}{\pi t^9}} * \exp\left(\frac{-e_s^2}{4at}\right) * \left(\frac{3}{2}\beta^2 - 3t_c\beta + \frac{t_c^2}{2}\right) \quad (16)$$

This leads to the solutions $\beta = 1 - \frac{\sqrt{6}}{3}$ and $\beta = 1 + \frac{\sqrt{6}}{3}$. The first one is on interest corresponding to a β value close to 0.18. The second characteristic time $t_{c_{2nde}}$, proportional to t_c , can then be defined (Eq.17).

$$t_{c_{2nde}} = \left(1 - \frac{\sqrt{6}}{3}\right) \frac{e_s^2}{2 * a} \quad (17)$$

This leads to the possibility to identify directly the thermal diffusivity from the second derivative of an experimental profile. Finding its maximum, α is then obtained by the expression (Eq.18). The thermal diffusivity can be identified without the exact knowledge of the imposed heat flux and the thermal conductivity of the sample. The only required term prior to the measurement is the sample thickness. However the time $t_{c_{2nde}}$ can be very short especially for thin sample and metallic materials. Equipments with high frequency in temperature measurement are required and the measure have to be well synchronized with the heating start

to get workable data.

$$\alpha = \left(1 - \frac{\sqrt{6}}{3}\right) \frac{e_s^2}{2t_{c_{2nde}}} \quad (18)$$

3 Experimental validation

3.1 Experimental setup

An experiment is designed to validate this method. The figure Fig.4 shows the experimental device used to heat the sample and record the temperature profile.

Fig. 4 Experimental setup

It consists of a 200W CO₂ laser (SYNRAD Firestar f200) working on the infrared wavelength range 10.2-10.7μm. The power can be adjusted from 5 to 100% in our tests. A meniscus lens is placed between the laser source and the heated sample to adjust the spot size at 8mm in diameter regarding our samples' dimensions. A red visible pointer aligned with CO₂ laser allows adjusting the sample position. The sample is placed on a low diffusivity material. A hole of

6mm in diameter is machined on the insulation plate to allow the temperature measurement on the sample rear surface and to prevent the lightning of the IR detector. The sample, the heat source and the IR detector are placed inside a closed chamber. This allows reducing air displacement around the sample and the influence of surrounding lights. It also limits the LASER reflections for safety reason.

A thermogram on the rear surface of the sample is recorded by an infrared camera FLIR SC7200 working on the wavelength range $[3.5 - 5.1\mu\text{m}]$. This allows reaching a frequency of 500Hz by measuring the entire back surface at room temperature. Measurements are performed for an integration time of $469\mu\text{s}$, suitable for the temperature range $[288 - 373\text{K}]$. The sample is covered with a mat black paint on all the faces. This allows obtaining the real temperature from the infrared measurement and reducing reflection losses from the LASER infrared heating. This paint is commonly use in our laboratory and has a high emissivity ($\epsilon=0.93$) which is nearly constant on the temperature range 293-373K. The calibration of the emissivity has been performed on the bandwidth $3\text{-}5\mu\text{m}$.

As the characteristic time can be very short for high diffusivity materials like metals, it is necessary to have a good synchronization of the heating start and the thermogram acquisition start. The temperature recording is then triggered by the lase switch on. The camera is linked to a function generator which send periodically pulses to the trigger input. The electrical circuit stays open while the lase is switch off. Starting to heat the sample induces to closed the circuit and then triggers the temperature recording.

3.2 Data processing

Here is described the procedure to obtain the characteristic time from raw data acquired with the IR camera. To illustrate this procedure, the temperature data from an ARMCO Iron sample chosen as reference material are used. More details are given in the following part on the samples and on the testing conditions.

Fig. 5 Rear surface thermograms on Armco Iron sample A3

A temporal temperature profile is determined by meaning the values measured on an area on the rear surface for each time. The figure Fig.5 shows four frames extracted from the IR measurement at 4 different times : before heating ($t=0s$), 0.15s before an identified characteristic time t_{c2nde} , at this identified characteristic time t_{c2nde} and 0.15s after this characteristic time t_{c2nde} . The chosen area is a

square of approximately 2mm in side corresponding to 144 pixels on the IR detector as represented on the thermograms. The sample is placed on an insulation material but heat diffusion still exists. To avoid the influence of this phenomenon on our measurement, the temperature is averaged on an area on the center of the sample.

Meaning the temperature on the 2mm square area allows reducing a part of the noise but insufficiently to get directly a suitable 2^{nde} derivative from the temperature data. Deriving two times a vector of noisy signal is not practicable because the effect of the noise is accentuated in the derivative. Consequently, the data need to be fitted with suitable functions. Then these functions can be derived analytically.

Before fitting the raw temperature profile, a 50Hz low pass filter is applied to remove the fluctuations from AC current and surrounding lights. Several functions have been tested on our following measurements and polynomial functions have been retained first because of their simplicity. The fitting of the experimental data are performed on these polynomial functions and on a custom function with a linear regression algorithm. The used custom function (Eq.19) is defined according to the analytical solution (Eq.14) of θ^* . A and B are the two parameters to be identified.

$$\theta^*(0, t) = A * \left(\sqrt{t} * e^{\frac{-B^2}{t}} - B\sqrt{\pi} * \operatorname{erfc}\left(\frac{B}{t}\right) \right) \quad (19)$$

The figure Fig.6a shows the raw data obtained on the ARMCO iron sample and the fitting temperature obtained with a 7th order polynomial function.

a. Full time variation

b. Close to the characteristic time $t_{c_{2nde}}$

c. 2nd derivative of the fitting functions

Fig. 6 Raw temperature profile fitting on Armco Iron sample A3

As illustrated by the residuals, a good correspondence is found between the measured data and the fitted values. The zoom view presented on the figure Fig.6b also confirms the good match close the characteristic time $t_{c_{2nde}}$.

As showed in the theoretical analysis, the approximated temperature θ^* and the real temperature measured increase similarly only at the first moments. Then the analytical solution moves apart from the real temperature value. For this reason, the fitting functions are not applied on a entire temperature record. Regarding the conclusions on the figure Fig.3, we make the choice to fit the temperature measurement until $3t_{c_{2nde}}$ approximately. This corresponds to a β value of 0.55 for which the real values and the analytical solution are still in good agreement. Because of the unknown of $t_{c_{2nde}}$ at the beginning of the fitting process, an iterative scheme is used to define the time range $0-3t_{c_{2nde}}$ according to the previous $t_{c_{2nde}}$ solution found. The convergence on the characteristic time is generally obtained after 7 iterations for a $3t_{c_{2nde}}$ variation lower than 2%.

The figure Fig.6c represents the second derivative of the 4 functions used to fit the raw data presented on Fig.6a. The maximum reached on each 2nde derivative curve corresponds to an identified characteristic time $t_{c_{2nde}}$. Each function give a characteristic time slightly different but in the same order of value. These values come from a test performed on the sample A3 described in the following part. The theoretical time $t_{c_{2nde}}$ should be 948ms and values from 956 to 1008ms are found experimentally depending of the used fitting function. The thermal diffusivity can be then directly calculated with the equation (Eq.18).

3.3 Thermal diffusivity of a standard material : ARMCO IRON

The method is firstly validated on ARMCO Iron samples because the thermal diffusivity of this material is well known. These thermal properties can be found in [18] regarding the temperature. An empiric model is given for temperatures in the range 20-770°C in (Eq.20) for T in °C. This leads to thermal diffusivity values from $2.04 \cdot 10^5 \text{ m}^2 \cdot \text{s}^{-1}$ at 293K to $1.89 \cdot 10^5 \text{ m}^2 \cdot \text{s}^{-1}$ at 353K.

$$\alpha_{Armco} = -2.058 \cdot 10^{-17} * T^4 + 3.449 \cdot 10^{-11} * T^2 - 4.057 \cdot 10^{-8} * T + 2.116 \cdot 10^{-5} \quad (20)$$

The method is applied on 4 samples with different thicknesses given in Table 1. The used micrometer has an accuracy of 0.01mm. For each sample, 25 heating process are applied to check the reproducibility of the method.

Sample	A1	A2	A3	A4
Thickness (mm)	9.08	10.43	14.52	20.01
Theoretical tc_{2nde} (ms) at T=293K	371	489	948	1800
Imposed heat flux density φ ($\text{W} \cdot \text{m}^{-2}$)	$5.1 \cdot 10^5$	$4.5 \cdot 10^5$	$3.1 \cdot 10^5$	$2.4 \cdot 10^5$

Table 1 Armco Iron samples

The initial sample temperature is closed to the room temperature around 293K. Before each test, the sample is cooled by natural convection and the next heating is applied when the temperature on the rear surface is nearly constant. In practice it corresponds to a difference between the sample temperature and the room temperature lower than 1K.

The heating power of the LASER is adjusted according to the thickness. The imposed heat flux densities are close to the values given in Table 1. Knowing pre-

cisely the imposed heat flux is not necessary to determine $t_{c_{2nde}}$. However choosing the LASER power is a compromise between a non negligible noise to signal ratio for a low heat flux density and a high temperature gradient in the sample for an important heat flux density.

An analytical analysis is performed in the discussion to help in the choice of the power to apply. The theoretical characteristic time $t_{c_{2nde}}$ are also given in Table 1 for each ARMCO iron sample. They are calculated for a thermal diffusivity of $2.04 \cdot 10^5 \text{ m}^2 \cdot \text{s}^{-1}$ corresponding to a temperature of 293K and for their respective thicknesses.

The figure Fig.7 shows the temperature evolution for three tests among twenty-five and for the four samples from A1 to A4. For each test, the raw data are plotted with the fitting curve obtained with the 7th order polynomial function. A good match is found between the data and the fitting function. Similar results are generally found on all the tests performed and for the other fitting functions. The identified characteristic times are also mentioned on the curves. These are obtained by derivating two times the 7th order polynomial functions.

Fig. 7 Fitting of three temperature profiles per ARMCO Iron sample - Zoom close to t_{c2nde}

The 2^{nde} derivative curves are showed for these tests on Fig.8. They are only plotted for the 7th order polynomial function and for the custom function to avoid the overload on the figures. As highlight on Fig.6, the identified t_{c2nde} changes a little bit with the choice of the fitting function. As well, some differences are

observed on the characteristic time according to the different tests performed but they looks staying in a narrow time range for each thickness.

Fig. 8 2nde derivative of three temperature profiles obtained on ARMCO Iron samples

The figure Fig.9 presents the different characteristic times from the 25 tests on the four samples. These are identified with the 7th order polynomial function

fitting. This demonstrate the reproducibility of the method even if the behaviour is slightly different according to the thickness of the sample.

Fig. 9 Distribution of the identified tc_{2nde} values with the 7th order polynomial function

The results given in Table 2 present the mean time tc_{2nde} and the standard deviation calculated for 25 tests performed. These are presented for the 4 fitting functions used. Generally the choice of the function has a minor influence on the results. The characteristic times obtained with the 6th order polynomial function don't give as good results as the other functions but the difference is slight. Poly-

nomial functions with a smaller order were also tested but were not sufficient to fit the temperature signal and then determined a consistent characteristic time.

The standard deviation on $t_{c_{2nde}}$ is higher on A3 and A4 in absolute value.

Sample	A1	A2	A3	A4
Theoric tc_{2nde} (ms) at T=293K	371	489	948	1800
6th polynomial				
Mean tc_{2nde} (ms)	451	539	981	1855
$\sigma_{tc_{2nde}}$ (ms) [%]	28.4 [6.3%]	31.9 [5.9%]	39.5 [4.0%]	83.3 [4.5%]
$\alpha_{mean} (*10^{-5} m^2.s^{-1})$	1.68	1.85	1.97	1.98
$\sigma_{\alpha} (*10^{-7} m^2.s^{-1})$ [%]	10.7 [6.4%]	10.9 [5.9%]	8.1 [4.1%]	8.9 [4.5%]
7th polynomial				
Mean tc_{2nde} (ms)	442	536	975	1841
$\sigma_{tc_{2nde}}$ (ms) [%]	21.6 [4.9%]	28.1 [5.2%]	37.2 [3.8%]	69.5 [3.8%]
$\alpha_{mean} (*10^{-5} m^2.s^{-1})$	1.71	1.86	1.98	1.99
$\sigma_{\alpha} (*10^{-7} m^2.s^{-1})$ [%]	8.4 [4.9%]	9.8 [5.3%]	7.5 [3.8%]	7.6 [3.8%]
8th polynomial				
Mean tc_{2nde} (ms)	440	539	977	1837
$\sigma_{tc_{2nde}}$ (ms) [%]	23.7 [5.4%]	29.4 [5.5%]	35.6 [3.6%]	66.9 [3.6%]
$\alpha_{mean} (*10^{-5} m^2.s^{-1})$	1.72	1.85	1.98	2.00
$\sigma_{\alpha} (*10^{-7} m^2.s^{-1})$ [%]	9.3 [5.4%]	10.2 [5.5%]	7.1 [3.6%]	7.4 [3.7%]
Custom function				
Mean tc_{2nde} (ms)	438	536	973	1851
$\sigma_{tc_{2nde}}$ (ms) [%]	26.7 [6.1%]	29.5 [5.5%]	37.9 [3.9%]	66.3 [3.6%]
$\alpha_{mean} (*10^{-5} m^2.s^{-1})$	1.73	1.86	1.99	1.98
$\sigma_{\alpha} (*10^{-7} m^2.s^{-1})$ [%]	10.5 [6.1%]	10.4 [5.6%]	7.8 [3.9%]	7.3 [3.7%]

Table 2 Identified values for Armco Iron samples - $\alpha = 2.05 * 10^{-5} m^2.s^{-1}$ at 293K

This can be explained by a lower temperature gradient versus time on the thicker samples and then a lower sensitivity. However the relative standard deviation is finally lower for the samples A3 and A4. This implies a best reproducibility on the calculation of α . For A3 and A4, the mean estimated values α_{mean} are closer to the theoretical value than the mean estimated values for the thinner samples. On α_{mean} , the relative difference is lower than 4% for these two samples whichever the fitting function. For these two thick samples, a standard deviation σ_α around 4% is also found on the estimated thermal diffusivity. The relative difference grows with the sample A2 on the mean thermal diffusivity identified but stays inferior to 10%. The error on α_{mean} is maximum (18%) for the sample A1 using the 6th order polynomial function. The standard deviation σ_α doesn't exceed 6.5% for A1 and A2 whichever the fitting function.

3.4 Measurements on low diffusivity materials

Thermal diffusivity identifications are performed on non-metallic materials which are under interest : 3 resins and alumina. The resins are materials currently developed for electrical and thermal insulation. These samples come from an industrial supplier and are around 4mm in thickness. The exact composition is unknown but the measured values are in the same range of the thermal diffusivity found on unreinforced epoxy resin [19]. The thermal diffusivity has also been calculated from the respective thermal conductivity, specific heat and density. The thermal conductivity is obtained from a ANTER UNITHERM 2021. The density is determined through the Archimede's principle on a weighing scale KERN AET500. The specific heat is calculated from a equilibrium with hot water in a Dewar calorime-

ter.

The alumina sample is a material used as grinding tool [20] [21] in our previous studies. The thermal diffusivity of sintered α -alumina is given at $1.11 \cdot 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ with an uncertainty of $2 \cdot 10^{-7} \text{ m}^2 \cdot \text{s}^{-1}$ in the literature [22].

The mean value of the identified thermal diffusivity and the standard deviation are presented on Table 3 for the four materials. Ten tests have been performed per material and the characteristic time $t_{c_{2nde}}$ is identified using a 7th order polynomial function.

Sample	Resin 1	Resin 2	Resin 3	Alumina
Thermal diffusivity ($\text{m}^2 \cdot \text{s}^{-1}$)	$1.30 \cdot 10^{-7}$	$1.87 \cdot 10^{-7}$	$4.55 \cdot 10^{-7}$	$1.02 \cdot 10^{-5}$
Standard deviation (%)	4.3	6.9	9.1	8.3

Table 3 Mean values identified on resins and alumina

For the 4 tested materials, the identifications are also quite reproducible. The standard deviation on the thermal diffusivity is higher than those obtained on ARMCO Iron samples. This is in the range of 4-9% depending on the tested materials. For the resins samples, the thermal diffusivities obtained show a difference between 10 and 15% compared to the values calculated with the thermal conductivity, the specific heat and the material density. This can be explained by the cumulative effect of the uncertainties on the three identified properties. For alumina, the mean value identified of $1.02 \cdot 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ is closed to the thermal diffusivity found in the literature. This shows that the proposed method is also suitable to identify low thermal diffusivities.

4 Discussion

Based on a new theoretical approach, our method allows identifying the thermal diffusivity of an isotropic material from a thermogramm. However the identification of a material property is always dependent on some experimental conditions and on the experimental setup. The BNM-LNE (France) [23] and the Austrian Research Centers [24] have used the GUM method [25] to characterize the uncertainties of their equipments to identify the thermal diffusivity by the flash method. This technique is used by laboratories developing metrology devices to estimate the accuracy of identified materials properties [26]. Here the method is not fully developed but some rules are proposed to choose the operating conditions which will reduce the uncertainty on the identified value with our equipment.

Regarding the propagation of uncertainty, the uncertainty on the thermal diffusivity is given by the equation (Eq.21) considering the equation (Eq.18). However the thickness of the sample and the characteristic time $t_{c_{2nde}}$ are not the only parameters to control. The heat flux imposed and the temperature gradient in the sample can have a non negligible influence on the estimation if the thermal diffusivity varies highly in a temperature range.

$$\frac{\Delta a}{a} = 2 \frac{\Delta e_s}{e_s} + \frac{\Delta t_{c_{2nde}}}{t_{c_{2nde}}} \quad (21)$$

4.1 Choice of the sample thickness

The sample thickness should be the first parameter to be chosen for a given material. Having a priori knowledge of the material thermal diffusivity, the sample thickness has to be fixed to obtain the characteristic time $t_{c_{2nde}}$ in the range of one second. To ensure the accurateness of the identification, the infrared signal

recording is triggered with the beginning of the laser heating. However, some delay exist because of the equipment. The recording frequency can not be higher than 500Hz because of the integration time and the size of the sample measured area. This induces a time delay which can reach 2ms between the laser heating start and the first temperature map of the sample. Finally, a time delay exists on the camera between the command of recording and the effectiveness of the operation. Nevertheless this time is lower than 1ms. Then a minimum time error of 3ms can be assumed.

There exist two source of error on the sample thickness. First, the measurement of this length is performed with a certified micrometer with an uncertainty of 20 μ m. Regarding the tested samples, this lead to a relative error inferior to 2% for samples with a thickness superior to 1mm. Secondly, the thickness is growing with the sample heating due to the thermal expansion.

In the case of the ARMCO iron, the thermal expansion is $12.10^{-6} \text{ K}^{-1}$ in the range 273-373°C. Considering that the maximum temperature increase of the sample will be around 50K, the sample thickness increase is overestimated considering an uncertainty of 0.06% which is negligible and independent of the thickness. The thermal expansion does not reach an increase of 1% of the volume for common materials in the same temperature variation. Then the suggested thickness according to the thermal diffusivity is given in the Table 4 for a characteristic time $tc_{2nde}=1s$.

Thermal diffusivity (m.s ⁻²)	10 ⁻⁴	10 ⁻⁵	10 ⁻⁶	10 ⁻⁷
Suggested Thickness (mm)	33	10.5	3.3	1.05
Corresponding materials	Or, Copper, Aluminium	Ferrous metals, Bronze, Lead	Glasses, Minerals	Wood, Resins, Thermoplastics

Table 4 Suggested thickness for a characteristic time tc_{2nde} of 1 second

4.2 Imposed heat flux

The imposed heat flux on the sample is one of the most critical parameter. Using a high power allows measuring a noticeable temperature variation on the rear surface and reducing the ratio noise/signal on the thermogram. However this induces a high thermal gradient between the heated surface and the rear surface. In case of a non negligible thermal diffusivity variation or material transformation in the temperature range, the identified value will be wrong or not usable. If the temperature variation is too small (low heat flux), the identification of the characteristic time is difficult due to the noise.

The equations (Eq.22), (Eq.23) and (Eq.24) are suitable to define the heat flux to impose for a given sample thickness and a priori knowledge of the material thermal conductivity.

$$\theta(e_s, tc_{2nde}) = \frac{2\varphi_0 * e_s}{\lambda} * \left(\sqrt{\frac{2tc_{2nde}}{\pi}} * \exp\left(\frac{-1}{tc_{2nde}}\right) - \operatorname{erfc}\left(\sqrt{\frac{1}{tc_{2nde}}}\right) \right) \quad (22)$$

$$\theta(e_s, 3tc_{2nde}) = \frac{2\varphi_0 * e_s}{\lambda} * \left(\sqrt{\frac{6tc_{2nde}}{\pi}} * \exp\left(\frac{-1}{3tc_{2nde}}\right) - \operatorname{erfc}\left(\sqrt{\frac{1}{3tc_{2nde}}}\right) \right) \quad (23)$$

$$\theta(0, 3tc_{2nde}) = \frac{\varphi_0 * e_s}{\lambda} * \frac{\sqrt{6tc_{2nde}}}{\pi} \quad (24)$$

The equation (Eq.22) and (Eq.23) gives the temperature value at the rear surface for the characteristic time $t_{c_{2nde}}$ and for $3t_{c_{2nde}}$. $3t_{c_{2nde}}$ is chosen because it is used as the upper limit of the time interval for the fitting process. These are directly obtained from (Eq.14). The equation (Eq.24) gives an approximation of the temperature reached on the heated surface for $t=3*t_{c_{2nde}}$. This equation gives the temperature variation for a semi-infinite wall with a constant heat flux [27]. Comparisons have been performed with a 1D finite difference model on several materials (ARMCO, alumina, phenolic resin). The temperature difference between the analytical model (Eq.24) and the finite difference model for a sample with a thickness e_s is negligible after $3t_{c_{2nde}}$.

Then the equation (Eq.25) gives an approximation regarding the difference in the order of magnitude for $\theta_0(3t_{c_{2nde}})$, $\theta_{e_s}(3t_{c_{2nde}})$ and $\theta_{e_s}(t_{c_{2nde}})$. This is suitable to choose the imposed heat flux. It allows choosing a value leading to a sufficient temperature variation at the thickness position and getting a reasonable temperature variation on the heated surface.

$$\theta_0(3t_{c_{2nde}}) \approx 5 * \theta_{e_s}(3t_{c_{2nde}}) \approx 100 * \theta_{e_s}(t_{c_{2nde}}) \quad (25)$$

In our tests, the imposed heat flux is calibrated to reach around 0.4°C on the rear surface at $t_{c_{2nde}}$ in the presented tests. This leads theoretically to a temperature elevation of 40°C at $3t_{c_{2nde}}$ on the heated surface. Depending to the material, the imposed heat flux could be higher but some problems may occur. Firstly the temperature elevation may cause a structural modification in the material. Secondly the assumption of constant thermal parameter becomes false because of the dependence with the temperature. However other experiments have been per-

formed with lower imposed heat flux on ARMCO Iron ($\theta^* \approx 0.18^\circ C$ at $t_{c_{2nde}}$) and the error on the identified thermal diffusivity was higher compared to the theoretical value. Using a low heating leads to increase the noise to signal ratio and then the standard deviation on the results grows.

5 Conclusion

An alternative method is proposed to the identification of the thermal diffusivity of isotropic material. An analytical solution is developed to get the temperature evolution of a sample surface, heating the opposite face at constant power. This solution is only valid in the first instant after the beginning of the sample heating but the analysis of its 2^{nde} derivative in the valid time domain leads to a new characteristic time $t_{c_{2nde}}$. Like the characteristic time given by Parker for the flash method, $t_{c_{2nde}}$ is proportional to the thermal diffusivity and the sample thickness.

Compared to the flash method, there are two main advantages regarding the experimental setting up. First, the flash method is based on a heating pulse which is considered infinitely short in the analytical solution. In experimental condition, most of laser equipments does not allow reaching heating time shorter than 1ms. In our method, the analytical solution is based on constant heating and there is no uncertainty regarding the pulse duration. Secondly, there is no need to know exactly the heat flux imposed on the surface. However this value has to be chosen in order to avoid a high thermal gradient and to be sufficient to limit the errors due to the noise on the thermogram. Concerning the heat losses and thermal noise, our experimental setup is embedded in an hermetic chamber to reduce convective

phenomena and surrounding radiations. Developing a room with inert atmosphere and controlled temperature is under investigation to improve the accuracy of the identification and to allow measurement at different temperatures.

Some good practice are given regarding the choice of the sample thickness and the heat flux density to impose. These are also based on analytical solutions but require to have an a priori knowledge on the material's thermal properties. The sample thickness is first chosen regarding the a priori diffusivity and a targeted characteristic time t_{c2nde} which should be around 1s. This limits the uncertainties due to the synchronization's error of heating and measurement. Then the heat flux imposed has to be chosen large enough so as to obtain a suitable ratio noise/signal. However the experimenter must pay attention that the thermal gradient in the sample has to be restricted in a temperature range where the thermal properties can be considered constant. This temperature range will change regarding the material and the transformations occurring at certain temperatures (phase change, chemical reaction,...) .

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Nomenclature

c_p	Specific heat	$\text{J.g}^{-1}.\text{K}^{-1}$
e_s	Sample thickness	mm
E	Heaviside function	
F	Laplace transform function	
G	Green function	
h	Convective coefficient	$\text{mW.m}^{-2}.\text{K}^{-1}$
p	Laplace Transform parameter	
q	Laplace Transform parameter	
T	Temperature	K
t	Time	s
$t_{1/2}$	Parker characteristic time	s
t_c	1rst derivative characteristic time	s
$t_{c_{2nde}}$	2nde derivative characteristic time	s
x	Thickness location	mm

Greek letters

α	Thermal diffusivity	$\text{m}^2.\text{s}^{-1}$
β	Adimensionned time	
ϵ	Emissivity	
λ	Thermal conductivity	$\text{mW.m}^{-1}.\text{K}^{-1}$
φ	Heat flux density	mW.m^{-2}
σ_α	Standard deviation of α	$\text{m}^2.\text{s}^{-1}$
ρ	Material density	kg.m^{-3}
θ	Temperature elevation	K
ξ	Green function parameter	

Subscripts

lim	Maximum reached temperature (flash method)
mean	Mean experimental value
ref	Reference value
∞	Room temperature

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