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Elissa El Rassy, Yann Billaud, Didier Saury. A direct method for the simultaneous characterization of thermal diffusivities of a bi-layer material consisting of a thin coating deposited on a substrate. *Applied Mathematical Modelling*, 2021, 91, pp.614-631. 10.1016/j.apm.2020.09.049 . hal-03029812

HAL Id: hal-03029812

<https://hal.science/hal-03029812>

Submitted on 26 Feb 2021

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A direct method for the simultaneous characterization of thermal diffusivities of a bi-layer material consisting of a thin coating deposited on a substrate

Elissa EL RASSY^{a,b}, Yann BILLAUD^a, Didier SAURY^a

^aInstitut Pprime UPR CNRS 3346 - CNRS \diamond ENSMA \diamond Université de Poitiers, 1 avenue Clément Ader, B.P. 40109, F-86961, Futuroscope Chasseneuil CEDEX, FRANCE;

^bLTEN – UMR CNRS 6607, Université de Nantes, CNRS, Rue Christian Pauc, BP 50609, 44306 Nantes cedex 3, France

Abstract

This work presents a method dedicated to the simultaneous identification of the thermal diffusivities of coatings or thin film materials. To ensure non-destructive thermal characterization of the coating, the present method also implies the identification of the substrate thermal properties, which may be orthotropic. The estimation of thermal diffusivities is based on the resolution of an inverse problem by minimizing the quadratic difference between the response of a 3D semi-analytical model and the measurements resulting from a single '3D flash method' type experiment, using a stochastic based optimization algorithm. A unique non-intrusive test, that consists in recording the temperature change cartography on one face of the sample by means of an infrared camera, is required. The evolution of the temperatures within the material is generated by a non-uniform and almost instantaneous excitation imposed by a CO₂ laser on the measured face. The difficulties related

to the control of the excitation, in terms of the distribution of the imposed flux or the energy absorbed by the material, are overcome by estimating the parameters associated to the excitation simultaneously with the thermal diffusivities. The developed method is applied to estimate the thermal diffusivity of a coating used in thermographic phosphor thermometry to measure wall surface temperatures and heat fluxes in combustion environments. The method is first evaluated on simulated data as a function of the measuring/excitation face in order to ensure the feasibility, the robustness and accuracy of the current method and to establish the best experimental configuration. Experimental results are then exploited and the estimation results are compared with other methods results.

Keywords: Bi-layer material, parameter estimation, flash method, inverse problem, thermal quadrupoles

1. Introduction

Coatings are used in many domains, they can serve as thermal (e.g. insulation) or fire protection), chemical (i.e. erosion, corrosion or oxidation prevention) or mechanical protection (e.g. ablation protection), or to improve optical properties (e.g. black painting to tend toward the emissivity of a black body). It can be also used in the frame of temperature measurement techniques, among those the most common techniques are namely, the "IR thermography" and the "phosphorescence thermometry". The latter is based on the phosphorescent properties of phosphoric mixture used to determine the surface temperature from the measurement of the radiation intensity emitted by this surface.

12 The present work aims to thermally characterize the thin layer, referred
13 hereinafter by "TPT" coating for "Thermographic Phosphor Thermometry",
14 deposited on surfaces of interest in internal combustion engines as the walls
15 of the combustion chamber. The thermal characterization (in particular the
16 thermal diffusivity) of such phosphorous layer is crucial in order to accurately
17 predict the intensity of heat transferred through this coat layer as well as the
18 temperature evolution profile at the surface of the covered material during
19 operating cycles.

20 However, as it is not desirable to separate the coating from its substrate,
21 the investigated method is based on a direct and simultaneous thermal char-
22 acterization of samples constituting layers.

23 In this context, and on the contrary to the thermal characterization of
24 isotropic or orthotropic monolayer materials [1–13] that had paid attention
25 to a large community, very few studies addressed the issues of coatings on
26 substrate materials. Some of these studies have been interested in the char-
27 acterization of only the thermal conductivity or diffusivity of (i) thin films
28 [14–16] or (ii) coatings on substrates materials [17–25], or (iii) 1D tempera-
29 ture dependent thermal diffusivity (i.e. $a(T)$) of a viscous intumescent coat-
30 ing with a moving boundary system [26]. In those works, a priori knowledge
31 of the substrate properties or a determination of these properties through
32 a previous experiment, is often required [14, 17, 18, 23, 24, 26–30]. Conse-
33 quently, any error in these properties will be propagated through the model
34 and results in an inaccuracy of the required estimation.

35 To overcome the difficulty to obtain separately each constituting compo-
36 nents, some authors try to identify the thermal diffusivities of the coating

37 without any knowledge about the substrate properties. This method relies on
38 a two-steps identification technique that allows such measurement but only
39 at very short time [31], limiting this latter one to relatively thick coatings.
40 Any estimation strategy applied for multilayers, or for all layers constituting
41 that multilayers material, and involving more than one step, may propagate
42 error throughout the various stages of estimation. For example, one can cite
43 the method of gobbé et al. [32] in which the in-plane and in-depth thermal
44 conductivities of a multilayers sample are estimated successively by the hot
45 wire and the hot strip methods, or a strategy developed by Li et al. [33]
46 in order to characterize both layers (i.e. the coating and the substrate),
47 by repeating the experiment/measurements several times. Very few studies
48 have been focused on simultaneous estimation of each isotropic layer thermal
49 diffusivities, in a multilayer or a coating on substrate bi-layered structure.
50 One can cite some works that have attempted to simultaneously estimate,
51 in cylindrical coordinates, the two-dimensional diffusivities of the anisotropic
52 coatings deposited on an isotropic substrate, but with very limited results
53 [27, 28].

54 The present study concerns the simultaneous estimation, i.e. using a
55 unique step, via a non-intrusive flash experiment, of the thermal diffusivity
56 of the coating and its substrate. The current case of a coating or a thin
57 layer deposited on a substrate is a particular case of bilayer materials in
58 which the thickness of the deposit is very small compared to the substrate.
59 The identification proposed in the framework of this study is based on a
60 direct estimation method that minimizes the quadratic difference between
61 the measurements resulting from a single 'flash' type experiment inspired

62 from [34] carried out by an IR camera, and the outputs of a direct pseudo-
63 analytical model based on the "3D thermal quadrupoles" method [35].

64 Considering the relative complexity and non-linearity of the direct model,
65 and in order to converge towards the global optimum (minimum) instead of
66 a local one, the desired parameters are identified by applying a hybrid op-
67 timization algorithm. The latter combines a global minimisation performed
68 by a particle-type swarm optimization algorithm (PSO) [36] followed by a
69 local minimisation carried out by a gradient based method.

70 First, the direct model and the estimation method are presented. The
71 estimation is then carried out on a bilayer material composed of a thin phos-
72 phorus layer deposited on a low diffusivity substrate. The feasibility and the
73 robustness of the overall method is demonstrated based on simulated noisy
74 data. The results obtained by considering two experimental configurations
75 and different assumptions concerning the thermal nature of the substrate
76 (isotropic or orthotropic), are then presented and discussed.

77 **2. Problem description**

78 *2.1. General principle*

79 The main aim of this part concerns the thermal characterization of a spe-
80 cific two-layers material constituted of a thin layer or coating deposited on an
81 isotropic or orthotropic material. The coating considered in this study is the
82 a phosphorescent material generally applied in the combustion chambers for
83 temperature measurements. In order to reproduce, as well as possible, the
84 experimental deposition of the TPT coating, the coating should be deposited
85 on a metallic sample, such as copper or aluminum. However, these materials

86 are highly diffusive and due to some experimental limitations related to the
87 achievable acquisition frequency of the handled IR camera, these substrates
88 necessitating a very high acquisition frequency (typically $> 1000 \text{ Hz}$) are
89 replaced by a substrate having a low thermal diffusion (e.g. HDPE = High
90 Density Polyethylene or polyamide) that requires a moderate acquisition fre-
91 quency ($\simeq 50 \text{ Hz}$). The identification of thermophysical properties of such
92 complex materials is generally based on the resolution of an inverse heat
93 conduction problem (IHCP).

94 *2.2. Inverse Heat Conduction Problem (IHCP)*

95 This section presents the general principle of inverse problems and each
96 element involved in the overall procedure. It contains general briefing of each
97 element of the general procedure and its current application in the present
98 method.

99 [Figure 1 about here.]

100 The IHCP general principle consists in comparing the "observables" Y^*
101 obtained from experimental or synthetic measurements with those resulting
102 from an analytical or numerical model ($Y(\beta)$) that must, as much as possible,
103 mimic the experiment. Thereby, the model has to provide outputs $Y(\beta)$ that
104 must i) be compatible with the measurements Y^* and ii) be dependent on the
105 parameters to estimate β . This last point is discussed further in the section
106 dedicated to the sensitivity analysis. This comparison is performed by means
107 of a cost function f , also called "objective function". As long as this function
108 f does not satisfy a certain criterion, the optimization algorithm adjusts the
109 input parameters β of the direct model. The process is repeated until the

110 procedure converges to the optimal set of parameters $\hat{\beta}$ with the admissible
111 values, i.e the one that minimizes the difference between the experiment and
112 the model.

113 For an experimental application, the major sections investigated in the
114 identification technique will be mainly:

- 115 • The experiment
- 116 • The direct model
- 117 • The comparison of observable quantities via a cost function
- 118 • The minimization via an identification algorithm

119 The overall estimation strategy concept is illustrated in Fig. 1 that presents
120 the connection between the elements involved in the inverse problem resolu-
121 tion. The various steps of the estimation strategy are detailed and discussed
122 in the following sections. The estimation method developed in this work is
123 referred as DSEH (Direct and Simultaneous Estimation using Harmonics).

124 *2.2.1. Experimental 3D flash method*

125 The experiment protocol corresponds to an unconventional and 3D flash
126 technique qualified as a non-intrusive method, both in terms of excitation
127 and measurements. In this method, the sample that should be thermally
128 characterized is subjected to a localized and non-uniform thermal excitation
129 using a CO₂ laser. The resulting surface temperature evolution cartography
130 is continuously measured, during the cooling phase. These measurements
131 are performed by an IR camera, at the front or rear faces of the material.

132 Figs. 2 represent the experimental setup and the equipment used to generate
133 the experimental data.

134 [Figure 2 about here.]

135 The specifications of the main devices are detailed hereafter:

- 136 • The infrared camera, that is a FLIR SC7000 having n adjustable fre-
137 quency (up to 200 Hz), and a resolution of 320×256 pixels (full frame).
- 138 • The laser is a CO₂ laser (DIAMOND GEM-Series by Coherent©),
139 having an adjustable power and duration time.

140 2.2.2. *Direct bi-layered model*

141 The identification method proposed in this study is based on a three-
142 dimensional analytical heat transfer model that mimics the flash technique.
143 This model describes the transient heat conduction within a homogeneous
144 and opaque bilayer material, being exposed to a non-uniform and almost
145 instantaneous thermal excitation.

146 As shown in Fig. 3, the two layers have the same dimensions in the XY
147 plane ($l_{x_1} = l_{x_2} = l_x$ and $l_{y_1} = l_{y_2} = l_y$). The lateral faces are assumed to be
148 thermally isolated, while the front and rear faces are subjected to convective
149 and radiation losses described by a linearized global heat exchange coefficient
150 that is assumed to be equal to a constant and uniform term on both sides,
151 $h_f = h_b = h$, with $h = 10 \text{ W} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$, a commonly accepted value for this
152 kind of experimental exercise.

153 In addition, at time $t = 0$, the system is supposed to be in thermal
154 equilibrium at T_∞ . It is important to underline that all the temperatures

155 considered in this model are relative to the temperature of the environment
156 (i.e. $T(x, t = 0) = 0$). The thermal resistance at the interface between the
157 two layers is neglected. In addition, the thermal excitation must be chosen
158 in such a way to generate a sufficient temperature elevation to be accurately
159 measured, but at the same time moderate enough to be able to consider
160 the thermophysical properties as constant and independent on temperature
161 variation. The system of partial differential equations that describes the heat
162 transfers within the bi-layered system, the conditions at the interfaces, and
163 the boundary and initial conditions are presented herebelow.

164 [Figure 3 about here.]

165 The set of differential partial equations modeling the associated heat
166 transfers inside the medium (layer 1 and 2) as well as the condition at in-
167 terface, and the initial and boundaries conditions (IC & BC) are presented
168 hereafter. In these equations, T refers to the temperature relative to the
169 ambient temperature (T_∞):

$$\text{layer 1 : } \frac{\partial T_1}{\partial t} = a_{x1} \frac{d^2 T_1}{dx^2} + a_{y1} \frac{d^2 T_1}{dy^2} + a_{z1} \frac{d^2 T_1}{dz^2} \quad \text{for } z \in [0, l_{z1}], t > 0 \quad (1)$$

$$\text{layer 2 : } \frac{\partial T_2}{\partial t} = a_{x2} \frac{d^2 T_2}{dx^2} + a_{y2} \frac{d^2 T_2}{dy^2} + a_{z2} \frac{d^2 T_2}{dz^2} \quad \text{for } z \in [l_{z1}, l_{z1} + l_{z2}], t > 0$$

$$\begin{aligned} \text{Interface: } \lambda_{z1} \frac{\partial T_1(z = l_{z1-})}{\partial z} &= \lambda_{z2} \frac{\partial T_2(z = l_{z1+})}{\partial z} \quad \text{for } z = l_{z1}, t > 0 \quad (2) \\ T_2(z = l_{z1+}) &= T_1(z = l_{z1-}) \quad \text{for } z = l_{z1}, t > 0 \end{aligned}$$

$$\text{BC1:} \quad -\lambda_{x_1} \frac{\partial T_1}{\partial x} = 0 \quad \text{for } x = 0 \text{ and } x = l_x, t > 0 \quad (3)$$

$$-\lambda_{y_1} \frac{\partial T_1}{\partial y} = 0 \quad \text{for } y = 0 \text{ and } y = l_y, t > 0$$

$$-\lambda_{z_1} \frac{\partial T_1}{\partial z} = -h_f \cdot T_1 + \phi_{x,y}^{ex}(t) \quad \text{for } z = 0, t > 0$$

$$\text{BC2:} \quad -\lambda_{x_2} \frac{\partial T_2}{\partial x} = 0 \quad \text{for } x = 0 \text{ and } x = l_x, t > 0 \quad (4)$$

$$-\lambda_{y_2} \frac{\partial T_2}{\partial y} = 0 \quad \text{for } y = 0 \text{ and } y = l_y, t > 0$$

$$-\lambda_{z_2} \frac{\partial T_2}{\partial z} = -h_b \cdot T_2 \quad \text{for } z = l_{z_1} + l_{z_2}, t > 0$$

$$\text{IC: } T_1(x, y, z) = 0, T_2(x, y, z) = 0 \quad \text{for } t = 0 \quad (5)$$

170 In the considered case, the thermal resistance at the interface between
 171 both layers is neglected. After getting the harmonics $\xi_{m,n}(z, p)$ by projection
 172 of the physical relative temperature $T(x, y, z, t)$ into space Fourier cosine and
 173 Laplace domain, the three dimensional analytical solution can be obtained
 174 using the thermal quadrupoles formalism as follows:

$$\begin{pmatrix} \xi_{m,n}(z = 0, p) \\ \phi_{m,n}^{ex}(p) - h_f \xi_{m,n}(z = 0, p) \end{pmatrix} = \begin{pmatrix} A_{m,n}(p) & B_{m,n}(p) \\ C_{m,n}(p) & D_{m,n}(p) \end{pmatrix} \times \begin{pmatrix} \xi_{m,n}(z = l_{z_1} + l_{z_2}, p) \\ h_b \cdot \xi_{m,n}(z = l_{z_1} + l_{z_2}, p) \end{pmatrix} \quad (6)$$

175 with:

$$\xi_{m,n}(z, p) = \frac{1}{l_x \cdot l_y} \int_{t=0}^{\infty} \int_{y=0}^{l_y} \int_{x=0}^{l_x} T(x, y, z, t) \cdot \cos\left(\frac{m\pi x}{L_x}\right) \cdot \cos\left(\frac{n\pi y}{L_y}\right) \cdot e^{-pt} dx dy dt \quad (7)$$

$$\phi_{m,n}^{ex}(p) = \frac{1}{l_x \cdot l_y} \int_{t=0}^{\infty} \int_{y=0}^{l_y} \int_{x=0}^{l_x} \phi_{x,y}^{ex}(t) \cdot \cos\left(\frac{m\pi x}{L_x}\right) \cdot \cos\left(\frac{n\pi y}{L_y}\right) \cdot e^{-pt} dx dy dt \quad (8)$$

176 and

$$\begin{pmatrix} A_{m,n}(p) & B_{m,n}(p) \\ C_{m,n}(p) & D_{m,n}(p) \end{pmatrix} = \begin{pmatrix} a_{m,n,1} & b_{m,n,1} \\ c_{m,n,1} & d_{m,n,1} \end{pmatrix} \times \begin{pmatrix} a_{m,n,2} & b_{m,n,2} \\ c_{m,n,2} & d_{m,n,2} \end{pmatrix} \quad (9)$$

177 where:

$$a_{m,n,i \in \{1,2\}} = \cosh(l_{z_i} \cdot K_{m,n,i}(p)) \quad (10)$$

$$b_{m,n,i \in \{1,2\}} = \frac{\sinh(l_{z_i} \cdot K_{m,n,i}(p))}{\lambda_{z_i} \cdot K_{m,n,i}(p)} \quad (11)$$

$$c_{m,n,i \in \{1,2\}} = \lambda_{z_i} \cdot K_{m,n,i}(p) \cdot \sinh(l_{z_i} \cdot K_{m,n,i}(p)) \quad (12)$$

$$d_{m,n,i \in \{1,2\}} = a_{m,n,i}(p) \quad (13)$$

178

$$K_{m,n,i}(p) = \sqrt{\frac{p}{a_{z_i}} + \alpha_m^2 \frac{a_{x_i}}{a_{z_i}} + \beta_n^2 \frac{a_{y_i}}{a_{z_i}}}; \quad \alpha_m = \frac{m\pi}{l_x}; \quad \beta_n = \frac{n\pi}{l_y}$$

179 The resulting analytical expressions of the rear ($l_z = l_{z_1} + l_{z_2}$) and front
180 ($l_z = 0$) face normalized harmonics are respectively:

$$\xi_{m,n}(z = l_{z_1} + l_{z_2}, p) = \frac{\phi_{m,n}^{ex}(p)}{C_{m,n}(p) + D_{m,n}(p) \cdot h_b + A_{m,n}(p) \cdot h_f + B_{m,n}(p) \cdot h_f \cdot h_b} \quad (14)$$

181

$$\begin{aligned} \xi_{m,n}(z = 0, p) &= \xi_{m,n}(z = l_{z_1} + l_{z_2}, p) \cdot [A_{m,n}(p) + B_{m,n}(p) \cdot h_b] \quad (15) \\ &= \frac{\phi_{m,n}^{ex}(p) \cdot (A_{m,n}(p) + B_{m,n}(p) \cdot h_b)}{C_{m,n}(p) + D_{m,n}(p) \cdot h_b + A_{m,n}(p) \cdot h_f + B_{m,n}(p) \cdot h_f \cdot h_b} \end{aligned}$$

182 These normalized harmonics are then projected into the real time domain
183 using De-Hoog inversion technique, and $\xi_{m,n}^{mod}(\beta, z = 0$ or $z = l_{z_1} + l_{z_2}, t)$ will

184 represent the model outputs involved in the inverse problem technique and
 185 estimation strategy.

186 Note that, the process under which the coating layer is deposited on the
 187 surface of the substrate encourage the consideration of a negligible contact
 188 resistance Rc at the interface between both layers. In Addition, assuming
 189 the excitation source can be modeled as $\phi_{x,y}^{ex}(t) = \phi_0 \cdot F_{x,y}(x, y) \cdot u(t)$ leads
 190 to $\phi_{m,n}^{ex}(p) = \frac{\phi_0 \cdot F_{m,n} \cdot u(p)}{l_x \cdot l_y} = R_{m,n} \cdot u(p)$, $R_{m,n}$ being the normalized Fourier
 191 cosine projection of the excitation shape and $u(p)$ the Laplace transform of
 192 the excitation time evolution.

193 2.2.3. *Cost function*

194 The cost function, also known as objective function, is the quadratic
 195 deviation between the measured signal and the signal predicted by the direct
 196 physical model. Thus, the estimator used for the minimization of the cost
 197 function for the front face, is written as follows:

$$\hat{\beta} = \arg \min[f(\beta)] = \min_{\beta} \sqrt{\sum_{m=0}^M \sum_{n=0}^N [\xi_{m,n}^{mod}(\beta, z = 0, t) - \xi_{m,n}^{exp}(t)]^2} \quad (16)$$

198 In this method, the considered harmonics are equally weighted. The
 199 first term of Eq.16, i.e. $\xi_{m,n}^{mod}(\beta, t)$, corresponds to the temporal normalized
 200 harmonics at the front face, achieved by the Laplace inversion (De Hoog
 201 Algorithm) which is applied to the direct model outputs (Eq.15). The sec-
 202 ond term, $\xi_{m,n}^{exp}(t)$, represents the observables issued from front face relative
 203 temperature measurement, projected in Fourier Cosine space:

$$\xi_{m,n}^{exp}(t) = \frac{1}{l_x \cdot l_y} \int_{y=0}^{l_y} \int_{x=0}^{l_x} T^{exp}(x, y, z, t) \cdot \cos\left(\frac{m\pi x}{L_x}\right) \cdot \cos\left(\frac{n\pi y}{L_y}\right) dx dy \quad (17)$$

204 Given that the harmonics of low spatial frequencies hold the largest quan-
 205 tity of information related to diffusivities, the exploited harmonics in this
 206 study will be the first even modes: $(m, n) = \{0, 2, 4, 6\} \times \{0, 2, 4, 6\}$. The
 207 odd harmonics are thus not considered in the estimation. In fact, here they
 208 can be considered as negligible (when compared to even ones), since the exci-
 209 tation spot is centered and exhibits revolution symmetries. Centering can be
 210 improved by IR images post treatments, whereas the revolution symmetry
 211 is strongly connected to the excitation source. The framing of the images
 212 should be performed in a way that guarantees these last assumptions.

213 The TPT coating is considered isotropic, i.e. $a_{x_{\text{coat}}} = a_{y_{\text{coat}}} = a_{z_{\text{coat}}} =$
 214 a_{TPT} , thus the parameters vector β that should be estimated is $\beta = [a_{x,1}, a_{y,1},$
 215 $a_{z,1}, a_{TPT}, R_{0,0}, R_{0,2}, R_{2,2} \dots R_{m,n} \dots R_{6,6}]$ for the case where the substrate is
 216 considered as orthotropic. In that case, 20 parameters must be estimated.
 217 However, for the case where the substrate is also considered as isotropic, the
 218 vector parameter becomes $\beta = [a_{HDPE}, a_{TPT}, R_{0,0}, R_{0,2}, R_{2,2} \dots R_{m,n} \dots R_{6,6}]$
 219 and then 18 parameters have to be identified simultaneously.

220 *2.2.4. Optimization algorithm*

221 Hybrid optimization algorithm is applied in the current study to mini-
 222 mize the cost function (Eq.16) and find the optimal parameter vector $\hat{\beta}$. The
 223 relatively complex estimation problem and the non-linear nature of the phe-
 224 nomenon require the use of a stochastic approach in order to seek the global
 225 minimum and avoid getting stuck into a local minimum. Thanks to a cou-
 226 pled stochastic-deterministic optimization algorithm, a global search of the
 227 minimum region is achieved by using a Particle Swarm Optimization (PSO)
 228 algorithm [36–38], followed by a local search of the cost function minimum

229 value, which carried out by the gradient based method (interior point method
230 [39]). All algorithm implementations details are well described in [2].

231 **3. Numerical Applications**

232 *3.1. Sensitivity analysis*

233 A study of reduced sensitivities is conducted here in order to determine
234 the most appropriate configurations for the estimation of the coating proper-
235 ties, with or without the simultaneous estimation of the substrate properties.

236 Reduced sensitivities are defined as:

$$Sr_{m,n}(\beta_j, t) = \frac{\partial Y(\beta, t)}{\partial \beta_j} \times \beta_j \Big|_{\beta_k \neq j} \quad (18)$$

237

238 The reduced sensitivities (Eq.18) have then similar units (and so similar
239 scale values). Thus, they can properly ensure a comparison of the different
240 parameters impacts on the model outputs [40, 41].

241 [Figure 4 about here.]

242 The classification of the 4 different cases that are investigated to find the
243 most suitable experimental configuration, is shown in Fig. 4a. The sensitivity
244 of the harmonic $\xi_{2,2}(t)$ to the coating in-depth thermal diffusivity, and for
245 the four possible configurations, is presented in Fig. 4b. It is important to
246 note that the sensitivity analysis that allows to compare different possible
247 experimental configurations, and the numerical applications represented in 3
248 require the values of the parameters β or at least approximative values, in
249 order to obtain synthetic data via the direct model (Eq.15). These values are

250 inspired from the literature [42] and from a study [43] that considered the
251 case of a high-speed thermographic phosphor thermometry used to control
252 the temperature increase and the time and position of flame impingement at
253 the piston surface. These values are gathered in table 1.

254 [Table 1 about here.]

255 An exploitation window with $l_x = l_y = 50 \text{ mm}$ has been proved, through
256 a preliminary study [44], to be suitable for such application, since it can
257 give a good accuracy/time ratio. The parameters involved in the thermal
258 excitation definition correspond to the total amount of energy deposited on
259 the material, $Q = 0.56 \text{ J}$, and to the laser radius $r = \frac{l_x}{9.55} (\approx 5 \text{ mm})$ (coherent
260 with previous experimental observations).

261 While remaining consistent in terms of radius value that can be experi-
262 mentally generated, this set of parameters can guarantee a temperature evo-
263 lution at the surface of the material: i) sufficient to be accurately measured
264 and ii) tolerable to avoid any risk of thermophysical properties modification
265 (i.e. temperature dependence of parameters) that can occur at high temper-
266 ature (typically $> 10 \text{ }^\circ\text{C}$).

267 In these numerical applications, the spatial distribution of the thermal
268 excitation has been parameterized by a cubic polynomial shape, which is
269 consistent with experimental observations and already tested in previous
270 monolayers applications [2]. This form allows us to calculate the form factors
271 $F_{m,n}$ in the Fourier cosine space.

272 The normalized shape of the excitation is described, along the x-axis,

$$F_x(x) = \frac{1}{r} \times \begin{cases} 1 - 3 \cdot \left(\frac{x}{r}\right)^2 - 2\left(\frac{x}{r}\right)^3 & \text{for } -r \leq x < 0 \\ 1 - 3 \cdot \left(\frac{x}{r}\right)^2 + 2\left(\frac{x}{r}\right)^3 & \text{for } 0 \leq x < r \\ 0 & \text{otherwise} \end{cases} \quad (19)$$

273 The cosine Fourier transform in the x-direction of the previous function

274 $F_x(x)$ is

$$\begin{aligned} F_m &= \int_0^{l_x} F_x(x - \frac{l_x}{2}) \times \cos\left(\frac{m \cdot \pi \cdot x}{l_x}\right) dx \\ F_m &= \frac{-24 \left(\cos\left(\frac{m \cdot \pi}{2}\right) \cdot \left(\cos(\alpha_m \cdot r) - 1 + \frac{\alpha_m \cdot r}{2} \cdot \sin(\alpha_m \cdot r) \right) \right)}{r^4 \cdot \alpha_m^4} \end{aligned} \quad (20)$$

The same function is used in the y-direction with $F_y(y)$. Consequently, the shape function associated with the laser beam, $F_{x,y}(x, y) = F_x(x) \cdot F_y(y)$, in the cosine-Fourier domain, leads to the following $F_{m,n}$ shape factor:

$$F_{m,n} = \frac{24^2}{r^8 \cdot \alpha_m^4 \cdot \beta_n^4} \cdot \left[\left(\cos\left(\frac{m \cdot \pi}{2}\right) \cdot \left(\cos(\alpha_m \cdot r) - 1 + \frac{\alpha_m \cdot r}{2} \cdot \sin(\alpha_m \cdot r) \right) \right) \cdot \left(\cos\left(\frac{n \cdot \pi}{2}\right) \cdot \left(\cos(\beta_n \cdot r) - 1 + \frac{\beta_n \cdot r}{2} \cdot \sin(\beta_n \cdot r) \right) \right) \right] \quad (21)$$

275 In that case, the normalized shape factor in the Fourier-cosine space $R_{m,n}$

276 is such as $R_{m,n} = \frac{\phi_0 \cdot F_{m,n}}{l_x \cdot l_y}$.

277 3.2. Numerical and Experimental configurations

278 Two possible experimental configurations are considered for numerical
279 validation in order to test the feasibility and the accuracy of the identification
280 method for both cases. The FF-FF (see Fig. 4a) configuration is the most

281 sensitive, as shown in Fig. 4b. The other configuration BF-BF (see Fig.
282 4a) corresponds to the simplest one, in terms of experimental conditions and
283 limitations. Their respective experimental protocols are illustrated in Fig. 5.
284 The FF-FF configuration (case (a) in Fig. 5) consists in depositing the source
285 heat flux $\phi_{x,y}^{ex}(t)$ on the surface of the coating and measuring the resulting
286 temperature evolution profile at the same side. On the contrary, the BF-BF
287 configuration (case (b) in Fig. 5) considers the case where the excitation
288 and the temperature profiles measurements are carried out at the substrate
289 surface side.

290 [Figure 5 about here.]

291 The excitation of the thin coating surface side (occurring in the config-
292 urations FF-FF and FF-BF) is not advisable, due to the degradation and
293 poor control of heat penetration depth reasons. The same observations can
294 be made for the temperature measurements at this surface (configurations
295 FF-FF and BF-FF) due to the ill-knowledge of the TPT surface emissivity.

296 3.3. *Parameters initialization*

297 In addition to the general parameters described in [36], the PSO algorithm
298 specifications and conditions as well as the stopping criteria, are tabulated
299 in the Table 2. The stopping criterion is a combination of several conditions.
300 The iterative minimization will be stopped (i) if the maximum number of
301 iterations is achieved, or (ii) if the number of stall iterations without any
302 significant change and with a best value of the cost function less than the
303 tolerance value, exceeds the maximum stall iterations. Furthermore, fol-
304 lowing the PSO principle, the swarms particles move in the domain field,

305 predefined by a upper and a lower bounds, seeking for the optimal solution
306 i.e. for the lowest value of the cost function (Eq.16). One can find also the
307 bounds of the parameters values that should be estimated in Table 2.

308 [Table 2 about here.]

309 *3.4. Numerical results and discussion*

310 The numerical exercise currently conducted in order to validate the iden-
311 tification feasibility and evaluate the robustness and the accuracy of the
312 estimation method, relies on "synthetic measurements" for both configura-
313 tions FF-FF (case (a)) and BF-BF (case (b)). These data are generated from
314 the direct model for which a random noise with a Gaussian distribution is
315 added, in order to mimic experimental conditions.

316 The overall procedure is shown in Fig. 6. This technique is used to
317 evaluate the feasibility of an estimation method, compare different methods
318 or adjust the estimation method parameters. The same model being used
319 twice, it is clear that the success in retrieving the parameter does not ensure
320 the success when applying the method to experimental data. The method
321 does not detect any errors in the model or mismatch between the model and
322 the experiment. However, this strategy may be considered as a preliminary
323 validation of the overall estimation strategy.

324 [Figure 6 about here.]

325 Once the model is validated, this strategy is used to check the feasibility of
326 the estimation procedure, in terms of parameters correlations, and adequacy
327 of optimization algorithm. In addition, it is used to perform parametric

328 studies in order to define the optimal algorithm parameters for each case
329 under consideration.

330 The numerical results for both experimental configurations (a) and (b)
331 and for both cases with isotropic or orthotropic substrate, and with or with-
332 out random noise added (up to 5%), are presented in Table 3 for a 2 mm
333 HDPE substrate covered by a 50 μm TPT coating. These calculations are
334 repeated for 3 mm HDPE substrate covered by a 300 μm TPT coating (see
335 Table 3) in order to be coherent with experimental applications, that follows
336 this section.

337 [Table 3 about here.]

338 Results in table 3 are presented in terms of relative deviation or error
339 between the parameters values used to generate the synthetic data and those
340 estimated using the current identification investigated in thin study. They
341 prove the feasibility of the identification method developed for such bi-layered
342 materials.

343 *3.5. Optimal configuration selection*

344 It can obviously be noticed in table 3 the small relative deviation between
345 the original values and the estimated ones for all treated cases, with and
346 without random Gaussian noise added. The comparison validate therefore
347 the robustness and accuracy of the current method. Not surprisingly, the
348 configuration (a)-FF-FF, gives theoretically better results compared to the
349 configuration (b)-BF-BF and notably without adding noise to the original
350 signal generated by the direct model (with a relative error $< 2.1 \cdot 10^{-3} \%$
351 without noise and $< 1.9 \%$ with 5 % noise for the 2 mm HDPE covered by

352 50 μm of TPT, and a relative error $< 1.0 \cdot 10^{-3}$ % without noise and < 1.9 %
353 with 5 % noise for the 3 mm HDPE covered by 300 μm of TPT). Despite
354 this, results obtained with the configuration (b)-BF-BF are also convincing
355 and promising, specially when adding a certain level of noise (with a relative
356 error $< 6.2 \cdot 10^{-2}$ % without noise and < 1.4 % with 5 % noise for the 2 mm
357 HDPE covered by 50 μm of TPT, and a relative error $< 1.0 \cdot 10^{-2}$ % without
358 noise and < 2 % with 5 % noise for the 3 mm HDPE covered by 300 μm of
359 TPT).

360 [Figure 7 about here.]

361 In fact, this latter configuration (BF-BF) is more suitable from the ex-
362 perimental points of view, as it limits the risk of coating degradation that
363 can take place within the configuration (a)-FF-FF. Adding to that, impulse
364 model is difficult to realize for the configuration (a)-FF-FF, due to the ill-
365 knowledge of the coating thermal characteristic time and the necessity of a
366 high acquisition frequency to detect the dynamic of heat transfer through
367 this thin layer. Nevertheless, the total acquisition time required for the con-
368 figuration (a)-FF-FF is quite lower than that required with the configuration
369 (b)-BF-BF. This intuitive observation is confronted by sensitivity study for
370 the observables to the diffusivities of both layers. The reduced sensitivities
371 for the mode ($m = 2, n = 2$), generally considered as the reference one [45],
372 are plotted in Figs. 7 for both configurations and for the case where both
373 layers are considered as isotropic.

374 This study proved that the identification of thermal diffusivities is possible
375 at short time for the case (a)-FF-FF, whereas the case (b)-BF-BF requires

376 a longer duration time, due to the substrate properties and its thickness.
377 Lastly, accounting all restrictions and risks that could be faced within the
378 configuration (a)-FF-FF and the good accuracy level that could be achieved
379 within the configuration (b)-BF-BF, this latter will be preferred for experi-
380 mental applications presented thereafter.

381 4. Experimental applications

382 Experimental applications have been carried out on samples constituted
383 of a HDPE layer of 3 *mm* thickness covered by a thermal phosphorescent
384 coating whose thicknesses are between 300 and 350 μm . These samples were
385 provided and manufactured by the "Institut Français du Pétrole et Energies
386 Nouvelles" (IFPEN). A photography of one of these samples is shown in Fig.
387 8a.

388 [Figure 8 about here.]

389 As already argued and demonstrated in 3.5, the configuration (b)-BF-BF
390 that consists of an excitation and temperature evolution measurement, both
391 conducted at the material rear face (substrate side), is used. In this case, the
392 substrate layer should be painted by a black thin layer of known emissivity,
393 as shown in Fig. 8b, in order to control the surface emissivity and to have
394 relatively high surface absorption of the laser beam that generates the heat
395 excitation.

396 The identification technique was conducted first on these samples, using
397 the values of the density ρ and heat capacity C that are found in the literature
398 (Table 4). In addition, the density ρ and heat capacity C were measured using

399 a calorimeter, a digital balance and a digital micrometer. The measured
400 values and the level of uncertainties are also indicated in Table 4. These
401 measures were also used for the identification and compared in the next
402 section to the results obtained with the data found in the literature. Note
403 that, all PSO algorithm specifications and stopping criteria, as well as the
404 particles seeking bounds, are the same as those used in the numerical exercise
405 and tabulated in the Table 2.

406 [Table 4 about here.]

407 Adding to these physical properties, the dimension of the exploitation
408 window, namely the size of the frames, $l_x \times l_y$, have to respect the compromise
409 between a sufficient size in order to respect the boundary conditions and
410 restricted size to avoid the degradation of the data (by dilution of the signal
411 in the background signal). Thus, the exploitation window is chosen such as
412 $l_x \times l_y = 35,8 \text{ mm} \times 37.5 \text{ mm}$ for the sample 1 and $l_x \times l_y = 22,8 \text{ mm} \times 24.7 \text{ mm}$
413 for the sample 2, while conserving a symmetric excitation and a centered spot,
414 as mentioned in section 2.2.3.

415 ***4.1. Experimental results and discussion***

416 The results corresponding to the sample 1 (3 mm HDPE ; 300 μm of TPT
417 coating) and sample 2 (3 mm HDPE ; 350 μm TPT coating) are tabulated
418 in Table 5. The variance-covariance matrix of the estimated parameters (see
419 Eq. A.7 in Appendix A for further details) is calculated and the standard
420 deviations of the results are presented into brackets in Tables 5, 6 and 7.

421 [Table 5 about here.]

422 Regarding the substrate layer, relatively thick compared to the coating
 423 layer and typically quasi-isotropic, its thermal diffusivities have been iden-
 424 tified using a "2D" reference method known as ENH [10, 31] (Estimation
 425 using Normalization of Harmonics). Results are also reported in Table 6 for
 426 in-plane diffusivities. Values obtained with the current DSEH method (Tab.
 427 5) for these same in-plane diffusivities are also reported in this table.

428 [Table 6 about here.]

429 The thermal conductivity of the TPT coating in both samples can be
 430 deduced with $\lambda_{TPT} = \rho \cdot C \cdot a_{TPT} = 0.525 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ for sample 1 and
 431 $0.540 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ for sample 2.

432 These estimations are repeated using the density ρ and heat capacity C
 433 values that are measured (Table 4) on sample 1. The results obtained for
 434 this sample are summarized in Table 7.

435 [Table 7 about here.]

436 First of all, Table 5 shows that the thermal diffusivities identified for
 437 both samples are close to each other, which can be considered as a promis-
 438 ing results. From Table 5 it can be also observed that the isotropic char-
 439 acter (i.e. $a_x \approx a_y \approx a_z$) of HDPE is verified for both samples. For
 440 sample 1, $diag(\bar{a}_{HDPE}) = [0.274, 0.259, 0.278] \text{ mm}^2 \cdot \text{s}^{-1}$ and for sample 2,
 441 $diag(\bar{a}_{HDPE}) = [0.265, 0.265, 0.279] \text{ mm}^2 \cdot \text{s}^{-1}$. Furthermore, these values
 442 are coherent with those found in the literature [42] ($a_{HDPE-[42]} \in [0.260 -$
 443 $0.288] \text{ mm}^2 \cdot \text{s}^{-1}$), and are in a very good agreement with results obtained
 444 using ENH method (see Table 6) for the substrate.

445 Regarding the TPT coating, estimated diffusivities values are found, using
 446 measured thermal properties, $\lambda_{TPT} = 0.500 \pm 0.077 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ for sample 1
 447 and $\lambda_{TPT} = 0.510 \pm 0.078 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ for sample 2, to be in good agreement
 448 with that retrieved by Benoit Fond team from Magdeburg: $\lambda_{TPT} = 0.47 \pm$
 449 $0.07 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ who conducted a contact thermal characterization method,
 450 the "hot disk" method [46]. Adding to that, the estimated values are in the
 451 same order of magnitude than those presented in [43]. It should be notice that
 452 the composition (i.e. solvent used) and the mixing ratio may vary according
 453 to the manufacturing process, and explain the slight discrepancy observed
 454 here.

455 Finally a small difference in the results can be observed when repeating
 456 the estimation procedure with the measured values of $\rho \cdot C$, as shown in Table
 457 7. It is also demonstrated that the influence of errors propagation from the
 458 a priori known properties on the estimation results, appears to be neglected.

459 **4.2. Results accuracy evaluation: consistency between experimen-**
 460 **tal and numerical results**

461 Finally, in order to illustrate the consistency of the method, evolution
 462 of the first experimental harmonics $\xi_{m,n}^{exp}(z = 0, t)$ are compared with the
 463 estimated harmonics $\xi_{m,n}^{est}(\hat{\beta}, z = 0, t)$ in Fig. 9, for the sample 1. The
 464 discrepancy between experimental and estimated data are represented by
 465 residual lines that illustrate the great fit between the experimental and the
 466 estimated signals. It should be noticed that the highest deviation is always
 467 observed in the normalized harmonic $\xi_{0,0}$ (mean field) which is often highly
 468 affected by the environmental changes.

469 [Figure 9 about here.]

470 5. Conclusion

471 The proposed identification method is applied on a specific degenerated
472 case of two-layer material constituted of a thin coating deposited on an
473 isotropic or orthotropic substrate. First, a numerical exercise considering
474 a TPT coating involved in phosphorescence thermometry and deposited on
475 an HDPE layer, is carried out using noisy synthetic data within two possible
476 experimental configurations. For some technical limitations, this coating was
477 deposited on a HDPE polymer substrate layer. This kind of deposit is usually
478 encountered in the engines internal combustion chambers for wall tempera-
479 ture measurements. Among the two experimental configurations tested, one
480 consists in exciting and measuring the temperature evolution at the coating
481 side while the other configuration considers the case where excitation and
482 measurements are conducted at the substrate surface side. Based on con-
483 vincing estimation results, sensitivity analysis, and experimental limitations,
484 the last configuration (excitation and measurement on the substrate side)
485 has been proved more convenient for such exercise. Then, the identification
486 has been experimentally carried out on two different samples and gave very
487 promising results. In addition, the values of the polymer diffusivities verify-
488 ing the isotropic character of this latter, and are in good agreements with
489 the literature values as well as with the ENH in-plane estimation method.
490 Regarding the coating, estimated values are in the same order of magnitudes
491 with those found in the literature, and in good agreements with other re-
492 search team results obtained using a contact characterization method. Each
493 element of the method, which allows the simultaneous estimation of the ther-
494 mal diffusivity tensors of each constituting layers, is described and discussed.

495 Among these elements, the pseudo-analytic model and the hypothesis on
496 which it is based, as well as the estimation method used to minimize the
497 discrepancy between the outputs of the model and the measurement, are
498 presented. The materials of the sample have been chosen in such a way to be
499 able to neglect the thermal contact resistance between layers. The samples
500 have been carefully prepared to reach a perfect contact between both layers.
501 In general, thermal contact resistance is significant and may dominate for
502 relatively highly heat conductors such as metals, or for relatively thin layer
503 (having thicknesses in the order of nanometers, which is not the case here
504 where TPT coatings have thicknesses between 50 to 300 micrometers). It
505 can be neglected for the association of relatively low heat conductors such as
506 the TPT coating deposited on polymers such as the HDPE substrate.

507 **Acknowledgments**

508 The Authors thank the European Union and the Nouvelle Aquitaine Dis-
509 trict for their financial support through the CPER/FEDER 2014-2020 pro-
510 grams. The Authors also thank IFPEN that manufactures and provides test
511 samples.

512 **Appendix A. Variance-Covariance Matrix**

513 The Variance-Covariance Matrix is frequently used as a testing tool for
514 characterizing the solutions dispersion, and is considered as one of the most
515 important estimator properties. Typically, this matrix quantifies the estima-
516 tions dispersion among the expected value. The best and the most accurate

517 estimator is the one having the lowest variance, in such a way the estima-
 518 tions $\hat{\beta}$ slightly vary when switching the input data. The deepest is the
 519 information extraction from observables Y^* , the lowest are the standard de-
 520 viations/variances of the estimations β . The estimation variance-covariance
 521 matrix is noticed $cov(\hat{\beta})$, of dimension $n_\beta \times n_\beta$, and is consistent with the
 522 ordinary least square (OLS).

523 When assuming that the measurement noise is non-correlated with a stan-
 524 dard deviation of σ_{noise} , and that variances are similar for all observations
 525 Y^* (homoscedasticity hypothesis), therefore one can apply the following cor-
 526 relation, given in [47]:

$$cov(\hat{\beta}) = \sigma_{noise}^2 [S^T S]^{-1} \quad (\text{A.1})$$

527 Where S is the sensitivity matrix defined in the following equations:

$$S_{m,n}(\beta_j, t) = \left. \frac{\partial Y(\beta, t)}{\partial \beta_j} \right|_{\beta_k \neq j} = \left. \frac{\partial \xi_{m,n}(\beta, t)}{\partial \beta_j} \right|_{\beta_k \neq j} \quad (\text{A.2})$$

528 In this study the measurement noise is assumed Gaussian, additive and
 529 constant in time, and it can be qualified by i.i.d (independent and identi-
 530 cally distributed). Based on the calculations developed by Ruffio in [45],
 531 when working with normalized harmonics $\xi_{m,n}$, the diagonal coefficients of
 532 the variance-covariance matrix are the variances $\sigma_{m,n}^2$ of harmonics and are
 533 given by:

$$\sigma_{m,n}^2 = \frac{\sigma_m^2}{4 \cdot (N_x \times N_y)} (1 + \delta_m) \cdot (1 + \delta_n) \quad (\text{A.3})$$

$$\delta_m = \begin{cases} 1 & \text{if } m = 0, \\ 0 & \text{otherwise} \end{cases} \quad \text{and} \quad \delta_n = \begin{cases} 1 & \text{if } n = 0, \\ 0 & \text{otherwise} \end{cases} \quad (\text{A.4})$$

534 $\sigma_{m,n}$ refers here to the standard deviation corresponding to each harmonic
 535 and are obtained by an IR camera having $N_x \times N_y$ pixels (depending on the
 536 exploitation window size at each treated case). The standard deviation of
 537 each pixel is $\sigma_m = 0.1$ °C. Therefore, Eq.A.3 entails:

$$\frac{\sigma_m^2}{4 \cdot (N_x \times N_y)} \leq \sigma_{m,n}^2 \leq \frac{\sigma_m^2}{(N_x \times N_y)} \quad (\text{A.5})$$

538 All harmonics are assumed to have the same standard deviations (the worst
 539 case scenario), equal to that corresponding to the mean field, i.e. $m=n=0$.

540 Consequently,

$$\sigma_{m,n}^2 = \sigma_{0,0}^2 = \frac{\sigma_m^2}{(N_x \times N_y)} = \frac{0.1^2}{(N_x \times N_y)} \quad (\text{A.6})$$

541 which finally leads to:

$$\text{cov}(\hat{\beta}) = \sigma_{m,n}^2 [S^T S]^{-1} = \frac{0.1^2}{(N_x \times N_y)} [S^T S]^{-1} \quad (\text{A.7})$$

542 **Nomenclature**

543 ***Latin Symbols***

543	\bar{a}	Thermal diffusivity tensor	$m^2 \cdot s^{-1}$
	a_x, a_y, a_z	Thermal diffusivities	$m^2 \cdot s^{-1}$
	C	Thermal Capacity	$J \cdot kg^{-1} \cdot K^{-1}$
	f	Cost function	(-)
	$F_{x,y}$	Shape function associated with the laser beam	(-)
	$F_{m,n}$	Fourier coefficients of the shape function	(-)
	h	Overall heat transfer coefficient	$W \cdot m^{-2} \cdot K^{-1}$
	l_x, l_y, l_z	Sample's dimensions	m
	M, N	Harmonics maximum indices	(-)
	p	Laplace variable	(-)
	$R_{m,n}$	Excitation factor	$W \cdot m^{-2}$
544	Rc	Thermal contact resistance	$K \cdot m^2 \cdot W^{-1}$
	r	Laser spot radius	m
	S	Sensitivity	K
	Sr	Reduced Sensitivity	K
	T	Temperature elevation	K
	T_∞	Environment temperature	K
	$u(t)$	Time shape function of the laser beam	(-)
	$X_m(x)$	Basis function in the x-plane	(-)
	$Y_n(y)$	Basis function in the y-plane	(-)
	Y^*	Observables	K
	$Y(\beta)$	Model outputs	K

545

546 ***Greek symbols***

α_m, β_n	Harmonic pulsations	$rad \cdot m^{-1}$
β	Parameter vector to be estimated	(-)
$\hat{\beta}$	Optimal parameters vector	(-)
ρ	Density	$kg \cdot m^{-3}$
$\lambda_x, \lambda_y, \lambda_z$	Thermal conductivities	$W \cdot m^{-1} \cdot K^{-1}$
547 σ	Standard deviation	(-)
$\xi_{m,n}(t)$	Normalized harmonics in time domain	K
$\xi_{m,n}(p)$	Normalized harmonics in Laplace domain	$K \cdot s$
ϕ_0	Amount of energy absorbed by the sample	J
$\phi_{m,n}^{ex}(p)$	Excitation in the Fourier and Laplace domains	$W \cdot m^{-2} \cdot s$
$\phi_{x,y}^{ex}(t)$	Excitation in the physical and time domains	$W \cdot m^{-2}$

548 ***Subscripts and Superscripts***

ex	Excitation
exp	Experiment
f,b	Front and back faces
549 m,n	Spatial Fourier modes
T	Transpose symbol
x,y	Cartesian coordinates

550 ***Abbreviations***

550 BF	Back face
551 Config	Configuration

DSEH	Direct and Simultaneous Estimation using Harmonics
ENH	Estimation using Normalization of Harmonics
ERH	Estimation using Ratio of Harmonics
FF	Front face
552 HDPE	High Density Polyethylene
OLS	Ordinary Least Square Estimator
PSO	Particle swarm optimization
Rel.	Relative
TPT	Thermographic Phosphor Thermometry

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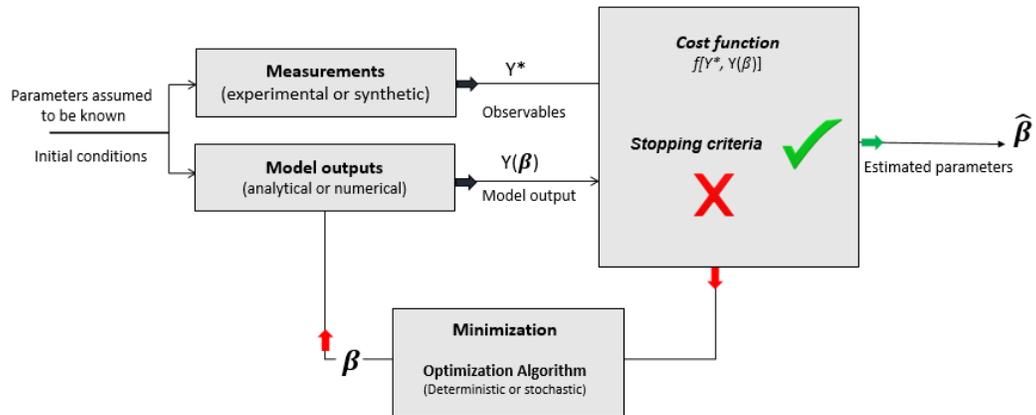


Figure 1: Inverse problem principle and main sections (steps).

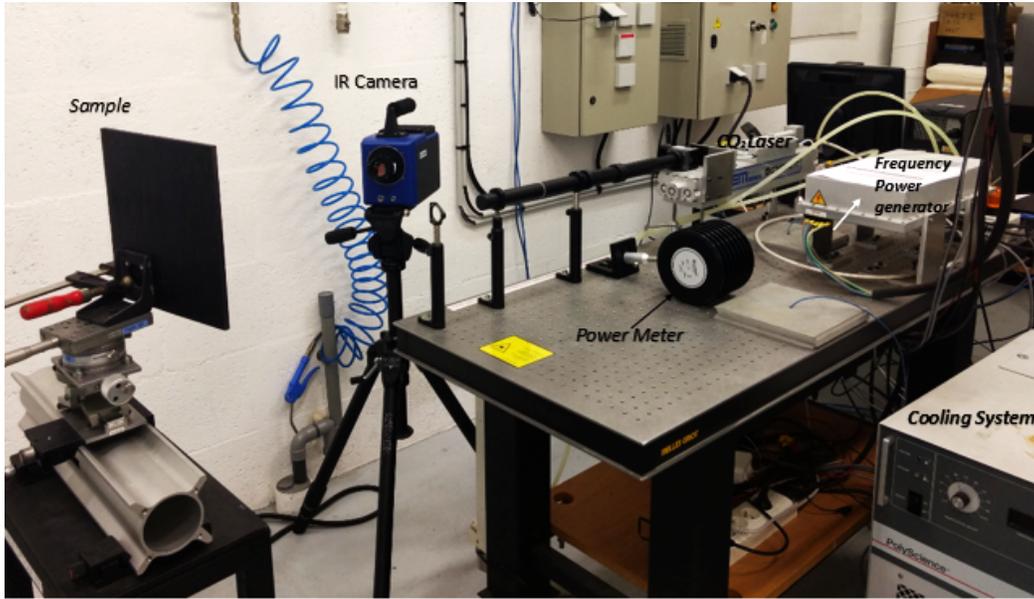


Figure 2: Experimental setup representing the front face flash method and including the main devices involved in the measurement procedure : the sample, CO_2 laser and IR camera.

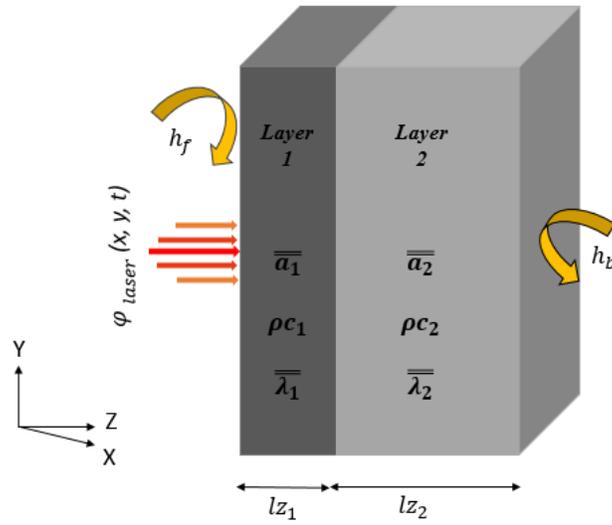


Figure 3: Homogeneous two-layer plane material subjected to non-uniform flash excitation at the front face.

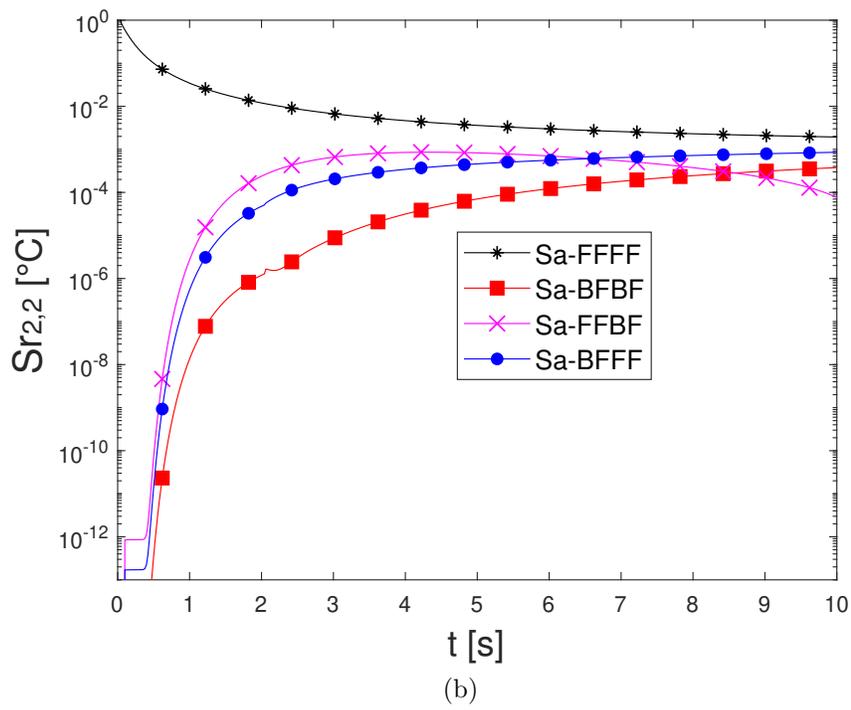
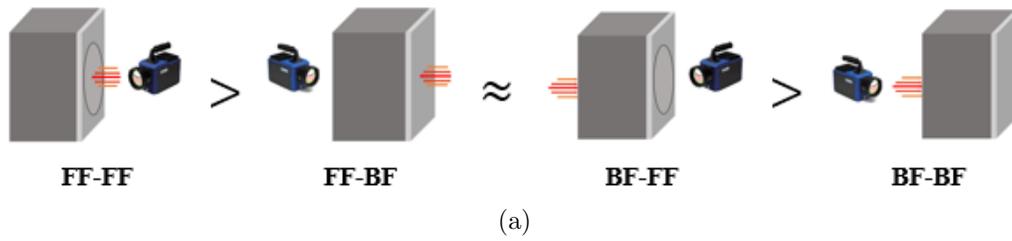


Figure 4: Sensitivity evolution of the harmonic $\xi_{2,2}(t)$ to the coating in depth thermal diffusivity, for the four possible configurations.

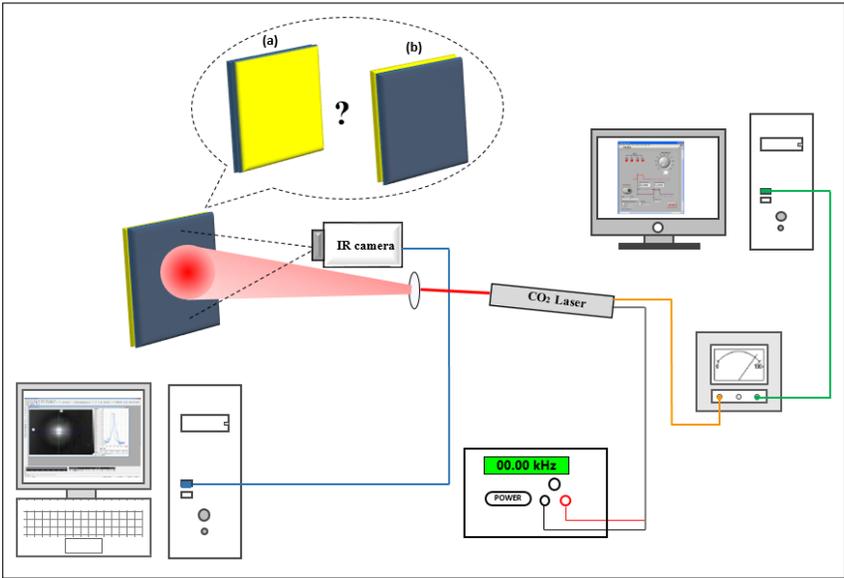


Figure 5: The numerically tested and compared experimental configurations, dedicated to the thermal characterization of the TPT coating.

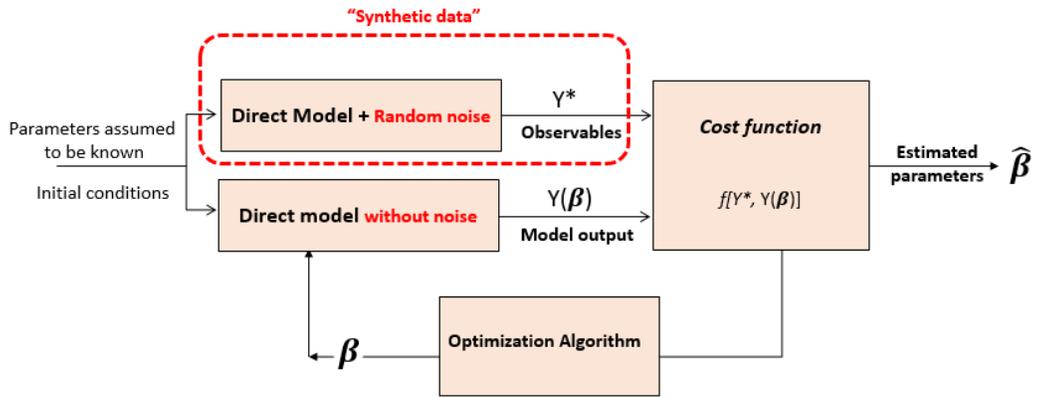
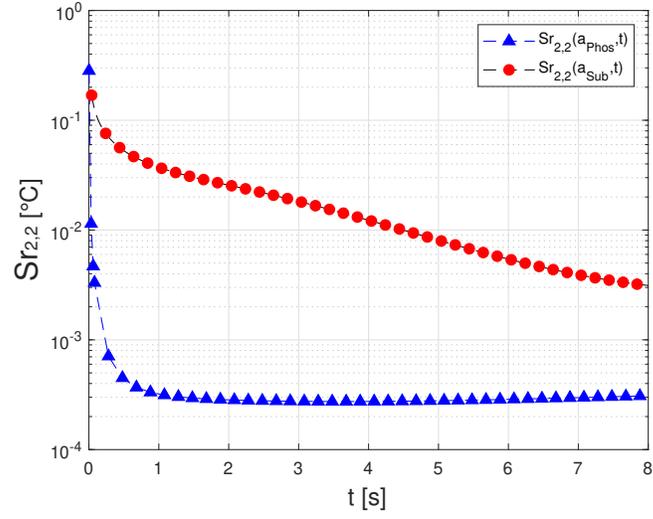
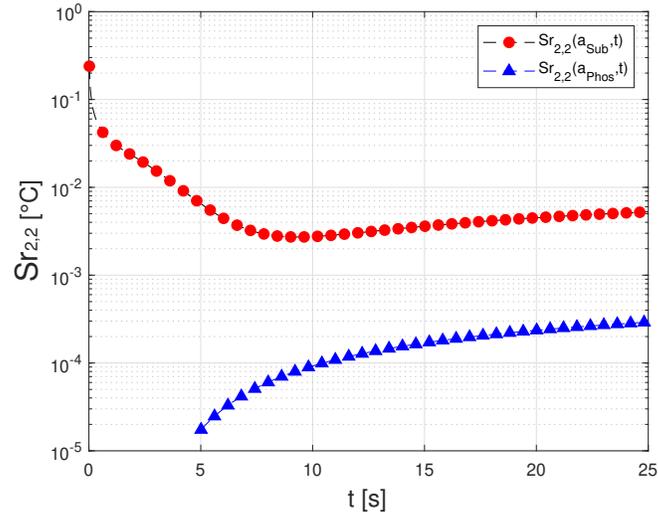


Figure 6: Numerical application principle



(a) Configuration(a)



(b) Configuration(b)

Figure 7: The evolution of normalized harmonics $\xi_{2,2}$ reduced sensitivities to the in-depth thermal diffusivities of both layers, and for both configurations (a) and (b).



(a) TPT coating over a HDPE substrate - Coating side



(b) Black painted HDPE - Substrate side

Figure 8: Experimental application on the two-layers material constituted by a TPT-coating deposited over HDPE substrate

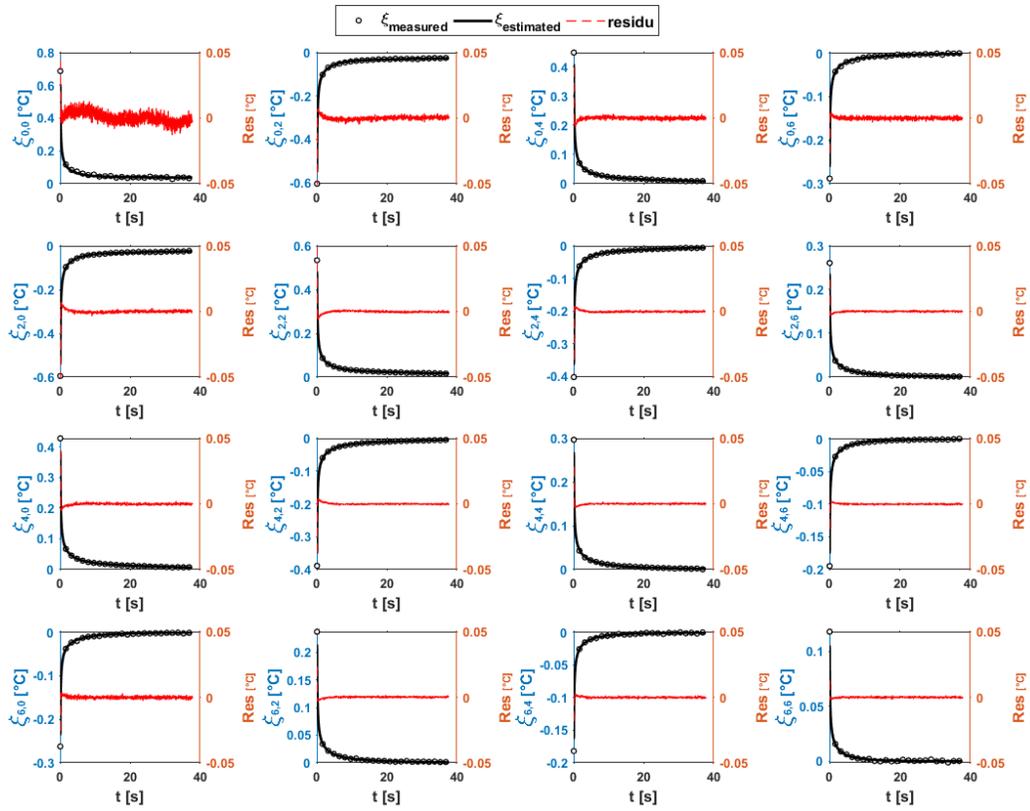


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Parameters	TPT coating [43]	HDPE [42]
a [$mm^2 \cdot s^{-1}$]	[0.30 - 1.00]	2.77
ρC [$kJ \cdot m^{-3} \cdot K^{-1}$]	1316	1805
l_z [mm]	[0.05 - 0.30]	[2.0 - 3.0]

Table 1: Model parameters values used to generate synthetic measures signals.

Conditions	Values
Bounds of $\overline{a_1}$ and $\overline{a_2}$	$[10^{-9}; 10^{-4}] m^2 \cdot s^{-1}$
Bounds of $R_{m,n}$	$[-100; +100] W \cdot m^2$
Number of PSO particles	50
Maximum iterations	$500 \times$ size of β
Maximum stop (stall) iterations	20
Tolerance value	10^{-8}
Maximum time	$+\infty$
Maximum stall time	$+\infty$
Minimum objective value	$-\infty$

Table 2: PSO specifications, and stopping criteria selected for the estimation procedure.

Configuration			Config.(a) / FF-FF		Config.(b) / BF-BF	
Case ($e_{TPT} = 50 \mu m / e_{HDPE} = 2 mm$)		noise level Rel. deviation %	0%	5%	0%	5%
1	isotropic substrate	$\frac{ \Delta a }{a}$	$5.37 \cdot 10^{-4}\%$	0.50%	$1.39 \cdot 10^{-3}\%$	0.51%
	isotropic coating	$\frac{ \Delta a }{a}$	$2.06 \cdot 10^{-3}\%$	1.81%	$6.18 \cdot 10^{-2}\%$	1.33%
2	orthotropic substrate	$\frac{ \Delta a_x }{a_x}$	$2.56 \cdot 10^{-4}\%$	1.40%	$1.69 \cdot 10^{-3}\%$	1.02%
		$\frac{ \Delta a_y }{a_y}$	$2.84 \cdot 10^{-4}\%$	0.66%	$1.64 \cdot 10^{-3}\%$	0.45%
		$\frac{ \Delta a_z }{a_z}$	$1.77 \cdot 10^{-4}\%$	0.16%	$2.83 \cdot 10^{-4}\%$	0.33%
	isotropic coating	$\frac{ \Delta a }{a}$	$5.61 \cdot 10^{-4}\%$	0.45%	$2.74 \cdot 10^{-2}\%$	0.42%
Case ($e_{TPT} = 300 \mu m / e_{HDPE} = 3 mm$)		noise level Rel. deviation %	0%	5%	0%	5%
1	isotropic substrate	$\frac{ \Delta a }{a}$	$1.22 \cdot 10^{-4}\%$	1.88%	$8.16 \cdot 10^{-4}\%$	0.84%
	isotropic coating	$\frac{ \Delta a }{a}$	$3.01 \cdot 10^{-4}\%$	1.24%	$2.83 \cdot 10^{-3}\%$	1.22%
2	orthotropic substrate	$\frac{ \Delta a_x }{a_x}$	$3.71 \cdot 10^{-4}\%$	0.68%	$6.13 \cdot 10^{-4}\%$	0.95%
		$\frac{ \Delta a_y }{a_y}$	$3.15 \cdot 10^{-4}\%$	0.69%	$6.19 \cdot 10^{-4}\%$	0.85%
		$\frac{ \Delta a_z }{a_z}$	$6.96 \cdot 10^{-4}\%$	1.21%	$1.51 \cdot 10^{-3}\%$	0.85%
	isotropic coating	$\frac{ \Delta a }{a}$	$9.20 \cdot 10^{-4}\%$	1.86%	$9.01 \cdot 10^{-3}\%$	1.88%

Table 3: Estimation results in function of the presupposed nature of the substrate (isotropic or orthotropic) for a 2 mm HDPE covered by a 50 μm TPT coating and a 3 mm HDPE covered by a 300 μm of TPT coating with $a_{HDPE} = 2.77 mm^2 \cdot s^{-1}$ and $a_{TPT} = 1 mm^2 \cdot s^{-1}$.

Parameters	Values from literature [43][42]	Measured values
$\rho_{HDPE} [kg \cdot m^{-3}]$	950	897 ± 16
$C_{HDPE} [J \cdot kg^{-1} \cdot K^{-1}]$	1900	1950 ± 58
$\rho_{TPT} [kg \cdot m^{-3}]$	2800	3131 ± 110
$C_{TPT} [J \cdot kg^{-1} \cdot K^{-1}]$	470	400 ± 12

Table 4: Values required for the experimental identification.

Estimated parameters	Sample 1	Sample 2
$a_{xHDPE} [mm^2 \cdot s^{-1}]$	0.274 ($\sigma = 1.41 \cdot 10^{-3}$, 0.52%)	0.265 ($\sigma = 9.50 \cdot 10^{-4}$, 0.36%)
$a_{yHDPE} [mm^2 \cdot s^{-1}]$	0.259 ($\sigma = 1.41 \cdot 10^{-3}$, 0.55%)	0.265 ($\sigma = 9.50 \cdot 10^{-4}$, 0.36%)
$a_{zHDPE} [mm^2 \cdot s^{-1}]$	0.278 ($\sigma = 1.9 \cdot 10^{-4}$, 0.069%)	0.279 ($\sigma = 1.4 \cdot 10^{-4}$, 0.049%)
$a_{TPT} [mm^2 \cdot s^{-1}]$	0.399 ($\sigma = 0.035$, 8.85%)	0.411 ($\sigma = 0.025$, 6.20%)

Table 5: Experimental results for sample 1 constituted of a 3 mm HDPE covered by a 300 μm TPT coating and sample 2 constituted of a 3 mm HDPE covered by a 350 μm TPT coating.

Estimated parameter ($\text{mm}^2 \cdot \text{s}^{-1}$)	Sample 1		Sample 2	
	DSEH	ENH	DSEH	ENH
a_{xHDPE}	0.274	0.277 $\sigma = 6.73 \cdot 10^{-3}$ (2.43%)	0.265	0.263 $\sigma = 3.92 \cdot 10^{-3}$ (1.49%)
a_{yHDPE}	0.259	0.257 $\sigma = 9.78 \cdot 10^{-3}$ (3.80%)	0.265	0.267 $\sigma = 6.34 \cdot 10^{-3}$ (2.37%)

Table 6: Comparison of thermal diffusivities of HDPE using DSEH and ENH methods.

Estimated parameters	Using literature values	Using measured values
$a_{xHDPE} [mm^2 \cdot s^{-1}]$	0.274 ($\sigma = 1.41 \cdot 10^{-3}, 0.52\%$)	0.273 ($\sigma = 1.41 \cdot 10^{-3}, 0.52\%$)
$a_{yHDPE} [mm^2 \cdot s^{-1}]$	0.259 ($\sigma = 1.41 \cdot 10^{-3}, 0.55\%$)	0.259 ($\sigma = 1.41 \cdot 10^{-3}, 0.55\%$)
$a_{zHDPE} [mm^2 \cdot s^{-1}]$	0.278 ($\sigma = 1.9 \cdot 10^{-4}, 0.069\%$)	0.278 ($\sigma = 1.9 \cdot 10^{-4}, 0.069\%$)
$a_{TPT} [mm^2 \cdot s^{-1}]$	0.399 ($\sigma = 0.035, 8.85\%$)	0.407 ($\sigma = 0.035, 8.69\%$)

Table 7: Experimental identification results using the measured values, compared to previous results using literature values of some parameters.