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Technical Note

Effect of a thin layer on the measurement of the thermal diffusivity of a material by a flash method

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1. Introduction

The *flash method* is a commonly used technique for the measurement of the thermal diffusivity of materials. In this method, the front face of the sample absorbs the flash energy and the rear face temperature variation is measured.

In practical cases, a coating is often deposited on the front or rear face of the sample in order to modify their radiative or electric properties: a metal deposit is used to make the faces opaque (case of a semi-transparent material) and a "black" paint on the front face can increase the energy absorption (case of a metallic material). The use of a black coating on the rear face can also increase its emissivity (case of a temperature measurement through an infrared detector), while a metallic coating on the same face (silver paint on a non-coated material) can be used to warrant the electrical contact of an open junction thermocouple.

The value of the diffusivity measured by the "*Flash method*" can be strongly affected by the use of such a coating. The aim of this paper is to study the effects of a paint or of a "*sputtered*" thin film on the thermal response of the material and to determine if these effects can be neglected.

2. Model

When the material is uniformly stimulated on its whole front face, heat transfer within the material can be considered one-directional (see Fig. 1). The model is given by the solution of the one-dimensional heat transfer equation [1]. It can be represented by a set of three quadrupoles [2]:

- A quadrupole associated with the "*coating*" ("*c*"), which is completely defined by the knowledge of its thermal conductivity λ_c, its specific heat capacity $(\rho C)_c$ and its thickness e_c. Its diffusivity is defined by: $a_c = \lambda_c/(\rho C)_c$. The coating can also be defined by its thermal resistance $R_c = e_c/\lambda_c$, its thermal capacitance $C_c = (\rho C)_c e_c$ and its characteristic time: $\tau_c = e_c^2/a_c$
- A "*contact resistance*" (R_{cr}) quadrupole that represents the imperfect contact between the coating and the material ("*m*").
- A quadrupole, which represents the material that must be characterized (conductivity λ_m, heat capacity $(\rho C)_m$, diffusivity $a_m = \lambda_m/(\rho C)_m$).

Each quadrupole is associated to a transfer matrix that links the Laplace transforms of both temperatures T and fluxes ϕ of the front (subscript "*o*") and back faces (subscript "*i*") of the material:

$$\begin{pmatrix} \bar{T}_0 \\ \phi_0 \end{pmatrix} = \begin{bmatrix} \mathscr{A} & \mathscr{B} \\ \mathscr{C} & \mathscr{D} \end{bmatrix} \begin{pmatrix} \bar{T}_1 \\ \phi_1 \end{pmatrix}$$

$$= \begin{bmatrix} \mathscr{A}_c & \mathscr{B}_c \\ \mathscr{C}_c & \mathscr{D}_c \end{bmatrix} \begin{bmatrix} 1 & R_{\mathrm{cr}} \\ 0 & 1 \end{bmatrix} \begin{bmatrix} \mathscr{A}_m & \mathscr{B}_m \\ \mathscr{C}_m & \mathscr{D}_m \end{bmatrix} \begin{pmatrix} \bar{T}_1 \\ \phi_1 \end{pmatrix}$$

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p is the Laplace variable and the bar denotes the Laplace transforms of temperatures and fluxes.

$\mathscr{A}$, $\mathscr{B}$, $\mathscr{C}$ and $\mathscr{D}$ depend on the heat conductivity, capacity and diffusivity of the material:

$$\mathscr{A}_i = \mathscr{D}_i = \cosh(q_i), \quad \mathscr{B}_i = \frac{1}{\lambda_i\sqrt{p/a_i}}\sinh(q_i) \quad \text{and}$$

$$\mathscr{C}_i = \lambda_i\sqrt{p/a_i}\,\sinh(q_i) \text{ with:}$$

$$q_i = e_i\sqrt{p/a_i} \quad \text{for } i = c \text{ or } m$$

In the case considered here, the heat losses are neglected and the front face stimulation is assumed to be a heat pulse in time. Q is the absorbed energy by a unit surface.

In the flash method, the rear face thermogram can be normalized by its maximum. The value of this maximum is given by the adiabatic temperature:

$$T_{\max} = Q/\big((\rho C)_c e_c + (\rho C)_m e_m\big) \tag{1}$$

The following equations define the normalized rear face thermogram:

$$\bar{T}_1/T_{\max} = \frac{C_c + C_m}{\mathscr{C}} \tag{2}$$

with:

$$\mathscr{C} = C_m p \frac{\sinh(q_m)}{q_m}\left[\cosh(q_c) + \frac{\sinh(q_c)}{q_c}\left(R_{\mathrm{cr}} C_c p + \frac{C_c}{C_m} q_m \coth(q_m)\right)\right] \tag{3}$$

where: $C_m = (\rho C)_m e_m$ is the capacitance of the medium and $R_m = e_m/\lambda_m$ its resistance.

In Eq. (3), the bracketed term appears as a correction term that takes into account the effect of both the coating and the contact resistance.

This model can be used for a direct simulation. We will see later the results given by such a model (see Section 4). Using an inverse technique, this model can also be used to estimate the contact resistance or thermal properties of the two layers material [3].

Nevertheless, it can also be used to develop an approached model and evaluate the effects of the coating on the diffusivity value that is measured by using a non-coated material model.

3. Approximated solution of the model

3.1. Series approximation

In order to obtain an approximated solution of the model that can be compared with the response of the non-coated material later on, a series expansion in p of the corrective term of Eq. (3), truncated to its first term, is implemented:

$$\mathscr{C} = (C_m + C_c)p\,\frac{\sinh(q_m)}{q_m}\left\{1 + p\frac{C_m \cdot C_c}{C_m + C_c}\left[\frac{R_c}{2}\left(1 + \frac{1}{3}\frac{C_c}{C_m}\right) + R_{\mathrm{cr}} + \frac{R_m}{3}\right] + \mathrm{O}\big(p^2\big)\right\} \tag{4}$$

If we assume that heat capacities of both, layer and coating are the same order of magnitude, which is the case for most non porous materials, and that the thickness of the layer is weak compared to the thickness of the material (then: $C_c \ll C_m$). The following equation can be derived:

$$\mathscr{C} \simeq (C_m + C_c)p\,\frac{\sinh(q_m)}{q_m}\left\{1 + pC_c\left(\frac{R_c}{2} + R_{\mathrm{cr}} + \frac{R_m}{3}\right)\right\} \tag{5}$$

This result clearly shows the importance of the capacitance effect of the layer even though these effects have been already taken into account by the reduction of the thermogram.

Two corrections can be introduced to take the effects of the coating into account:

- A time shift of the thermogram
- A modification of the characteristic time of the material

3.2. "Shifting" of the thermogram

As a first approximation, the effect of the coating can be interpreted by a shift of the thermogram. Eq. (5) can be considered as a first order expansion in p of the following expression:

$$\bar{T}_1/T_{\max} \simeq \big\{\bar{T}_1/T_{\max}\big\}_0 \exp(-pC_c(R_c/2 + R_{\mathrm{cr}} + R_m/2)) \tag{6}$$

where $\{\bar{T}_1/T_{\max}\}_0$ is the transient response of the non-coated material in the Laplace domain (for a first-order development $1/(1 + Kp) \simeq \exp(-Kp)$).

The delay duration is then equal to the quantity: $\tau_1 = C_c(R_c/2 + R_{\mathrm{cr}} + R_m/3)$. In the case where the coating resistance and the contact resistance can be neglected with respect to the material resistance, that is $R_c \ll R_m$ and $R_{\mathrm{cr}} \ll R_m$, this correction introduced on the thermogram corresponds to a time delay equal to $C_c R_m/3$.

This relation clearly shows that we have to be rebuke to the simple-minded idea that a coating can be neglected if its thermal resistance is weak compared to the resistance of the material.

3.3. *Modification of the diffusivity*

Another technique to obtain a more consistent thermogram is to modify the value of the diffusivity in order to obtain a thermogram close to the thermogram of a homogeneous material.

This correction can be obtained by a linear expansion around $k = 1$ of the expression given by equation:

$$\frac{\sinh(q_m k)}{q_m k} = \frac{\sinh(q_m)}{q_m} + (k-1)\big(\cosh(q_m) - \sinh(q_m)/q_m\big) + \mathrm{O}\big((k-1)^2\big) \qquad (7)$$

A development of this expression in q_m leads to:

$$\frac{\sinh(q_m k)}{q_m k} \simeq \frac{\sinh(q_m)}{q_m}\left[1 + (k-1)q_m^2/3\right] \qquad (8)$$

By an identification of Eqs. (5) and (8), one finds:

$$\left(\frac{e^2}{a}\right)_{\mathrm{es}} = \left(\frac{e_m^2}{a_m}\right)\left[1 + \frac{C_c}{C_m}\left(1 + \frac{3}{2}\frac{R_c}{R_m} + 3\frac{R_{\mathrm{cr}}}{R_m}\right)\right]^2 \qquad (9)$$

The time delay derived in Section 3.2 can be translated into an increase of the characteristic time of the medium. We just have to check if this analysis is fully justified in some practical cases.

4. Case of a two-coating material

The same development as those conducted in a case of a one-coating material has been derived for a two-coating material, by taking into account a coating on both, the rear and the front face of the material. Since the expression is quite more complicated in the general case, we make the assumption that the two deposited layers are similar (see Fig. 2).

The delay duration (See Section 3.2) becomes equal to $\tau_2 = 2\tau_1$, with the same assumptions.

Eq. (9) becomes:

$$\left(\frac{e^2}{a}\right)_{\mathrm{es}} = \left(\frac{e_m^2}{a_m}\right)\left[1 + \frac{C_c}{C_m}\left(2 + 3\frac{R_c}{R_m} + 6\frac{R_{\mathrm{cr}}}{R_m}\right)\right]^2 \qquad (10)$$

Thus, one can see that the effect on the diffusivity is emphasized by a two factor if the material is coated on its two faces.

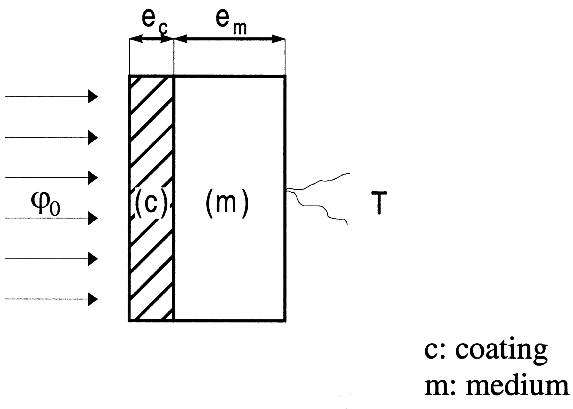

Fig. 1. Principle of the method.

5. Numerical results — one-coating material

To illustrate the preceding approach, a relative thick coating case is considered. The contact resistance is neglected. The thickness of the coating is quite large in order to magnify its effects and to test the different approximations.

Properties of the coating:

$\lambda_c = 0.6\ \mathrm{W\,m^{-1}\,K^{-1}}$ $R_c = 2.5 \times 10^{-4}\ \mathrm{K\,m^2\,W^{-1}}$
$(\rho c)_c = 4 \times 10^6\ \mathrm{J\,m^{-3}\,K^{-1}}$ $C_c = 600\ \mathrm{J\,m^{-2}\,K^{-1}}$
$e_c = 150 \times 10^{-6}\ \mathrm{m}$ $\tau_c = 0.15\ \mathrm{s}$

Two cases can be considered:

1. An insulating material (see Fig. 3)

$\lambda_m = 0.2\ \mathrm{W\,m^{-1}\,K^{-1}}$ $R_m = 2 \times 10^{-2}\ \mathrm{K\,m^2\,W^{-1}}$
$(\rho c)_m = 2 \times 10^6\ \mathrm{J\,m^{-3}\,K^{-1}}$ $C_m = 8000\ \mathrm{J\,m^{-2}\,K^{-1}}$
$e_m = 4 \times 10^{-3}\ \mathrm{m}$ $\tau_m = 160\ \mathrm{s}$
$t_{\mathrm{shift}} = 4.075\ \mathrm{s}$ $\dfrac{(e^2/a)_{\mathrm{est}}}{(e_m^2/a_m)} = 1.16$

2. A conductive material (see Fig. 4)

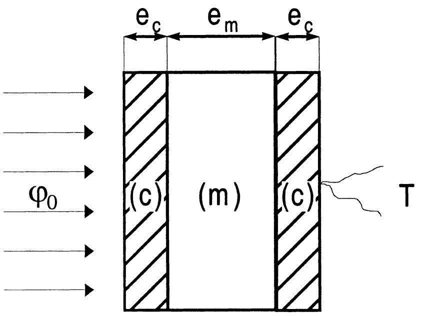

Fig. 2. Case of a two-coating sample.

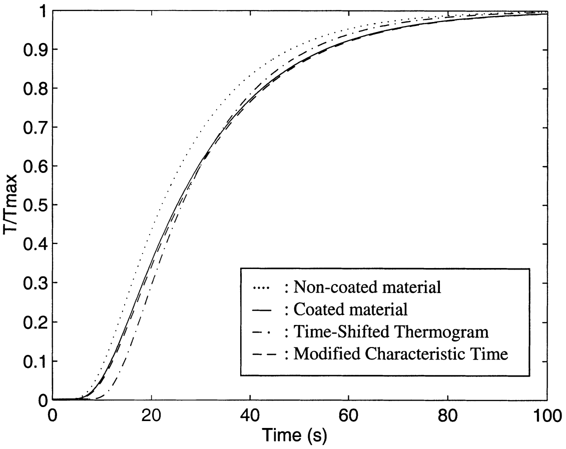

Fig. 3. Case of an insulating material.

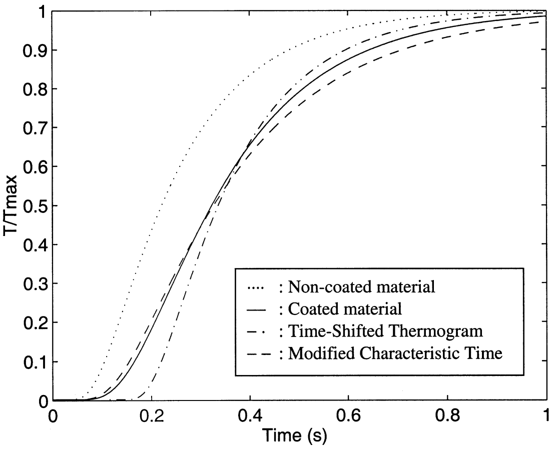

Fig. 4. Case of a conductive material.

$\lambda_m = 20\,\mathrm{W\,m^{-1}\,K^{-1}}$ $\qquad R_m = 2\times10^{-4}\,\mathrm{K\,m^2\,W^{-1}}$

$(\rho c)_m = 2\times10^6\,\mathrm{J\,m^{-3}\,K^{-1}}$ $\qquad C_m = 8000\,\mathrm{J\,m^{-2}\,K^{-1}}$

$e_m = 4\times10^{-3}\,\mathrm{m}$ $\qquad \tau_m = 1.6\,\mathrm{s}$

$t_{\mathrm{shift}} = 0.115\,\mathrm{s}$ $\qquad \dfrac{(e^2/a)_{\mathrm{est}}}{(e_m^2/a_m)} = 1.48$

In both cases, we compare the responses of the non-coated material (*dots*), of the two-layer material (*solid curve*) with the response obtained by a time-shift of the thermogram (*mixed*) and the response given by a modification of the characteristic time (*doted*).

In the two cases considered here, the correction obtained by a shift of the thermogram gives satisfactory result of the delayed time introduced on the half rise time of the thermogram. The modification of the characteristic time of the medium (see Eq. (9)) constitutes a better correction.

The errors on the diffusivity values are quite large — respectively, 15 and 50% in the case of an insulating material and a conductive material. This can be mainly explained by the low conductivity of the coating ($\lambda_c = 0.6$ W m^{-1} K^{-1}) and its large thickness ($e_c = 150$ µm).

Eq. (9) or (10) can be used to determine the thickness of the coating which has to be considered in order to reduce this error for instance to 1%. One finds that for both insulating and conductive materials, a 10 µm-thick coating for one-coating materials or a 5 µm-thick coating for each layer of two-coating materials allow to reach this result.

6. Conclusions

To neglect the effect of the coating on one face of the material, the time correction induced by the presence of the coating must be neglected with respect to the characteristic time of the non-coated material. That is:

$$\tau_1 = C_c(R_c/2 + R_{\mathrm{cr}} + R_m/3) \ll R_m C_m$$

This criterion can be decomposed into three criteria that have to be simultaneously met:

- A characteristic time low compared to the characteristic time of the material

  $$R_c C_c \ll R_m C_m$$

- A coating thermal capacitance neglected compared to the material capacitance

  $$C_c \ll C_m$$

- A low effect of the contact resistance

  $$R_{\mathrm{cr}} C_c \ll R_m C_m$$

These criteria remain the same if the material is coated on its two faces.

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