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Diffusivity measurement of semi-transparent media: model of the coupled transient heat transfer and experiments on glass, silica glass and zinc selenide

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Abstract

The aim of this theoretical and experimental study is to provide a complete methodology to estimate the intrinsic diffusivity of semi-transparent media from flash method experiments. A semi-analytical model describes the coupled conductive–radiative transient heat transfer in a slab. The relevance of the model used for the inversion is then investigated. Experimental results are presented for several semi-transparent samples: float glass, SiO_2 and ZnSe. Measurements of rear face temperature are obtained by optical way (infrared detection) for a wide temperature range (293–700 K), for several radiation boundary conditions (black or gold coatings). The results are compared with those obtained through an other set-up and an other experimental procedure.

1. Introduction

In the last decade, a number of authors took interest in the coupled conductive and radiative heat transfer and studied the transfer mechanisms in terms of the temperature response [1–6]. Within semi-transparent media such as window glasses, crystalline ceramics and insulated foams, energy can be transferred by radiation in addition to heat conduction. In this case of combined heat transfer, the measurement of the thermal intrinsic diffusivity becomes a challenge: in participating media, the measured diffusivity—estimated thanks to a purely conductive model—is an apparent one. It takes both conductive and radiative transfers into account.

In order to obtain the true thermal diffusivity (which must be determined without any contribution from radiant transmission), a model including both radiative and conductive effects has to be built in a first stage. The model presented in the first part is based on a kernel substitution technique [7]. It provides the temperature rise of the medium, which can absorb, emit and anisotropically scatter thermal radiation. The problem of parameter estimation is then studied in a theoretical way: estimations are performed on simulated noised thermograms. It is proved that the estimation of the phononic diffusivity is obtained with a good accuracy.

In a second part, transient measurements are performed by an optical way. A new original flash set-up has been developed in order to measure the rear face temperature of samples such as float glass, silica glass and zinc selenide. The flash experiment allows to reach the intrinsic diffusivity through an inverse data treatment using the right combined model. This method is then compared with the gold boundaries method [5] commonly used for the thermal diffusivity measurement of semi-transparent materials (where inversion is

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Nomenclature

a_{ph}	phononic diffusivity $\lambda/\rho c$ ($m^2\,s^{-1}$)
c	heat capacity ($J\,kg\,K^{-1}$)
e	slab thickness (m)
h	heat loss coefficient ($W\,m^{-2}\,K^{-1}$)
H	Biot number he/λ
i	intensity
i_b	black body intensity
N_{pl}	Planck number $\lambda\beta/4n^2\sigma T_0^3$
n	refractive index
p^*	reduced Laplace parameter $e^2 p/\alpha_{ph}$
Q	heat pulse energy surface density ($J\,m^{-2}$)
T	temperature (K)
t^*	reduced time $\alpha_{ph} t/e^2$
z	space coordinate orthogonal to the sample faces (m)
Greek symbols	
β	extinction coefficient ($\sigma_s + \kappa$) (m^{-1})
κ	absorption coefficient (m^{-1})
ε	emissivity
λ	thermal conductivity ($W\,m^{-1}\,K^{-1}$)
μ	$\cos(\theta)$
ϕ	heat flux density ($W\,m^{-2}$)
Φ	phase function
ρ	density ($kg\,m^{-3}$)
σ_s	scattering coefficient (m^{-1})
σ	Stefan Boltzmann constant
τ_0	gray optical thickness $(\kappa + \sigma_s)e$
τ	gray optical depth $(\kappa + \sigma_s)z$
θ	reduced temperature $T - T_0/(Q/\rho c_p e)$
ω_0	scattering albedo σ_s/β
Superscripts	
+	refers to the forward direction
−	refers to the backward direction
∧	refers to an estimated quantity

implemented using the classical conductive model in the case of reflective boundaries and small optical thickness of the sample).

2. Theory

2.1. Physical model and assumptions

The physical system is an uniform plane parallel gray medium confined within opaque walls. Diffuse gray emissivities and reflectivities are assumed. The coordinate system and the physical model are presented in the Fig. 1. The semi-transparent slab absorbs, emits and scatters anisotropically. It is initially at uniform temperature T_0 and receives a heat pulse stimulation Q on its front face (flash method). The variation of the rear-face temperature with respect to time t is then considered.

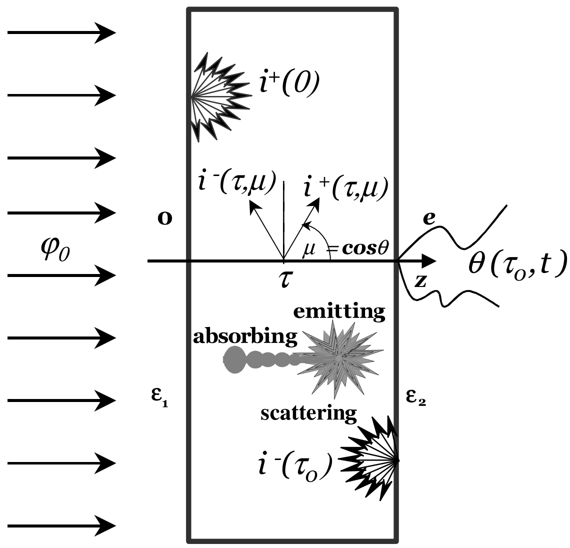

Fig. 1. Scheme of the medium.

2.2. Mathematical formulation and analytical approach

The governing equations of the coupled conductive–radiative transient problem considered are given by

$$\mu\frac{\partial i(\tau,\mu)}{\partial\tau} + i(\tau,\mu) = (1-\omega_0)n^2 i_b(\tau,\mu) + \frac{\omega_0}{2}\int_{-1}^{1} i(\tau,\mu')\Phi(\mu,\mu')\,d\mu' \quad (1)$$

$$\beta^2\lambda\frac{\partial^2\theta(\tau,t)}{\partial\tau^2} - \beta\frac{\partial q_r(\tau,t)}{\partial\tau} = \rho c\frac{\partial\theta(\tau,t)}{\partial t} \quad (2)$$

where μ is the cosine of θ, i is the radiation intensity, i_b the intensity of the black body, n the refractive index, $\tau = \beta z$ the optical depth, ω_0 the albedo, $\Phi(\mu,\mu')$ the phase function, β the extinction coefficient, T_0 the reference temperature, $\theta = (T - T_0)/\{Q/\rho c e\}$ the reduced temperature, q_r the radiative heat flux, ρ the density, c the calorific capacity and e the sample thickness.

In order to obtain a semi-analytical solution, the same method as described in [6] and [14], based on the exponential kernel technique [7] is performed then.

The Laplace transform $\widetilde{\theta}(p) = \int_0^{+\infty} \theta(t)\exp(-pt)\mathrm{d}t$ is applied and the dimensionless temperature is found to satisfy the following differential equation:

$$\frac{\mathrm{d}^4\widetilde{\theta}(\tau)}{\mathrm{d}\tau^4} - B\frac{\mathrm{d}^2\widetilde{\theta}(\tau)}{\mathrm{d}\tau^2} + C\widetilde{\theta}(\tau) = 0, \tag{3}$$

where the following coefficients are defined:

$$B = \delta^{*^2} + \frac{4a\tau_0^2(1-\omega_0)}{N_{\mathrm{pl}}} + p^* \quad C = \delta^{*^2} p^* \tag{4}$$

$$\delta^* = (b^2 - \omega_0 a a_1)^{1/2}\left(1 - \omega_0 a - \frac{1}{4}\omega_0 a a_2\left(\frac{3}{b^2} - 1\right)^2\right)^{1/2}\tau_0 \tag{5}$$

and (a, b) solutions of $\begin{cases} \left(\frac{1}{2} + \frac{a_2}{8}\right)b^4 - b^3 - \frac{3}{4}a_2 b^2 + \frac{9a_2}{8} = 0 \\ a = \frac{b}{2}, \quad b \in]0, 1] \end{cases}$ (6)

Coefficients a_1 and a_2 are the coefficients of the first terms of the Legendre polynomial series expansion of the phase function with $(a, b) = (3/4, 3/2)$ and $a_1 = a_2 = 0$ in the case of an isotropic scattering medium.

The solution of this fourth-order ordinary differential temperature equation is

$$\widetilde{\theta}(\tau) = \sum_{i=1}^{4} \alpha_i \mathrm{e}^{\gamma_i \tau} \tag{7}$$

where the α_i's are four constants determined from boundary conditions and the α_i's the four roots of the characteristic equation of Eq. (3). Appropriate radiative and energy conservation equations written at the boundaries ($\tau = 0$ and $\tau = \tau_0$) of the sample allow to calculate the four α_i's—see the quadrupole formulation presented in [8, Chapter 8]—and a numerical inversion of the Laplace transform yields the simulated rear face thermogram (temperature versus time curve).

2.3. Reduced model in view of parameter estimation problem

The media are assumed to be gray for the previous analytical approach to be valid or, if it is not the case, the non-grayness of materials is taken into account through mean radiative coefficients.

These strong assumptions will be discussed in Sections 2.4 (simulation of estimation) and 3.5 (experimental estimation) but they are already justified, at this point, by the following considerations:

- Heat transfer is of the coupled conductive and radiative type: as a consequence the radiative effects are not as important as in the case of a purely radiative heat transfer. Moreover the above model, which is a reduced model, is only used to simulate a temperature response (and not an intensity field) to a given heat flux stimulation: surface temperature is the only state variable that is observed.
- It is well known that the radiative properties of media may strongly depend on the wavelength but this happens mainly for gases and not for the solid condensed media we are interested in: these types of materials have a relatively smooth absorption or scattering coefficient.
- The purpose of the semi-analytical model presented here is not to be used as a direct model but to be implemented to invert temperature measurements in order to estimate a conductive parameter (the radiative parameters are not looked at, even if they have to be present in the model that is used for thermal diffusivity estimation).

The non-grayness effect on the parameter estimation will be investigated in the next section for a test case.

2.4. Theoretical study of parameter estimation

Before using a model for parameter estimation [9] from experiments, it is necessary to check:

- which parameters will be correctly estimated;
- which parameters will be poorly estimated but are necessary to take into account the kind of physical mechanisms involved in the experiment;
- which parameter must be eliminated because of a lack of sensitivity that can possibly make the estimation procedure impossible (depending on the robustness of the estimation).

A complete theoretical study using stochastic and statistic tools has already been performed [10]. In the case of an experiment on an absorbing emitting but non-scattering medium, the four parameters of the model are: the optical thickness τ_0, the Planck number N_{pl}, the phononic diffusivity a_{ph} and the Biot number H (in order to take the heat losses into account).

Several estimations on simulated noised thermograms have been carried out and the sensitivity curves have been studied. An example of parameter estimation is presented in Fig. 2. It corresponds to a simulated thermogram that has been corrupted by an additional uncorrelated gaussian noise of constant standard deviation equal to 0.5% of the maximum of the reduced thermogram. The real values of the parameters are $\tau_0 = 0.1$, $N_{\mathrm{pl}} = 0.4773$, $H = 0.05$, $a_{\mathrm{ph}} = 5 \times 10^{-7}$ m^2 s^{-1} and the estimated values are $\tau_0 = 0.10116$, $N_{\mathrm{pl}} = 0.48084$, $H = 0.050376$, $a_{\mathrm{ph}} = 4.9949 \times 10^{-7}$ m^2 s^{-1}. The sensitivity of the temperature signal to the phononic diffusivity is very high and appears to be uncorrelated

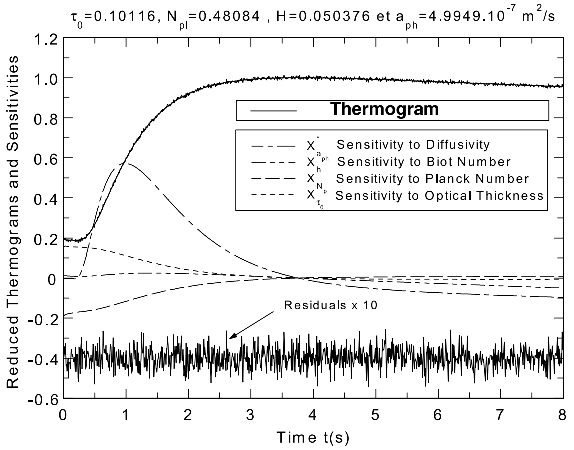

Fig. 2. Estimation on a noised thermogram and sensitivity curves.

with the other parameters. Therefore the estimation of the phononic diffusivity can be obtained with a good accuracy and the residuals between the simulated noised thermogram and the estimated reconstructed thermograms are very small.

The latter remark relative to the case presented in Fig. 2 can be made for all the cases (i.e. investigating the non-linearity of the parameter estimation problem towards the parameters has been studied in detail in [10] and [11]).

The relative error for the estimated diffusivity, calculated either through stochastic tools (covariance matrix of the linearized estimator) or through statistic tools (Monte Carlo simulations), does not exceed 2% even if some radiative properties (such as spectral absorption coefficient, internal emissivity of the opaque walls) are not known exactly.

2.4.1. Parameter estimation for a non-gray medium: test case on a silica glass

In order to test the ability of the semi-analytical model to determine—in the most general case of a non-gray medium—thermal diffusivity, estimation on a thermogram obtained with a multi-band numerical model has been performed. The absorption coefficient of a silica glass sample is given for six spectral intervals in Table 1 for a temperature of 773 K [5].

Results concerning the parameter estimation are presented in Table 2 where the nominal Planck number and the nominal optical thickness have been calculated with the mean Rosseland absorption coefficient calculated with the real six band spectrum.

The value of the estimated diffusivity is very close to the nominal diffusivity value. That proves the ability of the gray model to yield the intrinsic diffusivity of a non-gray medium in a least squares estimation using a thermogram produced by the a rear face flash experiment (here the simulated thermogram is obtained with a six bands model). This result is valid only if the radiative coefficients spectral variations are smooth (continuous variation with wavelength, case of the semi-transparent media studied here). It is interesting to note that this analysis (estimation of theoretical gray coefficients from a non-gray theoretical simulation) shows that the values obtained for the optical thickness are of the same order as those obtained with the mean Rosseland absorption coefficient.

Table 1
Silica absorption coefficients for a six bands model (non-gray approach)

Wavelength (μm)	Absorption coefficient (cm^{-1})
0.8–2.0	0.01
2.0–2.4	0.13
2.4–3.4	0.60
3.4–3.8	0.90
3.8–4.68	4.80
4.68–54.94	500

Table 2
Parameters estimation for a non-gray medium (with or without noise)

	τ_0	N_{pl}	a_{ph} (10^7)
Nominal	0.1733	1.051	5.7305
Without noise	0.22385	1.1697	5.7333
With noise	0.2237	1.1744	5.7344
$\sigma_\varepsilon^* = 0.005$	0.0017	0.0101	0.0027

This comparison between gray and non-gray behaviour *on temperature responses* has been already performed twice [8, p. 320–325]:

- using the results of Tan et al. [13] for front face non-gray simulations of a semi-transparent slab submitted to a Heat Flux Step;
- using numerical non-gray simulation of André [14] of the flash method performed on borosilicate and pure silica glass using rectangular spectral band model with up to 10 spectral intervals.

The conclusion is always the same:

- there always exists an equivalent gray coefficient that match the response of the non-gray sample. This is of course valid only because the temperature variations of the sample are confined around a mean constant temperature (unique emission spectrum). Each flash experiment at a given reference temperature T_0 is generating a very small temperature rise compared to T_0 (a few Celsius degrees).

- It is shown (but not explained) that this equivalent mean absorption coefficient is generally very close to the Rosseland one.
- It is shown that, in all cases, this parameter (the absorption coefficient deduced from the optical thickness) is necessary for the model (in order to reproduce physical effects that are clearly due to the radiation phenomenon) but it is not correlated with the thermal diffusivity, the parameter we want to measure (metrological concern).

We think that, as far as the parameter estimation problem is concerned, these three arguments justify the use of our reduced model for estimating thermal diffusivity (there is no concern in measuring the other radiative parameters).

Since this conductive–radiative transient model is able to estimate the phononic diffusivity of semi-transparent materials, transient temperature measurements have been performed in order to determine the phononic diffusivity of semi-transparent samples.

3. Experiments

The aim of the experiments is to measure the rear face transient temperature of the sample when its front face absorbs a heat pulse. The phononic diffusivity is estimated, from the thermogram, in a second stage.

The experimental set-up and the semi-transparent samples are described in this section. Results in term of transient temperature measurements and phononic diffusivity estimation are then presented. To conclude, a synoptic table is proposed in order to summarize the advantage of our method compared with the classic gold boundaries method [15].

3.1. Experimental set-up

The apparatus built in order to measure the phononic diffusivity from 300 to 700 K is based on two basic principles: excitation with flash lamps and temperature measurement through an infrared detector). The use of an infrared detector to measure the temperature is really interesting when compared against other techniques such as thermocouples (Bi_2Te_3 and $FeSi_2$): it permits to avoid the error associated with the intrusive nature of a contact measurement (fin-effect, contact resistance...). It also limits the two-dimensional effects (non-uniform rear face temperature of the sample).

The apparatus, presented in Figs. 3 and 4, is composed of

- a mobile system of three flash lamps for the excitation;
- an infrared HgCdTe detector for signal detection;
- an amplifier conditioner for the amplification and the filtering of the signal;
- a numerical oscilloscope for data acquisition.

The sample is set in a measurement cell located inside a double wall quartz bell. This bell fulfills the following functions. The inner chamber can be evacuated in order to allow measurements under semi-vacuum condition (5 Pa). Hot air is flowing through the space between the inside walls in order to maintain the base initial temperature level of the sample (from 300 to 700 K). The quartz walls are transparent to the flash excitation in the visible region.

Radiative shields limit the radiative heat losses.

3.2. Samples

The samples chosen for these experiments are absorbing and emitting materials (no scattering): float glass, pure silica, zinc selenide. Their transmission ranges, their optical and thermal properties are different.

Float glass, that has been previously studied [12,15], presents a low thermal conductivity ($\lambda = 1\ \mathrm{W\,m^{-1}\,K^{-1}}$) and a transparency zone in the visible and in near infrared up to 2.5 μm.

Silica is a material without impurities. Its conductivity is close to the conductivity of glass.

Zinc selenide, usually used for optical lenses, has been chosen for its wide transmission range.

The sample must be thick enough to avoid problems due to the shape and duration of the excitation, to the influence of the coatings and to non-linearity, but it must be thin enough to have a significant rear face temperature rise.

By using samples with two different thicknesses (silica and zinc selenium) we are able to prove that the value of the measured diffusivity is really intrinsic (it must not depend on the sample thickness).

For the experiments, the sample is either coated with gold (highly reflecting) or sprayed with a black paint (highly absorbing and emitting). If the boundaries are highly reflecting and the sample optical thickness small the internal radiative effects remain low. Black boundaries (without gold coating) emphasize the radiative effects.

It is important to point out here that, even in the case of gold boundaries, the three faces of the cylindrical samples are coated with a thin layer of black paint in order to increase the energy absorption of the sample to provide a significant rear face response (large enough absorption of the visible light emitted by the flash lamp).

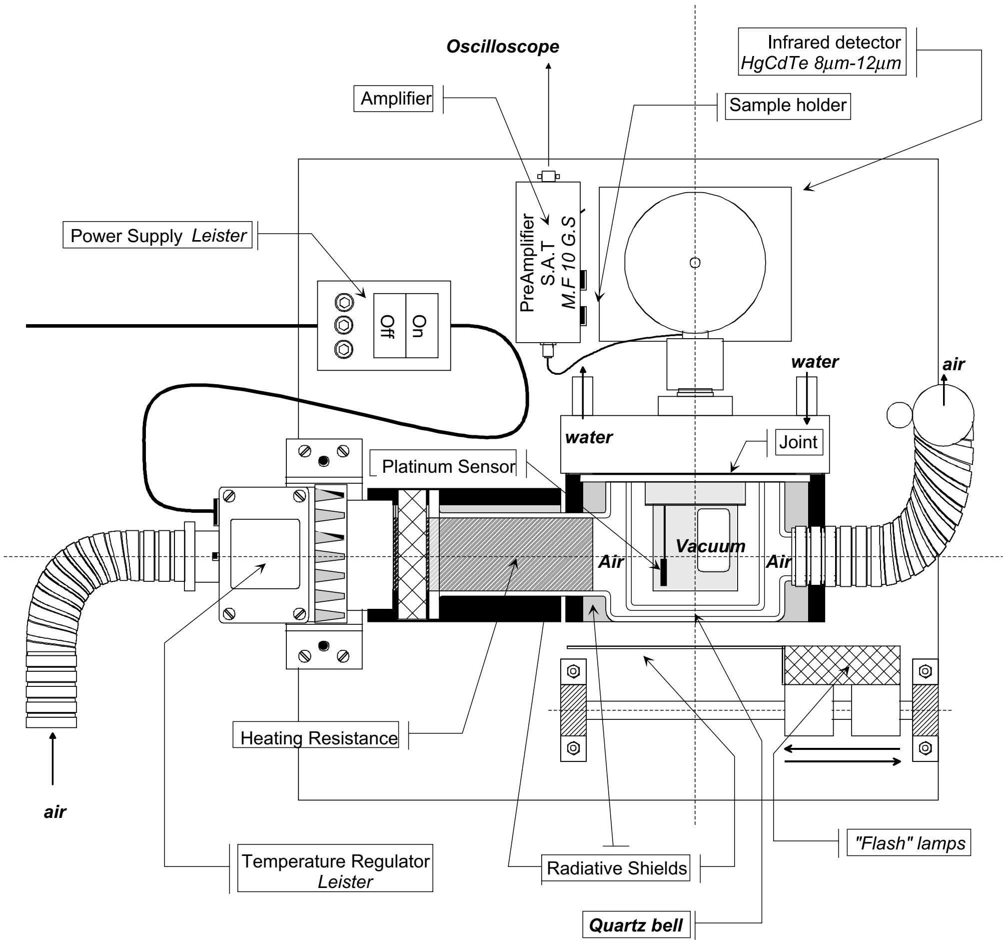

Fig. 3. Experimental apparatus.

3.3. Measurement of the transient rear-face temperature: experimental thermograms

The temperature evolution of the rear face is presented for the most significant cases (Figs. 5 and 6): sample of silica or zinc selenium, 4 mm thick and black boundaries, that is to say when the radiative transfer is emphasized. First, we put the stress on the fact that the signal over ratio is very good without any filtering of the signal (wide band pass filter larger than 100 kHz).

If attention is focused on the very short times, especially for the zinc selenium sample (see Fig. 6), we can notice that there is a temperature jump. This temperature jump increases with the nominal temperature. This phenomenon could be explained by the effect of radiative heat transfer (in the case of purely conductive transfer, this temperature jump does not exist). In this case, a purely conductive model will not be able to estimate the phononic diffusivity: it is necessary to take the radiative effects into account thanks to the coupled conductive–radiative model.

3.4. Parameter estimation procedure

From the rear face transient temperature measurements, different models are used to estimate the diffusivity:

- the conductive model which involves two parameters: the diffusivity and the Biot number;
- the coupled conductive–radiative model which involves four parameters: the optical thickness, the Planck number, the phononic diffusivity and the Biot number.

It is interesting to study the experimental residuals, which are the difference between the experimental thermogram and the thermogram calculated with the estimated parameters, are scrutinized.

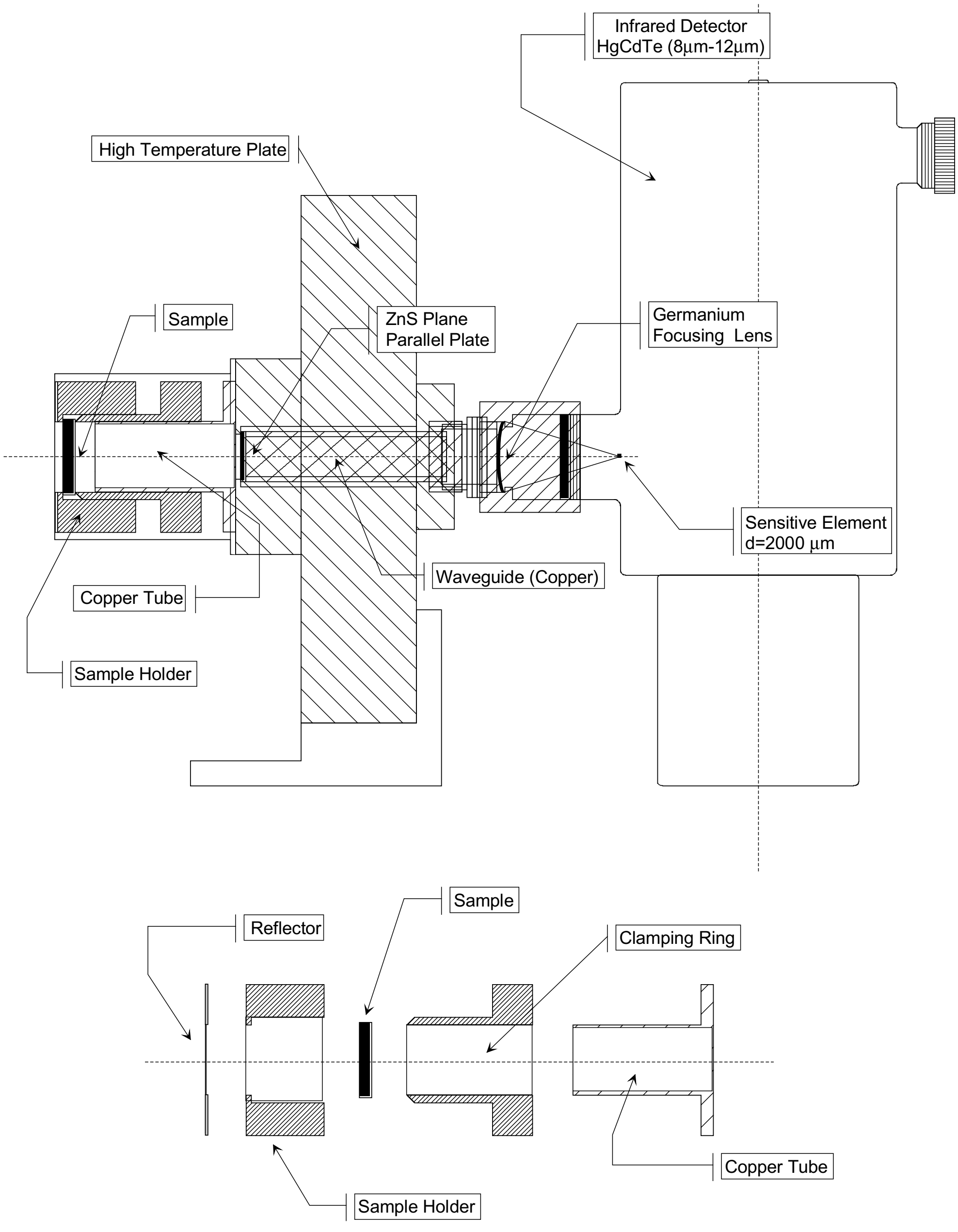

Fig. 4. Experimental apparatus (zoom).

Fig. 7 presents the residuals obtained for the selenium zinc thick (4 mm) sample in the case of black boundaries. The experimental residuals are very bad for the conductive model, especially for very short times: this confirms the previous simulations shown in Section 2.4 (the conductive model is unable to reproduce the temperature jump at early times). The residuals are very low for the coupled conductive–radiative model: this shows that this model seems to be appropriate to yield precise estimations of the phononic diffusivity.

3.5. Experimental estimation of the diffusivity

We present now experimental estimations of the phononic diffusivity for three materials and two thicknesses (except for float glass where only the results for a 2 mm sample are presented) and different temperature levels. Both models—purely conductive and coupled radiative–conductive—have been used for these estimations. Estimations have been made for gold-coated or black painted samples. Each experimental point

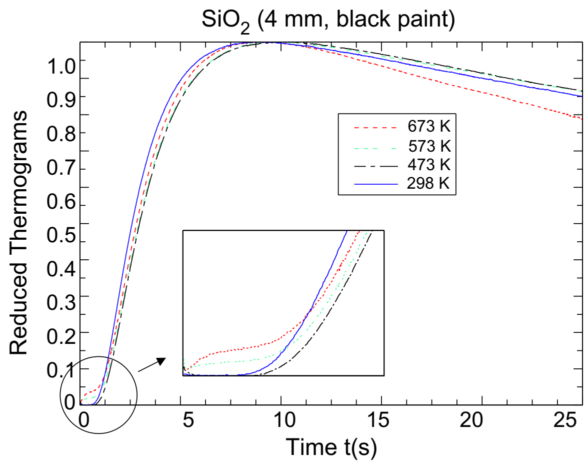

Fig. 5. Experimental thermograms for SiO_2 sample (4 mm, black paint).

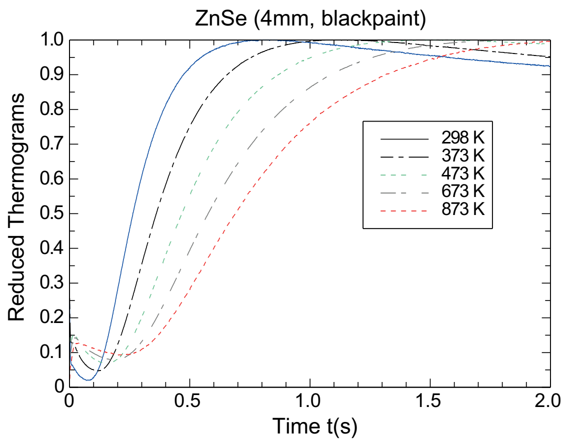

Fig. 6. Experimental thermograms for ZnSe sample (4 mm, black paint).

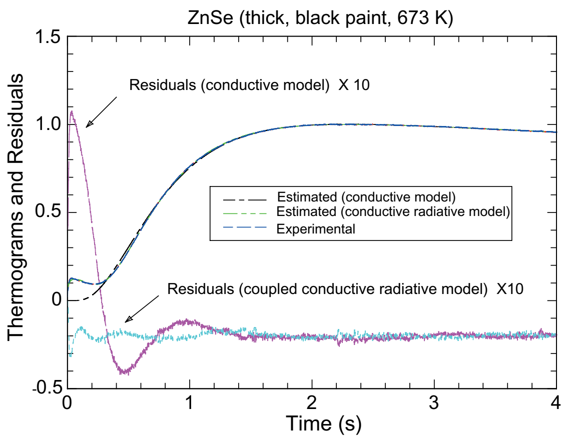

Fig. 7. Experimental and estimated thermograms and residuals.

presented in the following estimated diffusivity versus temperature curves (Figs. 8–12) corresponds to an average of at least four estimations for the same sample. Experimental estimations obtained previously by André [15] for the same golden-coated samples using the purely conductive model for inversion are also shown on these figures where each point corresponds to only one experiment here.

3.5.1. Float glass

Fig. 8 presents the thermal diffusivity of float glass versus temperature. This variation of the diffusivity presents a classical shape: it first decreases with temperature, presents then a minimum for a temperature range around 600 K and finally increases. These results are the same as those found with another experimental set-up (temperature measurement made by thermocouple $FeSi_2$) for gold-coated sample and estimation made with a purely conductive model [15]. For this range of temperature, there is no significant radiative effect for the rear face thermogram; that is why we can not notice any discrepancy between the diffusivity estimated with the conductive or the combined model from experiments on gold-coated or black painted samples.

3.5.2. Silica

In the case of silica samples (see Figs. 9 and 10) an interesting phenomenon begins to appear for the diffusivity at high temperatures. One can notice in Fig. 10 a small difference between the diffusivity estimated in the case of black boundaries with the conductive and the conductive–radiative model. Since the radiative effects

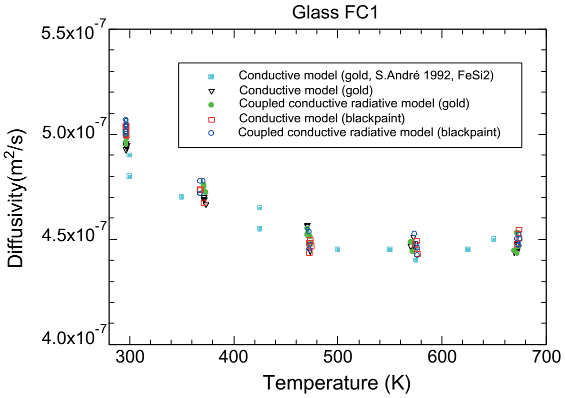

Fig. 8. Diffusivity of the float glass FC1.

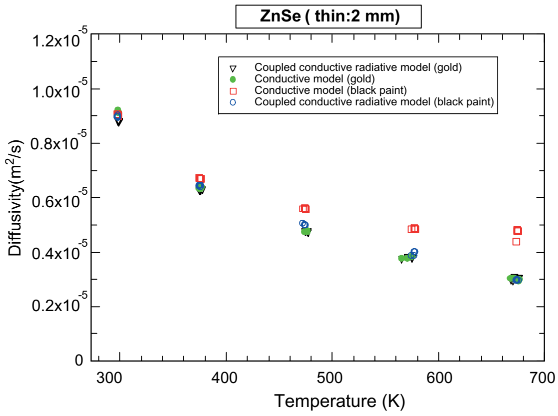

Fig. 11. Diffusivity of ZnSe (2 mm thick sample).

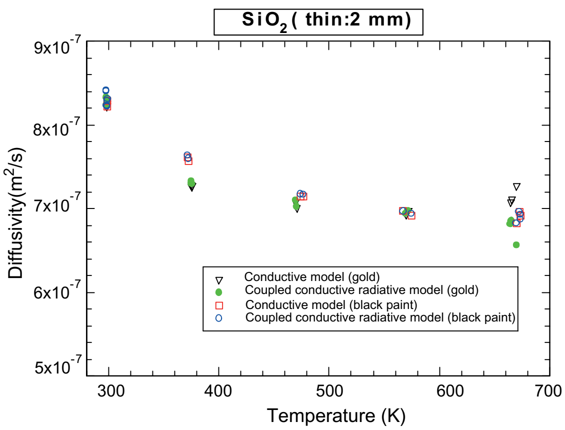

Fig. 9. Diffusivity of SiO_2 (2 mm thick sample).

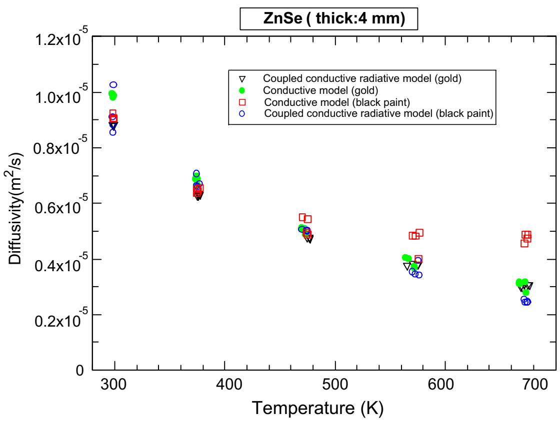

Fig. 12. Diffusivity of ZnSe (4 mm thick sample).

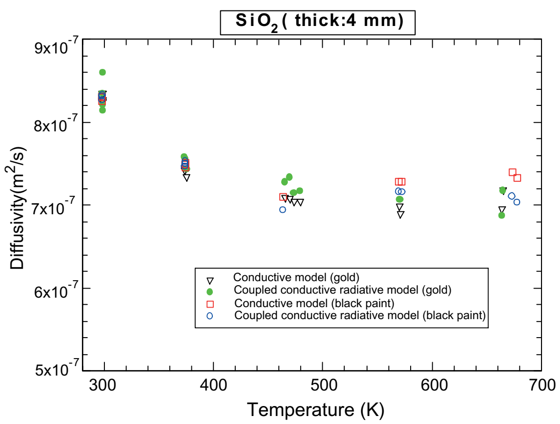

Fig. 10. Diffusivity of SiO_2 (4 mm thick sample).

becomes significant (they can be noticed on the thermogram plotted in Fig. 5), the conductive model is unable to estimate the diffusivity properly for high temperatures: it overestimates its value. This effect is less important for the thin sample results shown in Fig. 9.

Comparisons of the results presented for the two thicknesses (Figs. 9 and 10) show that the values of the diffusivity estimated through the use of the coupled radiative–conductive model are nearly identical (with a maximum difference of 3% for higher temperatures). This thickness independence of the estimated diffusivity shows that our estimation technique yields the true phononic diffusivity that must be an intrinsic parameter.

3.5.3. Zinc selenium

In the case of the zinc selenium samples–see the estimated diffusivities shown in Figs. 11 and 12 for the two thicknesses–the radiative effects are more important than those observed for the silica samples (see the very significant temperature jump in Fig. 6): there is no doubt that the use of the conductive model on the black painted samples yields an overestimation of its diffusivity. In this

Table 3
Comparison between the gold boundaries method and the black boundaries method

	Gold boundaries method	Black boundaries method
Modeling	Model with two parameters	Model with four parameters
Estimation	True ? thermal diffusivity	True thermal diffusivity
Experiments	Gold	Black paint
Cost	150$/sample	5$/spray cartridge
Time	3 days–4 weeks	1/2 day
Easy	No	Yes

case the estimated value does not correspond to the phononic diffusivity but to an apparent diffusivity including the effects of radiative transfer.

Comparison of both thickness results (gold-coated samples with inversion by both models or black painted samples with inversion by the coupled model) shows that the estimated diffusivity (provided by the same model) does not differ by more than 3% or 4% for the two different thicknesses. This leads to the same conclusion as for the silica sample: the intrinsic phononic diffusivity seems to be reached by the coupled model.

3.6. Comparison between the classic gold boundaries and the black boundaries methods

The black boundaries method which consists in spraying black paint on the sample and in estimating the thermal diffusivity value with the combined conductive radiative model presents several advantages. It provides the thermal diffusivity value with a good accuracy (<5%). It also avoids gold coatings. Moreover, it is quite impossible to know, in the case of gold boundaries method, if the radiative effects could be neglected. As a result, the diffusivity is overestimated and is not the real diffusivity but an apparent one.

The comparison, between the classic gold boundaries method and the black boundaries method, is summarized in Table 3 in terms of modeling, estimation and experimental point of view.

4. Conclusion

In this paper, a complete methodology for determining the phononic diffusivity of semi-transparent materials is proposed:

- A semi-analytical model permits to obtain the thermal response of the media in the case of transient combined conductive–radiative heat transfer and a theoretical study demonstrates the ability of the model to estimate the phononic diffusivity of semi-transparent media in an accurate way.
- A new experimental set-up is developed in order to measure, with an infrared detector, the evolution of the rear-face temperature of semi-transparent samples such as glass, silica and zinc selenium.

The quality of the thermograms (excellent signal/noise ratio) stems from the experimental equipment that has been used (radiative excitation and detection). The measurements, made by an optical way, allows to avoid all the difficulties encountered with the classical apparatus using contact thermocouples for temperature measurements (thermocouple intrusive effects caused by a high temperature difference between its hot and cold junctions, electrical contact with the sample that must be covered with a metal paint, offset problem for the amplifier caused by the temperature gradient).

The black boundaries method proposed here could supplant the classical gold boundaries method (less expensive, quicker and more precise).

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