Parametric estimation of thermo-radiative properties of materials based on harmonic excitation.

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Abstract

A sample device for the measurement of emissivity, thermal conductivity and diffusivity of opaque materials is described. The measurement method is based on the use of a harmonic excitation of a material sample plate. The front face of the sample is heated periodically and its temperature is measured using a thermocouple. The rear face temperature is obtained by infrared thermography. Half the surface is covered by a black paint of known emissivity used as a reference to compute the sample surface directional emissivity. Thermal conductivity and diffusivity are simultaneously identified from both real and imaginary parts of the heat transfer function. Results are presented for a thermally thick sample of PVC. The identified thermophysical properties are in agreement with literature value and reproducible results are obtained. Moreover, some limitations of the method are considered. First, the black-paint layer influence on the heat transfer cannot be neglected in all the frequency range of measurement. Secondly, an accurate estimation of the thermal conductivity cannot be obtained without an exact knowledge of the heat transfer coefficient.

Key Words: emissivity; radiative properties; thermal conductivity; thermal diffusivity; infrared thermography.

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1. INTRODUCTION

Thermal conductivity, thermal diffusivity and emissivity are all essential thermophysical properties of materials. The importance of these properties for development of materials ranges from determining safe operating temperatures, to predicting the rate of heat dissipation from many components [1]. Therefore, the development of new experimental methods and the improvement of existing methods to simultaneously measure these thermo-radiative properties is still necessary.

We can find in the literature various conductivity measurement methods. The steady state methods, of which the best known is the hot-plate method, allow direct access to conductivity but generally involve a strong temperature gradient inside the sample; the parameter obtained is an average conductivity on the corresponding interval of temperature. In addition, measurements take a long time [2]. The transient state methods give access to diffusivity using the heat capacity value.

Moreover, numerous techniques allowing thermal properties measurements have been developed [3]. The transient plane source (TPS) technique is well known for the measurement of thermal parameters. Photoacoustic and photopyroelectric techniques are two measurement methods using a laser source as a periodic heating device and allow simultaneous estimation of the thermophysical parameters [4].

Several methods allowing emissivity measurements have been developed. This parameter can be directly measured using its definition: ratio of the sample emitted power to the blackbody one. Indirect emissivity measurement using the relationship between reflectivity and emissivity, based on Kirchhoff’s first law, has found many applications also. Particularly, M. Siroux et al (1998) [5] developed a periodic radiometry technique. It is shown that thermal modulation of a sample allows direct and indirect emissivity measurements even at ambient temperature, with ordinary equipment.

In the present work, an improved periodic radiometry technique allowing measurements of the directional emissivity, thermal diffusivity and thermal conductivity of opaque materials, poly(vinylchloride) (PVC) at ambient temperature is described. The method is based on the use of an harmonic thermal excitation and an infrared thermography measurement. This method was initiated a few years ago by S. Matteï [6] in our laboratory. The aim of this paper is to give a complete description of the updated experimental device, along with the thermal model, numerical treatments and to test the performances of the overall procedure. The advantage of this kind of method is to allow a simultaneous thermo-radiative characterization.

2. THE EXPERIMENTAL SET-UP

2.1. Principle of the periodical radiometric method proposed

The experimental configuration used is improved relatively to that developed by S. Matteï et al [6]. In fact, because of the complexity of the previous device, we propose a simplified technique for emissivity measurement (Fig. 1). In this configuration, the experimental set-up is composed of four parts:
- Sample-holder
- Heating source (thermoelectric device)
- Detection system (infrared camera)
- Control system
2.2. Sample-holder

As shown in Fig. 2, the sample is fixed on a Duralumin plate. A thermal grease of high conductivity is applied on the contact surfaces between the sample and the metallic plate to ensure good thermal exchanges between various elements. The front side of the Duralumin plate is fixed on a thermoelectric device and the heat sink. Half the area of this face is coated with black paint of 0.97 emissivity [5].

![Diagram](image)

**Fig. 1.** Schematic view of the experimental set-up

**Fig. 2.** Sample-holder

2.3. Modulated heating source

The front side of the Duralumin plate is periodically heated. The temperature of the source is modulated around room temperature and is measured with a K-type thermocouple, which is inserted at 2 cm depth in the Duralumin plate. The multi-harmonic excitation, which is a sum...
of sinusoids of various frequencies, is obtained using a thermoelectric device. A power amplifier provides the power applied to this thermoelectric device.

2.4. Detection system

The infrared detector facing the rear face of the sample is an AGEMA infrared camera (Thermovision 870). Its specifications are given in table I.

<table>
<thead>
<tr>
<th>Spectral response</th>
<th>Temperature range</th>
<th>Accuracy</th>
<th>Thermal Sensitivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 to 5 µm</td>
<td>-20 to 500 °C</td>
<td>±2°C</td>
<td>0.1°C at 30 °C</td>
</tr>
</tbody>
</table>

The infrared camera converts electromagnetic thermal energy radiated from an object into an electronic video signal. This signal is amplified and transmitted via an interconnecting cable to a display monitor. The electromagnetic energy radiating from the object being scanned is focused by an infrared lens into a mirror that is oscillated by a motor. The oscillating mirror and motor are synchronized in such a way that four fields produce an interlaced frame each having 100 horizontal scanning lines. 70 of these lines are used as active imaging lines per field, or 280 per frame. By using National Instruments DAQ Board, model PCI-6024E, the maximum sampling frequency can be fixed to 1.25 MHz, which allows a frame acquisition rate of about 2Hz on a PII 350MHz PC. Each sample is converted in a 12-bit numerical data using the digital-analog converter of the DAQ Board.

2.5. Control system

National Instruments DAQ Board, model PCI-6034E is used to control the heating power provided to the thermoelectric element and to perform the acquisition of thermocouple temperature. The K-type thermocouple is plugged into a conditioning module (model 5B37 from Analog Device) allowing to amplify the thermocouple signal and to perform the cold-junction compensation. Temperature data are acquired at a rate of 1Hz. Each temperature data is the average of 25 samples acquired at a rate of 1 kHz, in order to increase the signal-to-noise ratio.

One of the analog outputs of the DAQ Board is used to control the voltage supplied by the power amplifier. These two devices are connected through a conditioning module (model 5B49 from Analog Devices). The whole system is controlled using an application developed with the LabView environment.

3. EMISSIVITY MEASUREMENTS

3.1. Experimental protocol

In the configuration presented on Fig. 1, for obtaining thermal conductivity and diffusivity of materials we need temperature values on the rear face and on the front face of the sample. As it was shown in paragraphs 2.3 and 2.4, the temperature of the front-side face is measured with a K-type thermocouple and the infrared camera is used to obtain the temperature of the rear face. An infrared camera measures the radiosity of the target body surface. A temperature can be computed from this radiosity using the camera calibration curve; however, this temperature is not the real temperature of the body surface, but a blackbody equivalent one, which means the temperature of an ideal blackbody generating the same radiation energy as
the real body. In order to estimate the real temperature of the body surface two informations are needed, the surface emissivity and the ambient irradiation of the body.

The ambient irradiation can be considered as a static perturbation. Thus, when using a periodical method, its influence on experimental measurements can be neglected. On the contrary, the surface emissivity value must be known to compute the surface temperature. One classical procedure is to cover partially the target surface with a coating of known emissivity and to obtain an estimation of the uncovered surface by means of the ratio between the measured radiosities.

The measured radiative flux is the sum of an emitted flux and a reflected one. The reflected flux is not modulated, thus it can be separated from the emitted one. If \( K \) is the transfer function of the infrared detection system (including geometrical parameters), the infrared output signal component at \( f \) is given by:

\[
\left| \tilde{U}_s (\theta, f) \right| = K \varepsilon_s (\theta) \sigma \tilde{T}_s^4 (f) \quad (1)
\]

\[
\left| \tilde{U}_{\text{ref}} (\theta = 0, f) \right| = K \varepsilon_{\text{ref}} (\theta = 0) \sigma \tilde{T}_{\text{ref}}^4 (f) \quad (2)
\]

where \( \varepsilon_s (\theta) \) is the directional emissivity of the sample at the emission angle \( \theta \), \( \varepsilon_{\text{ref}} (\theta = 0) \) is the normal directional emissivity of the reference black paint, \( \sigma \) is the Stefan-Boltzmann constant. \( \tilde{U}_s \), \( \tilde{U}_{\text{ref}} \) are, respectively, the sample and the reference voltage amplitude, measured by the IR camera detector.

If temperature amplitudes are small, we can express the two voltage amplitudes, \( \tilde{U}_s \), \( \tilde{U}_{\text{ref}} \), using the corresponding temperature measurements, given by the camera piloting software:

\[
\tilde{U}_i (f) = C \cdot 4 \tilde{T}_{i, \text{camera}}^3 (f) \Delta T_{i, \text{camera}} (f) \quad (3)
\]

with \( C \) a proportionality constant that does not depend on frequency \( f \), \( i = \{s, \text{ref}\} \), \( \Delta T_i = \left| \tilde{T}_i (f) \right| \) is the temperature amplitude and \( \bar{T} \) is the mean temperature.

The directional emissivity is given by:

\[
\hat{\varepsilon}_s = \varepsilon_{\text{ref}} \cdot \frac{\tilde{T}_{s, \text{camera}}^3 \Delta T_{s, \text{camera}}}{\tilde{T}_{\text{ref, camera}}^3 \Delta T_{\text{ref, camera}}} \quad (4)
\]

The uncertainty on the emissivity estimation is computed from a first order Taylor expansion.

\[
\frac{u^2 (\varepsilon_s)}{\varepsilon_s^2} = \frac{u^2 (\varepsilon_{\text{ref}})}{\varepsilon_{\text{ref}}^2} + A_s + A_{\text{ref}} \quad (5)
\]

with:

\[
A_i = \frac{u^2 (\Delta \bar{T}_{i, \text{camera}})}{(\Delta \bar{T}_{i, \text{camera}})^2} + 9 \frac{u^2 (\bar{T}_{i, \text{camera}})}{(\bar{T}_{i, \text{camera}})^2} \quad (6)
\]

with \( i = \{S, \text{ref}\} \).
The approximate statistical confidence bounds for the estimated value of emissivity with 95.5\% confidence level is given by:

\[ e_s = \hat{e} \pm 2 \times u(e) \]  

(7)

3.2. Results

Measurements have been performed using a PVC sample (44×44×5.2 mm³). Half the surface of the sample is coated with black paint of 0.97 emissivity [7]. Different modulation frequencies ranging from 0.5 mHz to 12.5 mHz were used. More details concerning the excitation signals used will be given section 4. The emissivity values and their corresponding uncertainties were computed using relation (4) to (7).

Fig. 3 represents the evolution of the total directional emissivity of the PVC computed at each excitation frequency.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{Fig3.png}
\caption{PVC normal directional total emissivity values obtained from several measurements}
\end{figure}

At low frequency, we notice a good agreement between the experimental values and those found in the literature [6] (\(\varepsilon = 0.98\)). We also notice an over estimation of the emissivity which monotonically increases with the excitation frequency. This can be explained by the presence of the black paint layer as developed in section 3.3.

3.3. Influence of the black paint layer

In order to study the influence of the black paint layer, a numerical simulation of the experimental set-up thermal response has been performed. For that, we computed the heat transfer function of the experimental set-up using two configurations. The quadrupole theory was used to obtain these heat transfer functions [8].

In the first configuration we consider only the duraluminum plate, the grease layer, and the sample plate. This allows to compute the heat transfer function denoted \( \tilde{H}_{\text{PVC}} \). The geometrical and thermophysical characteristics of all layers are given in table II. In the second configuration, a fourth layer associated to the coating layer is added. As the thermal
conductivity and diffusivity of the coating are unknown, we used the properties of PVC. Indeed, the black paint can be considered as a polymeric material, so its properties are probably close to the PVC ones. This second configuration allowed to compute a heat transfer function denoted as \( \tilde{H}_{\text{black paint}} \).

In Fig. 4 we plotted the ratio between the modulus of \( \tilde{H}_{\text{PVC}} \) and \( \tilde{H}_{\text{black paint}} \) heat transfer functions in the frequency domain of measurement.

![Fig. 4. Ratio of sample and black paint transfer function amplitude](image)

We considered two black paint layer thicknesses (100 µm and 200 µm). In the low frequency domain \( (f \leq 10^{-3} \text{ Hz}) \), the ratio between the two heat transfer function modulus is close to one. In this domain, the influence of the coating on heat transfer can be neglected. So, we can state that the temperature amplitudes on the PVC and the black paint surfaces are almost the same.

On the contrary, in the high frequency domain \( (f > 10^{-3} \text{ Hz}) \), the ratio between the two heat transfer functions modulus increases significantly for both black paint thicknesses. This indicates that the temperature amplitudes on the PVC and the black paint surfaces cannot be considered as equal in this frequency range. Moreover, this explains that emissivity values greater than one are obtained in the high frequency range. Thus, the PVC surface emissivity can be computed only at the lowest frequency. The obtained value of the PVC normal emissivity is 0.98 ± 0.03.

Consequently, this value was used to compute the temperature amplitudes on the PVC surface at each frequency.

### 4. THERMAL CONDUCTIVITY AND DIFFUSIVITY MEASUREMENTS

#### 4.1. Thermal heat transfer model

In periodic mode, the front-side temperature \( T_{FS} \) measured by the thermocouple inserted into the Duralumin plate is modulated (Fig. 5):

\[
T_{FS}(t) = \bar{T} + \sum_{i=1}^{N} \Delta T_i \sin(2\pi f_0 (2^{i-1})t)
\]  

(8)
where $t$ is time, $\Delta T_i$ is temperature amplitude, $f_0$ is the fundamental modulation frequency, and $N$ is the number of excitation frequencies ($N = 5$).

We compute the theoretical thermal transfer function by the relationship [8]:

$$
\tilde{H}(f) = \frac{\tilde{T}_{BS}(f)}{\tilde{T}_{FS}(f)}
$$

where $\tilde{T}_{BS}(f)$ and $\tilde{T}_{FS}(f)$ are the complex temperatures of, respectively, the rear side and the front side of the sample (Fig. 5). $\tilde{H}$ is a complex number.

Fig. 5. Schematic view of the experimental thermal exchange model.

The relationship between the front-side temperature/flux and the rear one in the experimental set-up (Fig. 5) is given by [8]:

$$
\begin{bmatrix}
\tilde{T}_{FS}(f) \\
\tilde{\phi}_{FS}(f)
\end{bmatrix} = [Q_1] \begin{bmatrix}
\tilde{T}_0(f) \\
\tilde{\phi}_0(f)
\end{bmatrix}
$$

$$
\begin{bmatrix}
\tilde{T}_{BS}(f) \\
\tilde{\phi}_{BS}(f)
\end{bmatrix} = [Q_2] \begin{bmatrix}
\tilde{T}_0(f) \\
\tilde{\phi}_0(f)
\end{bmatrix}
$$

where $\tilde{\phi}_{FS}(f)$ and $\tilde{\phi}_{BS}(f)$ are, respectively, the Fourier transforms of front side flux and rear side one; $\tilde{T}_0(f)$ and $\tilde{\phi}_0(f)$ are, respectively, the Fourier transforms of air temperature and flux on the rear face ($\tilde{T}_0(f) = 0$); and $[Q_1]$, $[Q_2]$ are complex matrices defined by:

$$
[Q_1] = [Q_D] \cdot [Q_{Gr}] \cdot [Q_S] \cdot [Q_h]
$$

$$
[Q_2] = [Q_h] = \begin{bmatrix}
1 \\
0 \\
1/h \\
1
\end{bmatrix}
$$

The indices D and S correspond, respectively, to the half of the front plate (made of duralumin) and of the sample.

$$
[Q_1] = \begin{bmatrix}
\cosh(q_i x_i) & \sinh(q_i x_i) \\
k_i q_i & k_i q_i \\
k_i q_i \sinh(q_i x_i) & \cosh(q_i x_i)
\end{bmatrix}
$$
with \( i = \{D,S\} \)

\( k \) is the thermal conductivity, \( x \) is the material thickness and \( q \) is given by:

\[
q_i = \sqrt{\frac{2j\pi f}{a_i}}
\]

(14)  

where \( a \) is the thermal diffusivity, \( f \) the frequency and \( j^2 = -1 \).

\( [Q_{Gr}] \) is the quadrupole associated to the grease layer, which is supposed to have no inertia.

\[
[Q_{Gr}] = \begin{bmatrix} 1 & R_{Gr} \\ 0 & 1 \end{bmatrix}
\]

(15)  

where \( R_{Gr} \) is the thermal resistance and is estimated to be \( R_{Gr} = 118 \times 10^{-6} \text{ m}^2\text{KW}^{-1} \) [9].

\( [Q_b] \) is the quadrupole associated to the heat transfer coefficient \( h \) (which is fixed to \( 8 \text{ Wm}^{-2} \text{ K}^{-1} \)) between the air and the rear side sample.

The theoretical heat transfer function is then obtained using equations (9) to (15):

\[
\tilde{H}(s) = \frac{1}{A_1 \cosh(st_s) + A_2 \sinh(st_s)}
\]

(16)  

where

\[
t_i = \frac{x_i}{\sqrt{a_i}}
\]

\[
s = \sqrt{2\pi f}
\]

\[
b_i = \frac{k_i}{\sqrt{a_i}}
\]

\[
A_1 = (1 + hR_{Gr}) \cosh(st_d) + \frac{h}{s b_D} \sinh(st_d)
\]

(18)  

\[
A_2 = \left(s b_S R_{Gr} + \frac{h}{s b_S}\right) \cosh(st_d) + \left(\frac{b_S}{b_D}\right) \sinh(st_d)
\]

(19)  

The complex experimental transfer function is calculated using equation (20) [10].

\[
H(f) = \frac{\text{FFT}(T_{BS}(t)) \cdot \text{FFT}(T_{FS}(t))^*}{\text{FFT}(T_{FS}(t))^2}
\]

(20)  

\( \text{FFT}(T_{FS}(t))^* \) is the complex conjugate of \( \text{FFT}(T_{FS}(t)) \).

The experimental transfer function amplitude and phase are given by, respectively, the \( H \) modulus and argument at each excitation frequency. The empirical criterion used for the sample study is the application of several frequencies to obtain information on the experimental transfer function phase between -\( \pi \) and 0.
4.2. Conductivity and diffusivity identification

In this study, a parameter estimation technique was applied to estimate both thermal conductivity and diffusivity. The identification procedure consists in finding the set of parameters $B$ that minimises the following criterion:

$$S(\hat{B}_{k,u}) = \sum_{i=1}^{N} \left[ (H_{\text{real}}(f_i) - \tilde{H}_{\text{real}}(f_i))^2 + (H_{\text{imag}}(f_i) - \tilde{H}_{\text{imag}}(f_i))^2 \right]$$  \hspace{1cm} (21)$$

where $H_{\text{real}}(f_i)$ and $\tilde{H}_{\text{real}}(f_i)$ are, respectively, the real parts of the experimental and theoretical transfer function, $H_{\text{imag}}(f_i)$ and $\tilde{H}_{\text{imag}}(f_i)$ are, respectively, the imaginary parts of the experimental and theoretical transfer function. $\hat{B}$ is the vector of two estimated parameters ($b_S$ and $t_S$) and $N$ is the number of experimental data. The variance-covariance matrix of the estimated parameters vector $\hat{B}$ can be approximated as:

$$\text{cov}(\hat{B}) = \sigma^2 (m.m^T)^{-1}$$  \hspace{1cm} (22)$$

where $m = \left( \frac{\partial \tilde{H}}{\partial \hat{B}} \right)$ represents the Jacobian matrix and $m^T$ the Jacobian transpose.

$\sigma$ represents the standard deviation of the measurements and is estimated by:

$$\sigma^2 = \frac{S(\hat{B})}{N - q}$$  \hspace{1cm} (23)$$

where $N$ is the number of experimental data and $q$ is the number of identified parameters. The variance-covariance matrix can be written as:

$$\text{cov}(\hat{B}) = \begin{bmatrix} \sigma_{b_S}^2 & \sigma_{b_S, t_S} \\ \sigma_{t_S, b_S} & \sigma_{t_S}^2 \end{bmatrix}$$  \hspace{1cm} (24)$$

Then the thermal conductivity and diffusivity of the sample are obtained from:

$$\begin{cases} \hat{k} = \frac{x_S \hat{b}_S}{\hat{t}_S} \\ \hat{\alpha} = \left( \frac{x_S}{\hat{t}_S} \right)^2 \end{cases}$$  \hspace{1cm} (25)$$

where $x_S$ is the sample thickness.

The corresponding uncertainties are obtained from:

$$\begin{cases} u(\hat{k}) = \hat{k} \sqrt{\frac{u^2(x_S)}{x_S^2} + \frac{\sigma_{b_S}^2}{\hat{b}_S^2} + \frac{\sigma_{t_S}^2}{\hat{t}_S^2}} \\ u(\hat{\alpha}) = 2\hat{\alpha} \sqrt{\frac{u^2(x_S)}{x_S^2} + \frac{\sigma_{t_S}^2}{\hat{t}_S^2}} \end{cases}$$  \hspace{1cm} (26)$$
where \( u(x_s) \) is the uncertainty of measurement of the sample thickness and is assumed to be equal to \( \frac{50 \times 10^{-3}}{\sqrt{3}} \) mm.

The approximate statistical confidence bounds for the estimated thermal conductivity and diffusivity are:

\[
\begin{align*}
\hat{k} &= k \pm 2 \times u(\hat{k}) \\
\hat{a} &= a \pm 2 \times u(\hat{a})
\end{align*}
\] (27)

at a confidence level of 95%.

4.3. Thermal properties of PVC

In the configuration shown on Fig. 5, the measurements on a PVC sample were achieved for a large frequency range \( 0.5 \text{ mHz} \leq f \leq 12.5 \text{ mHz} \). The choice of the excitation frequencies is semi-empirical, based on the knowledge of approximate values of the thermal properties for the studied sample. The properties of all materials used in the computations are presented in table II.

**Table II.** Values of the thermal parameters used in the computation.

| Layers                              | Thicknesses     | Parameters (at 20°C)      | Values             |
|-------------------------------------|-----------------|---------------------------|
| Duralumin plate [11]                | 5 mm            | Thermal conductivity      | 164 \( \text{Wm}^{-1}\text{K}^{-1} \) |
|                                     |                 | Thermal diffusivity       | \( 6.66 \times 10^{-5} \) \( \text{m}^2\text{s}^{-1} \) |
|                                     |                 | Density                   | 2787 \( \text{kg.m}^{-3} \) |
|                                     |                 | Specific heat             | 883 \( \text{Jkg}^{-1}\text{K}^{-1} \) |
| PVC [2,6]                           | 5.2 mm          | Thermal conductivity      | 0.167 \( \text{Wm}^{-1}\text{K}^{-1} \) |
|                                     |                 | Thermal diffusivity       | \( 1.18 \times 10^{-7} \) \( \text{m}^2\text{s}^{-1} \) |
| Black paint                         | 200 µm or 100 µm| Thermal conductivity      | 0.167 \( \text{Wm}^{-1}\text{K}^{-1} \) |
|                                     |                 | Thermal diffusivity       | \( 1.18 \times 10^{-7} \) \( \text{m}^2\text{s}^{-1} \) |
| Thermal grease [9]                  | -               | Contact resistance        | \( 118 \times 10^{-6} \) \( \text{m}^2\text{KW}^{-1} \) |
| Thermal exchange on the rear of the sample | -              | Exchange coefficient      | 8 \( \text{Wm}^{-2}\text{K}^{-1} \) |

Fig. 6 and 7 show the amplitude spectra of the signal power for each excitation frequency on the front and rear sides of the sample.
The identification of thermal conductivity and diffusivity was performed simultaneously on the real and the imaginary parts of the two transfer functions. The modulus and the phase of the theoretical transfer function was calculated with the identified values and compared to the experimental ones. A good agreement between theoretical and experimental transfer functions is observed (Fig. 8 and 9).
The results obtained are presented in table III.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
<th>Uncertainty</th>
<th>Relative uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>(k \text{ (Wm}^{-1}\text{K}^{-1}))</td>
<td>0.167</td>
<td>0.017</td>
<td>10.18%</td>
</tr>
<tr>
<td>(a \text{ (m}^2\text{s}^{-1}))</td>
<td>(1.199 \times 10^{-7})</td>
<td>(0.038 \times 10^{-7})</td>
<td>3.17%</td>
</tr>
</tbody>
</table>

The relative accuracy is 10.18% for thermal conductivity and 3.17% for diffusivity. These uncertainties originate only from measurement noise and the subsequent identification procedure. The uncertainties in supposedly known parameters are not considered. This point will be discussed further in this paper.

The values obtained by this method are consistent with those found in the literature: \(k = 0.16 - 0.18 \text{ Wm}^{-1}\text{K}^{-1}\) and \(a = 1.1 - 1.2 \times 10^{-7} \text{ m}^2\text{s}^{-1}\) [2, 6].
4.4. Reproducibility study

Several measurements were carried out to study the reproducibility of the measurements. First, four successive experiments were performed on the same sample (measurement nos 1-4). Later, two experiments were realized using the same sample in the same experimental conditions (measurement nos 5-6). Finally, three experiments were conducted after removing, cleaning and setting up the sample again (measurement nos 7-9). The statistical dispersion of modulus and phase of the experimental transfer function, calculated from these experiments, are given in table IV.

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>$H$ modulus mean</th>
<th>$H$ modulus standard deviation</th>
<th>$H$ phase (rad)</th>
<th>$H$ phase standard deviation (rad)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$5 \times 10^{-4}$</td>
<td>0.780</td>
<td>0.008</td>
<td>-0.324</td>
<td>0.018</td>
</tr>
<tr>
<td>$1 \times 10^{-3}$</td>
<td>0.714</td>
<td>0.006</td>
<td>-0.619</td>
<td>0.031</td>
</tr>
<tr>
<td>$2 \times 10^{-3}$</td>
<td>0.555</td>
<td>0.012</td>
<td>-1.085</td>
<td>0.043</td>
</tr>
<tr>
<td>$4 \times 10^{-3}$</td>
<td>0.337</td>
<td>0.014</td>
<td>-1.683</td>
<td>0.051</td>
</tr>
<tr>
<td>$8 \times 10^{-3}$</td>
<td>0.163</td>
<td>0.009</td>
<td>-2.427</td>
<td>0.065</td>
</tr>
</tbody>
</table>

The modulus and phase transfer function standard deviations vary from one frequency to another. The maximal relative variation remains lower than 5.52% for the modulus and 5.56% for the phase. These values are admissible for the experimental reproducibility of the results. Fig. 10 and 11 show the identified values of the thermal conductivity and diffusivity with their respective identification uncertainties for all experimental measurements concerning the PVC sample.

![Fig. 10. PVC thermal conductivity values obtained from several measurements](image-url)
The mean value of the thermal conductivity and diffusivity for the several measurements are respectively, \( k = 0.175 \text{ W.m}^{-1}\text{K}^{-1} \) and \( a = 1.14 \times 10^{-7} \text{ m}^2\text{s}^{-1} \). We have calculated the experimental standard deviations of the thermal conductivity (\( \sigma(\hat{k}_1, \hat{k}_2, ..., \hat{k}_9) \)) and diffusivity (\( \sigma(\hat{a}_1, \hat{a}_2, ..., \hat{a}_9) \)) for the nine experimental measurements. These two results are compared to the mean values of the identification procedure standard deviation for thermal conductivity \( \sigma_{\hat{k}} \) and diffusivity \( \sigma_{\hat{a}} \).

The standard deviation of the several measurements of the thermal conductivity values \( \sigma(\hat{k}_1, \hat{k}_2, ..., \hat{k}_9) = 1.13 \times 10^{-2} \text{ Wm}^{-1}\text{K}^{-1} \) is lower than the average \( \sigma_{\hat{k}} = 4.91 \times 10^{-2} \text{ Wm}^{-1}\text{K}^{-1} \) of the individual thermal conductivity uncertainties of all measurements. This is also the case for thermal diffusivity: \( \sigma(\hat{a}_1, \hat{a}_2, ..., \hat{a}_9) = 5.59 \times 10^{-9} \text{ m}^2\text{s}^{-1} \) is lower than \( \sigma_{\hat{a}} = 7.55 \times 10^{-9} \text{ m}^2\text{s}^{-1} \).

We can say that in these conditions the whole measurement system (experimental device and measurement method) gives reproducible results.

### 4.5. Limitation of the method

The results presented in this paper were obtained for one value of the exchange coefficient \( (h = 8 \text{ Wm}^{-2}\text{K}^{-1}) \). Indeed, this value of the exchange coefficient allows to obtain thermophysical properties in good agreement with those found in the literature \([2, 6]\). Several identifications were performed using values of \( h \) ranging from 6 to 10 \text{ Wm}^{-2}\text{K}^{-1}. Table V shows the values of the thermal conductivity obtained, thermal diffusivity and the ratio between the exchange coefficient and the thermal conductivity. For each value of \( h \), \( a \) is constant. However, \( k \) is strongly dependent on the exchange coefficient. Finally, we can observe that the ratio \( \frac{h}{k} \) remains constant. This is an indication that only this ratio has a significant influence in the thermal model, as in fact could be expected.

Therefore the accuracy on the conductivity measurement is limited by the accuracy to which \( h \) is known. Since part of this coefficient represents a convective exchange, its value as obtained from classical correlations cannot be considered very accurate; an independent calibration of this value appears necessary.
Table V. Values of identified parameters for different values of exchange coefficient \( h \)

<table>
<thead>
<tr>
<th>Identified parameters</th>
<th>( h = 6 , \text{Wm}^{-2}\text{K}^{-1} )</th>
<th>( h = 7 , \text{Wm}^{-2}\text{K}^{-1} )</th>
<th>( h = 8 , \text{Wm}^{-2}\text{K}^{-1} )</th>
<th>( h = 9 , \text{Wm}^{-2}\text{K}^{-1} )</th>
<th>( h = 10 , \text{Wm}^{-2}\text{K}^{-1} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal conductivity ( k ) (Wm(^{-1})K(^{-1}))</td>
<td>0.125 ± 0.012</td>
<td>0.146 ± 0.015</td>
<td>0.167 ± 0.017</td>
<td>0.187 ± 0.019</td>
<td>0.208 ± 0.021</td>
</tr>
<tr>
<td>Thermal diffusivity ( a ) (( \times 10^{-7} ) m(^2)s(^{-1} ))</td>
<td>1.196 ± 0.038</td>
<td>1.197 ± 0.038</td>
<td>1.199 ± 0.038</td>
<td>1.200 ± 0.038</td>
<td>1.202 ± 0.038</td>
</tr>
<tr>
<td>( \frac{h}{k} ) (m(^{-1} ))</td>
<td>48.00</td>
<td>47.95</td>
<td>47.90</td>
<td>48.13</td>
<td>47.62</td>
</tr>
</tbody>
</table>

5. CONCLUSION

We have studied a PVC sample of known thermophysical properties in order to validate the experimental device and measurement method. The different experiments performed showed a good reproducibility of the experimental results. The thermal conductivity and diffusivity values for the sample of PVC given by the experiment are in agreement with those indicated in the literature [2, 6].

The above results show that it is possible to obtain the emissivity for opaque materials at room temperature and low frequency. However, various improvements should be made on the device to reduce the uncertainties on the thermal conductivity and to avoid the influence of the black coating on measurements in the high frequency range. Finally, in the studied configuration, it is not possible to identify the thermal conductivity without knowing the exchange coefficient value. Thus, future improvements should be focused on this particular problem.
References