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Measurement of thermal diffusivity with a modified Angstrom method

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Measurement of thermal diffusivity with a modified Angstrom method

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Abstract. A methodology has been developed to measure the thermal diffusivity of high thermal conductivity solid materials. The basic idea traces back to the Ångström method, but the implementation differs in some points. Originally the specimen was free to the environment, here the specimen is thermally insulated to reduce, and control, the effects of natural convection. An appropriate analytical solution is used to calculate temperature distribution. The original two-point approach is replaced by a parameter estimation technique to consider multiple measurement points and reduce the final uncertainty. The methodology is used for a pure copper bar. The results are compared with the data available in the literature. The measured thermal diffusivity is $1.16 \pm 0.07 \cdot 10^{-4}$ m^2/s while the most reliable data available in the literature is $1.17 \pm 0.04 \cdot 10^{-4}$ m^2/s. The agreement of the present value with those available in the literature is excellent. This makes the use of this simple analytical approach attractive.

1. Introduction

Knowledge, within acceptable levels of uncertainty, of thermal diffusivity is currently of relevance, considering the potential for predicting the thermal behaviour of complex systems offered by numerical calculation procedures and the availability of new materials.

Electronics cooling, for example, shows an evolution that directly involves the knowledge of the thermophysical properties of materials, and in particular of thermal diffusivity. First, there is a widespread awareness that in many electronics thermal management applications involving time-varying workloads, ignoring the non-steady state behaviour of the systems certainly leads to oversized cooling [1] and that the driver of this time behaviour is thermal diffusivity [2]. Furthermore, new alternative materials to Al are proposed for the fabrication of finned heat sinks and other heat transfer components (alumina Al2O3, aluminium nitride AlN, beryllium oxide BeO etc.). With these new materials, in addition to the ever-present temperature dependence, small differences in alloy composition are sufficient to have large variations in thermophysical properties. For example, for Al2O3 (alumina) the thermal conductivity strongly depends on the Al concentration and can vary from 20 to 30 W·m^{-1}·K^{-1}. For AlN (aluminium nitride) the thermal conductivity ranges from 170 to 220 W·m^{-1}·K^{-1}. For BeO (beryllium oxide) it varies from 190 to 250 W·m^{-1}·K^{-1}, especially with temperature. Furthermore, while the thermal properties of pure metallic elements are well known [3], e.g. aluminium, those of alloys (e.g. Al 6060, 6061, 6063 used for heat sinks in electronics) can vary over a wide range, depending on the composition of the alloy. Measuring Al's thermal properties is further made troublesome by their variability with temperature.

A thermal property is generally determined by experimentally reproducing a situation, if possible a simple one, for which it is known how the property itself is related to some directly measurable quantities: temperature, thermal flows, etc. Thermal diffusivity must necessarily be determined in a non-stationary thermal condition since it characterizes this regime. These methods are based on measuring the temperature response to a time-varying heat source. In [4] an extensive discussion on the different related measurement methods and applications is proposed. Their classification distinguishes between transient and periodic temperature methods. In transient methods, thermal diffusivity is estimated from the temperature response of a sample to a sudden change in input heat. An example is the Flash method. In periodic temperature methods, thermal diffusivity is estimated from the response of a sample to a heat input that varies periodically with time. Examples include the 3ω-method and the Ångström method.

Conversely, methods that can be used to determine thermal conductivity involve measurements under steady-state conditions. However, these schemes are conveniently used only for materials with small thermal conductivity (less than 1 $W \cdot m^{-1} \cdot K^{-1}$), such as non-metallic materials. In the case of metallic materials, steady-state measurement methods, although conceptually very simple, can nevertheless be affected by uncertainties, even high ones. Therefore, in this case it is usual preferable to measure the thermal diffusivity in a non-steady state and possibly, through knowledge of the value of thermal capacity per unit volume, to trace back to that of the thermal conductivity. According to Abeles et al [5] the well-known calorimetric methods allow to measure the thermal capacity per unit of volume with an accuracy better than 1%, even at high temperatures (typically density and specific heat are, respectively, measured by the immersion method and by differential scanning calorimetry).

An extensive review of the main transient methods used to measure thermal diffusivity is reported by Danielson and Sidles [6]. Among these, processes in which a periodic thermal regime is reproduced deserve special attention, in particular those based on the Ångström method [7].

In this paper, a methodology for measuring the thermal diffusivity (and other thermal properties) of high conductivity solid materials is discussed. The basic idea goes back to the Ångström method [8], but the implementation differs in some points (see the section "Experimental data" for a discussion of the differences). The methodology is used for a highly pure copper bar. The results are compared with the data available in the literature and with the results obtained with a methodology [9] based on the use of a specifically developed FEM code [10]. In [9] an analytical solution was also used, but in that case the coupling with the environment was not considered. Necessarily, a simplified analytical approach gives results that differ from the most reliable data available in the literature, as we were able to measure in [9]. However, the complexity of the required FEM code [10] makes the use a relatively simple analytical approach attractive.

2. Experimental data

The basic idea of the present methodology for measuring the thermal diffusivity of high conductivity solid materials dates back to the Ångström method [8], but the implementation differs in some points.

In the Ångström technique [8], a "periodic square" thermal load is applied to one end of the specimen, while its entire length is exposed to the surrounding environment. A temperature wave propagates along the rod. In this wave propagation the phase shifts and the amplitude decreases. With measurements at two points spaced along the rod, the thermal diffusivity is determined.

Here, as in the case of the Ångström method [8], the data reduction is based on an analytical solution to the thermal problem. However, the square heating, which characterizes the Ångström method, is replaced by a monochromatic thermal wave produced by Joule heating. This simplifies the comparison between analytical solution and experiment.

A further specificity of Ångström's original method is the absence of thermal insulation on the specimen. This is because the tests are intended for a range of high temperatures, where the thermal radiation at the surface is high enough to contrast the characteristic fluctuations of natural convection. However, at relatively low temperature (less than 100°C), typical when using metals in finned heat sinks, the role played by thermal radiation is not sufficient to contrast the effect of natural convection, which may increase experimental uncertainty. To underline the order of magnitude of the situation, for a specimen temperature of about 450°C, the heat transfer coefficients are 13.4 $W\cdot m^{-2}\cdot K^{-1}$ for convection and 31.5 $W\cdot m^{-2}\cdot K^{-1}$ for radiation, respectively. For a specimen temperature of about 35°C, the heat transfer coefficients are 6.8 $W\cdot m^{-2}\cdot K^{-1}$ for convection and 5.8 $W\cdot m^{-2}\cdot K^{-1}$ by for radiation, respectively. Since here the focus was on low-temperature applications, the specimen was thermally insulated to reduce and control the effects of natural convection.

The role played by an external material in thermal contact with the specimen, such as a layer of expanded rubber, has been discussed by Eichhorn [11]. A parameter is proposed to evaluate whether this material, although having a non-negligible thermal capacity, does not participate and can be neglected in the mathematical model of the specimen. This parameter should be lower than 1. Here it is 0.24. For this reason, the presence of the insulating material has been considered in the boundary term and neglected in the unsteady term of the model.

An analytical solution is presented able to cover the data trend. The two-points approach of the original Ångström method is replaced by a more recent parameter estimation technique, using the analytical solution. In this way, a large number of measurement points can be considered, and the final uncertainty is reduced.

For an application of this modified Ångström method to the measurement of thermal diffusivity in high thermal conductivity materials, the data collected by Pagliarini and Piva in [9] are used here. The required data are essentially temperatures measured with thermocouples along a specimen. For a better understanding of the following data reduction procedure, a brief description of the system is provided. For further details please refer to the original article [9].

A cylindrical sample (12.8 mm diameter) of pure copper (99.9% purity) was examined (figure 1). One end of the sample is heated with a sinusoidal power by dissipating electrical energy in a constantan wire spirally wound on the surface; the other end is kept at a constant temperature by immersing the specimen in a thermostatic bath (free length between heater and bath 206.1 mm). The specimen is covered with an insulating layer of expanded rubber (8.9 mm thick) to reduce heat transfer to the environment. The temperature of the specimen is measured with T thermocouples glued to its surface.

The thermal power, variable over time with a sinusoidal law (period 50 s) around an average value:

$$\mathrm{Q(t')} = \mathrm{Q_m}\left[1 + \sin\left(\omega\, \mathrm{t'} - \frac{\pi}{2}\right)\right] \tag{1}$$

is produced by dissipating the pulsed voltage:

$$\mathrm{V(t')} = \mathrm{V_m}\left[\sin\left(\frac{\omega}{2}\mathrm{t'}\right)\right] \tag{2}$$

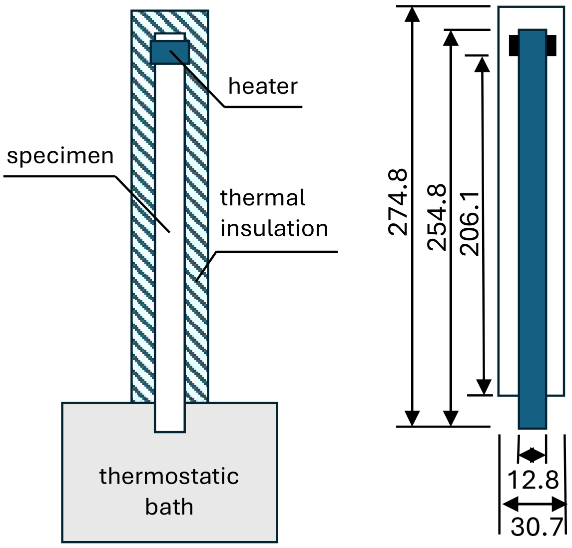

Figure 1. System and specimen schematics.

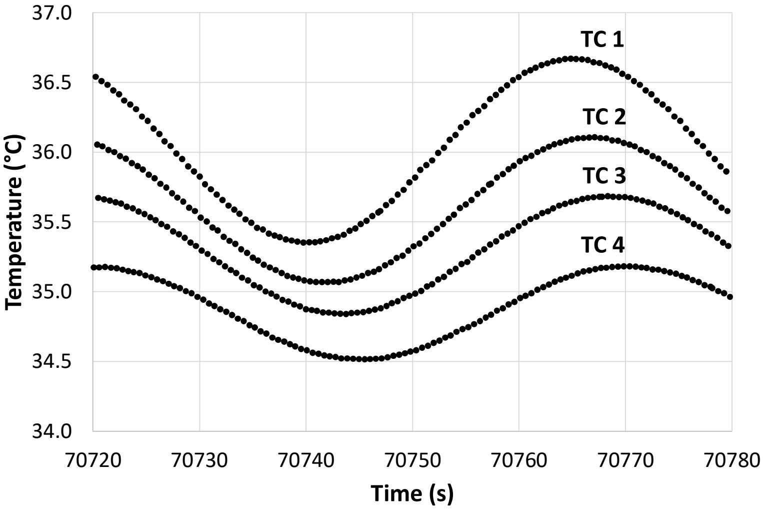

Figure 2. Time distribution of temperature in thermocouples 1-4.

In [9] the temperature data was used directly as a voltage signal, without any conversion. Here the same data is converted into temperature with a thermocouple calibration curve; then these temperatures are processed.

After a sufficiently long transient heating (never less than 10 hours), for each thermocouple a temperature composed of an average and an oscillating part is experienced, given as:

$$\mathrm{T}'(\mathrm{x}_i',\mathrm{t}') = \mathrm{T}_{\mathrm{m},i}'(\mathrm{x}_i') + \mathrm{A}_i(\mathrm{x}_i')\sin\big(\omega\,\mathrm{t}' - \varphi_i(\mathrm{x}_i')\big) \tag{3}$$

In figure 2 the time distribution of temperature data is shown for the first four thermocouples. The amplitude reduction and phase delay superimposed on a decreasing mean temperature are clearly readable.

In [12] a different methodology is proposed in which the heater with steady positive contribution, equation (1), is replaced by a source based on the Peltier effect, capable to oscillate around zero power. However, as will be shown in the "Result" paragraph, the stationary gradient of temperature along the specimen due to the steady power contribution did not introduce difficulties in data reduction and calculations.

3. Data reduction

For each thermocouple, in the steady-periodic regime the temperature can be decomposed into an average part and an oscillating part, equation (3). To determine its unknown parameters T'_m, A and φ, a numerical code has been developed that allows to sinusoidally interpolate the temperature values collected for each thermocouple. Specifically, we search for the values of the parameters that minimize the sum of the squares of the deviations of the experimental values from the corresponding theoretical ones. In more detail, given the interpolation function T', where T'_m, A and φ are the parameters sought, once the experimental temperature values Y_i in a number of n successive instants of time are known, the function to be minimized is:

$$S = \sum_{i=1}^{n} \frac{1}{\sigma_i^2} (Y_i - T'_i)^2 \tag{4}$$

where σ_i^2 is the variance associated with each experimental observation.

The solution of this research was obtained using the Gaussian linearization method described in [13]. The solution starts with the choice of trial values for the unknown parameters and then proceeds with the search for successive approximations of the corrections that lead to the minimization of equation (4). The search is stopped when for each of the parameters Π_i:

$$\frac{\Pi_i^{k+1} - \Pi_i^k}{\Pi_i^k} \leq 10^{-5} \tag{5}$$

In table 1 data is shown for the first eight thermocouples. In figure 3 amplitude, A_i, and phase delay, φ_i, are shown. The figure covers the free length of the rod (206.1 mm); the amplitude decreases exponentially and at the end of the rod it is reasonably equal to zero. The phase delay increases linearly.

In figure 4 the average temperature is shown. This parameter decreases linearly along the rod. This linear trend means that the coupling with the environment is barely appreciable, thus confirming the assumption of a limited participation of the thermal insulation in the oscillating heating.

The results of the data reduction for the first eight thermocouples are reported in table 1. Although there are more than eight thermocouples glued on the wall of the rod, only the first eight are reported because for the following ones the amplitude becomes less than 0.1 °C, clearly without statistical significance.

4. Theoretical analysis

The problem under consideration is schematized as a steady-periodic diffusion in a 1D semi-infinite body (fin approximation for negligible transverse temperature gradients).

To theoretically analyse the experimental problem of heat transfer in periodic regime, for the specimen under examination the energy balance equation for the cylinder thin fin must be written:

Table 1. Temperature data reduction based on equation (3).

TC n.	1	2	3	4	5	6	7	8
x'_i (mm)	6.2	16.5	26.1	35.5	46.0	55. 7	66.2	85.3
T'_{mi} (°C)	36.03	35.60	35.27	34.86	34.46	34.12	33.70	32.94
A_i (°C)	0.66	0.52	0.42	0.33	0.26	0.21	0.16	0.10
φ_i (s)	15.11	16.82	18.39	20.10	22.25	23.86	25.83	29.67

$$\frac{\partial T'}{\partial t'} = \frac{k}{\rho c}\frac{\partial^2 T'}{\partial x'^2} - \frac{4K}{\rho c D}(T' - T'_a) \tag{6}$$

where for the surface facing the environment the heat transfer coefficient K includes both conduction in the layer of thermal insulation and convection coupled to radiation from the surface towards the environment.

The domain of interest is between the end of the heater and the beginning of the immersion zone in the thermostatic bath. Here the boundary conditions are, respectively:

$$x'=0 \qquad T' = T'_1 + \Delta T'_2 \cos(\omega t') \tag{7}$$

$$x'\rightarrow+\infty \qquad T' = T'_a \tag{8}$$

The problem is then decomposed into its stationary and periodic components:

$$T'(x', t') = T'_m(x') + T'_p(x', t') \tag{9}$$

In this way, the energy balance equation (equation (6)) can be separated into a stationary part and a periodic part. Regarding the periodic part we obtain:

$$\frac{\partial T'_p}{\partial t'} = \frac{k}{\rho c}\frac{\partial^2 T'_p}{\partial x'^2} - \frac{4K}{\rho c D}\left(T'_p - T'_a\right) \tag{10}$$

with the boundary conditions:

$$x'=0 \qquad T'_p(0, t') = \Delta T'_2 \cos(\omega t') \tag{11}$$

$$x'\rightarrow+\infty \qquad T'_p(x' \rightarrow +\infty, t') = 0 \tag{12}$$

Equation (10) is rewritten as a function of the following dimensionless variables:

$$T_p = \frac{T'_p - T'_a}{\Delta T'_2} \qquad t = \frac{t'}{P} \qquad x = \frac{x'}{D}$$

turning in:

$$\frac{\partial T_p}{\partial t} = \mathrm{Fo}\frac{\partial^2 T_p}{\partial x^2} - 4\,\mathrm{Bi}\,\mathrm{Fo}\,T_p \tag{13}$$

with:

$$\mathrm{Fo} = \frac{\alpha P}{D^2} \qquad \mathrm{Bi} = \frac{KD}{k}$$

and the boundary conditions:

$$x=0 \qquad T_p(0,t) = \cos(2\pi\, t) \tag{14}$$

$$x\to+\infty \qquad T_p(x \to +\infty, t) = 0 \tag{15}$$

The solution to this problem is obtained analytically using the complex temperature method, as proposed by Arpaci [14]. The time-dependent real temperature is replaced by the corresponding complex temperature:

$$T_p(x,t) = F(x)\exp(i\, 2\pi\, t) \tag{16}$$

where the variable F belongs to the complex field:

$$T_p(x,t) = \left(F_{Re}(x) + i\, F_{Im}(x)\right)\exp(i\, 2\pi t) \tag{17}$$

Substituting equation (16) in equation (13) we get:

$$i\, 2\pi\, F = \mathrm{Fo}\frac{d^2 F}{\partial x^2} - 4\,\mathrm{Bi}\,\mathrm{Fo}\, F \tag{18}$$

$$x=0 \qquad F(0) = 1 \tag{19}$$

$$x\to+\infty \qquad F(x \to +\infty) = 0 \tag{20}$$

The solution of equation (18) is given by:

$$F(x) = \exp(-(\beta_{Re}+i\,\beta_{Im})x) \tag{21}$$

where:

$$(\beta_{Re}+i\,\beta_{Im})^2 = +\left(4\,\mathrm{Bi} + i\,\frac{2\pi}{\mathrm{Fo}}\right) \tag{22}$$

Equation (22) gives:

$$\beta_{Re}\beta_{Im} = \frac{\pi}{\mathrm{Fo}} \tag{23}$$

$$\beta_{Re}^2 - \beta_{Im}^2 = 4\,\mathrm{Bi} \tag{24}$$

The solution of the periodic component of the temperature distribution is finally:

$$T_p(x,t) = \exp(-\beta_{Re}\, x)\exp\left(i\, 2\pi\left(t - \frac{\beta_{Im}}{2\pi}\, x\right)\right) \tag{25}$$

5. Results

To validate the measurement procedure, data already collected for a 99.99% pure copper specimen [9] were used. There the data collected as voltage were processed directly, assuming that for a narrow temperature range the voltage-temperature correlation of the thermocouples T

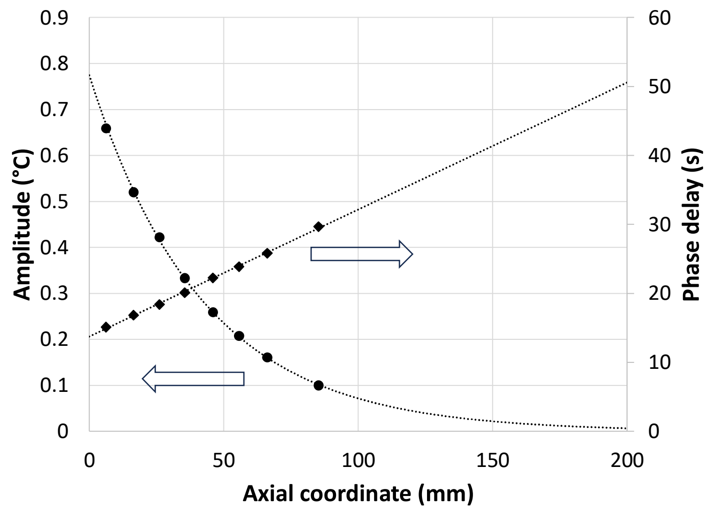

Figure 3. Amplitude (●) and phase delay (♦) distribution along the specimen.

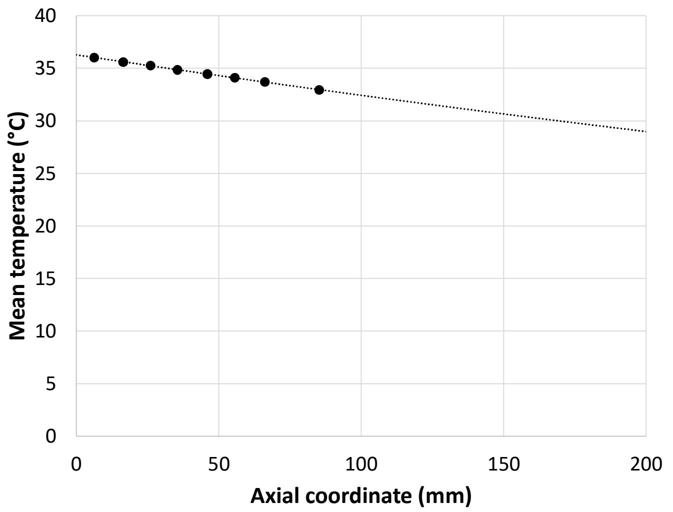

Figure 4. Average temperature distribution along the specimen.

was linear and no conversion into temperature was necessary. Here after calibrating the thermocouples, it was found that in the temperature range under examination a proper voltage-temperature correlation requires a parabolic, and therefore non-linear, correlation. For this reason, the original data were converted into temperature and reprocessed with the parameter estimation technique described in section 3. Results of these calculations are of values amplitude and phase delay of the temperature waves for the different thermocouples.

The parameter estimation technique discussed in section 3 was also used to calculate, from the amplitude and phase delay data, the parameters β_{Re} and β_{Im} in equation (21). The thermal diffusivity α of the specimen is then evaluated with equation (23) of the analytical model proposed in section 4, different from the one used in [9], which did not consider the thermal coupling with the environment.

The results of thermal diffusivity obtained by applying the parameter estimation method described in section 3 are compared in table 2 with those obtained in [9] and with others available

Table 2. Thermal diffusivity values for pure copper.

	$\alpha\ 10^4$ m^2/s	$\varepsilon_a\ 10^4$ m^2/s
Present solution	1.16	0.07
Toulukian [3]	1.17	0.05
Gandia and Lopez-Baeza [15]	1.20	0.13
Leden et al [16]	1.17	0.04
Pagliarini and Piva [9]: numerical	1.17	0.04
Pagliarini and Piva [9]: analytical	1.14	0.07

in the literature. The agreement of the present value with those available in the literature is excellent. Therefore, the methodology based on the amplitude and phase delay of the periodic signal allows to model the experimental details with adequate precision.

The expanded uncertainty of the measured parameters is estimated at the 95% confidence level, following the procedure proposed in [17]. In the estimation of thermal diffusivity with equation (23), the parameters involved are: diameter, D, period, P, real and imaginary part of the complex temperature, β_{Re} and β_{Im}. The corresponding standard uncertainties are: $\varepsilon_D = 0.2$ mm, $\varepsilon_P = 0.055$ s, $\varepsilon_{\beta Re} = 0.001$ and $\varepsilon_{\beta RIm} = 0.001$. The last two come from interpolating the amplitude and phase delay of the oscillatory part of the temperature along the rod using the parameter estimation technique. In this application a standard uncertainty of 0.03°C is assumed for temperature measurements (see [9] for details). The combined standard uncertainty assigned to the calculated parameter Π is then estimated using the root-sum-square propagation rule.

6. Concluding remarks

A variant of the Ångström method is used to measure the thermal diffusivity of a specimen with high thermal conductivity. The first variant consists of having the specimen thermally insulated to reduce the thermal coupling with the environment. Second, the two-point measurement method has been replaced by a parameter estimation procedure, which allows the result to be based on many data, with lower uncertainties. Third, a monochromatic thermal wave produced by Joule heating is used. A simplified mathematical model has been solved analytically.

The measurement procedure was validated on a high purity copper sample, in order to refer to literature data for comparison. The results allowed to consider the procedure reliable.

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7. Nomenclature

Symbols

A	K	Amplitude
Bi	-	Biot number (K·D)/k
c	$J\cdot kg^{-1}\cdot K^{-1}$	Specific heat
D	m	Diameter
F	-	Complex dimensionless temperature
Fo	-	Fourier number $(\alpha\cdot P)/L^2$
i	-	Imaginary number
k	$W\cdot m^{-1}\cdot K^{-1}$	Thermal conductivity
K	$W\cdot m^{-2}\cdot K^{-1}$	Heat transfer coefficient
P	s	Period
Q	W	Thermal power
S	-	Objective function
t’	s	Time
t	-	Dimensionless time
T’	K	Temperature
T	-	Dimensionless temperature
V	V	Voltage
x’	m	Axial coordinate
x	-	Dimensionless axial coordinate
Y	K	Measured temperature

Greek symbols

α	m^2/s	Thermal diffusivity
β	-	Complex number
ε	-	Uncertainty
ρ	kg/m^3	Density
σ^2	K^2	Temperature variance
ω	rad/s	Angular frequency $2\pi/P$
Π	-	Generic parameter
φ	rad	Phase delay

Subscripts

a	Ambient
Im	Imaginary part
m	Mean
p	Periodic
Re	Real part

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The author wishes to acknowledge Giorgio Pagliarini for the long and always fruitful cooperation.