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Thermophysical Parameters of Optical Glass BK 7 Measured by the Pulse Transient Method[1]

Ľ. Kubičár,[2,3] V. Vretenár,[2] and U. Hammerschmidt[4]

This paper is focused on the pulse transient method. The theory of the method and the measuring regime (time window) are analyzed. The results of the analysis are verified on borosilicate crown glass BK7, which is a candidate for a standard for thermal conductivity. Thermal contact and surface effects affect the length of the time window in which the evaluation procedure is applied. The one-point evaluation technique is compared with the results of the fitting procedure that uses the time window found by difference analysis. The values of the thermal conductivity, thermal diffusivity, and specific heat were found to be $1.05\,\mathrm{W\cdot m^{-1}\cdot K^{-1}}$, $0.548\times10^{-6}\,\mathrm{m^{-2}\cdot s^{-1}}$, and $767\ \mathrm{J\cdot kg^{-1}\cdot K^{-1}}$, respectively, using the one-point evaluation technique.

KEY WORDS: optical glass BK 7; pulse transient method; specific heat; thermal conductivity; thermal diffusivity.

1. INTRODUCTION

Modern technology is looking for measuring methods that provide reliable data of thermophysical properties of a broad class of materials, preferably on small-sized specimens over a short time. Recently, a class of transient methods was broadened by new experimental arrangements that show several advantages compared with classical methods [1–3]. Some of them, depending on the experimental arrangement, yield a full set of thermophysical parameters, namely, specific heat, thermal diffusivity, and thermal conductivity or thermal effusivity. Moreover, the specimen size can be

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[2] Institute of Physics SAS, Dúbravska cesta 9, 84228 Bratislava, Slovakia.
[3] To whom correspondence should be addressed. E-mail: fyzikubo@savba.sk
[4] Physikalisch-Technische Bundesanstalt, Postfach 3345, 38023 Braunschweig, Germany.

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chosen from a wide range; thus, inhomogeneous and porous materials can be examined. Data reliability can be improved to a great extent by elimination/minimization of the heat losses from the specimen surface, and the thermal contact and heat capacity of the measuring probe effects that become active during the measuring process. The experimental arrangements under discussion here involve measuring probes that are embedded in the material to suppress the above-mentioned interfering effects.

Transient methods are based on the generation of a dynamic temperature field inside the specimen. They utilize a small heat disturbance in the form of a pulse of heat or a heat flux in the form of a stepwise function [1]. From the temperature response to this disturbance, the thermophysical properties can be calculated according to the model used. Next we will concentrate on the pulse transient method [4]. Three factors influence the uncertainty of the pulse transient method, namely, the measuring time during which the temperature field is developed inside the material under test, the geometry of the specimen, and the properties of the heat source. The optimal experimental setup requires such a specimen size that the temperature field will not be disturbed during that particular time period when the temperature response is highly sensitive to the thermophysical properties of the tested material.

The present study deals with measurements of the thermophysical parameters of the optical glass BK7 by the pulse transient method. Influence of the major interfering effects are discussed, namely, the constriction effect caused by the heat source construction and the surface effect caused by heat losses from the outer specimen surface. The one-point evaluation is compared with the fitting within the time window that is estimated by difference analysis. The difference analysis extends the specimen thickness range where thermophysical data are stable.

2. THEORY

The principle of the method is outlined in Fig. 1 [4]. The specimen consists of three parts (I, II, III). A planar heat source is clamped between the first and second parts. The heat pulse is produced due to Joule heating from the electrical resistance of the planar source. One junction of a thermocouple is placed between the second and third parts. This sensor measures the temperature response to the heat pulse. The ideal model of the pulse transient method yields the relation,

$$T(h,t) = \frac{Q}{c_{\mathrm{p}}\rho\sqrt{\pi at}} \exp\left(\frac{h^2}{4at}\right) \tag{1}$$

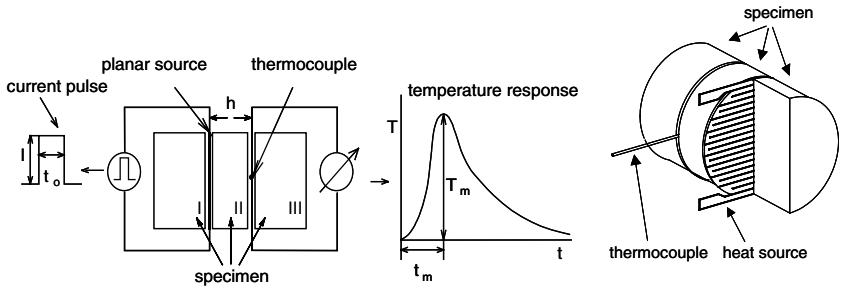

Fig. 1. Principle of the pulse transient method (left) and the experimental setup (right). A part of the specimen is 'cut out' to see the structure of the heat source (right).

for the transient temperature $T(h,t)$ at the thermocouple junction. Here, $Q = RI^2t_0$ is the energy of the heat pulse, R is the electrical resistance of the heat source, t_0 is the width of the current pulse, ρ is the specimen density, c_p is the specific heat of the specimen, a is the thermal diffusivity of the specimen, and h denotes the distance between the heater and temperature sensor. Two different procedures for determination of the thermophysical properties mentioned above can be used, namely, the one-point procedure where the maximum of the temperature response is taken as the input and the fitting procedure where the function (Eq.(1)) is fitted to the observed data within the time window of the temperature response [5].

The one-point procedure (standard procedure) deals with the following relations [4]:

Specific heat c_p:

$$c_p = Q/(\sqrt{2\pi e}\rho h T_m) \tag{2}$$

Thermal diffusivity a:

$$a = h^2/(2t_m) \tag{3}$$

Thermal conductivity λ:

$$\lambda = ac_p\rho = Qh/(2\sqrt{\pi e}t_m T_m) \tag{4}$$

T_m is the maximum of the temperature response at time t_m, and e denotes the Euler number .

3. INFLUENCE OF DISTURBING EFFECTS

Equations (1)–(4) correspond to the ideal model of the method. The use of the ideal model is a prerequisite to obtain reliable data in

intercomparison measurements; otherwise, additional parameters involved in the measuring process have to be compared. The ideal model represents an unbounded specimen in which a two-dimensional heat source of the same material acts. An ideal thermal contact exists between the heat source and the specimen. The temperature sensor has similar properties as the heat source. In addition, the ideal model assumes a heat pulse in the form of Dirac's δ-function. However, heat loss from the specimen surface (outer boundary effect) and the heat source construction (inner boundary effect: constriction effect) influence the measuring process.

The effect of heat losses from the specimen surface is demonstrated in Fig. 2. The ideal model assumes planar isotherms. However, due to heat losses from the outer specimen surfaces the shape of isotherms starts to deform. Reliable data require the shape of the isotherms to be planar in the surroundings of the thermometer. The deformation of the isotherms at the location of the thermocouple depends on the diameter of the specimen, on the thermophysical properties of the specimen, and on the distance between the heat source and the thermometer [4]. Therefore, by using several specimens of different thicknesses and diameters, the data stability interval can be located and, thus, reliable data of the thermophysical properties can be determined.

The heat source contributes to the constriction thermal contact resistance due to its construction (cf. Fig. 3.), which usually plays a predominant role in the heat transport from the heat source into the

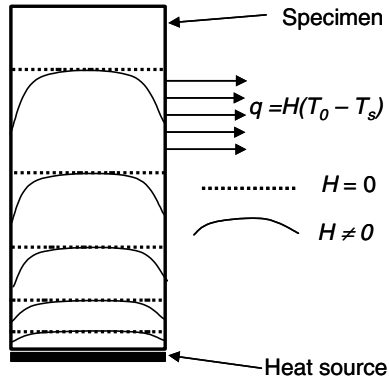

Fig. 2. Deformation of the temperature field for a planar heat source and a specimen in the form of a cylinder. H—heat loss coefficient, T_s—specimen surface temperature, T_0—surrounding temperature.

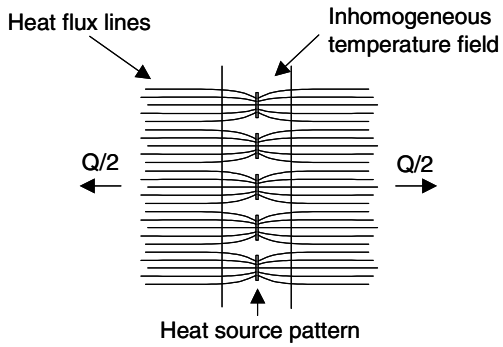

Fig. 3. Constriction contact resistance due to the construction of the heat source as shown in Fig. 1.

specimen when one uses the setup shown in Fig. 1. The heat flux lines are deformed around every conducting path, i.e., the temperature gradient is higher near the contact compared with that inside the specimen. Reliable data require that the part of the specimen volume that is penetrated by the deformed temperature field is small in comparison to that penetrated by the non-deformed temperature field. By varying the specimen thickness, again, a data stability interval can be found, i.e., reliable data of the thermophysical parameters can be determined.

4. EXPERIMENT

The measurements were performed on BK7, a borosilicate crown glass fabricated by Schott AG, Mainz, Germany, which is a candidate reference material for thermal conductivity. This glass is a very common material for optical components in the visible range. It is known for good scratch resistance, a very low amount of inclusions, and is almost bubble-free. Because of stable chemical properties, no special treatment is required to grind and polish the material.

A specimen set consists of three parallelepipeds with a uniform cross section of $30 \times 30\,\mathrm{mm}^2$ and thicknesses $h_I = 15\,\mathrm{mm}$, h, and $h_{III} = 15\,\mathrm{mm}$ (see Fig. 1). The density is determined from the mass and the volume of all three parts of the specimen ($\rho = 2510 \pm 2\,\mathrm{kg \cdot m^{-3}}$). A series of part-II-samples of different thickness h were used as listed in Table I. The heat source is made of 22-μm thick nickel foil. The conducting line is etched in a form of a meander. Its electrical resistance is $1.8\,\Omega$. A chromel–alumel thermocouple with a diameter of 70 μm was used for measuring the temperature response. A heat sink paste (Glammorgan Middland) was applied

between all parts of the setup to improve mutual thermal contacts. The measurements were performed using the instrument RT 1.02, Institute of Physics, SAS, Slovakia. Experimental parameters are listed in Table I. The heat pulse width t_0 was chosen to meet the criterion of Dirac's δ-function, i.e., no correction has to be used [4]. A set of measurements was performed using various heat pulse widths and heat pulse energies. The heat pulse energy was limited from the lower side by sufficient ratio of the temperature response to the temperature drift and from the upper side by suppression of the nonlinear effects. Thermophysical data must be stable within the above-specified heat pulse energy range. All measurements were made at a temperature of 25 °C.

5. RESULTS AND DISCUSSION

Variations of the values of thermophysical properties are presented in Fig. 4 where the determined values for the thermal diffusivity in the upper, specific heat in the middle, and thermal conductivity at the bottom are plotted as a function of specimen thickness. The one-point procedure (standard procedure) was used for data evaluation using Eqs. (2)–(4) [4]. Data sets of unusually high scatter were discarded. The box indicates the data stability interval. Variations of the specific heat and thermal diffusivity should approach zero within the data stability interval. The effect of contact constriction occurs for a small specimen thickness, while heat losses from the outer specimen surface shifts data for a thickness $h =$ 15 mm. A correction procedure was applied to the data to analyze the heat loss effect [4]. Corrected data are shown in Fig. 4 (circled points) when the heat loss coefficient reaches $H = 1.2\,\mathrm{W \cdot m^{-2}}$. Thus, a data stability interval

Table I. Experimental Parameters of the Measurements of BK 7

Specimen thickness h (mm)	Heat pulse width t_0 (s)	Heat pulse energy Q ($10^4\,\mathrm{W \cdot m^{-2}}$)	Surrounding atmosphere
2.0 ± 0.1	0.8	$2.1 - 4$	Air
4.0 ± 0.1	1.8	$3 - 5$	Air
6.0 ± 0.1	2.5	$3.4 - 5.2$	Air
8.0 ± 0.1	4.0	$3.5 - 5$	Air
10.0 ± 0.1	6.0	$5 - 6$	Air
15.0 ± 0.1	15	$3.6 - 6.7$	Air
15.0 ± 0.1	15	$3.6 - 6.7$	Vacuum

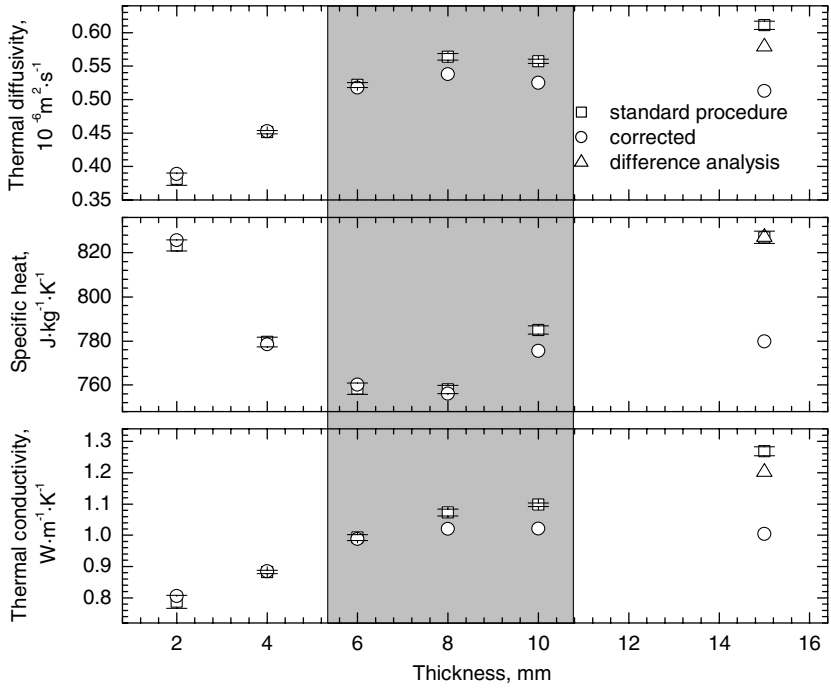

Fig. 4. Variations of the thermal diffusivity, specific heat, and thermal conductivity with specimen thickness at 25 °C. Data stability interval is indicated by a box. Error bars indicate uncertainty given by statistics.

might be longer for a thicker specimen when an additional parameter, the heat loss coefficient H, is introduced into the model.

A test of the ideal model was made for a specimen thickness $h =$ 15 mm due to the use of a vacuum to suppress conductive and convective heat losses from the specimen surface. Differences in the determined values in an air environment and in vacuum for specific heat ($c_{\text{air}} =$ $826.5\,\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$, $c_{\text{vac}} = 828\,\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$) and thermal diffusivity ($a_{\text{air}} =$ $0.603\times10^{-6}\,\text{m}^2\cdot\text{s}^{-1}$, $a_{\text{vac}} = 0.600\times10^{-6}\ \text{m}^2\cdot\text{s}^{-1}$) were found to be negligible. This result indicates that deformation of the isotherms is not caused by air convection that is characterized by Newton's law $q = H(T_{\text{s}} - T_0)$ (T_s—temperature of the specimen surface, T_0—surrounding temperature, q—density of heat flow) [4]. Another mechanism of heat loss, e.g., thermal radiation, is probably active during the measuring process.

A difference analysis was used to estimate the time window for data evaluation [6]. The difference analysis is based on fitting the temperature

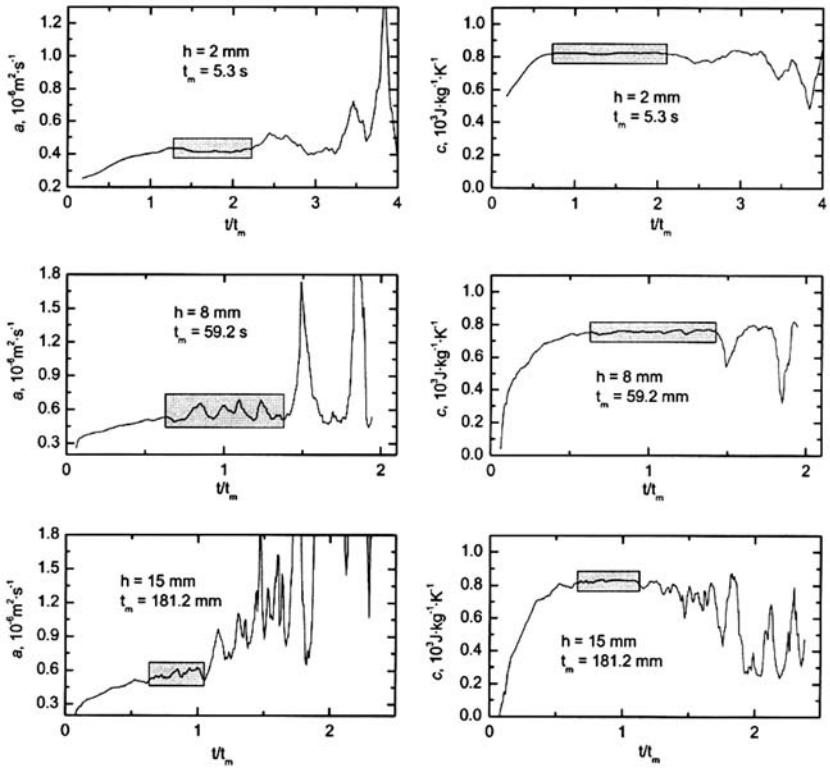

Fig. 5. Thermal diffusivity (left) and specific heat (right) as obtained by difference analysis for different specimen thicknesses. Boxes indicate data stability interval. The maximum of the temperature response is shown for every specimen thickness.

function (Eq.(1)) found for the ideal model to the experimental dataset within a small time interval. A time interval of $\Delta F = at/h^2$ is chosen that was continuously moved over the scanned temperature response. The corresponding values of the specific heat and the thermal diffusivity are shown in Fig. 5 for three specimen thicknesses, 2, 8, and 15 mm. The fitted values are plotted as a function of the dimensionless time t/t_{m} where t is the time of the center of the corresponding small time interval and t_{m} is the time of the maximum of the temperature response.

The basic criterion for accurate measurements is the non-disturbed temperature field. Its characteristic shape is generated by the heat source used. For small values of t/t_{m} (small penetration depth [2]), the temperature field within the specimen is affected by the construction of the heat source, while for large values of t/t_{m} (large penetration depth), it is affected

by heat losses at the specimen surface [4]. The time window can be estimated for every specimen thickness within which the fitted data are stable. Here, data stability is given by slopes of the specific heat and thermal diffusivity curves that approach zero. The individual windows are indicated in Fig. 5 by boxes and listed in Table II for every specimen thickness together with the corresponding thermophysical properties obtained from fitting within the time window ($a_{\text{fit}}, c_{\text{fit}}$) and from the standard procedure ($a_{\text{stand}}, c_{\text{stand}}$).

The location and length of the time window are affected by the sensitivity coefficients [7] and by the degree of the linear correlation between them [8] when the ideal model is used. For the case of the real model, the time window is shortened by the heat source effect at the beginning and by the surface effect at its end [9]. The heat source effect can be found for every specimen thickness for short times, i.e., small penetration depth (see Fig. 5). The outer surface effect can be found for a specimen thickness $h = 15\,\text{mm}$ only (cf. Fig. 5). For long times (outside the time window), the curves should fluctuate around a value which corresponds to the time window when heat losses do not disturb the measurement. For a specimen thickness $h = 15\,\text{mm}$ and long times (Fig. 5), unequivocally, both the curve of the specific heat and the curve of the thermal diffusivity deviate from the value given by the data stability interval. The use of a larger cross-section of the specimen might suppress this effect as follows in Fig. 2. The deformation of isotherms at the location of the temperature sensor is reduced due to an increase of the specimen diameter.

The standard procedure uses the time of the maximum of the temperature response, $t/t_{\text{m}} = 1$, for data evaluation. Figure 4 indicates the relation between the data stability interval and the dimensionless time t/t_{m}. There is a clear discrepancy between the standard procedure that uses the time $t/t_{\text{m}} = 1$ and the fitting procedure that is applied within the time window for specimen thicknesses $h = 2\,\text{mm}$ and $h = 15\,\text{mm}$. For both thicknesses, the time $t/t_{\text{m}} = 1$ is outside or just on the boundaries of the data stability interval, especially for $h = 2\,\text{mm}$. The true value for $h = 2\,\text{mm}$ should be sought well above $t/t_{\text{m}} = 1$; unfortunately, for $t/t_{\text{m}} \gg 1$ the correlation between the sensitivity coefficients is high. Time $t/t_{\text{m}} = 1$ for a specimen thickness $h = 15\,\text{mm}$ is just at the upper side of the data stability interval. Table II indicates that the thermal diffusivity is more affected by heat losses (surface effect) than is the specific heat, provided that the standard procedure is used.

The data stability interval can be prolonged by using a thicker specimen providing that the difference analysis is used (Fig. 5). However, this is valid for only the thermal diffusivity and thermal conductivity. Variations in the derived values of specific heat were detected for larger specimen thicknesses.

Table II. Time Windows for Specimen Thicknesses of 2, 8, and 15 mm; Thermal Conductivity and Specific Heat Results Obtained by Standard Technique (a_{stand}, c_{stand}) and by Fitting (a_{fit}, c_{fit})

Specimen thickness (mm)	Thermal diffusivity ($10^{-6}\,m^2 \cdot s^{-1}$)					Specific heat ($J \cdot kg^{-1} \cdot K^{-1}$)				
	Time window (s)					Time window (s)				
	Start	End	Width	a_{fit}	a_{stand}	Start	End	Width	c_{fit}	c_{stand}
2.0	6.0	11.0	5.0	0.423 ± 0.008	0.377 ± 0.008	3.1	12.3	9.2	828 ± 10	823 ± 10
8.0	34.0	86.8	52.8	0.562 ± 0.011	0.572 ± 0.011	33.6	88.8	55.2	761 ± 9	759 ± 9
15.0	102	206	104	0.562 ± 0.011	0.608 ± 0.012	108	219	111	827 ± 10	827 ± 10

Table III. Mean Values of the Thermophysical Property Data and Standard Deviations of Optical Glass BK7 at 25 °C

	Specific heat ($J \cdot kg^{-1} \cdot K^{-1}$)		Thermal diffusivity ($10^{-6} m^2 \cdot s^{-1}$)		Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	
Procedure	$\bar{c}$	δc	$\bar{a}$	δa	$\bar{\lambda}$	$\delta\lambda$
Standard	767	13	0.548	0.02	1.05	0.05
Correction	764	10	0.526	0.01	1.01	0.02
Difference analysis	769	13	0.544	0.02	1.04	0.04

Table IV. Dimensionless Characteristics of the Time Window

	$h = 6$ mm		$h = 8$ mm		$h = 10$ mm	
Time window	$\bar{F}$	ΔF	$\bar{F}$	ΔF	$\bar{F}$	ΔF
Start	0.33	0.051	0.28	0.028	0.32	0.035
End	0.67	0.058	0.67	0.070	0.58	0.015
Width	0.34	0.049	0.39	0.073	0.26	0.040

The origin of these variations has to be studied in more detail due to the use of a broader range of specimen thicknesses and cross sections.

6. CONCLUSIONS

Thermophysical properties, i.e., specific heat, thermal diffusivity, and thermal conductivity of optical glass BK7 measured by the pulse transient method are presented. Variation of the specimen thickness was used to locate the proper specimen thickness window and time window in which thermophysical data are stable. Two procedures were used for the determination of the thermophysical data, namely, the one-point procedure (standard procedure) that is based on the maximum of the temperature response and the fitting procedure when the maximum $t/t_m = 1$ is outside the time window that is estimated by the difference analysis. The correction procedure was used as an extension to the one-point procedure to correct for the surface effect. Table III lists mean values and standard deviations of the thermophysical properties. A dimensionless window given by the Fourier number $F = at/h^2$ was estimated from the specimen thickness window ($h = 6$, 8, and 10 mm) as shown in Fig. 4. The mean values and standard deviations of the characteristics of the dimensionless window are listed in Table IV. Clearly, the heat losses from the specimen surface

are active for a specimen thickness $h = 10\,\text{mm}$ as the end of the time window is postponed to shorter times which causes a smaller time window.

The estimated window should be valid for all materials when the pulse transient method is used with a specimen with a cross section of $30 \times 30\,\text{mm}^2$. Nevertheless, the validity of this dimensionless time window has to be verified on more materials representing a wide range of different thermal conductivities.

The origin of the variations in the derived values for the specific heat and of the data shift in thermal conductivity and thermal diffusivity for a specimen thickness $h > 15\,\text{mm}$ has to be studied in more detail. Here, a broader range of specimen thickness and cross sections is needed.

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