

THERMOPHYSICAL PARAMETERS MEASURED BY CLASSIC AND TRANSIENT METHODS.

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Abstract

The present contribution analyses experimental data that were obtained by Guarded Hot Plate method for measuring thermal conductivity, DSC for measuring specific heat, Flash method for measuring thermal diffusivity and Pulse Transient method for measuring thermal conductivity, thermal diffusivity and specific heat and the published data. Data on homogenous materials (BK7, PMMA and Stainless Steel A310) and heterogeneous materials (composite SiC – C/C and porous aerated autoclaved concrete) are analysed. Agreement for homogenous and disagreement for heterogeneous materials on thermophysical data was found using classic and transient methods.

Key words: transient methods, thermal diffusivity, thermal conductivity, specific heat

1 Introduction.

Classic methods use steady-state measuring regime for determination of the thermal conductivity (guarded hot plate method), equilibrium regime for determination of the specific heat (adiabatic calorimetry) and dynamic regime for determination of the thermal diffusivity (Flash method) and transient methods for measuring thermal conductivity, thermal diffusivity and specific heat. The present contribution deals with differences in values of the thermophysical parameters measured by classic methods and by transient methods. The analysis will be performed considering homogeneous, heterogeneous and anisotropic materials that in modern technology are often used. The results of analysis will be verified by measurements of thermophysical parameters using pulse transient method on homogeneous and heterogeneous materials.

2 Thermophysical parameters.

A broad range of classic measuring methods needs to be included into our analysis considering measuring regime and material structure. Generally, the classic methods include steady state measuring methods for determination of the thermal conductivity λ , equilibrium methods for determination of the specific heat c and finally dynamic methods that are used for determination of the thermal diffusivity a . Data consistency can be tested by relation $\lambda = ac\rho$ (ρ is density) when one uses different methods for determination of the individual parameters. One might be astonished whether data consistency relation can be used when data coming from different measuring regimes are

considered. In contrary to classic methods some transient methods give all three parameters within a single measurement. Then a question arises how good is agreement in data coming from various methods. The experiments have shown that for homogeneous, isotropic materials data consistency is satisfactorily fulfilled for classic methods. Good agreement was found among data given by classic and by transient methods for homogeneous materials even sometimes difficulties can be found considering measurement accuracy [2]. Nevertheless clear differences in data exist when porous or anisotropic materials are inspected by classic and by transient methods [3].

3 Experiment

Pulse transient method (see Fig. 1) was used for measurements verification to find agreement or differences in experimental data [4]. Then the following relation were used for determination of the thermophysical parameters:

thermal diffusivity

$$a = \frac{h^2}{2t_m}, \quad (1)$$

specific heat,

$$c = \frac{Q}{\sqrt{2\pi \exp(1)} \rho h T_m}, \quad (2)$$

and the thermal conductivity

$$\lambda = ac\rho = \frac{Qh}{2\sqrt{2\pi \exp(1)} T_m t_m}, \quad (3)$$

where ρ is density, $Q = RI^2 t_0$ and other parameters are elucidated in Fig. 1.

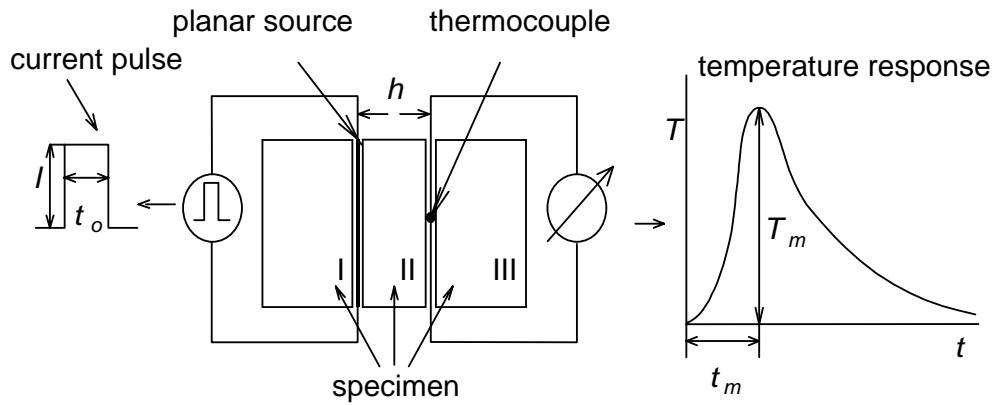


Fig. 1.: Principle of the pulse transient method.

4 Results and discussion

Homogeneous materials. Table 1 specifies density and the geometry of the tested materials. The geometry of the specimen set-up was chosen to eliminate the surface and

the contact effect [5]. Table 2 gives an overview on recommended values of thermophysical parameters that were obtained by classic methods as well as measured values by pulse transient. Recommended data on BK 7 are taken from catalogue lists only. A difference in density was found between the tested specimen and the values specified on the catalogue lists ($\rho = 2100 \div 2300 \text{ kg m}^{-3}$). The difference between the values given by classic and by transient methods on Stainless steel A 310 and Perspex is within several percents that might correspond to accuracy of the measurements.

Table 1: Geometry of the tested homogeneous materials.

Material	Density ρ [kg m^{-3}]	Dimension [mm]	Specimen thickness h [mm]	
			part II	part I/III
Stainless steel A 310	7902	$\text{Ø } 20$	4.9	10/8
Glass BK 7	2510	30x30	8	15/15
Perspex	1184	$\text{Ø } 30$	6	15/15

Table 2: Published, recommended, measured values and deviations of thermophysical parameters of homogeneous materials

Parameter	Data type	Stainless steel A310	BK7 [5]	Perspex
Thermal diffusivity $10^{-6} [\text{m}^2 \text{sec}^{-1}]$	Recommended	3.45 [6]	–	0.11 [7, 8]
	Measured	3.35	0.546	0.1102
	Deviation	- 3.00 %	–	- 3.3 %
Specific heat [$\text{J kg}^{-1} \text{K}^{-1}$]	Recommended	471.0 [6]	710 – 910	1460.0 [7, 8]
	Measured	469.0	767.9	1430.0
	Deviation	- 0.4 %	–	- 2.1 %
Thermal conductivity [$\text{W m}^{-1} \text{K}^{-1}$]	Recommended	12.8 [6]	1.114 – 1.3	0.19 [7, 8]
	Measured	12.4	1.05	0.187
	Deviation	- 3.23 %	–	- 4.9 %

Heterogeneous materials. Table 3 specifies density and the geometry of the tested materials. The geometry of the specimen set-up was chosen to eliminate the surface and the contact effect on experimental data [5]. Two materials were chosen for the thermophysical analysis namely composite C/C–SiC and the autoclaved aerated concrete (AAC). While former material is composed by C fibers and by SiC matrix, the latter one belongs to family of the porous material having porosity of 80 %. A broad range of pores distribution contains the skeleton of the AAC. The predominant part of these pores is open while small fraction of micropores can be closed.

Table 3: Geometry of the tested heterogeneous materials.

Material	Density ρ [kg m^{-3}]	Dimension [mm]	Specimen thickness h [mm]	
			part II	part I/III
Composite C/C-SiC	2020	$\text{Ø } 9.85$	9.15	15 / 15
Aerated autoclaved concrete	512	150x150	15	40 / 40

Table 4 gives an overview on values of thermophysical parameters that were obtained by classic methods (in parenthesis) [9] as well as by pulse transient on composite C/C - SiC. The composite is strongly anisotropic. Two different orientations were chosen for analysis namely the parallel orientation to fibers and the transversal one. A difference of several percent was found between the published and the measured data in transversal

orientation to fibers of the thermal conductivity. Large differences in transport parameters (thermal conductivity and thermal diffusivity) were found between parallel and transversal orientation to the fibers. In addition a large difference exists between the specific heat values measured in parallel and transversal direction to the fibers. This indicates that specific heat is a transport parameter when transient techniques are used for anisotropic composites. The measured value can be ascribed for that case to apparent values. Effective values of specific heat given by DSC agree within several percent with that one found by pulse transient in parallel direction to the fibers. A relation between the effective value and the apparent value of the specific heat has to be found on the base of the macrostructure model.

Table 4: Published and measured thermophysical parameters of Composite C/C-SiC, T = 25°C.

Parameter	Transversal to fibers	Parallel to fibers	Difference
Thermal diffusivity $10^{-6} [\text{m}^2\text{sec}^{-1}]$	6.25	10.78	+72 %
Specific heat $[\text{J kg}^{-1} \text{K}^{-1}]$	(710)		
	816	718.9	-12 %
Thermal conductivity $[\text{W m}^{-1} \text{K}^{-1}]$	(10.5) 10.24	15.73	+54 %

Table 5 gives an overview on values of thermophysical parameters of AAC that were obtained by classic methods as well as by a variant of the transient method (in parenthesis) [10] and by pulse transient method [3]. An agreement between the published and the measured values might be limited due to not specified moisture content. A significant difference in thermophysical parameters was found by pulse transient when different atmospheres were used. Again, gas in pores influenced specific heat values. Mixing rule was used to test the contribution of gas in pores to the volume specific heat. Calculated value in parenthesis corresponds to skeleton when no contribution from pore content exists (vacuum). Contributions of gases to volume specific heat for porosity of 80 % are negligible, below 1 %. However, measured specific heat values change around 11 % using different atmospheres. In addition measured specific heat value lowers with growing thermal conductivity of the surrounding atmosphere and tends to reach the recommended value. Gas in pores significantly influences the heat transport through

Table 5: Published, recommended and measured thermophysical parameters of AAC, T = 25°C. Data in parenthesis are calculated for skeleton in vacuum.

Atmosphere	Thermal diffusivity $10^{-6} [\text{m}^2\text{sec}^{-1}]$	Thermal conductivity $[\text{W m}^{-1} \text{K}^{-1}]$	Specific heat $[\text{J kg}^{-1} \text{K}^{-1}]$	Volume specific heat $[\text{J m}^{-3} \text{K}^{-1}]$
Vacuum	0.20	0.115	984	(5.04x10 ⁵)
Air, Humidity~30 %	0.277 [10] 0.26	0.135 [10] 0.141	840 [11] 924	(5.04x10 ⁵) +1.27x10 ³
Helium	0.40	0.200	867	(5.04x10 ⁵) +7.5x10 ²

porous materials. Thermophysical data of used gasses are given in Table 6. The most reliable data on specific heat can be obtained for atmosphere having high thermal conductivity and low heat capacity. Then pores are shortcut for heat flux and heat transport correspond to model of quasi-homogeneous continuum. However, relation between the effective value and the apparent value of the specific heat has to be found on the base of the macrostructure model.

Table 6: Thermophysical data of air and helium [12].

Atmosphere	Density [kg m ⁻³]	Thermal diffusivity [m ² sec ⁻¹]	Specific heat [J kg ⁻¹ K ⁻¹]	Thermal conductivity [W m ⁻¹ K ⁻¹]
Helium	0.179	161.0×10 ⁻⁶	5234	0.151
Air	1.29	15.8×10 ⁻⁶	1227	0.025

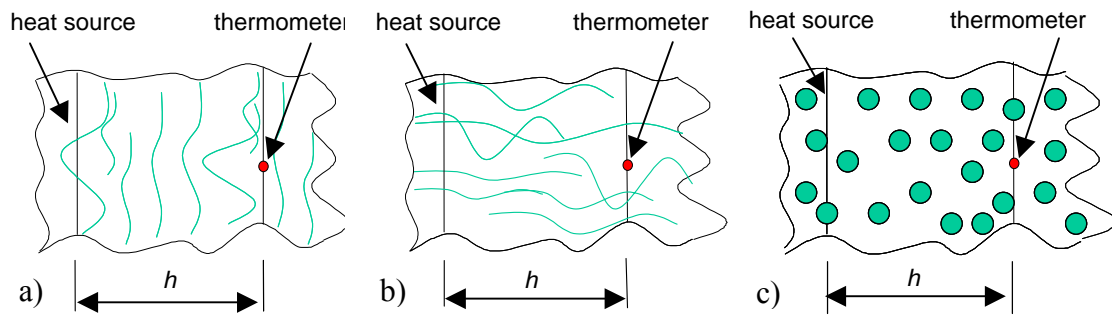


Fig. 2.: Model of the heat transport for in-homogeneous materials when pulse transient method is used. Composite SiC – C/C with orientation of fibres perpendicular (a) or parallel (b) to the heat flux, porous material (c).

Fig. 2a,b indicates the role of the fibers in the heat transport. Fibers composed parallel to the heat transport do not influence the dynamic of the heat transport while fibers composed suppress heat transport. Similar process indicates Fig. 2c for pores filled by high thermal conductivity gas having low heat capacity or by empty pores. Suppressed dynamic of the heat transport lowers magnitude of the temperature response (see Fig. 10) thus specific heat apparently grows considering relation (2).

5 Conclusions.

The paper discusses differences between values of the thermophysical parameters measured by classic and by transient methods. Analysis showed that agreement between data coming from single parameter and multiparameter methods can be found for homogeneous materials, only considering various measuring regimes. For heterogeneous materials data on specific heat differs between classic and transient methods.

The result of the analysis was verified by measurements of thermophysical parameters by pulse transient method on homogeneous and heterogeneous materials. Data given by steady state, equilibrium and dynamic measuring regime were inter-compared. Measurements of stainless steel A 310 and Perspex showed a satisfactorily agreement in data on specific heat (2.1 %), thermal diffusivity (3.3 %) and thermal conductivity (4.9 %). Data consistency $\lambda = ac\rho$ was satisfactorily fulfilled. Variations in published data on

optical glass BK 7 were found for densities and thermophysical parameters. Thus additional intercomparison has to be made between classic and transient methods.

Clear differences in specific heat data given by classic and by transient methods in heterogeneous materials were found. Data consistency is not fulfilled. Better agreement in specific heat data was found for measurements in parallel orientation to the fibers for composite C/C-SiC and in helium atmosphere for porous AAC. Carbon fibers as well as helium atmosphere have high thermal conductivity. Heterogeneity for composite is created by oriented fibers and for AAC by a broad distribution of the pore size. While for former case the role of fibers is not clear, in the latter helium the pores might represent a shortcut for heat flux skeleton. Relation between the effective value of the specific heat given by classic methods and the apparent value given by transient methods has to be found on the base of the macrostructure model.

The presented study might give recommendation for material standard preparation used for multiproperty and single property measurement methods that are often based on different measuring regimes. Clearly homogeneous materials can be used for standard preparation, only. A satisfactory agreement in data on thermophysical parameters was found in the thermal conductivity range 0.19 – 12.8 W/m K using classic – single parameter and pulse transient – multiparameter methods.

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