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Thermophysical Properties of Heterogeneous Structures Measured by Pulse Transient Method[1]

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This paper discusses differences in thermophysical parameters (thermal conductivity λ, thermal diffusivity a, and specific heat c) that can be found when experimental methods with different measuring regimes are used. Two classes of methods are compared, namely, classical methods using steady-state, equilibrium, and dynamic measuring regimes and transient methods. The data consistency formula $\lambda = ac\rho$ gives a picture on data reliability when single-parameter methods are used. Results of analysis are verified on published, recommended, and measured data by transient methods considering homogenous materials (stainless steel A 310, BK 7, Perspex) and heterogeneous materials (composite C/C–SiC, aerated autoclaved concrete). Satisfactory agreement on data for the thermophysical parameters was found on homogenous materials only.

KEY WORDS: heterogeneous materials; homogeneous materials; pulse transient method; specific heat; thermal conductivity; thermal diffusivity.

1. INTRODUCTION

Modern technology is looking for measuring methods that give reliable data of thermophysical parameters of broad classes of materials, preferably on small specimens in a short time. Attention is focused on measuring

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techniques that are used with materials in the form of bulk. Classical measuring methods such as guarded hot plate, adiabatic and drop calorimetry, DSC, flash, and Angström methods give one parameter only. All of the above mentioned methods have some limitations regarding specimen form, measuring regime, surrounding atmosphere, and/or temperature range.

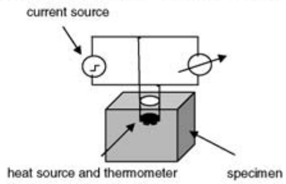
Recently a group of methods known as contact transient methods (CTM's) started to be used and have shown several advantages in comparison to classical methods [1]. The main advantage of the transient methods is that some of them, depending on the experimental arrangement, give a full set of thermophysical parameters, namely, specific heat, thermal diffusivity, and thermal conductivity or effusivity. Moreover, the specimen size can be chosen over a broad range; thus, materials can be examined that possess significant heterogeneity or porosity. The measuring regime of thermal analysis can be used, and various surrounding atmospheres can be applied when using appropriate experimental apparatus. However, introduction of these methods in everyday life underlined the importance of the development of a standard transient method.

Transient methods are based on generation of a non-stationary temperature field inside the specimen. The measuring process can be described as follows. The temperature of the specimen is stabilized. A small disturbance is applied to the specimen. From the temperature response to this small disturbance, the thermophysical parameters can be calculated according to the model used. Technically, the non-stationary temperature field is generated by the passage of an electric current through an electrical resistance that can be designed in the form of a wire, strip, foil, or a ball. A pulse of the heat or a heat flux in the form of a step-wise function can be used for generation of the dynamic temperature field. A temperature sensor either unified with the heat source (one-probe method) or placed apart from the heat source (two-probe method) measures the temperature response. Depending on the geometry of the specimen and the heat source and on the method for the temperature field generation, one or more thermophysical parameters can be obtained. Table I gives a summary of the basic experimental arrangements of the transient methods. The drawings show the shape of the probes, method for the generation of the temperature field, and the list of measured parameters. The same specimen setup can be used for several transient methods.

Implementation of the transient methods into various laboratories opened a question on how good is the agreement among data coming from various methods. This is especially important for multiparameter methods where individual parameters can be determined by independent single-parameter methods usually working with different measuring regimes. The

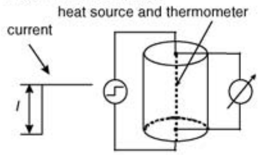
Table I. Transient Methods

Hot Ball Method – Needle Probe



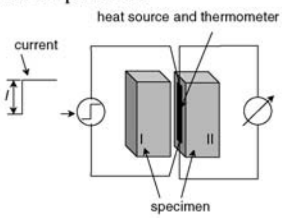
thermal conductivity

Hot Wire Method



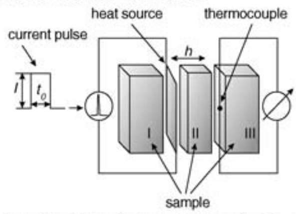
thermal conductivity

Hot Strip Method



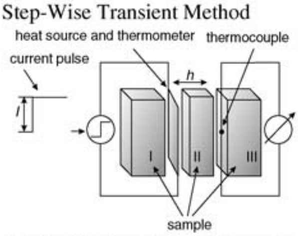
thermal conductivity

Pulse Transient Method



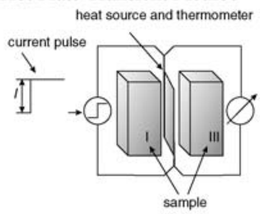
specific heat, thermal diffusivity, and thermal conductivity

Step-Wise Transient Method



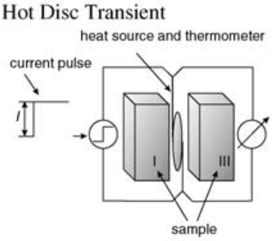
specific heat, thermal diffusivity, and thermal conductivity

Hot Plate Transient Method



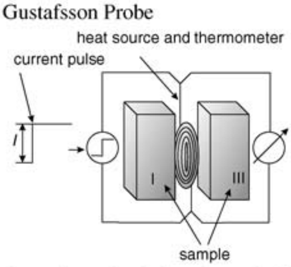
thermal effusivity

Hot Disc Transient



thermal conductivity, thermal diffusivity, and specific heat

Gustafsson Probe



thermal conductivity, thermal diffusivity, and specific heat present contribution deals with differences in values of the thermophysical parameters measured by classical methods and by transient methods.

Definitions of thermophysical parameters will be reviewed considering steady-state, equilibrium, and non-stationary measuring regimes, and possible variations of the measured values will be indicated. The analysis will be performed considering homogenous, anisotropic, and heterogeneous materials, which are often used in modern technology. The results of analysis will be verified by measurements of thermophysical parameters using a pulse transient method on homogenous and heterogeneous materials.

2. DEFINITION OF THERMOPHYSICAL PARAMETERS

A broad range of classical measuring methods needs to be included into any analysis considering the measuring regime and material structure. Generally, the classical 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 the relation $\lambda = ac\rho$ (ρ is density) when different methods are used for the determination of the individual parameters. This relation is valid for opaque solids only where heat transport by radiation is negligible. Contrary to classical methods, some transient methods can give all three parameters within a single measurement. Then a question arises on how good is the agreement in data coming from various methods. One might be surprised on whether the data consistency relation can be used when data coming from different measuring regimes are considered.

The experiments have shown that, for homogenous, isotropic materials, data consistency is satisfactorily fulfilled for classical methods. A critical analysis of the data consistency relation was performed on Pyroceram for a broad range of temperatures [2]. Good agreement was found among data determined by classical and transient methods, although sometimes difficulties can be found considering measurement accuracy for a broad range of materials [3]. Nevertheless, clear differences in data exist when porous or anisotropic materials are tested by both classical and transient methods [4].

Table IIa gives an overview on the most common experimental methods and measuring regimes when isotropic materials are investigated. A detailed analysis of Table IIa shows that, for homogenous, even anisotropic materials, satisfactory data consistency can be obtained, when any structural changes or chemical reactions are excluded. This is critical for specific heat data as its classical definition is made in a more general

Table IIa. Thermophysical Parameters and Measuring Principles Used for Homogenous (Isotropic/Anisotropic) Materials

Measuring regime	Parameter	Comment
Steady state	Thermal conductivity $\vec{q} = -\bar{\bar{\lambda}} grad(T)$	Experimental methods are operating with one-dimensional stationary temperature fields of axial or radial symmetry. Structural changes or chemical reactions have to be excluded.
Equilibrium	Specific heat $c_p = \frac{H_2 - H_1}{V\rho(T_2 - T_1)}$, H_1, H_2 – enthalpy ρ– density V - volume	A change of heat capacity resulting from a change of temperature can include various contributions (phononic, electronic, chemical reaction, structural change, etc.). Adiabatic and drop calorimeters and DSC are most frequently used.
Parameter is calculated	Thermal diffusivity $a = \frac{\lambda}{c_p \rho}$	Any structural change or chemical reaction has to be excluded, otherwise data consistency is violated.
Transient	Some of the methods give three parameters, i.e., specific heat, thermal diffusivity, and thermal conductivity.	Methods are based on non-stationary temperature field. Flash and Angström methods can be included into this class. Structural changes or chemical reactions have to be excluded.

way, i.e., it might include some structural changes. Thus the above-mentioned limitation regarding materials should be considered as a condition for standard material preparation when transient methods are used.

A significantly different picture can be found in Table IIb,c which is related to heterogeneous (porous, composite) materials and to classical as well as transient methods. Experimentally, specimen sizes are targeted for which measured parameters are not functions of any dimension. This criterion results in effective values of the thermophysical parameters. Data consistency is fulfilled when classical methods are used. However, a difference exists for data for the effective specific heat depending on whether equilibrium and transient methods are used. For the latter case, i.e., the case of transient methods, the specific heat is a apparent transport parameter. An apparent value of the specific heat is obtained that is different from that determined by equilibrium methods. This was confirmed by measurements of thermal transport properties of cast gamma-TiAl alloys using the Gustafsson probe where significant anisotropy was found [6].

Table IIb. Measuring Regimes and Parameters When Using Classical Methods for Heterogeneous (Porous, Composites) Materials

Measuring regime	Parameter	Comment	
Steady state	Effective value of thermal conductivity	Experimental methods are operating with one-dimensional temperature fields of axial or radial symmetry. Any structural changes or chemical reactions have to be excluded. Experimentally, specimen size is targeted for which measured parameter is not a function of any dimension. The effective value of the thermal conductivity is a complicated function of the macrostructure. Theoretically, the effective value can be calculated using an appropriate model. For special stochastic model the value is given in Ref. 5. $\sum_{i=1}^{n} w_i \frac{\lambda_i - \lambda_{\text{eff}}}{1+\frac{1}{g}\frac{\lambda_i-\lambda_{\text{eff}}}{\lambda_{\text{eff}}}} = 0,$	w_i – volume fraction of the i-th component that has thermal conductivity λ_i, g – threshold of percolation
Equilibrium	Effective value of the specific heat	A change of heat capacity resulting a change of temperature can include various contributions (phononic, electronic, chemical reaction, structural change, etc.). Adiabatic and drop calorimeters and DSC are most frequently used. Experimentally, specimen size is targeted for which measured parameter represents an effective value regarding macrostructure. Theoretically, the effective value of the specific heat can be calculated using a mixing rule. $c_{\text{p,eff}} = \sum_{i=1}^{n} \frac{M_i}{M} c_{\text{p},i} M = \sum_{i=1}^{n} M_i$	M_i– mass fraction of the i-th component that has specific heat $c_{\text{p},i}$
Parameter is calculated	Effective value of the thermal diffusivity	Any structural changes or chemical reactions have to be excluded, otherwise data consistency is violated. $a_{\text{eff,calc}} = \frac{\lambda_{\text{eff}}}{\bar{\rho} c_{\text{p,eff}}}$ $\bar{\rho} = \sum_{i=1}^{n} V_i \rho_i$	V_i, ρ_i – volume of the ith component

Table IIc. Measuring Regimes and Parameters When Using Transient Methods for Heterogeneous (Porous, Composites) Materials

Measuring regime	Parameter	Comment
Transient	Effective value of the thermal diffusivity	Methods are based on generation of a nonstationary temperature field. Flash and Angström methods can be included in this class. Any structural changes or chemical reactions have to be excluded. Experimentally, specimen size is targeted for which measured parameter is not a function of any dimension. The effective value of the thermal diffusivity is a complicated function of the macrostructure. Theoretically for a special stochastic model, the value is calculated by [5] $\sum_{i=1}^{n} w_i \frac{a_i - a_{\text{eff}}}{1+\frac{1}{g}\frac{a_i - a_{\text{eff}}}{a_{eff}}} = 0,$ w_i - volume fraction of the i-th component that has thermal diffusivity a_i, g - threshold of percolation
	Effective value of the thermal conductivity	Methods are based on generation of the nonstationary temperature field. Any structural changes or chemical reactions have to be excluded. Experimentally, specimen size is targeted for which measured parameter is not a function of any dimension. The effective value of the thermal conductivity is a complicated function of the macrostructure. Theoretically for a special stochastic model, the value is calculated by [5] $\sum_{i=1}^{n} w_i \frac{\lambda_i - \lambda_{\text{eff}}}{1+\frac{1}{g}\frac{\lambda_i - \lambda_{\text{eff}}}{\lambda_{\text{eff}}}} = 0,$ w_i - volume fraction of the i-th component that has thermal conductivity λ_i, g - threshold of percolation
	Apparent value of specific heat $c_{\text{app}} \neq c_{\text{eff}}$	Apparent value is measured by transient methods. Any structural changes or chemical reactions have to be excluded. Experimentally, specimen size is targeted for which measured parameter is not a function of any dimension. Theoretically, the apparent value using, i.e., special stochastic model can be calculated according [5] to $c_{\text{app,calc}} = \frac{1}{\rho}\frac{\lambda_{\text{eff}}}{a_{\text{eff}}}$

Theoretically, effective values can be calculated using thermophysical properties of the individual components and a selected model. Transport properties such as thermal conductivity or thermal diffusivity are a complicated function of the macrostructure while a simple mixing rule exists for specific heat calculations when classical methods are used. A more complicated situation exists when measuring methods based on non-stationary temperature fields are used. A relation between the effective and apparent values of the specific heat has to be determined using an appropriate macrostructure model.

3. EXPERIMENTAL DATA

The pulse transient method, shown in Fig. 1, was used for verification measurements to find agreement or differences in experimental data [7]. The specimen consists of three pieces where a planar heat source and a thermocouple are placed between the contact surfaces of the individual specimen parts. An electrical current produces a heat pulse in the planar electrical resistance to generate a non-stationary temperature field within the specimen. The temperature change (temperature response) is measured by a thermocouple. Then the thermal diffusivity can be determined using the relation,

$$a = \frac{h^2}{2t_{\mathrm{m}}}, \tag{1}$$

the specific heat using

$$c = \frac{Q}{\sqrt{2\pi\, exp(1)}\rho h T_{\mathrm{m}}}, \tag{2}$$

and the thermal conductivity using

$$\lambda = ac\rho = \frac{Qh}{2\sqrt{2\pi\, exp(1)}T_{\mathrm{m}} t_{\mathrm{m}}}, \tag{3}$$

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

3.1. Homogeneous Materials

Table III specifies the density and geometry of the tested materials. The geometry of the specimen setup was selected to minimize surface

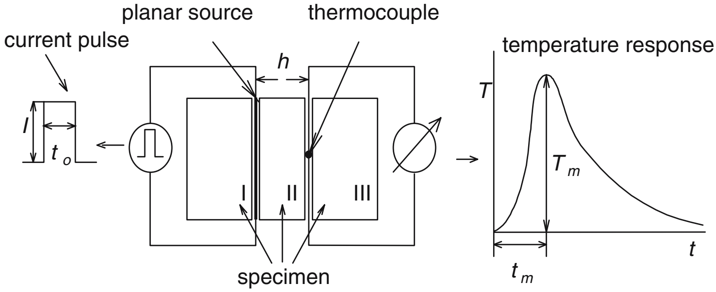


Fig. 1. Principle of the pulse transient method.

Table III. Geometry of the Tested Homogenous Materials

Material	Density ρ (kg·m^{-3})	Cross-section (mm)	Specimen thickness h (mm)	Thickness of specimen parts I/III (mm)
Stainless steel A 310	7902	Ø 20	4.9	10/8
Glass BK 7	2510	30×30	8	15/15
Perspex	1184	Ø 30	6	15/15

and contact effects. Table IV gives a summary of recommended/published values of thermophysical parameters that were obtained by classical methods as well as measured values using the pulse transient method. Recommended data for BK 7 includes values of thermal conductivity that were obtained by a comparative steady-state method using Pyrex and Pyroceram as certified materials [11]. The data obtained by the pulse transient method are within the range of the values found by the comparative steady-state method; however, the differences are too large (15%) and could be caused by a radiation effect due to data dependency on specimen thickness. Therefore, additional analysis needs to be carried out to find the reason for these differences. A full study made by different methods is also underway. The difference between the values given by classical and by transient methods on stainless steel A 310 and Perspex is within several percent, which might correspond to the uncertainty of the measurements.

Table IV. Published, Recommended, and Measured Values and Deviations of Thermophysical Parameters of Homogenous Materials, $T = 25°C$

Parameter	Data type	Stainless steel A310	BK7	Perspex
	Recommended	3.45 [8]	0.525 – 0.550 [9]	0.11 [12, 13]
Thermal diffusivity	Measured	3.35	0.546 [10]	0.110
(10^{-6} $m^2 \cdot s^{-1}$)	Deviation	−2.9%	–	+ 0.0%
	Recommended	471 [8]	760 – 810 [9]	1460 [12, 13]
Specific heat	Measured	469	767.9 [10]	1430
($J \cdot kg^{-1} \cdot K^{-1}$)	Deviation	−0.4%	–	−2.1%
	Recommended	12.8 [8]	1.02 – 1.18 [9, 11]	0.19 [12, 13]
Thermal conductivity	Measured	12.4	1.05 [10]	0.187
($W \cdot m^{-1} \cdot K^{-1}$)	Deviation	−3.1%	–	−1.6%

3.2. Heterogeneous Materials

Table V specifies both the density and geometry of the tested materials. The geometry of the specimen setup was selected to minimize surface and contact effects on experimental data. Two materials were chosen for the thermophysical analysis, namely a C/C–SiC composite and an autoclaved aerated concrete. The C/C–SiC composite was prepared by the liquid silicon infiltration process (LSI) using carbon fibers 7 μm in diameter. The final stage of the LSI process leads to a composite that was composed of a SiC matrix of 2.8% porosity and 60% carbon fibers. Details on composite preparation are published elsewhere, together with a study of variations of the physical properties on process parameters [14]. Autoclaved aerated concrete belongs to a family of porous materials having a porosity of 80 vol%. A broad range of pore distributions contains the skeleton of the autoclaved aerated concrete. The predominant part of these pores is open while a small fraction of micro-pores can be closed.

Table VI includes values of the thermophysical parameters that were obtained by classical methods (in parentheses) [14] as well as those obtained by the pulse transient method for the C/C–SiC composite. The composite is strongly anisotropic and two different orientations were chosen for analysis, namely, the parallel orientation to fibers and the transverse one. A difference of several percent was found between the published and the measured data for thermal conductivity in the transverse orientation to fibers. Large differences in transport properties (thermal conductivity and thermal diffusivity) were found between parallel and transverse orientations to the fibers. In addition, a large difference exists between the specific heat values measured in the parallel and transverse directions to

Table V. Geometry of the Tested Heterogeneous Materials

Material	Density ρ ($kg{\cdot}m^{-3}$)	Cross-section (mm)	Specimen thickness h (mm)	Thickness of specimen parts I/III (mm)
Composite C/C–SiC	2020	Ø 9.85	9.15	15/15
Aerated autoclaved concrete	512	150 × 150	15	40/40

Table VI. Published and Measured Thermophysical Parameters of C/C–SiC Composite, $T = 25°C$

Parameter	Transverse to fibers	Parallel to fibers	Difference
Thermal diffusivity (10^{-6} $m^2{\cdot}s^{-1}$)	6.25	10.78	+72%
Specific heat ($J{\cdot}kg^{-1}{\cdot}K^{-1}$)	(710)[14] 816	718.9	−12%
Thermal conductivity ($W{\cdot}m^{-1}{\cdot}K^{-1}$)	(10.5) [14] 10.24	15.73	+54%

the fibers. This indicates that the specific heat is an apparent 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 those found by the pulse transient method in the parallel direction to the fibers. A relation between the effective and apparent values of the specific heat must be found on the basis of a macrostructure model.

Table VII contains values of the thermophysical parameters of autoclaved aerated concrete that were obtained by classical methods as well as by a variant of the transient method (in parentheses) [15] and by the pulse transient method [4]. Agreement between the published and measured values might be limited due to the uncertainty in moisture content. A significant difference in thermophysical properties was found by the pulse transient method when different environments were used. Again, the gas in pores influenced the specific heat values. A mixing rule was used to test the contribution of the gas in pores to the volumetric specific heat. The calculated value in parentheses corresponds to the skeleton when no contribution from the pores exists (vacuum). The contributions of gases to the volumetric specific heat for a porosity of 80 vol% are negligible,

Table VII. Published and Measured Thermophysical Parameters of Autoclaved Aerated Concrete, $T = 25°C$. (Values in parentheses are calculated for skeleton in vacuum.)

Atmosphere	Thermal diffusivity (10^{-6} m^2·s^{-1})	Thermal conductivity (W·m^{-1}·K^{-1})	Specific heat (J·kg^{-1}·K^{-1})	Volumetric specific heat (J·m^{-3}·K^{-1})
Vacuum	0.2	0.115	984	(5.04×10^5)
Air, humidity ~30 %	(0.277) [15] 0.26	(0.135) [15] 0.141	(840) [16] 924	1270
Helium	0.4	0.2	867	750]

i.e., less than 1%. However, measured specific heat values change by about 11% using different gases. In addition, the measured specific heat value decreases with increasing thermal conductivity of the surrounding atmosphere and tends to reach the recommended value. The gas in pores significantly influences the heat transport through porous materials. Thermophysical properties for the gasses used here are given in Table VIII. The most reliable data on specific heat can be obtained for a gas having a high thermal conductivity and low heat capacity. Then the pores are a shortcut for heat flux and heat transport corresponding to a model of a quasi-homogeneous continuum. However, the relation between the effective and apparent values of the specific heat has to be determined on the basis of a macrostructure model.

4. CONCLUSIONS

The paper presents a study of transient methods. Some of them belong to the multiparameter measuring methods. Analysis of the measuring principles of thermophysical parameters showed that agreement between data coming from single-parameter and multi-parameter methods can be found for homogenous materials, only considering various

Table VIII. Thermophysical Properties for Air and Helium [17]

Atmosphere	Density (kg·m^{-3})	Thermal diffusivity (10^{-6} m^2·s^{-1})	Specific heat (J·kg^{-1}·K^{-1})	Thermal conductivity (W·m^{-1}·K^{-1})
Helium	0.179	161	5234	0.151
Air	1.29	15.8	1227	0.025

measuring regimes. For heterogeneous materials, data on the specific heat differs between classical and transient methods.

The results of the analysis were verified by measurements of thermophysical parameters with the pulse transient method on homogenous and heterogeneous materials. Measurements of stainless steel A 310 and Perspex showed a satisfactory agreement for specific heat (2.1%), thermal diffusivity (2.9%), and thermal conductivity (3.1%). Variations in published data on optical glass BK 7 were found for densities and other thermophysical properties. Thus, additional inter-comparisons must be made between classical and transient methods. Data determined by steady-state, equilibrium, and dynamic measuring methods were inter-compared and the consistency $\lambda = ac\rho$ was satisfactorily fulfilled.

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

This study might give recommendations for standard material preparation used in multi-parameter and single-parameter measurement methods that are often based on different measuring regimes. Clearly, only homogenous materials can be used for standards. Satisfactory agreement in data on thermophysical properties was found for the thermal conductivity range of 0.19 to 12.8 $W \cdot m^{-1} \cdot K^{-1}$ using classical — single-parameter and pulse transient — multi-parameter methods.

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