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Thermal Properties of Phase Change Materials

O. Zmeškal · L. Dohnalová

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Abstract This contribution deals with a study of the thermal properties of systems which consist of different types of materials: poly(methyl methacrylate) (PMMA), thermal insulator Aerotherm®, and phase change material Micronal®. The aim of this study is to evaluate the ability of these materials to decrease the working temperature of solar cells and therefore to increase their quantum efficiency. Step-wise and pulse transient methods were used for the determination of thermal parameters, such as the thermal conductivity, thermal diffusivity, specific heat, heat source quality, and heat losses. For the calculation of thermophysical parameters, a fractal theory of physical fields was used. For the determination of the parameters of the fractal model, a multiparametric regression method was used. The experimental and theoretical results are in good agreement.

Keywords Heat losses · Heat source quality parameter · PCM— phase change material · Specific heat · Thermal conductivity · Thermal diffusivity · Thermophysical properties

1 Introduction

Thermal parameters such as the thermal conductivity, thermal diffusivity, specific heat, heat source quality, and heat losses are important characteristics of solid-state materials [1,2]. Usually single phase materials are used for experimental studies [3,4]. In this work the material base was extended for a paraffin wax mixture in gypsum (Micronal® PCM), the material where a phase change were observed. According to

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O. Zmeškal (✉) · L. Dohnalová
Institute of Physical and Applied Chemistry, Faculty of Chemistry, Brno University of Technology, Purkyňova 118, 61200 Brno, Czech Republic
e-mail: zmeskal@fch.vutbr.cz

the composition of the mixture, three phase change temperatures can be obtained, namely, at (21, 23, and 26) °C [5]. On the contrary to commonly used methods, such as described in [6–8], we used modified transient step-wise and pulse methods [9,10] which were based on the fractal theory of a thermal field [11]. Experimental data were obtained during a heat process evoked by an external energy supply applied to the *planar heat source*. The first type of measurements was made at a constant reference temperature and variable heat power, the second type was based on a constant power and a variable reference temperature. The aim of this work was to determine thermophysical parameters of material with a phase change and compare them with those obtained on phase stable materials.

The analysis of the experimental results of a phase change material (PCM) showed that in this case, it was not an ideal surface source ($D = 2$), because its contact with the material surface was larger ($D \approx 2.6$), during the phase changes, and when the heat source is not only heating but also the phase changes, and the source dimension is larger during an intensive phase change and is even greater than three ($D > 3$). After converting thermal parameters for the ideal surface of the heat source, we get the real values which are tabulated for basic materials. The analysis of the experimental results for Aerotherm® showed that in this case it was not an ideal surface source ($D = 2$), because its thermal contact with the material surface was poor ($D \approx 1.9$).

Comparing with a material with tabulated values of thermophysical parameters, the principal of our method was demonstrated. The value of the thermal diffusivity characterized by our method was in very good agreement with the tabulated value. This method gives reproducible and correct results. That is the reason of using this method for other materials.

2 Theory

Thermophysical properties of materials [3,4] can be derived from a theory of fields based on the fractal character of space [12–14]. This theory is based, in general, on the theory of fields. The special cases of this theory are in agreement with the classical theories derived from the evaluation of the system of differential equations [6] for different boundary conditions: type of heat source (point, linear, and planar), diameter of heated material (infinite, defined shape, and size), type of heating (step-wise, pulse transient method), and type of material (homogeneous, heterogeneous) [15]. To the specimen, a rectangle (Dirac) pulse and step-wise heat pulse was applied, which caused an increase of temperature in the specimen. The dependence of the temperature increase on the given power with time was measured. From the fundamental measured temperature–time dependences, the thermophysical parameters of some materials were derived [3,4] along with the influence of boundary conditions.

The fractal theory characterizes generally how the temperature of a measured material is changed as a function of the heat supplied to the material. The trend of the temperature evolution with the amount of heat can be expressed by the parameter K (so-called fractal measure), and by the parameter D (so-called fractal dimension). The fractal measure K gives information about the average value of the physical quantity (about the distribution of the temperature in the sample). The fractal dimension

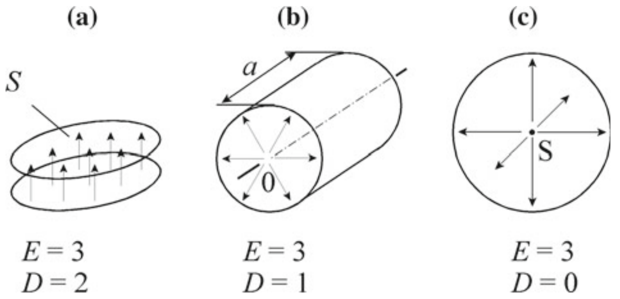

Fig. 1 Heat flow geometry for (a) plane-parallel, (b) cylindrical, and (c) spherical coordinates in Euclidean space

expresses the character of the heat transfer from the heat source into the material. The values of the fractal dimension $D = 0, 1, 2, 3$ describe the properties of the ideal heat sources (shown in Fig. 1); the other (non-integer) values characterize the real heat sources, $D \in \langle 0, 3 \rangle$ and the heat flow from the material to the surroundings.

In the ideal case the value of the fractal dimension for a planar heat source and cylindrical sample is $D = 2$. In comparison with this ideal situation, a D value higher than two ($D > 2$) means that the heat source has a larger surface than a planar one, the surface of the heat source is inhomogeneous, and it has good thermal contact with the material. A lower value of the fractal dimension ($D < 2$) suggests a smaller surface of the heat source in comparison with a planar one, the surface of the heat source is also inhomogeneous, and it has bad thermal contact with the material.

2.1 General Fractal Heat Transfer Model

The model was extracted from the fractal theory of a physical field [11]. The dependence of the difference of the temperature ΔT on the radius r of space-time for the thermal field can be written for this model as

$$\Delta T(r) = -\frac{\hbar c}{k_{\mathrm{B}}} \frac{K r^{D-E+2}}{D(D-E+2)}, \tag{1}$$

where $\hbar$ is the modified Planck constant, c is the speed of heat propagation, k_{B} is the Boltzmann constant, and E, D, K represent the Euclidean dimension, fractal dimension, and fractal measure, respectively.

On the basis of this equation, we distinguish two possible modes of the temperature distribution in a fractal medium. For a thermally non-conductive material with bad thermal contact with the heat source, the $(E - D)$ value is higher than 2, $E - D > 2$. In the special case in vacuum with the point heat source placed in 3D, in Euclidean space, we obtain $D = 0$. The prevailing means of heat transport occurs by radiation.

For a real gas, liquid, or solid material, $E - D < 2$. In the special case (ideal gas or solid with ideal crystalline structure), all atoms or molecules represent heat sources; so in 3D Euclidean space, we obtain where $D = E$ where $E = 3$. The dominant heat transport is, in this case, realized by convection and/or conduction.

The fractal measure K (in m^{-D}) characterizes the density of the fractal structure in the space. The fractal measure represents in 3D the average density of the thermal quanta in space for a point heat source in vacuum ($D = 0$) and the number of thermal quanta for an ideal gas or solid with an ideal crystalline structure ($D = E$).

If we define the heat source quality parameter as $s = (E - D)/2 = (a - a_0)/a$ and the power of the heat source in relation to the thermal conductivity of the real specimen characterized by fractal dimension D as

$$\frac{P}{\lambda} = -\frac{K\hbar c}{k_\mathrm{B}} \frac{1}{E - 2s} \left(\frac{\pi}{1 - s}\right)^s, \tag{2}$$

the change of temperature, dependent on time t after step-wise heating with power P, can be expressed by

$$\Delta T(t) = \frac{P(4\pi at)^{1-s}}{2\pi\lambda} \exp\left[-\left(\frac{h^2}{4at} + \frac{4at}{R^2}\right)\right], \tag{3}$$

where h is the thickness of the measured sample ($r^2 = h^2 - c^2t^2 + 4a_0t$ in notation of Eq. 1).

The thermal conductivity of a real material can be written as $\lambda = c_p\rho a$, where ρ is its mass density and c_p is the specific heat.

2.2 Method of Evaluation of Transient Measurement Data

The described regression method can be used for the determination of the parameters of a real fractal model which is described by Eq. 3,

$$\begin{aligned} y(t) = \ln\Delta T(t) &= \ln\left(\frac{P(4\pi a)^{1-s}}{2\pi\lambda}\right) + (1 - s)\ln t - \frac{h^2}{4at} - \frac{4at}{R^2} \\ &= m_0 + m_1x_1 + m_2x_2 + m_3x_3, \end{aligned} \tag{4}$$

where $x_1 = \ln t$, $x_2 = 1/t$, and $x_3 = t$. From the parameters m_0, m_1, m_2, and m_3, the thermophysical and other parameters of the measured system can be determined: the heat source quality $s = (E - D)/2 = 1 - m_1$ (or the fractal dimension D of the heat source in 3D space, $E = 3$), the heat loss parameter proportional to the measured sample diameter $R = h/\sqrt{m_2m_3}$, the thermal diffusivity $a = -h^2/4m_2$, and the thermal conductivity $\lambda = (P/2\pi)\left(-\pi h^2/m_2\right)^{m_1} \mathrm{e}^{-m_0}$. From the last two quantities and the known mass density ρ, the specific heat $c_p = \lambda/(\rho a)$ can be calculated.

3 Experimental

3.1 Measured Materials

A material with known thermophysical parameters—poly(methyl methacrylate) (PMMA) was first measured. The samples shown in Fig. 2 were disc-shaped with

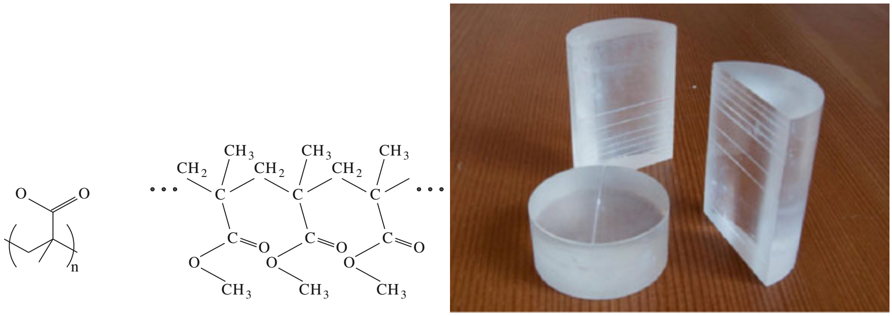

Fig. 2 PMMA—chemical structure (*left* image) and real specimen (*right* image)

a radius $R = 15\,\text{mm}$ and a thickness $h = 6\,\text{mm}$. The density of the material was $\rho = 1184\,\text{kg} \cdot \text{m}^{-3}$, the thermal diffusivity $a = 1.12 \times 10^{-7}\,\text{m}^2 \cdot \text{s}^{-1}$, the specific heat $c_p = 1450\,\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$, and the thermal conductivity $\lambda = 0.193\,\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$.

A studied material known as Aerotherm® [16] consists of aerogel (better insulator) with functional components of 3M glass balls (thin-walled glass microspheres containing rarefied air). Its main function is thermo-reflection and thermo-insulation.The disc-shaped sample had a radius $R = 15$ mm and a thickness $h = 16\,\text{mm}$. The density of the material was $\rho = 574\,\text{kg} \cdot \text{m}^{-3}$, and the thermal conductivity $\lambda = 0.047\,\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ (for an application thickness of 0.8 mm to 1.0 mm, table value).

The third part of the measurements was done on a PCM material (Micronal®) [5] (Fig. 3). This PCM is a composite material used for passive temperature stabilization. PCM technology is based on the use of latent heat from a phase change. The microcapsules of a solid polymer of a size from 2 µm to 20 µm are filled by wax with a predominant amount of hexadecane, see Fig. 3. According to the composition of the mixture of paraffin waxes, Micronal® PCM shows phase change temperatures at (21, 23, and 26) °C. The disc-shaped sample had a radius $R = 15\,\text{mm}$ and a thickness $h = 15\,\text{mm}$.

The time response of the specimen to the step-wise heat pulse applied from HP 6625a power supply to a heat source (20 µm thick Ni foil in Kapton®, $d = 30\,\text{mm}$) was measured. From the dependences of the temperature changes, the thermophysical parameters were determined.

The method presented in Ref. [8] was modified for better determination of the thermal parameters.

3.2 Experimental Arrangement

The Thermophysical Transient Tester 1.02 was used for measurements of responses to a heat pulse. It was developed at the Institute of Physics, Slovak Academy of Science [17]. The setup of the experimental arrangement is described in [8].

The first experiment was realized with a constant reference temperature $T_0 = 20\,°\text{C}$ and different heat powers (see Fig. 4, left). The heat power was increased every hour (or half an hour) and the responses were used for the determination of heat parameters

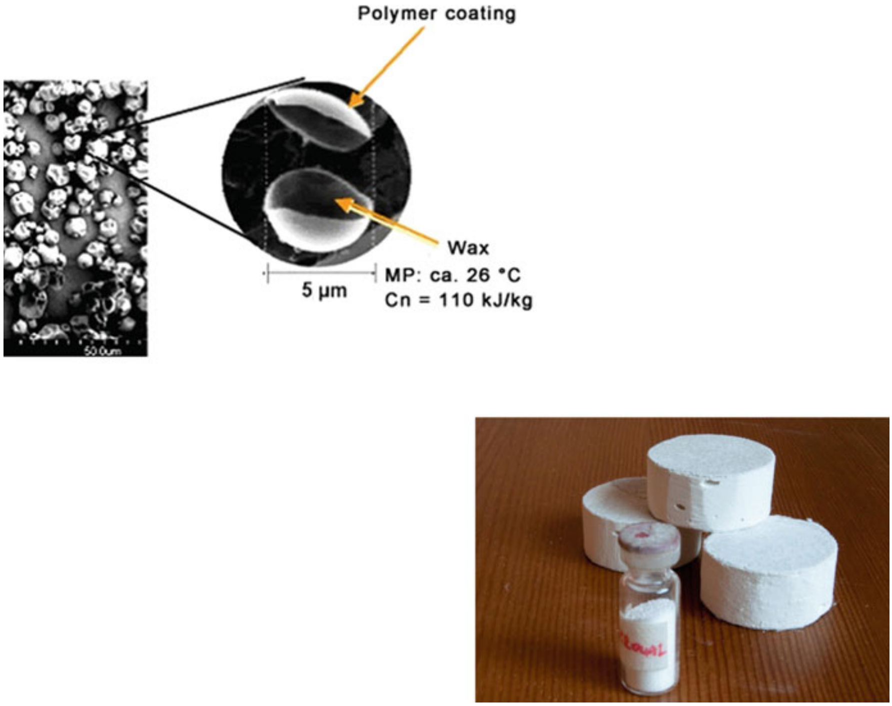

Fig. 3 Structure of microcapsules with phase change wax of Micronal® PCM [5] and real specimen (microcapsules of Micronal® PCM in gypsum)

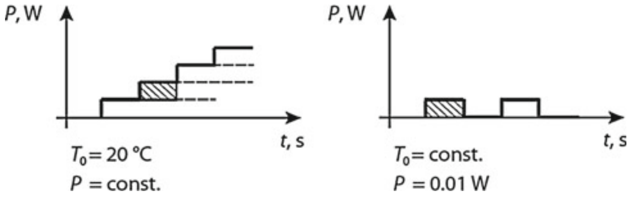

Fig. 4 Character of the heat pulse with variable power (*left*) and variable reference temperature (*right*)

after each applied pulse. This method was applied for all materials under study; we have here only the results for Aerotherm® (see Table 3).

The second experiment was realized for different initial conditions (steady state) within the temperature interval $T_0 \in \langle 20, 40 \rangle$ °C. The measurements were carried out at first in a 1 h cycle. That means that the temperature of the specimen was stabilized for 4 h; then the sample was treated by heating and cooling (1 h treatments, see Fig. 4, right).

The initial temperature of the sample was stabilized by a thermostat before each heating/cooling cycle. The sample temperature was measured by a thermocouple (K-type). This experiment was performed at first on the material with known thermophysical parameters (PMMA). Then the experiment with the same arrangement was applied to the specimen consisting of Micronal®.

4 Results

4.1 Parameter D

The parameter D defines a fractal dimension that gives information about the quality of the heat source. For all studied materials the fractal dimension was calculated by dragging a multi-parametric linear regression through the different positions in the time responses of the temperature changes. The average values were found for both heating and cooling processes for PMMA and PCM (Micronal®) as for $D > 2$. This suggests that the heating is drained away by a larger area than is the area of an ideal heat source. The fractal dimension for the PCM depends more dramatically on the temperature in the interval between 23 °C and 26 °C. It is caused by additional cooling or heating resulting from the phase change. The average values for Aerotherm® were found for $D < 2$. This suggests that the heating is drained away by a smaller area than is the area of an ideal heat source. Outside the phase change temperature, the value of the fractal dimension is again approximately 2, which characterizes the plane heat source. The approximated values of the fractal dimension equal to two, $D = 2$ (see Tables 1, 2, 3), were used for the calculation of the thermophysical parameters [18]. The analysis of experimental results showed that in neither case was there an ideal surface source ($D = 2$) (see Fig. 5), because its contact with the material surface was larger for PMMA ($D \approx 2.6$), and a smaller area for Aerotherm® ($D \approx 1.9$) and for Micronal® for the phase change temperature ($T > 28\,°\mathrm{C}$) $D \approx 6.2$ during the phase change. Here the heat source is a source not only for heating, but also for the phase transformation; the dimension of the source is greater when the intensive phase change is greater than three ($D > 3$), which suggests a very strong phase change.

Table 1 Thermal parameters calculated from measured characteristics during heating and cooling processes of PMMA ($h = 0.006\,\mathrm{m}$, $\rho = 1184\,\mathrm{kg \cdot m^{-3}}$)

PMMA	T_0 (°C)	a ($\mathrm{mm^2 \cdot s^{-1}}$)		R (m)		λ ($\mathrm{W \cdot m^{-1} \cdot K^{-1}}$)		c_p ($\mathrm{kJ \cdot kg^{-1} \cdot K^{-1}}$)	
		Up	Down	Up	Down	Up	Down	Up	Down
20 °C	19.98	0.069	0.051	0.042	0.034	0.136	0.118	1.653	1.943
22 °C	22.08	0.064	0.111	0.042	0.056	0.133	0.201	1.751	1.531
23 °C	23.06	0.104	0.063	0.060	0.041	0.142	0.144	1.570	1.713
24 °C	24.03	0.081	0.097	0.052	0.063	0.162	0.203	1.865	1.768
25 °C	25.01	0.065	0.072	0.043	0.046	0.171	0.155	2.137	1.881
25 °C	25.14	0.083	0.068	0.046	0.038	0.165	0.153	1.533	1.895
26 °C	25.97	0.127	0.069	0.091	0.045	0.224	0.146	1.466	1.959
27 °C	26.93	0.082	0.049	0.051	0.033	0.175	0.114	1.897	1.947
28 °C	27.82	0.227	0.095	0.125	0.057	0.274	0.175	1.018	1.564
29 °C	28.65	0.112	0.071	0.097	0.047	0.197	0.151	1.593	1.824
30 °C	29.78	0.042	0.067	0.053	0.049	0.174	0.155	3.219	1.863
35 °C	34.46	0.045	0.059	0.026	0.054	0.093	0.163	1.911	1.927

Recalculated dimension of heat source $D = 2$, heat power $P = 0.01\,\mathrm{W}$

Table 2 Thermal parameters calculated from characteristics measured during heating and cooling process of Micronal® ($h = 0.014\,\text{m},\ \rho = 693\,\text{kg} \cdot \text{m}^{-3}$)

Micronal®	T_0 (°C)	a ($\text{mm}^2 \cdot \text{s}^{-1}$)		R (m)		λ ($\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$)		c_p ($\text{kJ} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$)	
		Up	Down	Up	Down	Up	Down	Up	Down
18 °C	18.07	0.156	0.155	0.062	0.064	0.381	0.395	3.527	3.690
20 °C	20.09	0.129	0.129	0.056	0.053	0.309	0.324	3.235	3.633
21 °C	21.11	0.262	0.388	0.136	0.096	0.557	1.119	3.394	4.280
22 °C	22.02	0.180	0.171	0.080	0.084	0.609	0.537	4.397	4.894
23 °C	22.85	0.124	0.186	0.081	0.090	0.462	0.884	4.536	5.434
24 °C	23.80	0.078	0.060	0.028	0.037	7.847	1.523	1.703	12.000
25 °C	24.69	0.099	0.058	0.075	0.049	0.474	0.325	6.841	7.746
26 °C	25.67	0.242	0.416	0.048	0.047	2.263	6.786	24.338	19.012
27 °C	26.62	0.215	0.740	0.032	0.061	48.424	23.109	35.453	44.985
27 °C	27.48	0.328	0.077	0.105	0.045	1.962	0.673	10.615	10.369
28 °C	28.21	0.204	0.229	0.062	0.099	0.279	0.380	2.052	2.310
29 °C	29.16	0.217	0.264	0.064	0.067	0.313	0.364	2.315	2.029

Recalculated dimension of heat source $D = 2$, heat power $P = 0.017\,\text{W}$

Table 3 Thermal parameters calculated from measured characteristics of Aerotherm® ($h = 0.014\,\text{m},\ \rho = 574\,\text{kg} \cdot \text{m}^{-3}$)

Aerotherm®	P (W)	T_0 (°C)	a ($\text{mm}^2 \cdot \text{s}^{-1}$)	R (m)	λ ($\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$)	c_p ($\text{kJ} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$)
21 °C	0.083	20.6	0.22847	0.043	0.439	2.769
22 °C	0.087	21.7	0.21652	0.039	0.387	2.575
23 °C	0.090	23.0	0.21302	0.039	0.383	2.584
26 °C	0.162	26.4	0.21212	0.037	0.341	2.311
29 °C	0.213	28.7	0.21641	0.038	0.349	2.319
32 °C	0.167	31.9	0.20464	0.035	0.301	2.116
38 °C	0.099	38.1	0.14595	0.024	0.163	1.609

Recalculated dimension of heat source $D = 2$, the heat power P is a parameter of experiment

4.2 Thermal Diffusivity a

The thermal diffusivity a was studied for the thermal system with a reference material PMMA (results in Table 1), and on Micronal® PCM (results in Table 2)—both measurements were carried out using the variable reference temperature method (see Fig. 4, left); for Aerotherm® PCM (results in Table 3)—measurements were carried out using the variable power method (see Fig. 4, right). The comparison of the dependences of the thermal diffusivities of the materials under study on temperature is shown in Fig. 6. The determined diffusivity of PMMA is less than the theoretical value (dashed line) for both up and down heating steps. The value of the thermal diffusivity a for other studied materials is higher than that for PMMA. It implies that the rate of heat flow through the material is higher. The thermal diffusivity for PMMA and

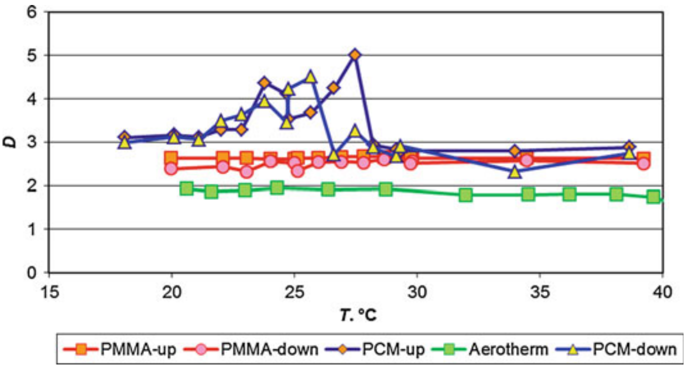

Fig. 5 Dependence of fractal dimension of heat source (heat source quality parameter $s = (E - D)/2 = (a - a_0)/a$) on temperature for PMMA, Aerotherm®, and Micronal® measured by multi-parametric regression method—fitting the whole transient response

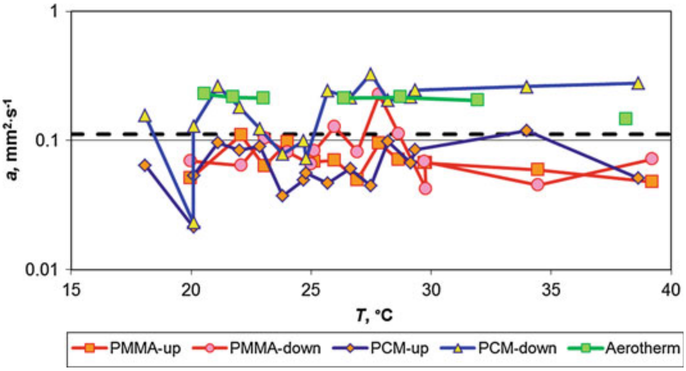

Fig. 6 Dependence of thermal diffusivity on temperature for PMMA, Aerotherm®, and Micronal® measured by modified multi-parametric regression method. PMMA (table value)—*dashed line*

Micronal® PCM is approximately two times higher than is the theoretical value, for Aerotherm® PCM, the value is about 2.5 times higher.

4.3 Parameter of Thermal Losses R

The thermal losses R were studied on pellets with a diameter $2R = 30\,\mathrm{mm}$ (PMMA—see the results in Table 1, Micronal® PCM—see the results in Table 2, and Aerotherm®—see the results in Table 3). The comparison of the dependences of thermal losses of the measured materials on temperature is shown in Fig. 7. The values of thermal losses R of all materials under study are approximately 2.5 times larger than the loss corresponding to the radius of the sample (dashed line). It follows that the losses play an important role during the heating of the sample.

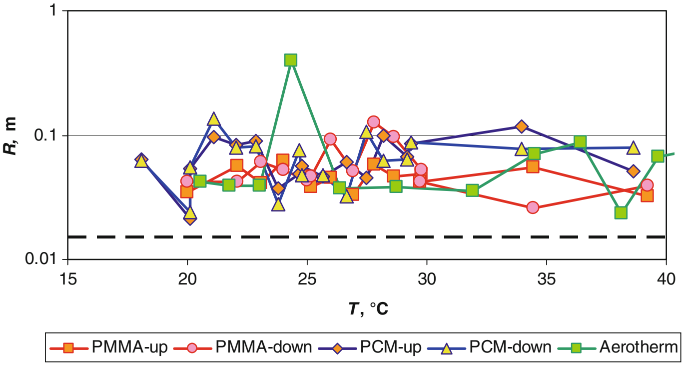

Fig. 7 Dependence of loss parameter on temperature for PMMA, Aerotherm®, and Micronal® measured by modified multi-parametric regression method. Radius of the sample—*dashed line*

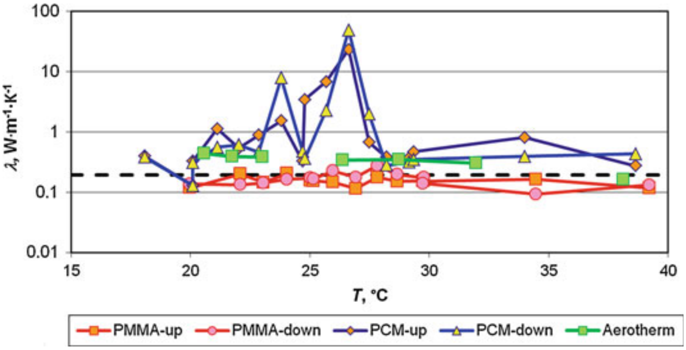

Fig. 8 Dependence of thermal conductivity on temperature for PMMA, Aerotherm®, and Micronal® measured by modified multi-parametric regression method. PMMA (table value)—*dashed line*

4.4 Thermal Conductivity λ

The results for the thermal conductivities λ of studied materials are summarized in Table 1 (PMMA), Table 2 (Micronal® PCM), and Table 3 (Aerotherm®). The comparison of the temperature dependences of the thermal conductivities of the studied materials is shown in Fig. 8. The value for PMMA is approximately the same as the table value (dashed line); for Aerotherm® and Micronal® PCMs, the values are higher.

The results of the thermal conductivity for PMMA are consistent with the theoretical values revaluated relative to the ideal planar source: $\lambda = 0.193\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$. The values of the thermal conductivity for Micronal® in gypsum after the phase change temperature are closer to paraffin ($\lambda = 0.25\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$) than to gypsum ($\lambda = 0.17\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$).

For Aerotherm® the reported thermal conductivity $\lambda = 0.047\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ (for an application thickness from 0.8 mm to 1.0 mm); for measured bulk materials the resulting values are about ten times larger, even though the components have

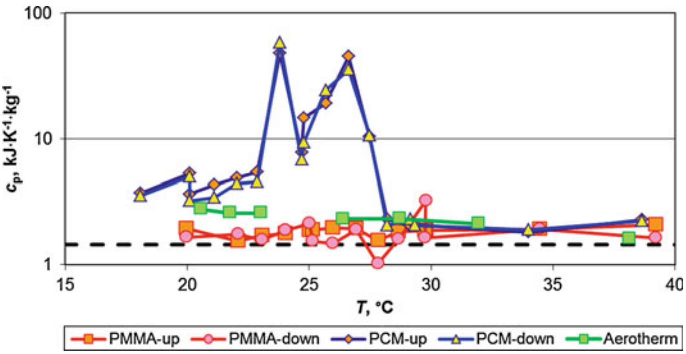

Fig. 9 Dependence of specific heat on temperature for PMMA, Aerotherm®, and Micronal® measured by modified multi-parametric regression method. PMMA (table value)—*dashed line*

very low thermal conductivity (silica aerogel $\lambda = 0.02\,\mathrm{W} \cdot \mathrm{m}^{-1} \cdot \mathrm{K}^{-1}$, fiberglass $\lambda = 0.04\,\mathrm{W} \cdot \mathrm{m}^{-1} \cdot \mathrm{K}^{-1}$.

4.5 Specific Heat c_p

The results of specific heat c_p measurements for all studied materials (PMMA, Aerotherm®, and Micronal® PCM) are summarized in Tables 1, 2, and 3. The comparison of the temperature dependences of specific heats of the measured materials is shown in Fig. 9. Experimental values are close to theoretical ones for Aerotherm®. Based on this experimental fact, we can state that there is no phase change in the used temperature range.

In the case of Micronal® PCM, the calculated values were about ten times higher than the theoretical ones. Thus, a more precise method must be developed for materials with such a phase change temperature.

The results of the heat capacity of PMMA are again in agreement with theoretical values revaluated to the ideal planar source: $c_p = 1.45\,\mathrm{kJ} \cdot \mathrm{kg}^{-1} \cdot \mathrm{K}^{-1}$. The values of the heat capacity for Micronal® in gypsum at a temperature above the phase change temperatures lie between the value of paraffin ($c_p = 2.90\,\mathrm{kJ} \cdot \mathrm{kg}^{-1} \cdot \mathrm{K}^{-1}$) and the value of gypsum ($c_p = 1.09\,\mathrm{kJ} \cdot \mathrm{kg}^{-1} \cdot \mathrm{K}^{-1}$). The heat capacity of Aerotherm® from our measurements is about four times greater than the tabulated value of the components (silica aerogel, $0.84\,\mathrm{kJ} \cdot \mathrm{kg}^{-1} \cdot \mathrm{K}^{-1}$; glass, $0.84\,\mathrm{kJ} \cdot \mathrm{kg}^{-1} \cdot \mathrm{K}^{-1}$).

5 Conclusion

Thermal properties of investigated materials were measured by two methods: (1) using constant reference temperature and variable power and (2) using constant power and variable reference temperature. The results of both methods were comparable and, therefore, both can be used for the determination of thermal parameters of materials. Experimental results showed relatively good conformity with theoretical values of thermal parameters for materials without phase changes. From the measured and calculated dependences of thermophysical parameters of PCM materials, the process of the phase change can be characterized. The values of thermophysical parameters were dramatically changed during the phase change. The values of the thermal conductivity and the specific heat increased significantly. A closer study of this process will be necessary. However, a more precise method must be developed for materials with a phase change temperature.

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