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EFFICIENCY OF THE NEEDLE PROBE TEST FOR EVALUATION OF THERMAL CONDUCTIVITY OF COMPOSITE MATERIALS: TWO-SCALE ANALYSIS

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Abstract: The needle probe test, as a thermal conductivity measurement method, has become very popular in recent years. In the present study, the efficiency of this methodology, for the case of composite materials, is investigated based on the numerical simulations. The material under study is a two-phase composite with periodic microstructure of "matrix-inclusion" type. Two-scale analysis, incorporating micromechanics approach, is performed. First, the effective thermal conductivity of the composite considered is found by the solution of the appropriate boundary value problem stated for the single unit cell. Next, numerical simulations of the needle probe test are carried out. In this case, two different locations of the measuring sensor are considered. It is shown that the "equivalent" conductivity, derived from the probe test, is strongly affected by the location of the sensor. Moreover, comparing the results obtained for different scales, one can notice that the "equivalent" conductivity cannot be interpreted as the effective one for the composites considered. Hence, a crude approximation of the effective property is proposed based on the volume fractions of constituents and the equivalent conductivities derived from different sensor locations.

Key words: thermal conductivity, composite, periodic microstructure, homogenization

1. INTRODUCTION

The methods for determination of the thermal conductivity of materials can be classified into two categories, namely: steady-state and transient heat transfer methods [1]. The significant factor that should be taken into account when choosing a method is time needed to perform the test. Steady-state methods usually require more time than transient ones, due to the fact that the material should be in a steady state during measurement procedure. One of the transient heat transfer methods is the needle probe test which has become very popular in recent years. Within this method a steel needle probe, as a heating source, embedded in the material specimen is utilized. During the test, the probe is heated and the temperature changes (for both heating and cooling phases) are recorded. In general, the probe can be considered as a transient-line heat source for which an analytical solution has been introduced by Carslaw and Jaeger [2].

The needle probe method has been utilized to find thermal conductivity of various materials, i.e., snow [3], materials from food industry [4], soils [5]–[7].

Nonetheless, [8] suggests that the probe test can also be used for composite materials with heterogeneous microstructure such as concrete. Due to the heterogeneity of the medium and contrast between thermal conductivities of its constituents, it can be presumed that the results of the test may be strongly affected by the location of the sensor. Therefore, in this paper the efficiency of the needle probe test, based on the numerical simulations, is examined.

The material under study is a periodic two-phase composite with the morphology of "matrix-inclusion" type. Two types of inclusions, with severely different thermal conductivities, are investigated. Different volume fractions of inclusions, obtained by the successive increase of inclusion diameter, are considered. Two positions of the sensor are simulated: in the center of the inclusion and at the largest distance from the inclusions. Acquired temperature versus time data are fit to the analytical solution [2] and the equivalent conductivity is evaluated. The efficiency of the test is assessed by comparison of the equivalent and the effective conductivities. The latter is determined based on the micromechanics approach — solution of the appropriate boundary value problem stated for the single unit cell.

The paper is organized as follows. In Section 2, the problem of evaluating effective thermal conductivity for periodic composites is briefly outlined. In addition, results of numerical simulations, based on the micromechanical approach, are presented. Section 3 provides a theory background concerning the problem of temperature increase of an infinite linear constant heat source within an infinite medium. Section 4 presents the results of the numerical simulation of the needle probe test, namely the equivalent thermal conductivities. In Section 5, the results of the thermal conductivities are compared and the efficiency of the needle probe method for composite materials is discussed. Final conclusions end the paper.

2. EFFECTIVE THERMAL CONDUCTIVITY OF COMPOSITE MATERIALS

2.1. FORMULATION OF THE PROBLEM

The primary objective of the micro-macro passage is to define, for a given heterogeneous medium, which possesses the property of statistical homogeneity, an 'equivalent' homogeneous material that has the same 'average' properties. In other words, the micromechanics approach is aimed at establishing an equivalent macroscopic description of a given physical process based on the description of the same phenomenon at the level of micro-inhomegeneities. Material constants of equivalent macroscopic description are referred to as the effective properties.

The micro-macro passage, i.e., derivation of a macroscopic description from that at the micro-level, consists in transforming the latter, through an appropriate averaging (over representative volume element – RVE) procedure, into a framework in which only the macroscopic variables (averaged variables) are employed. The complete transformation is obtained if and only if the boundary conditions between RVE and the rest of the material are specified. These boundary conditions should reflect, as closely as possible, the actual state of RVE within the medium considered [9]. Incorporation of specific boundary conditions in the local description is often referred to as the "closing hypothesis" [10]. In the paper, the composites with matrixinclusion morphology are considered. The investigation is limited to the case of periodic microstructure which means that the structure of composite is reconstructed based on the single unit cell being RVE (Fig. 1). This type of microstructure implies, in addition, periodic boundary conditions at the peripheries of the unit cell.

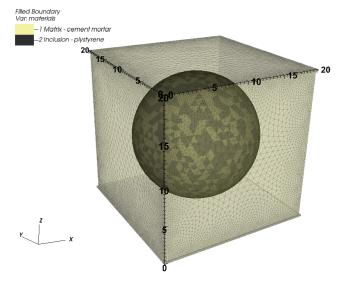


Fig. 1. Periodic unit cell for composites considered: 1 – matrix, 2 – spherical inclusion

In what follows the micro-macro transition for the case of steady-state heat flow process in a heterogeneous medium of volume V is briefly outlined. The micro-scale description of heat flow process consists of: constitutive equation (Fourier's law) for each phase of composite

$$f_i = -k \frac{\partial T}{\partial x_i} \quad \text{in } V \tag{1}$$

and the heat flux preservation law

$$\frac{\partial f_i}{\partial x_i} = 0 \quad \text{in } V \tag{2}$$

where f_i is an x_i component of the heat flux vector, T is the temperature, and k is the thermal conductivity coefficient. Note that matrix and inclusions are characterized by their own values of thermal conductivity k. The flux is said to be continuous at the interfaces between constituents.

The temperature at any point within the volume V can be defined as [11]:

$$T = \left\langle \frac{\partial T}{\partial x_i} \right\rangle (x_i - z_i^c) + \left\langle T \right\rangle + \overline{T} \ . \tag{3}$$

In the equation above <> is the volume averaging operator, $\langle \partial T/\partial x_i \rangle$ is the macroscopic gradient of temperature. The coordinates z_i^c specify the location of the centroid of RVE, while \overline{T} is so-called corrector term. The presence of corrector function in equation (3)

is the result of local heterogeneity of the medium considered. This function is constrained by $\langle \overline{T} \rangle = 0$, which can be formally proved by averaging expression (3).

Substituting relation (1), together with (3), in equation (2), one obtains

$$\begin{cases}
-\frac{\partial}{\partial x_i} \left(k \left\langle \frac{\partial T}{\partial x_i} \right\rangle + k \left\langle \frac{\partial \overline{T}}{\partial x_i} \right\rangle \right) = 0 \text{ in } V \\
\overline{T} - \text{locally periodic on } \partial V
\end{cases} \tag{4}$$

Noting that the boundary value problem (BVP) (4) possesses a linear form thus \overline{T} depends linearly on the macroscopic gradient, i.e.,

$$\overline{T} = A_i \left\langle \frac{\partial T}{\partial x_i} \right\rangle \tag{5}$$

where A_j is the solution of BVP (4) corresponding to the macroscopic gradient with its j-component equal to one and the remaining components equal to zero. Then, the formula which relates the local value of temperature gradient to its macroscopic counterpart can be established as

$$\frac{\partial T}{\partial x_j} = P_{ij} \left\langle \frac{\partial T}{\partial x_i} \right\rangle. \tag{6}$$

In the equation above, $P_{ij} = \left(\delta_{ij} + \frac{\partial A_i}{\partial x_j}\right)$ is referred

to as the localization operator. Relation (6) now allows the macroscopic heat flux to be expressed as a function of the macroscopic temperature gradient, i.e.,

$$\langle f_i \rangle = -k_{ij}^{\text{eff}} \left\langle \frac{\partial T}{\partial x_i} \right\rangle \tag{7}$$

where $k_{ij}^{\text{eff}} = \langle kP_{ij} \rangle$ represents the effective thermal conductivity coefficient of the composite.

It should be noted that in the case of random media, effective thermal conductivity k^{eff} is the ensemble average of the aforementioned local fields [11], [12].

2.2. NUMERICAL EVALUATION OF EFFECTIVE THERMAL CONDUCTIVITY BASED ON MICROMECHANICS

Effective thermal conductivities are determined for the matrix-inclusion composites considered using FEM. Two types of inclusions with severely different thermal conductivities are assumed, i.e., corresponding to the parameters of granite and polystyrene foam, respectively. Thermal conductivity coefficient of the matrix is constant and corresponds to the cement mortar parameter (k = 1.0 W/(mK)). The material constants for inclusions are listed in Table 1. The problem is investigated taking into account different volume fractions of the inclusions, namely: 20%, 25%, 30%, 35% and 40%. This is executed by the successive increase of inclusion diameter.

Each time the effective property is evaluated the boundary value problem (4) is solved. The solution is obtained using FlexPDE software [13]. As mentioned in the previous subsection, periodic boundary conditions, for the corrector term \overline{T} , are prescribed at the peripheries of RVE (Fig. 1). An example distribution of local fields (temperature and the heat flux) within periodic unit cell is shown in Fig. 2. Effective properties determined for both different conductivities and volume fractions of inclusions are summarized in Table 1.

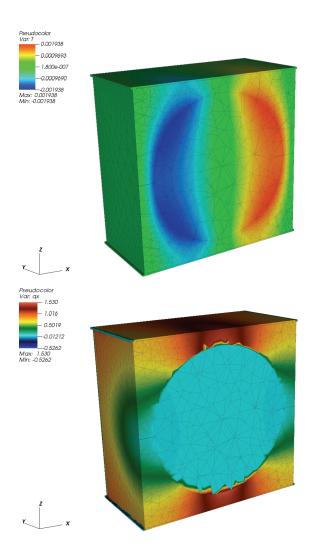


Fig. 2. Example distribution of local fields within periodic unit cell; (a) temperature field, (b) heat flux

Type of inclusion	Thermal conductivity k [W/(mK)]	Volume fraction of inclusions						
		0.20	0.25	0.30	0.35	0.40		
		Effective thermal conductivity k^{eff} [W/(mK)]						
Granite	2.365	1.200	1.255	1.312	1.371	1.433		
Dolystyrona foom	0.029	0.741	0.692	0.626	0.570	0.515		

Effective thermal conductivities for different types and volume fractions of inclusions

3. THEORY OF THE NEEDLE PROBE TEST

The needle probe test theory is based on the solution of heat transport problem induced by line heat source placed within an infinite, isotropic and homogenous medium of constant thermal diffusivity D. Thermal diffusivity D is defined as

$$D = \frac{k}{c_V} \tag{8}$$

where c_V represents volumetric heat capacity of the medium.

The solution of this problem corresponding to the case where the heat is produced from time t = 0 at a constant rate q per unit length of probe is presented, for instance, in [2]. The temperature increase is governed by the following expression [2]

$$\Delta T = -\frac{q}{4\pi k} \operatorname{Ei}\left(-\frac{r^2}{4Dt}\right),\tag{9}$$

where q is the heat input rate [W/m], r [m] is the radial distance from the line heat source, k and D are the thermal conductivity [W/(mK)] and the thermal diffusivity [m²/s] of the medium, respectively. The temperature change during cooling phase is [2]

$$\Delta T = \frac{q}{4\pi k} \left[-\operatorname{Ei}\left(-\frac{r^2}{4Dt}\right) + \operatorname{Ei}\left(-\frac{r^2}{4D(t-t_h)}\right) \right], \quad (10)$$

where t_h denotes the heating time [s]. The Ei symbol, which is present in the equations above, stands for the exponential integral, the special function on the complex plane, which is defined by the following expression [14]

$$-\operatorname{Ei}(-\alpha) = \int_{\alpha}^{\infty} \frac{e^{-t}}{t} dt . \tag{11}$$

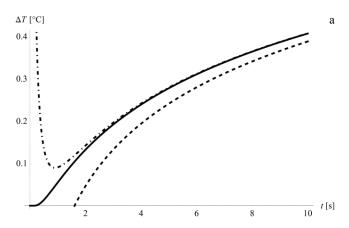
The function given by equation (11) can be approximated by the series expansion [15], i.e.,

$$-\operatorname{Ei}(-\alpha) = -\gamma - \ln \alpha - \sum_{n=1}^{\infty} \frac{(-\alpha)^n}{nn!}$$
 (12)

Table 1

where $\gamma = 0.5772$... is Euler's constant.

In Fig. 3, the temperature increases ΔT for two different time intervals, namely 0–10 seconds (Fig. 3a) and 50–60 seconds (Fig. 3b), are graphically presented. The results are obtained with the use of thermal properties usually observed for clayey soils [6], i.e., $D = 0.4 \text{ [mm}^2/\text{s]}$ and k = 1.5 [W/(mK)] as well as the standard radius of the needle probe [8], i.e., r = 1.2 [mm].



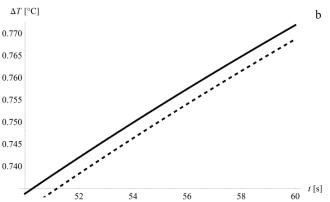


Fig. 3. The temperature increase ΔT for two different time intervals. Continuous line – analytical solution (eq. (9)), dashed line – approximation by the series expansion omitting the sum in eq. (12), dot-dashed line – approximation by the series expansion with one term of the sum (eq. (12))

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The continuous line corresponds to the analytical solution given by equation (9). The dashed and dot-dashed lines represent the approximations of equation (9) with the use of series expansion – equation (12) in which: the terms beyond $\ln \alpha$ are omitted and one element of the sum is only taken into account, respectively. It can be seen that for early times (0–10 s) the difference between analytical solution and approximations is quite significant. For longer times (50–60 s) the difference between analytical solution and approximations becomes negligibly small. Note that in Fig. 3b the continuous as well as dot-dashed lines overlap.

Therefore, as the coefficient $\alpha = r^2/(4Dt)$ becomes smaller and smaller – for long times – the terms beyond $\ln \alpha$ in the series expansion of Ei (eq. (12)) become negligibly small and then ΔT is linearly related to $\ln t$. In other words, equation (11) can be approximated as

$$\Delta T \approx \frac{q}{4\pi k} \ln t + C, \quad 0 < t < t_h, \tag{13}$$

where C is a constant. If a similar approach is applied, in addition, to the cooling phase it can be shown that the measurement data can be fit to the following equations [8]

$$T = m_0 + m_2 t + m_3 \ln t \tag{14}$$

for heating, and

$$T = m_1 + m_2 t + m_3 \ln \left(\frac{t}{t - t_h} \right)$$
 (15)

for cooling phase. In the equations above, m_0 can be treated as the ambient temperature, m_2 is the rate of background temperature drift, and m_3 is the slope of a line that relates temperature increase to $\ln t$. Since equations (14) and (15) are long time approximations it is proposed to ignore early time data – only the final 2/3 of all the data collected (during heating and cooling) are used for fitting [8]. While the parameters m_0 , m_1 , m_2 and m_3 are found (here the least squares analysis using Mathematica [14] is performed), the conductivity is evaluated using the following relation

$$k = \frac{q}{4\pi m_2}. (16)$$

4. NUMERICAL SIMULATION OF THE NEEDLE PROBE TEST FOR COMPOSITE WITH PERIODIC MICROSTRUCTURE

In this section, a numerical 3D model of the needle probe test is presented. The main aim of the

test is to identify the field of temperature in the medium investigated and to evaluate thermal conductivity based on the results obtained. The problem is solved using finite element method in Flex-PDE software.

4.1. STATEMENT OF THE PROBLEM

The material considered is a periodic two-phase composite with the morphology of "matrix-inclusion" type. The inclusions are postulated to be of a spherical shape. The structure of the composite is generated by periodicity from the unit cell (Fig. 1) being a cube with sphere located in the center. The total dimensions of the volume considered are $10 \times 10 \times 12$ cm. An example 3D view of the model is presented in Fig. 4.

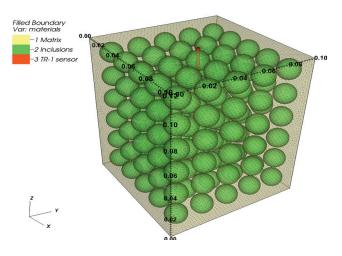


Fig. 4. An example 3D model

The needle is modeled as composed of a homogeneous material with a volumetric heat source of the power of about 9 kW/m³. This corresponds to the power of the original TR-1 sensor with the linear heat source of 4 W/m [8]. Even though the simplified geometry of the needle does not reflect the complexity of the real TR-1 sensor, it does not have any qualitative influence on the results of the study. The diameter of the needle is 2.4 mm. The thermal properties, i.e., thermal conductivity coefficient and volumetric heat capacity, have been estimated using the glycerin sample as the reference one, for calibration. The thermal conductivity coefficient for the needle is assumed as 10 W/(mK) and volumetric heat capacity as 2.5 MJ/(m³K).

The mathematical description constitutes the following system of equations

$$c_{V3} \frac{\partial T}{\partial t} - k_3 \nabla^2 T = R[H(t) - H(t - t_n)] \text{ in } \Omega_3,$$

$$c_{Vi} \frac{\partial T}{\partial t} - k_1 \nabla^2 T = 0 \text{ in } \Omega_3 \quad i = 1, 2,$$
(17)

where T denotes temperature, R stands for volumetric heat source of the needle, H(t) – Heaviside function, Ω_3 is volume occupied by the needle, and Ω_i is volume occupied by matrix (i = 1) or inclusions (i = 2), respectively.

Dirichlet type of boundary condition is prescribed: the room temperature is postulated. The problem is non-stationary. The t_h heating stage is followed by the "cooling" phase (no heat source). All the simulations are performed using FEM, namely FlexPDE software. Typical volume discretization consists of approximately 1 million elements. The 3D meshing operation is carried out by using second order finite elements.

Due to the complexity of the problem studied, the considerations have been limited to the two particular cases, corresponding to the extreme positions of the sensor (Fig. 5):

- the needle passes through the inclusions and the temperature sensor is located in the center of the inclusion (case "1"),
- the needle is placed in the matrix and sensor is at the longest distance from the inclusions (case "2").

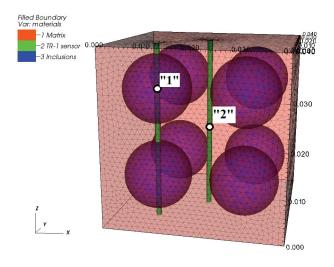


Fig. 5. Positions of the sensor considered in numerical simulations

4.2. RESULTS OF CALCULATIONS

A series of tests have been performed in order to obtain the temperature versus time distributions. For every volume fraction of the inclusions, the temperature diagrams are acquired for the two extreme positions of the sensor. Figure 6 presents an example tem-

perature distribution in the cooling phase (t = 200 s) for one of the cross-sections of the model.

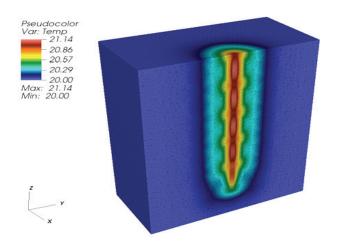


Fig. 6. Temperature distribution in the model with 25% of the polystyrene foam inclusions

The temperature results are compared for two situations investigated. In Figure 7, temperature versus time curves for the medium with 25% of polystyrene foam inclusions are shown.

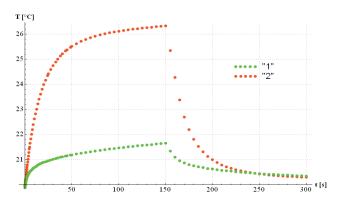


Fig. 7. Temperature curves for the medium with 25% polystyrene foam balls;
"1" – sensor in the center of the inclusion,
"2" – sensor at the longest distance from the inclusion

It can be noticed that the temperature is strongly affected by the position of the sensor. The more heterogeneous the medium is, the stronger the effect is observed.

4.3. EVALUATION OF THE EQUIVALENT THERMAL CONDUCTIVITY

Based on the temperature distributions mentioned in the previous section, the 'equivalent conductivity' coefficient value is calculated using the analytical solution presented in Section 3 (equations (14) and (15)). This implies that initially heterogeneous medium is replaced by the equivalent homogeneous one which leads to the same temperature distribution. Figures 8 and 9 show the fitting procedure for the temperature curves and the result of equivalent thermal conductivity coefficient for the composite with 25% of polystyrene foam inclusions.

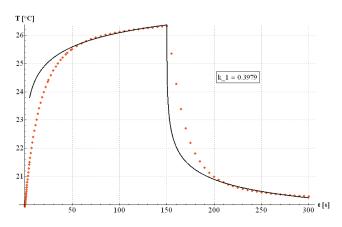


Fig. 8. Temperature distribution and fitting curve for the sensor position "1"

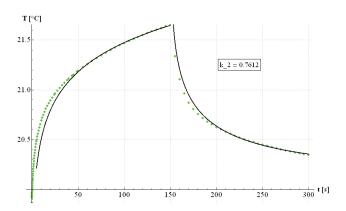


Fig. 9. Temperature distribution and fitting curve for the sensor position "2"

It is obvious that the "equivalent" conductivity does not represent a material property since it depends on the position of the sensor on the contrary to the "effective" conductivity which is the material property.

5. EVALUATION OF THE EFFECTIVE THERMAL CONDUCTIVITY BASED ON THE NEEDLE PROBE TEST

Due to the fact that the medium is statistically homogeneous, an average value of the adequate number of measurements should correspond to the effective value of the thermal conductivity parameter. According to the Central Limit Theorem, for the significance level of 5%, the number of trials satisfies the following inequality

$$n \ge \left(\frac{1.96\sigma}{k^{\text{eff}}\varepsilon}\right)^2 \tag{18}$$

where n is the number of trials (independent measurements), σ corresponds to the standard deviation which in this case can be evaluated by the difference between maximal and minimal value of equivalent thermal conductivity coefficient, $k_{\rm eff}$ is the effective thermal conductivity of the medium and ε is assumed as the tolerance error. For the medium with 40% granite inclusions the number of trials is estimated as (see Tab. 2)

$$n \ge \left(\frac{1.96 \cdot (1.452 - 1.383)}{1.433 \cdot 0.05}\right)^2 = 4. \tag{19}$$

Nonetheless, for the medium with 40% of polystyrene foam inclusions the number of trials equals 1240. It is obvious that for the material with inclu-

Table 2

Values of the thermal conductivity coefficients

261	Volume	"1"	"2"	k eff "1+2"	k _{eff}	$\Delta k_{ m eff}$
Material	fraction of inclusions	Thern	[%]			
	20%	1.219	1.188	1.194	1.200	0.51%
	25%	1.271	1.237	1.246	1.255	0.75%
Granite	30%	1.326	1.289	1.300	1.312	0.87%
	35%	1.385	1.339	1.355	1.371	1.13%
	40%	1.452	1.383	1.411	1.433	1.53%
	20%	0.496	0.819	0.755	0.741	1.80%
	25%	0.398	0.751	0.663	0.682	2.86%
Polystyrene foam	30%	0.313	0.732	0.606	0.626	3.12%
	35%	0.246	0.685	0.531	0.570	6.80%
	40%	0.204	0.649	0.471	0.515	8.62%

sions of particularly different thermal properties, the number of trials is extremely high, which makes the test ineffective and useless. Therefore, the modification of the test interpretation is needed. Crude evaluation of the expectation value defined as

$$k_{"1+2}^{\text{eff}} = \phi k_{"1"} + (1-\phi)k_{"2"}$$
 (20)

finally leads to a good approximation of the effective thermal conductivity. The formula presented above uses the volume fractions of the constituents, i.e., ϕ – volume fraction of inclusions and $(1 - \phi)$ – matrix volume fraction and equivalent thermal conductivities corresponding to the needle in the inclusion ("1") and needle in the matrix ("2").

6. CONCLUSIONS

In the paper, the efficiency of the needle probe test for evaluation of thermal conductivity for composite materials has been studied. The considerations are limited to the case of periodic composites with "matrix-inclusion" morphology. The composites of various inclusion volume fractions and different thermal conductivities of constituents have been investigated. The efficiency of the needle probe method was analyzed based only on the numerical simulations. The following conclusions can be drawn from the present study:

- (i) the temperature distribution is strongly affected by the location of the sensor; different temperature versus time functions were obtained for the sensor embedded in the center of the inclusion and the one placed at the longest distance from the inclusions (Fig. 7),
- (ii) the more heterogeneous the medium is, the more significant the difference in the temperature distribution is,
- (iii) the "equivalent" conductivities, determined by fitting (eqs. (14) and (15)) to the temperature versus time data, also depend on the sensor location,
- (iv) the "equivalent" conductivities cannot be interpreted as the effective ones,
- (v) the "equivalent" conductivities are influenced by the inclusion volume fractions as well as the contrast in thermal conductivities of the composite constituents,
- (vi) the crude approximation of the effective property is proposed based on the volume fractions of constituents and the equivalent conductivities derived from different sensor locations (eq. (20)),

(vii) the current study, as a preliminary one, did not take into consideration the influence of the "scale" effect (e.g., the influence of the inclusion to needle diameter ratio, increasing the number of inclusions maintaining the same volume fraction, etc.) on the results obtained.

(viii) the results of the present study can be treated as a base for future research in this area in order to derive a generalized approach for determination of thermal conductivity of heterogeneous materials.

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