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A study of thermal diffusivity of carbon-epoxy and glass-epoxy composites using the modified pulse method

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Abstract Transient heat transfer is studied and compared in two plane-parallel composite walls and one EPIDIAN 53 epoxy resin wall acting as a matrix for both composites. The first of the two walls is made of carbon-epoxy composite; the other wall is made of glass-epoxy composite, both with comparable thickness of about 1 mm and the same number of carbon and glass fabric layers (four layers). The study was conducted for temperatures in the range of 20–120 °C. The results of the study of thermal diffusivity which characterizes the material as a heat conductor under transient conditions have a preliminary character. Three series of tests were conducted for each wall. Each series took about 24 h. The results from the three series were approximated using linear functions and were found between $(0.7–1.35)\times10^{-7}\,\mathrm{m^2/s}$. In the whole range of temperature variation, the thermal diffusivity values for carbon-epoxy composite are from 1.2 to 1.5 times higher than those for the other two materials with nearly the same thermal diffusivity characteristics.

Keywords: Thermal diffusivity; Pulse method; Flash method; Thermophysical properties; Carbon-epoxy composite; Glass-epoxy composite; Epoxy resin

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Nomenclature

a	–	thermal diffusivity, m^2/s
c	–	specific heat, J/(kgK)
d	–	diameter, mm
l	–	thickness of the investigated sample, m
t	–	time, s
T	–	temperature, °C, K
T_0	–	reference temperature, °C, K
x	–	Cartesian coordinate, m

Greek symbols

$\Theta = T - T_0$	–	temperature, K
Θ_∞	–	temperature increase of the opaque samples after the time of the temperature equalisation on its opposite surfaces, respectively, K
Θ_1	–	temperature on the front face of the sample
Θ_2	–	temperature on the rear face of the sample
ρ	–	density, kg/m^3
$\tau = l^2/(\pi^2 a)$	–	characteristic time, s

1 Introduction

The aim of this study was to compare heat transfer characteristics in carbon-epoxy and glass-epoxy composites used in aviation engineering. Competence in calculating heat flux densities and heat transfer fluxes as well as temperature maximum values is of a critical significance in the design and in-service performance of aerostructure components exposed to high thermal loads [1,2,3]. This refers mainly to the fasteners that fasten heat engines to composite housings. When heat transfer in the wall is transient, the physical quantity that has a determinative influence on the heat transfer enhancement is thermal diffusivity, $a(T)$. A typical composite wall consists of a matrix and the other component – reinforcement, placed in the matrix – with much better mechanical properties. The matrix in both walls considered here was made of polymer, whereas carbon fibres or glass fibres made up the reinforcement. Since each wall has a non-uniform structure, the thermal diffusivity values determined are their substitutes and relate to the wall as a whole (with certain thickness, reinforcement structure, ...).

Measurements of thermal diffusivity of selected composite walls were made using the modified pulse method [6,7,8]. Two of the walls chosen for the study were made of carbon-epoxy and glass-epoxy composites, and the third one, the polymer wall constituted the matrix for the two composite

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walls. Temperature characteristics of the equivalent thermal diffusivity of the carbon-epoxy and glass-epoxy composite samples were determined in the temperature range of 290–393 K (17–120 °C).

2 Brief description of the method used to study transient heat transfer in composite materials and the experimental setup

The principle of determining the thermal diffusivity of solids using the modified pulse method consists in measuring the change in the difference between the temperatures of the front and rear faces of the plane-parallel and adiabatic sample treated with radiation pulse on one of the extreme surfaces. Then the appropriate program is used to process the resulting course of temperature difference changes, and the end result of this procedure is the sought value of thermal diffusivity. This method has been described in detail in [6,7], and its outline is illustrated in Figs. 1 and 2. The procedure of determining thermal diffusivity a using the modified pulse method is as follows:

- Theoretical identification of the temperature distribution as a function of time in the material of plane-parallel, opaque sample and determination of the temperature difference on its extreme surfaces based on the adopted one-dimensional model of heat transfer. This dependence has the following form:

$$\Delta\Theta(t) = \Theta_1(t) - \Theta_2(t) = \Theta(0,t) - \Theta(l,t) = 4\Theta_\infty \sum_{n=1}^{\infty} \exp\left[-\frac{(2n-1)^2 t}{\tau}\right] \tag{1}$$

where: Θ_∞ is a sample temperature rise after the transient process of temperature equalization inside its volume; Θ_1 and Θ_2 are the changes in temperature on the front and rear faces of the sample, respectively, and

$$\tau = \left[(l/\pi)^2\right] a^{-1} \tag{2}$$

is the characteristic time. If the relation (1) can be replaced by

$$\Delta\Theta_{n=1}(t) = 4\Theta_\infty \exp\left(-\frac{t}{\tau}\right) \tag{3}$$

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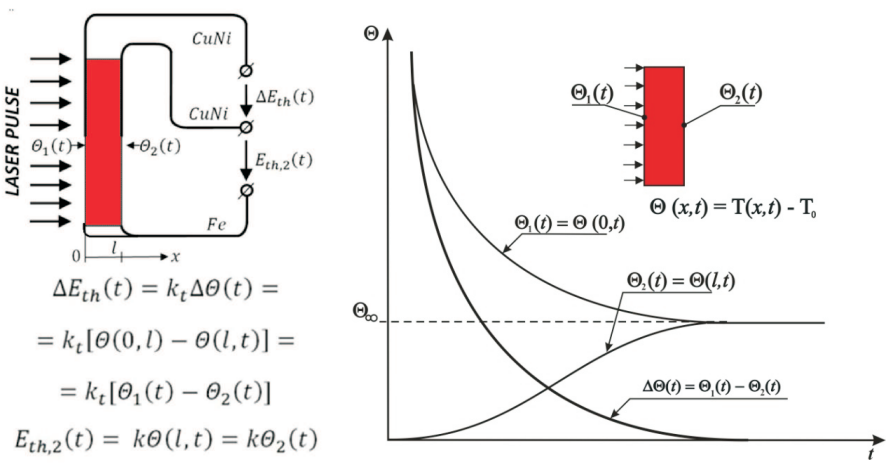
the resulting error is less than 1%. Expression (3) was used on the basis of the changes $\Delta\Theta'(t)$, obtained from the experiment, for determining the characteristic time, τ, and the thermal diffusivity, a, of the investigated sample (Fig. 2).



a) b)

Figure 1: a) The principle of temperature $\Theta_2(t)$ and temperature difference $\Delta\Theta(t)$ measurements between the extreme surfaces of the same sample: k_t – Seebeck constant of thermocouple 'CuNi – material of the sample'; k – known value of the Seebeck constant of thermocouple 'Fe – CuNi'; b) typical plot of changes in temperature on the front $\Theta(0,l)$ and rear $\Theta(l,t)$ faces of the sample and the difference between these values $\Delta\Theta(t)$ on the extreme surfaces of the sample after the absorption of radiation energy of the laser pulse on the front face [4–7], ΔE_{th} – e.m.f. of the differential thermocouple, $\Delta E_{th,2}$ – e.m.f. of the 'Fe-CuNi' thermocouple, $T(x,t)$ – temperature distribution in the sample $(0 \le x \le l)$ at time t.

- Experimental recording of the changes in temperature difference $\Delta\Theta'(t)$, between the front and the rear face of the sample, induced by the one-dimensional process of temperature equalization in the sample, after the laser shot onto the front face.
- Identification of the proper curve $\Delta\Theta(t)$, from the set that was the theoretical solution of this problem, with curve $\Delta\Theta'(t)$ obtained from the experiment; the sought value of thermal diffusivity a was subject to change in the identification process.

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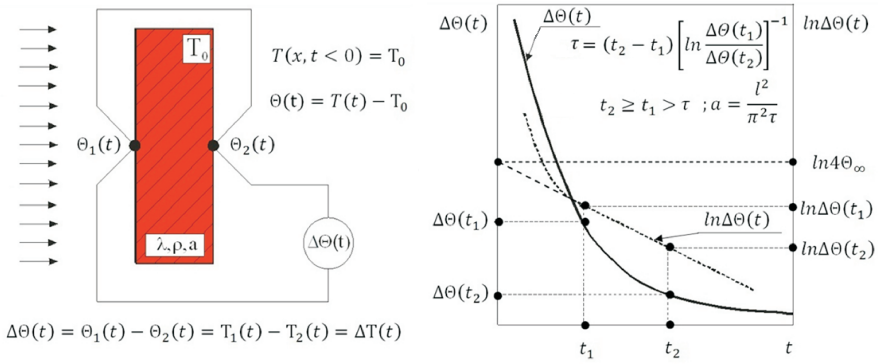

Figure 2: The principle of determining thermal diffusivity a by the modified pulse method, based on the changes in temperature difference $\Delta\Theta(t) = T_1(t) - T_2(t) = \Theta_1(t) - \Theta_2(t)$ on the extreme surfaces of the sample [6,7].

3 Measurements and results

Carbon-epoxy and glass-epoxy composite samples used for measuring their equivalent thermal diffusivity as well as the epoxy resin sample (matrix) used for measuring thermal diffusivity using the modified pulse method were made at the Polish Air Force Academy in Dęblin. Data relating to the size of the samples, their structure and the method of preparation for the tests are summarised in Tab. 1 and shown in Fig. 3.

Table 1: Characteristics of the composite samples and the epoxy resin sample (matrix).

Sample material	No. of layers of fabric in the sample	Sample thickness l, mm	Sample diameter d, mm
Epoxy resin EPIDIAN – 53 (hardener Z1)	0	1.64	12
Carbon-epoxy composite (carbon fabric 160 g/m^2, plain weave, amount of resin necessary for the optimum supersaturation 230g/m^2)	4	1.03	12
Glass-epoxy composite (glass fabric 170 g/m^2, plain weave, amount of resin necessary for the optimum supersaturation 230g/m^2)	4	0.98	12

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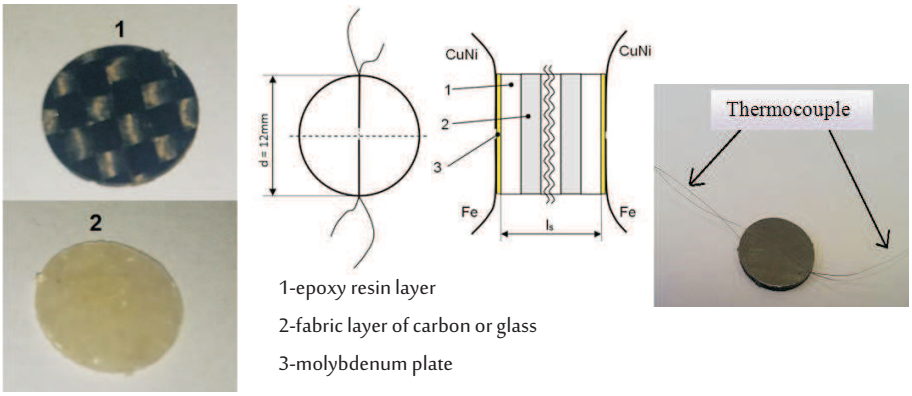
Preparation of the samples consisted in attaching the Mo planes to their extreme surfaces with glue (Fig. 3). These plates ensured that the requirement for the sample material to be opaque for the laser pulse radiation was satisfied. The heat transfer model used in the measurements imposed this requirement. Very thin films of the adhesive were, like the material of the matrix, made of epoxy resin EP 53. The bonding between the molybdenum plates and the samples took place at room temperature for approximately 24 h. The plates had the same diameters as the material of the samples ($d = 12$ mm) and the thickness of 0.07 mm. Thermoelectrodes, Fe and CuNi [9], used for measuring temperature $\Theta_2(t)$ and temperature difference $\Delta\Theta(t)$ were electrically welded to the Mo plates (Fig. 3).



a) b) c)

Figure 3: a) Samples for thermal diffusivity tests: 1. sample 'EP53 resin strengthened with four layers of carbon fabric'; 2. sample 'EP53 resin strengthened with four layers of glass fabric'; b) Preparation of the samples for measurements of their substitute thermal diffusivity; c) sample with Mo plates bonded to its extreme surfaces, to which 50 μm diameter Fe and CuNi thermoelectrodes were welded electrically.

Thermal diffusivity was determined for discrete temperature values for sample T_0 thermostated in a vacuum furnace. The values of thermal diffusivity were defined on the basis of the analysis of experimentally determined changes in temperature difference between the extreme surfaces of the adiabatic sample after the laser shot on its front face. Suitable software was used for that purpose [7].

Thermal diffusivity results from the investigations of carbon-epoxy and glass-epoxy composites and epoxy resin EP53 sample, respectively, are presented in Figs. 4, 5 and 6. Figure 7 shows a typical final record of measure-

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ment data processing, in the form of change characteristics $\Delta\Theta(t)$, $\Delta\Theta'(t)$, $\Theta_2'(t)$ and $\ln\Delta\Theta(t)$ for the determination of substitute thermal diffusivity of epoxy resin EPIDIAN 53 wall at the sample thermostating temperature $T = 48.6\,^{\circ}\mathrm{C}$ (third measurement series).

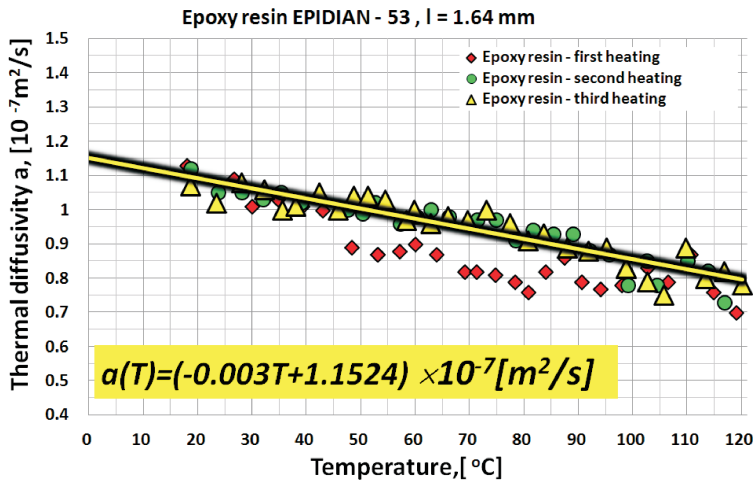

Figure 4: Summarised final temperature characteristics of thermal diffusivity of the epoxy resin EPIDIAN 53 sample after three heating series (changes approximated after the third heating series).

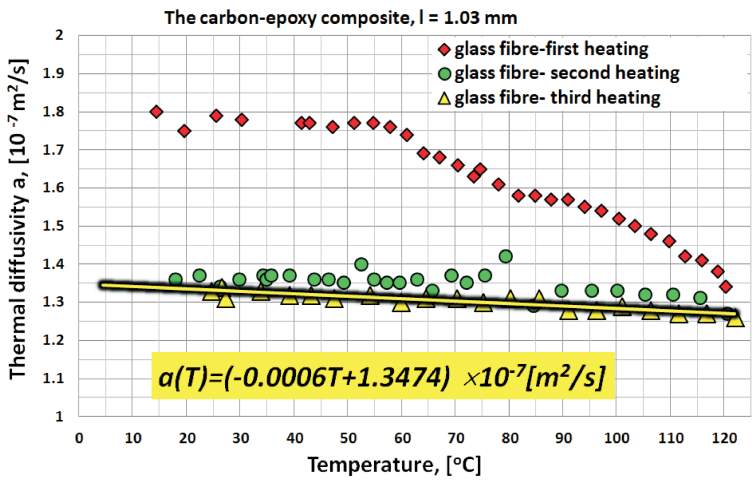

Figure 5: Summarised final temperature characteristics of thermal diffusivity of the carbon-epoxy sample after three heating series (changes approximated after the third heating series).

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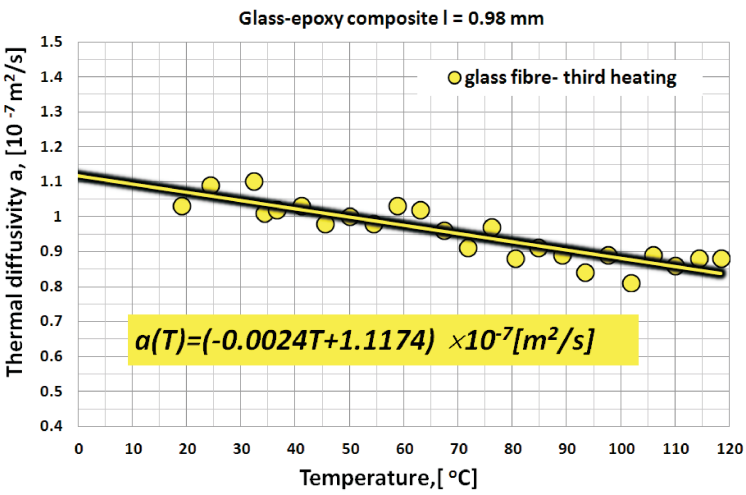

Figure 6: Final temperature characteristic of thermal diffusivity of the glass-epoxy sample after third heating.

None of the investigated samples was subject to thermal treatment prior to tests. Three test series were performed for each of the walls. A single test series consisted of two stages. The first stage included heating the sample at a temperature range from room to about 120 °C, with measurements taken every 20 min. Following the first stage, the sample placed in the vacuum furnace was subject to free cooling until it reached the initial room temperature, which constituted the second stage of the test. The cooling process started with switching off the furnace but in the vacuum. Each test series lasted approximately 24 h. The time interval between the series was equal to the duration of the measurement series.

Table 2 summarises the formulas for approximation of thermal diffusivity temperature characteristics of the samples. The approximation formulas refer to the samples after the third heating series (15–120 °C) for all the measurement of $a(T)$.

The error made while measuring thermal diffusivity using the modified pulse method was estimated on the basis of relationship $a = l^2/(\pi^2\tau)$ and is

$$\left|\frac{\Delta a}{a}\right| = 2\left|\frac{\Delta l}{l}\right| + \left|\frac{\Delta \tau}{\tau}\right|, \tag{4}$$

where Δl is sample thickness measurement error; $\Delta\tau$ is characteristic time measurement error.

The total error made while measuring thermal diffusivity using the modified pulse method, calculated from relationship (4), was estimated to be less

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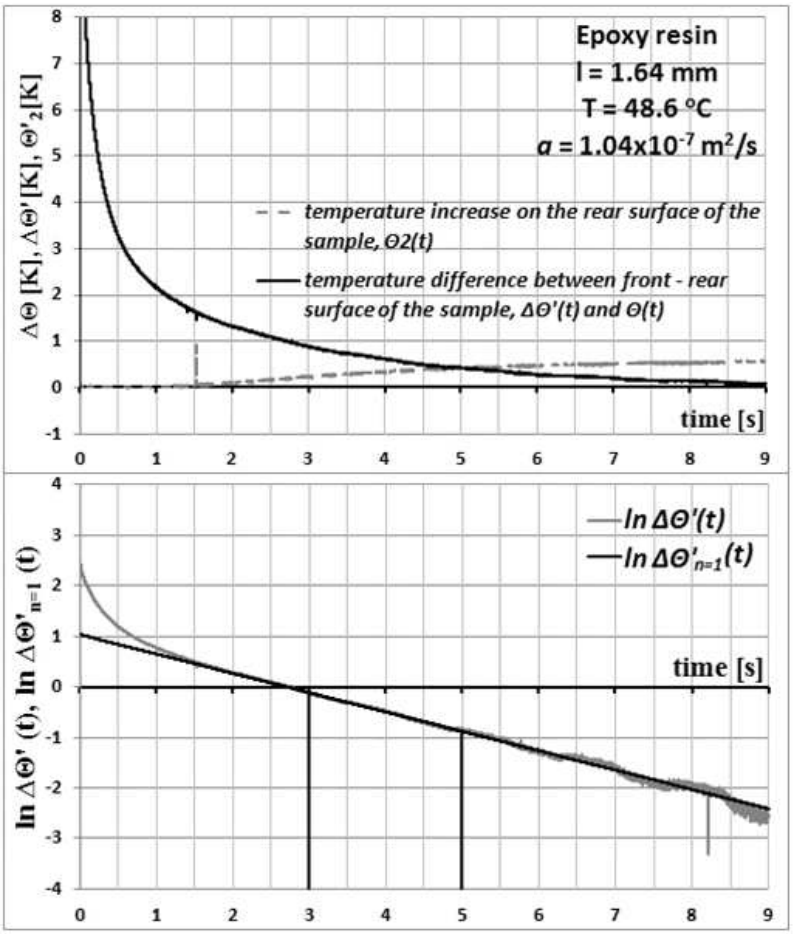

Figure 7: An example of the data processing final printout for thermal diffusivity of epoxy resin EPIDIAN 53 wall at the sample temperature $T = 48.6$ °C (third measurement series).

than 4%. The sample thickness measurement error depends on the thickness of the sample and was estimated to be equal to $2|\Delta l/l| \cong 2 \times 10^{-2}$, where $l \cong 1$ mm and $\Delta l \cong 10^{-2}$ mm. The error component $|\Delta\tau/\tau|$ related to the characteristic time is defined as a sum of:

- component, related to the fluctuations of the signal in the time interval $t_1 \div t_2$, which is determined through numerical processing of signal $\Delta\Theta'(t)$, recorded in the memory of the data acquisition and processing system, always less than 0.4×10^{-2};

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Table 2: Approximation formulas for thermal diffusivity temperature characteristics of the polymer composites and the epoxy resin.

Material	Approximation formula for thermal diffusivity $a(T)$ temperature characteristics (after the third measurement series; temperature T in °C)
Epoxy resin EPIDIAN – 53 ($l = 1.64$ mm, $d = 12$ mm)	$a(T) = (1.1524–0.003T) \times 10^{-7}$ m^2/s
Carbon-epoxy composite 4 layers of carbon fabric ($l = 1.03$ mm, $d = 12$ mm)	$a(T) = (1.3474–0.0006T) \times 10^{-7}$ m^2/s
Glass-epoxy composite 4 layers of glass fabric ($l = 0.98$ mm, $d = 12$ mm)	$a(T) = (1.1174–0.0024T) \times 10^{-7}$ m^2/s

- and error component for determining the characteristic time τ, made because of heat loss from both surfaces of the sample, which on the basis of [8] it was estimated to be less than 10^{-2}.

4 Summary

This paper has given an account of comparative research of transient heat transfer in two plane-parallel walls, one made of carbon-epoxy composite and the other made of glass-epoxy composite, with comparable thicknesses and the same number of carbon and glass fabric layers (four layers). Epoxy resin EPIDIAN 53 comprised the matrix for both walls.

Thermal diffusivity is the 'goodness-of-fit' criterion for the material as a heat conductor under transient heat transfer conditions. Its values, determined here for both composites and, for comparison, for the matrix material are summarised in the form of thermal diffusivity temperature characteristics in the figure below. The results of the study are initial results therefore the temperature characteristics has been approximated by linear functions. This type of approximation has proved to be appropriate for the carbon-epoxy composite, whereas for the epoxy resin and glass-epoxy composite the accurate plot for the characteristic $a(T)$ will have to be verified.

Thermal diffusivity temperature characteristics presented in Fig. 8 refers to the third series of measurements and hence to the third heating of the samples from the room temperature to about 120 °C. From a practical point

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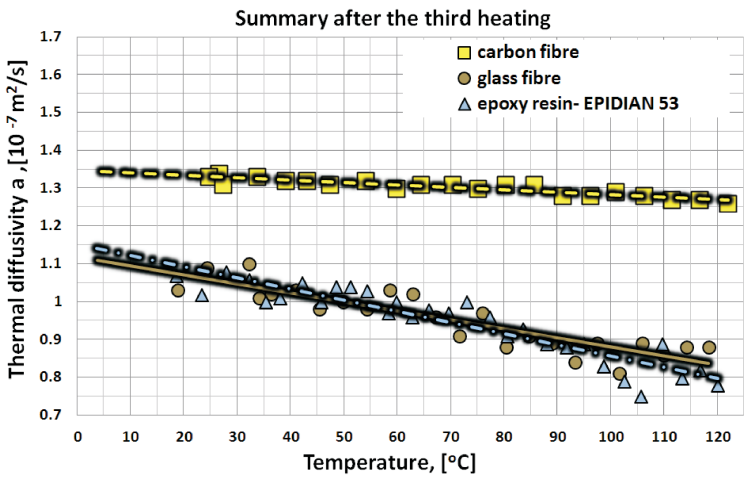

Figure 8: Final temperature characteristics of thermal diffusivity of the epoxy resin, carbon-epoxy and glass-epoxy composites after third heating.

of view, thermal diffusivity temperature characteristics were repeatable as early as after two measurement series.

On the basis of the initial results, thermal diffusivity temperature characteristics of the epoxy resin EPIDIAN 53 matrix and the glass-epoxy composite have been found to be nearly identical in terms of the plots and values. Thermal diffusivity temperature characteristic of the carbon-epoxy composite sample differs significantly from the temperature characteristics of the epoxy resin and glass-epoxy samples. Compared with the two other materials, the thermal diffusivity plot for the carbon-epoxy composite has only a slight inclination and the values are 1.2 to 1.5 times higher.

The advantage of the composite wall with carbon fabric is that owing to very high thermal conductivity and diffusivity of carbon fibres, the temperature in such a wall equalizes very quickly in the direction parallel to the wall surface. Also in the perpendicular direction, heat transfer both in the steady and transient states is even more intensive that expected based on the thermophysical parameters of the components of this composite wall.

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