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Increasing the Precision of Thermal Properties Measurement by the Periodic Heating Method

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The development of new thermal insulation materials, technologies and methods for the buildings construction allows for decrease in wasteful consumption of energy resources. Therefore, the knowledge of thermophysical properties is one of the most important tasks because the determination of these characteristics with high accuracy can help to achieve high energy efficiency.

The method and technical device are proposed in this work and can be used for increasing the measurement accuracy and choosing the optimal mode parameters for producing thermal insulation materials. The method for the thermophysical properties measurements is based on the periodic heating method (the third kind regular mode method) and the temperature oscillations made by Peltier element. The mathematical and physical models were developed and the automation of the measurement installation was made with the help of graphical programming environment LabView.

The equipment was calibrated by experiments with standard material - Plexiglas and the obtained data proved the appropriateness of developed mathematical model. After that the number of experiments with the nanomaterial "Nanographite" was performed. With the help of proposed methodology and the experimental setup the strong dependence between thermal diffusivity of "Nanographite" and moisture content was revealed. This fact will be used in further investigations of this new material.

1. Introduction

The knowledge of thermophysical properties (TPP) of producing thermal insulation materials and their dependence on temperature is necessary because it will allow to choose optimal mode of equipment operation. It will help to decrease the level of defects and to increase the efficiency and competitiveness of products. The developers of new materials can also use TPP as a data source for new production equipment design (Każmierczak-Balata et al, 2008). The development of new thermal insulation materials, technologies and methods for the buildings construction allows to decrease wasteful consumption of energy resources. Therefore, the knowledge of thermal properties is one of the most important tasks because the determination of these characteristics with high accuracy can help to achieve high energy efficiency.

The promising direction in this area is application of the periodic heating method. The theory of this method was significantly influenced by many well-known scientists (Ponomarev, 2012). One of its main benefits is an opportunity to register easily the phase shift of temperature waves in time, because time becomes the main physical value in the experiment. Since the time is the most precisely determined value, the accuracy of measurement results will be high.

The goal of this research is to increase the accuracy of TPP determination for thermal insulation materials with the help of choosing optimal modes of measurements. In this case the controllable parameter is the period of temperature oscillation in a sample.

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2. The method and the experimental setup

2.1 The proposed methodology

Analysis of the reviewed methods and tools for determining the thermophysical properties of the materials have shown that none of them doesn't consider the dependence between experiment mode and test material. In the process of determining the thermal characteristics it is necessary to change the parameters of the experiment according to the the characteristics of the sample, such as the heating power, the temperature difference, the period of the temperature wave.

Based on this, the authors of this work propose measurement methodology that provides accurate determination of thermophysical characteristics by choosing the optimal parameters of the thermophysical experiment.

This article discusses the method of choosing rational modes of thermal diffusivity measuring by the third kind regular mode method (periodic heating method), namely the relation of the delay time to the period of the temperature oscillations in the sample.

In this work we consider the case (Ponomarev et al., 2008) when the thermal diffusivity is found from the relation (1):

$$a = \frac{(x_2 - x_1)^2 \cdot \tau_0}{4 \cdot \pi \cdot [\tau_l(x_2, x_1)]^2} \tag{1}$$

where τ_1 is the lag time of temperature oscillations with period τ_0 at a depth x_2 compared with on the surface $x_1=0$ of the sample.

The root mean square ratio error δa of a measurement of thermal diffusivity a is a function

$$\delta a = f(\,\delta x, \delta \mathcal{P}_{max}, \psi) \tag{2}$$

where $\Psi = \frac{\tau_l}{\tau_0}$ is a relation of the lag time of harmonic oscillations in the point with coordinate *x* to the

period τ_0 of harmonic oscillations.

According to the graphs in the Figure 1, which are built from the obtained mathematical expressions (1,2), the minimum of the error δa corresponds to the same value of parameter ψ for different values of x. Therefore, it's necessary to control the value of ψ choosing the appropriate period of the temperature oscillations to keep the minimum level of the relative error δa .

The period of temperature oscillations will be different for each material, therefore we propose the method of measurements (Figure 2) which takes into account this factor.

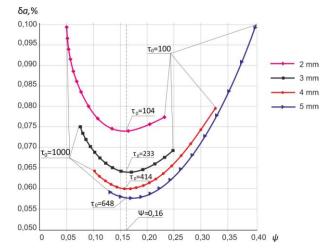


Figure 1: The dependence $\delta a=f(\psi)$ for different value of x (x=2...5 mm, $\tau_0=100...1,000$ s), where x- the thickness of the sample, τ_0 - the period of temperature oscillation, $a=1.09 \cdot 10^{-7} m^2/s$, $\Delta x=0.05$ mm, $\Delta \vartheta = 0.1^{\circ}$ C, $\vartheta_{max} = 10^{\circ}$ C

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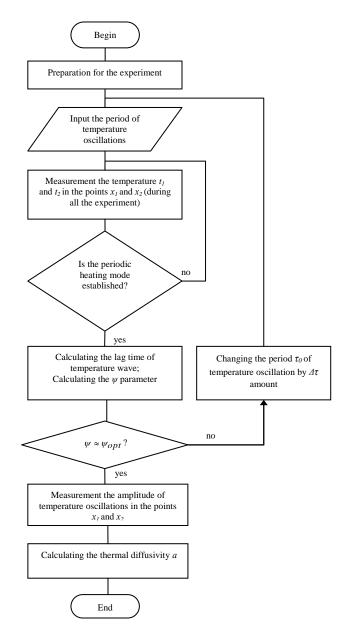


Figure 2: Algorithm of the thermal diffusivity measurement. Preparation for the experiment: making the flat sample from the investigated material; placing the sample on the surface of the Peltier element; placing the thermocouples in the points $x_1 u x_2$ of the sample; covering the sample with the layer of thermal insulation

2.2 The experimental setup

The processes of data obtaining and heat mode setting are completely automated and controlled by a personal computer. The scheme of the experimental setup is shown in the Figure 3. The Peltier element is connected to the power unit through the relay's contacts K1 and K2. The commutation of the relay proceeds by using discrete inputs and outputs of the board USB 6008 and the measurement of the temperature analogue channels 24-bit of the board NI USB 9111A.

The application of the Peltier element allowed to cancel liquid thermostat. It has positive influence on decreasing the size and weight of measurement installation.

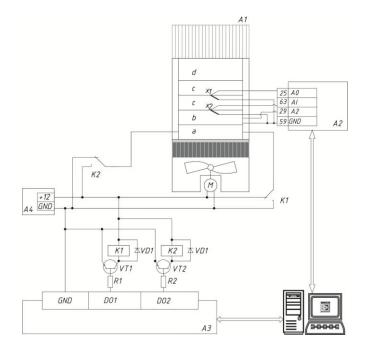


Figure 3: The scheme of the experimental setup. . A1 – measurement tool, A2 – data acquisition board NI USB 9111A, A3 – board NI USB 6008, A4 – power unit

2.3 The software

The program is designed in LabView environment and aimed at monitoring and controlling the process of the thermophysical experiment for determining the thermal diffusivity of solid, powder and liquid substances. It allows to choose the period of harmonic oscillation of the temperature in a sample at the expense of periodic changing the voltage supply polarity of the Peltier element, which is located under the sample. The thermal diffusivity is calculated from the phase differences of thermocouples signals. Hot junctions of the thermocouples are located inside the sample at a given distance from each other. The software can be used in study process by students for educational purposes or researches in the fields of physics and engineering.

When the program and the measurement setup are launched the value of ψ is determined experimentally. After that operator chooses the appropriate value of the heat effect period so that the parameter ψ is equal to 0.16. At the same time, the determination of temperature lag, which is measured at different points of the sample, is performed with the help of standard LabView tools. The front panel of the program is shown in the Figure 4.

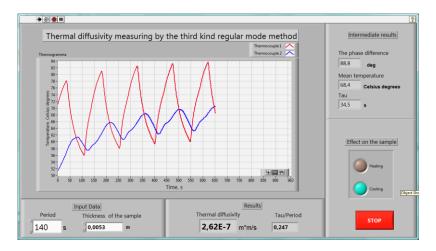


Figure 4: The front panel of the program for thermal diffusivity measurement

3. The experiments and discussion

The equipment was calibrated by experiments with standard material – Plexiglas (PMMA) and the obtained data proved the appropriateness of the mathematical model. Table 1 presents the results of the experimental setup calibration. We used $a=1.09 \cdot 10^{-7} \text{ m}^2/\text{s}$ as an actual value of the thermal diffusivity of PMMA.

The experiments have shown that the error of the thermal diffusivity measurement decreases with the increase in the temperature oscillation period. So when ψ =0.16 the temperature oscillation period reaches its minimum. Further increase in temperature oscillation period is considered to be undesirable as the duration of experiment can increase significantly.

Recently the presented experimental setup is used for determining thermophysical properties of carbon nanomaterials which are produced on the base of Tambov State Technical University. Particularly, a number of experiments with the material "Nanographite" were performed. It is crystalline flake graphite with the diameter from 10 to 100 μ and an average thickness is 3 - 5 nm. "Nanographite" is produced in the form of paste in water or organic solvents with the mass content of nanographite from 6 % to 10 %. This material is used in electrotechnical industry and it is important to know its thermal diffusivity for modelling nonstationary temperature field. The results of the experiment are shown in the Table 2.

Experience shows that adding Nanographite increases considerably the thermal diffusivity of material.

Thermal diffusivity depends significantly on moisture content (Table 3).

Reducing the moisture content increases the thermal diffusivity. According to the fact that the tested material had density close to the bulk, we should consider that increasing pressure on the material will also increase the thermal diffusivity.

4. Conclusions

The capability of temperature wave method (the third kind regular mode) is quite wide. The experiments performance is possible even with the small size samples. The periodic heating method is quite reliable, low cost and effective (Gonzalez-Mendizabal et al., 1998). Nowadays it is being actively developed. However, some issues of its use require looking for further solution such as advances in measurement devices and the methods of thermophysical properties determination.

Indicator	Period, s			
Indicator	40	60	80	160
Delay time, s	19	24.6	23	25
Parameter w	0.48	0.41	0.28	0.16
Thermal diffusivity, m ² /s	0.88·10 ⁻⁷	0.83·10 ⁻⁷	1.2·10 ⁻⁷	1.11·10 ⁻⁷
Ratio error, %	20	25	10	2

Table 1: Experimental data

Table 2: The results of the experiment with "Nanographite". The thickness of the sample is 0.0053 m, the material humidity is 40 %

Indicator	Period, s			
	140	160	180	200
Parameter ψ	0.19	0.18	0.16	0.15
Mean temperature, °C	68	69	69	68
Thermal diffusivity, m ² /s	4.18	4.39	4.99	4.30

Table 3: The results of the experiments with wet and dry "Nanographite". The thickness of the sample is 0.0053 m, mean temperature t = 68 °C, $\psi = 0.16$

N≌	Thermal diffusivity, m ² /s			
IN≚	Humidity 70 %	Humidity 0 %		
1	2.60·10 ⁻⁷	7.38·10 ⁻⁷		
2	2.71·10 ⁻⁷	7.43·10 ⁻⁷		
3	2.6·10 ⁻⁷	7.38·10 ⁻⁷		
4	2.65·10 ⁻⁷	7.4·10 ⁻⁷		
5	2.62·10 ⁻⁷	7.37·10 ⁻⁷		
Mean value	2.64·10 ⁻⁷	7.39·10 ⁻⁷		

The application of equal mode parameters for the thermal physical properties determination of different materials can lead to the significant errors in output data. The proposed methodology can help to optimize the experiment process and to increase the accuracy of measurements.

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