

Measuring the Thermal Diffusivity of Multi-Layered Graphene Nanoplatelets (GNPs) by a Periodic Heating Method

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Abstract

The present paper describes ways of increasing the accuracy of measurements of thermophysical properties (namely, thermal diffusivity) of materials. The knowledge of these characteristics is very important for modeling industrial processes, since their high-accuracy determination can reduce wasteful consumption of energy resources while developing novel thermal insulation materials, technologies, as well as methods for building construction.

The method proposed herein is based on periodic heating and can be used to improve the measurement accuracy and find the optimal parameters for producing thermal insulation materials. Periodic temperature oscillations were generated by means of the Peltier element. Mathematical and physical models were developed, and the measurement setup was automated using the LabView graphical programming environment. The analysis of possible error sources was performed, and the ways of decreasing error values were suggested.

The equipment was experimentally calibrated using a standard material (Plexiglas), and the obtained data proved the appropriateness of the developed mathematical model. Moreover, a number of tests were performed with a nanomaterial, namely, graphene nanoplatelets (GNPs). The proposed method and the experimental measurement setup made it possible to reveal a strong dependence between the thermal diffusivity of the GNPs and the moisture content. This finding will be considered in further studies concerning the above-mentioned nanomaterial.

Keywords

Periodic heating; thermal insulation materials; graphene nanoplatelets (GNPs); thermal diffusivity; measurement error.

Nomenclature

H – thickness of the sample, m; x_1, x_2 – coordinates of thermocouple position, m; a – thermal diffusivity, m^2/s ; T_c – ambient temperature, K; T_1, T_2 – temperatures of the sample, K; K_1, K_2 – transfer coefficients of the thermocouples, mV/K ; h – distance between the thermocouples, m; K_3, K_4 – transmission coefficients of the data acquisition board; f – frequency of temperature oscillations, Hz; τ_d – delay time, s; τ_0 – period of temperature oscillations, s; δa – relative error of thermal diffusivity measurements; δx – relative error of length measurements; ϑ_{\max} – maximum amplitude of temperature oscillations, K; $\delta \vartheta_{\max}$ – relative error of maximum amplitude measurements; Δx – absolute error of length measurements, m; $\Delta \vartheta$ – absolute error of temperature amplitude measurements, K; $\Psi = \frac{\tau_d}{\tau_0}$ – relationship between the delay time of harmonic oscillations at the point with coordinate x (τ_d) and the period of harmonic oscillations (τ_0); ψ_{opt} – optimal ψ value; $\Delta \tau$ – error of time measurements, s; $\vartheta_{\max 1}, \vartheta_{\max 2}$ – amplitudes of temperature oscillations at the thermocouple position, K; Δh – error of h measurements, m; $\Delta \vartheta$ – absolute error of temperature measurements, K; φ_1, φ – phases of temperature oscillations; Δ_{ADC} – random error introduced by the data acquisition board; U_{inp} – input voltage range for the analog-to-digital converter, mV; n – resolution of the analog-to-digital converter; Δ_{emf} – disturbance caused by external magnetic fields.

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1. Introduction

The knowledge of thermophysical properties of thermal insulation materials and their temperature dependence is necessary, since it will allow for choosing the optimal operating mode for equipment, reducing defect levels and raising the efficiency and competitiveness of products, the thermophysical properties of which appear to be the most important quality parameters. Developers of novel materials can also consider these properties as a data source for new production equipment design [1]. Manufacturing advanced thermal insulation materials, technologies and methods for building construction makes it possible to reduce wasteful consumption of energy resources. In this regard, the high-accuracy determination of the thermophysical characteristics of those materials may help in achieving high energy efficiency.

The application of the periodic heating method is a promising direction in this area. The opportunity to easily record phase shifts of temperature waves in time is among its main benefits, since the time becomes the main physical value obtained experimentally. Taking into account that this value is determined in the most precise manner, the accuracy of measurement results will be high.

A periodic heating method can be performed in different ways. One of them is based on using an electron beam to generate thermal waves in the sample [2]. The electron source and the sample under study represent a vacuum diode. The temperature of the sample is determined from the intensity of the electron beam. However, the sample should be made of an electrically conductive material. This fact limits the application of this method [2].

In case of temperature wave generation by electromagnetic radiation, the electrical properties of a test material are not critical. Moreover, laser radiation is frequently used as a temperature wave source. In this case, thermocouples and photoelectric sensors can be employed to register temperature changes.

The output radiation with power changing in accordance with the Periodic Law is one of the main disadvantages of the laser method. It negatively affects the accuracy of measuring the thermophysical properties. Developing continuous laser radiation-based systems which provide the highest stability of the output power complicates the construction of measurement instruments due to the use of mechanical modulators [2]. The need for an additional heater to

control the average temperature of the sample is the principal drawback of these systems.

The simplest way to excite temperature oscillations in a sample is through changing the power of the electric heater in contact with a sample or through switching the coolant flow surrounding one of sample surfaces [3].

In case of the photothermal method for temperature wave generation, the sample is heated periodically by light radiation with modulating light flux intensity, wherein the radiation is partially absorbed by the environment [4]. The need for several radiation photo detectors to record temperature and temperature wave phases (due to inconsistency of their sensitivity in different spectral ranges) during thermophysical measurements in a wide temperature range is the disadvantage of this method.

In case of the thermoelectric method, the Peltier element is employed to generate temperature oscillations [1].

In a reverse polarity situation, the current flowing through the Peltier element may control both sample cooling and heating, thereby giving the possibility to set the average sample temperature above or below the ambient temperature [5]. High power consumption in achieving significant temperature differences is the principal disadvantage of the Peltier element. In order to increase these differences, it is recommended to use a radiator and a fan that would direct the heat away from the heating side of the element.

In view of the aforementioned, the aim of the present research was to develop an advanced methodology for determining the thermal diffusivity of multi-layered graphene nanoplatelets (GNPs) and use it for improving the accuracy of measurements.

2. Material and Method

2.1. Material. In the present research, we investigated the thermal diffusivity of multi-layered GNPs – a two-dimensional allotropic carbon modification formed by a layer of carbon atoms with a thickness of one atom and having unique strength and thermal conductivity [6]. This material was produced as aqueous paste (diameter 10-100 μm , and average thickness 3–5 nm) at “NanoTechCenter” Ltd. (Tambov, Russia) according to the methodology described by Tkachev and co-authors [7].

2.2. Physical and Mathematical Models. To determine the thermal diffusivity, the experiment parameters should be changed according to the sample

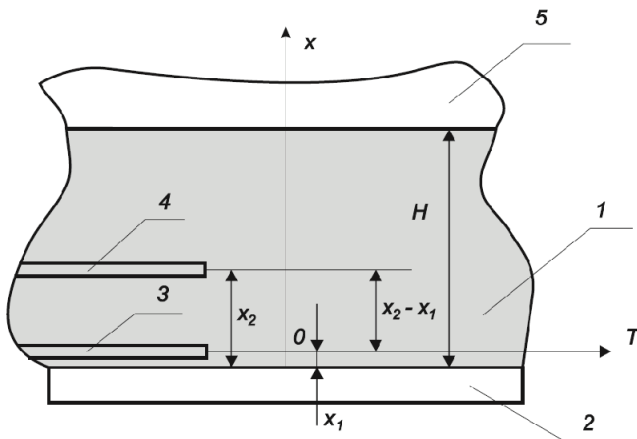


Fig. 1. The physical model of the proposed method:
1 – investigated sample; 2 – Peltier element as a periodic heating source; 3 and 4 – thermocouples;
5 – thermal insulation layer

characteristics such as heating power, temperature difference, and period of temperature wave [8].

In this regard, the authors of the present work propose a periodic heating-based measurement methodology that considers the relationship between the delay time and the period of temperature oscillations in the sample and provides an accurate determination of the thermal diffusivity by choosing optimal parameters of a thermophysical experiment. The physical model of this process is shown in Fig. 1.

It is considered herein that the thermal diffusivity (a) may be expressed as follows [9]

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

where τ_l is the time lag for temperature oscillations of period τ_0 at depth x_2 compared to surface $x_1 = 0$ of the sample.

The relative error (δa) of the thermal diffusivity measurement can be found as the following function

$$\delta a = f(\delta x, \delta \vartheta_{\max}, \psi). \quad (2)$$

As can be seen from the graphs (Fig. 2) built on the basis of mathematical expressions (1) and (2), the minimum δa corresponds to the same ψ value at different x values. Therefore, it is necessary to control the ψ value by choosing the appropriate period of temperature oscillations in order to keep the minimum level for δa . Since the period of temperature oscillations may differ for different materials, this fact should be taken into account during the measurements.

2.3. Analysis of Error Sources. To construct a measuring device according to the developed mathematical model and to determine the thermal diffusivity of the material under study with minimal errors, it is necessary to elucidate the sources of possible deviations of measurements from the actual thermal diffusivity values [10].

As a rule, all measurement errors can be of the following types: procedure (i.e., method errors), subjective (i.e., operator's errors), instrumental, and data processing.

In the developed mathematical model of the temperature field, the following simplifications and assumptions were made:

- 1) the temperature field induced in the sample is one-dimensional and depends only on the time and coordinate x ;
- 2) there are no internal heat sources inside the layers of the material under study;
- 3) the thermal diffusivity variation in the material can be neglected during the measurements;
- 4) the thermal contact resistance between the material layers, heater and thermocouples is negligible;
- 5) the heat capacity of the thermocouple placed in the sample can be neglected as well.

2.3.1. Preventing Procedure Errors during the Experiment. The validity of the aforementioned assumptions and preventive actions were considered while designing the measuring device in order to decrease the procedure errors caused by various simplifications.

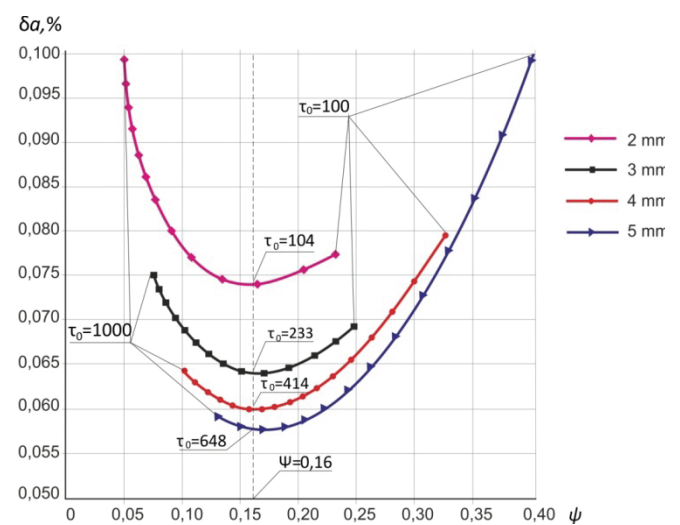


Fig. 2. The dependence $\delta a = f(\psi)$ for different x values
($x = 2\text{--}5\text{ mm}$, $\tau_0 = 100\text{--}1,000\text{ s}$), where x – the thickness of the sample, τ_0 – the period of temperature oscillation,
 $a = 1.09 \cdot 10^{-7} \text{ m}^2/\text{s}$; $\Delta x = 0.05\text{ mm}$; $\Delta \vartheta = 273.25\text{ K}$; $\vartheta_{\max} = 283.15\text{ K}$

To reduce the heat flow into the environment through the sample side surfaces, flat protective heaters or insulation materials are usually used. In the present research, a layer of the “Evropleks” foam insulation material (thermal conductivity: $0.039 \text{ W/(m}\cdot\text{K)}$) was employed.

A source of thermal energy may arise inside the material during its heating or cooling as a result of chemical reactions or structural and phase changes. To prevent errors caused by these factors, the material must be tested in order to confirm the absence of such phenomena in a predetermined temperature range. This can be done, for example, by using a differential scanning calorimeter.

If the temperature change in the material layer is negligible during the experiment, the value of the heat flow from the heater to the material layer should also be insignificant. When the range of temperature oscillations is $5\text{--}10^\circ\text{C}$, the thermal conductivity and thermal diffusivity changes of most polymers do not exceed 0.1% (Ponomarev et al., 2012). In this case, the thermophysical properties can be considered constant throughout the entire experiment.

Besides, the thermal contact resistance must be reduced. The sample surface should be carefully prepared and not have convexities and concavities, and the roughness must not exceed a critical value of $2.5 \mu\text{m}$ (Ponomarev et al., 2008). Moreover, it is recommended to use a thermally conductive paste, especially single-walled carbon nanotubes-based one (thermal conductivity: $> 2 \text{ W/(m}\cdot\text{K)}$). To reduce the lubricant layer during the measurement procedure, a down-force not leading to the deformation of the sample should be applied.

To decrease errors caused by the thermocouple capacity, the sensors should have minimal size and weight. In this case, the heat capacity of the transducer would be so low that the thermal inertia of the sensor

can be disregarded. In this paper, we describe a measuring device comprising sensors with protective tubes (diameter: 0.5 mm , length: 50 mm). The time constant of such transducer does not exceed 0.2 s ; that is why its inertial properties may be ignored.

2.3.2. Instrumental Errors. Modeling the error caused by the disadvantages of the measuring equipment (i.e., instrumental error) can be easily represented as a scheme consisting of interconnected measuring tools with transmission coefficients (or transfer functions) [11].

The measurement setup included two temperature measuring channels and one time measurement channel. Furthermore, before the experiment, the sample thickness (h) between the hot junctions of thermoelectric transducers was determined.

The scheme of the setup for measuring the thermal diffusivity is shown in Fig. 3.

The measuring cell containing the sample was kept at ambient temperature T_c . Sample temperatures T_1 and T_2 were measured by using thermoelectric transducers (chromel-alumel thermocouples) with transmission factors K_1 and K_2 . The measurements were performed at discrete-time moments τ defined by the control program. The hot junctions of the thermocouples were located at distance h from each other. Time values were determined using standard functions of the LabView programming environment. The output signals of the thermocouples came to the NI USB 9162 (24-bit) analog input data acquisition board having transmission coefficients K_3 and K_4 . The board comprised a built-in cold junction temperature compensation.

The h value was measured by using a caliper with an error (Δh) of $0.5 \cdot 10^{-4} \text{ m}$.

As can be seen from formula (1), the thermal diffusivity of the sample depends on $h = x_2 - x_1$, period of temperature oscillations τ_0 and delay time

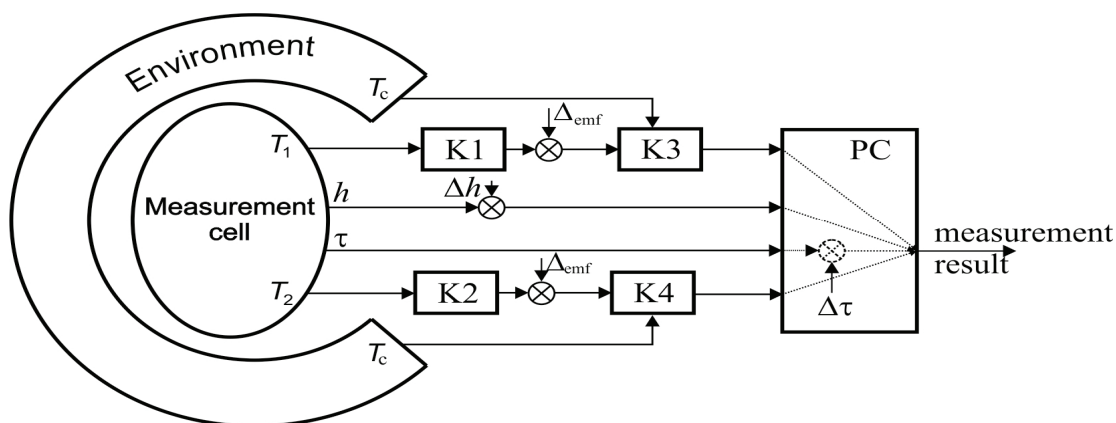


Fig. 3. The scheme of measuring setup for thermal diffusivity measurements

$\tau_d(x_2, x_1) = \tau_d(x_2) - \tau_d(x_1)$ of the temperature wave. However, it would be incorrect to mention that the metrological characteristics of the thermocouples and the analog channels of the data acquisition board do not affect the accuracy of the thermal diffusivity determination.

First, the conductors of thermoelectric converters and protective fittings possess their own heat capacity that commonly affects the inertial properties of the thermocouple. However, due to the small dimensions (e.g., diameter of the protective tube 0.5 mm) and the weight, the time constant of the thermocouple does not exceed 0.2 s, which is much lower than the expected value of the period of temperature oscillations. Consequently, the influence of the thermocouple thermal inertia on the result of the thermal diffusivity measurement can be neglected.

Second, disturbances from external magnetic fields and internal noise always affect the measured signal. This fact leads to waveform distortion (Fig. 4, where: 1 – the analog input signal from the data acquisition board, without noise and interference; 2 – the same signal with an upper boundary of random and systematic errors; and 3 – the signal with a lower boundary of random and systematic errors).

In this case, the error of the time parameter measurement can be determined from the following dependence

$$\Delta\tau = \frac{\Delta T}{\frac{\partial f(\tau)}{\partial \tau}}, \quad (3)$$

where $f(\tau)$ is the function for the described temperature oscillations.

The temperature oscillations at predetermined points of the sample may differ from harmonics, but using the “Extract Multiple Tone Information VI” tools of the LabView programming environment makes it possible to decompose any periodic signal into Fourier series in order to determine the amplitude, frequency and phase of each harmonic, as well as the phase difference for the first harmonic of the temperature oscillations at defined sample points.

The limit of the delay time measurement error caused by the random error of the temperature measurement (Fig. 4) can be expressed as follows

$$\Delta\tau = \frac{\Delta\vartheta}{\frac{\partial [\vartheta_{\max 1} \{\cos(2\pi f\tau + \varphi_1)\}]}{\partial \tau}} + \frac{\Delta\vartheta}{\frac{\partial [\vartheta_{\max 2} \{\cos(2\pi f\tau + \varphi_2)\}]}{\partial \tau}}, \quad (4)$$

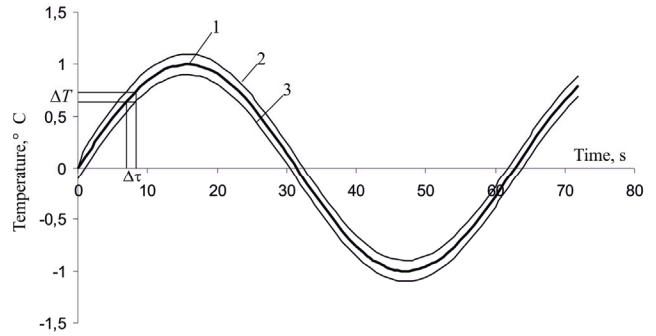


Fig. 4. The influence of temperature measurement errors on the time parameter measurement error

or

$$\Delta\tau = \frac{\Delta\vartheta}{2\pi f} \left\{ \left[\vartheta_{\max 1} \sin(2\pi f\tau + \varphi_1) \right]^{-1} + \left[\vartheta_{\max 2} \sin(2\pi f\tau + \varphi_2) \right]^{-1} \right\}, \quad (5)$$

where $\Delta\vartheta = \Delta T$ is the absolute error of the temperature measurement, $\vartheta_{\max 1}$, $\vartheta_{\max 2}$ are the amplitudes of temperature oscillations at the position of the thermocouple hot junctions, f is the oscillation frequency, and φ_1 , φ_2 are the oscillation phases.

The random error introduced by the data acquisition board represents quantization error $\Delta_{\text{ADC}} = U_{\text{inp}}/2^n$, where U_{inp} is the input voltage range for the analog-to-digital converter (ADC) ($U_{\text{inp}} = 140$ mV), n is the ADC resolution (24 bits). Considering that transfer coefficients $K1$ and $K2$ for two channels of the chromel-alumel type thermoelectric transducer are the same and equal to $K1 = K2 \cong 0.041$ mV/K, the absolute error of the temperature measurement was evaluated as follows

$$\Delta T = \frac{\Delta_{\text{ADC}}}{K1} = \frac{8.3 \cdot 10^{-6}}{0.041} \approx 2 \cdot 10^{-4}. \quad (6)$$

The analysis showed that the random error of the temperature measurements is mainly caused by an error arising due to the electromagnetic interference, the value of which was found to be ± 0.005

$$\Delta T = \frac{\Delta_{\text{ADC}}}{K1} + \Delta_{\text{emf}} = 5.2 \cdot 10^{-3}, \quad (7)$$

where Δ_{emf} are the disturbances from external magnetic fields

$$\Delta\tau = \frac{5.2 \cdot 10^{-3}}{2\pi f} \left\{ \left[\vartheta_{\max 1} \sin(2\pi f\tau + \varphi_1) \right]^{-1} + \left[\vartheta_{\max 2} \sin(2\pi f\tau + \varphi_2) \right]^{-1} \right\}. \quad (8)$$

According to the calculations, when the oscillation period is equal to 160 s, and $\vartheta_{\max 1}=10$ K, $\vartheta_{\max 2}=1$ K, the error of the delay time determination reaches 10 s. This error decreases with increasing the amplitude of temperature oscillations and the accuracy of its measurement.

As mentioned before, the time parameter (phase difference) measurement was carried out using the LabView tools. During this process, different kinds of delays are possible. The delay time is a random variable and corresponds to the time spent on performing the “processor-memory” cycle that includes command execution time and irregular delays associated with switching memory banks, command and data caching, heap regeneration, etc. Interrupt handlers from multiple external devices contribute to these delays: from HDD (hard disk drive) and FDD (floppy disk drive), from video card, network card, timer, etc. It also includes delays obtained as a result of the operative system switching to the other tasks.

To assess the delay time for the measurement process according to Godovsky [11], a test was developed using the LabView program, which determined that the standard deviation of time measurements with standard functions does not exceed $1 \cdot 10^{-3}$ s, and therefore this error component can be ignored.

2.3.3 Data Processing Errors. In case of indirect measurements performed by using a simplified algorithm, errors are sometimes associated with calculation errors, and not with measurement errors. For example, these errors may be encountered while integrating the dependencies by the rectangular method [1, 8]. Since these methods are not considered herein, it was assumed that the data processing errors are limited to rounding errors.

2.3.4 Sources of Subjective Errors. The sources of subjective errors (*i.e.*, operator's errors) arising during the measurements taken by the periodic heating method are as follows [12]:

- 1) mistakes and errors committed while fabricating sample parts and placing them into a measuring cell of the device in a wrong way prior to experiments;
- 2) errors coming from the heating (Peltier) element and thermocouples;
- 3) overabundant (or insufficient) coating of the thermal paste coating put to reduce the contact thermal resistance;
- 4) incorrect initial data input on the front panel of the program for controlling thermal diffusivity measurements;
- 5) incorrect connection of temperature transducers, relay switches and power unit to the data acquisition board, heater, etc.

To eliminate or at least decrease such errors and properly handle the measurement setup, a detailed experimental algorithm, user's manual and operator's guide should be developed.

3. Experimental Procedure

3.1. Experimental Setup. The processes of data obtaining and heat mode setting were completely automated and controlled with a personal computer. The scheme of the experimental setup is shown in Fig. 5. The Peltier element was connected to the power unit by means of relay contacts K1 and K2. The commutation of the relay was carried out by using discrete inputs and outputs of the USB 6008 board, and the temperature was measured using 24-bit analog channels of the NI USB 9111A board.

The developed measuring device allows for measuring the thermal diffusivity in a range $5 \cdot 10^{-8} - 1 \cdot 10^{-6} \text{ m}^2/\text{s}$.

Employing the Peltier element eliminates the use of liquid thermostats. Moreover, this element plays a positive role in decreasing the size and weight of the measurement setup (Mannella et al., 2014).

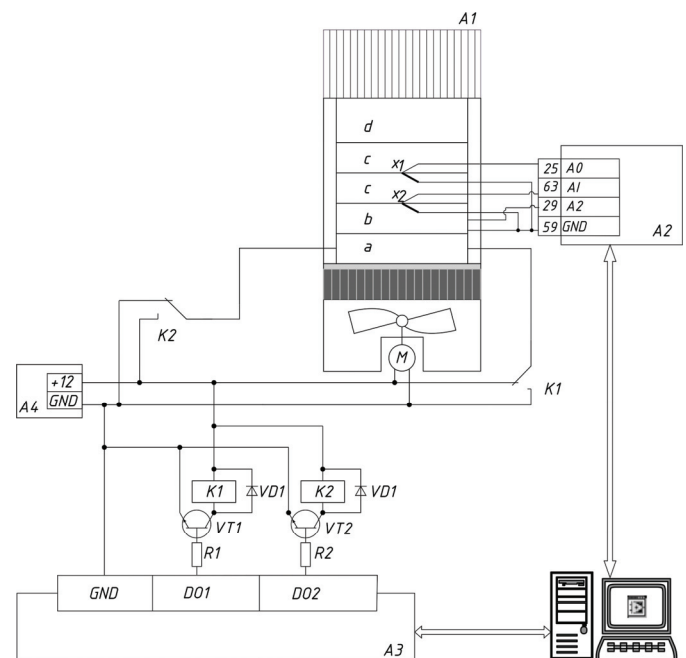


Fig. 5. The scheme of the experimental setup:
 A1 – measurement tool; A2 – data acquisition board NI USB 9111A; A3 – board NI USB 6008; A4 – power unit;
 a, b – Peltier elements; c – investigated sample;
 d – thermal insulation

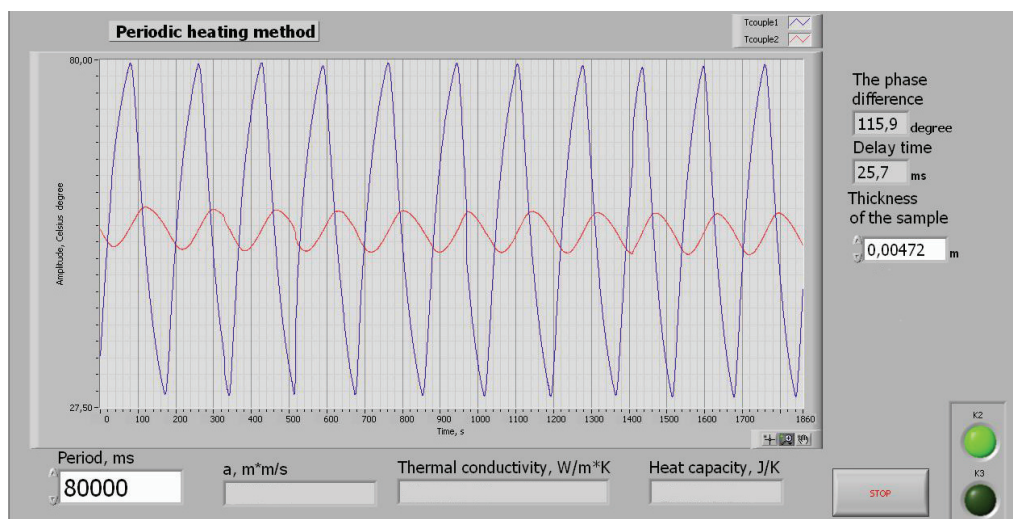


Fig. 6. The front panel of the program for thermal diffusivity measurement

3.2. Software. The program was designed in the LabView environment and aimed at monitoring and controlling experiments for determining the thermal diffusivity of solid, powder and liquid substances. It allows for choosing the period of temperature harmonic oscillations in a sample at the expense of periodic changing of the voltage power supply polarity of the Peltier element located under the sample. The thermal diffusivity is calculated based on the phase difference between the signals from the thermocouples. The thermocouple hot junctions are placed inside the sample at a certain distance from each other. Students can use this software for research or educational purposes in the fields of physics and engineering.

When the program and the measurement setup are launched, the ψ value is determined experimentally. After that, the operator chooses the appropriate value of the heating process period, so parameter ψ is equal to 0.16. At the same time, the determination of the temperature lag measured at different sample points is performed by using standard LabView tools. The front panel of the program is shown in Fig. 6.

4. Results and Discussion

The equipment was calibrated by experiments with a 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. In the present research, $a = 1.09 \cdot 10^{-7} \text{ m}^2/\text{s}$ was used as an actual value of the PMMA thermal diffusivity.

The experiments demonstrated that the error of the thermal diffusivity measurement depends on the period of temperature oscillations. Thus, when $\psi = 0.16$, δa reaches its minimum. A further increase in the temperature oscillation period is considered to be undesirable, since the duration of the experiment can be prolonged significantly.

Currently, the experimental setup presented herein is used for determining the thermal diffusivity of the GNPs. The experiment results are presented in Table 2.

As it is known, the larger is the amount of the material added, the higher is its thermal diffusivity.

In addition, the thermal diffusivity significantly depends on the moisture content (Table 3). Table 3 shows that thermal diffusivity increases when moisture content decreases. Five measurements for the humidity of the sample were conducted. After each measurement, the material was removed from the measuring cell, and a new portion of this material was uploaded. As can be seen from Table 3, the measurement results are different for the samples having the same humidity.

These findings demonstrate that there are some random factors affecting the measurement accuracy.

Table 1

Experimental data

| Indicator | Period, s | | | |
|--|----------------------|----------------------|---------------------|----------------------|
| | 40 | 60 | 80 | 160 |
| Delay time, s | 19 | 24.6 | 23 | 25 |
| Parameter ψ | 0.48 | 0.41 | 0.28 | 0.16 |
| Thermal diffusivity, m^2/s | $0.88 \cdot 10^{-7}$ | $0.83 \cdot 10^{-7}$ | $1.2 \cdot 10^{-7}$ | $1.11 \cdot 10^{-7}$ |
| Ratio error, % | 20 | 25 | 10 | 2 |

Table 2

The results of the experiment with GNPs. 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, $^{\circ}\text{C}$ | 68 | 69 | 69 | 68 |
| Thermal diffusivity, m^2/s | $4.18 \cdot 10^{-7}$ | $4.39 \cdot 10^{-7}$ | $4.99 \cdot 10^{-7}$ | $4.30 \cdot 10^{-7}$ |

Table 3

The results of the experiments with wet and dry GNPs. The thickness of the sample is 0.0053 m, mean temperature $t = 68\text{ }^{\circ}\text{C}$, $\psi = 0.16$

| № | Thermal diffusivity, m^2/s | |
|------------|--|----------------------|
| | Humidity 70 % | Humidity 0 % |
| 1 | $2.60 \cdot 10^{-7}$ | $7.38 \cdot 10^{-7}$ |
| 2 | $2.71 \cdot 10^{-7}$ | $7.43 \cdot 10^{-7}$ |
| 3 | $2.60 \cdot 10^{-7}$ | $7.38 \cdot 10^{-7}$ |
| 4 | $2.65 \cdot 10^{-7}$ | $7.40 \cdot 10^{-7}$ |
| 5 | $2.62 \cdot 10^{-7}$ | $7.37 \cdot 10^{-7}$ |
| Mean value | $2.64 \cdot 10^{-7}$ | $7.39 \cdot 10^{-7}$ |

It can be assumed that the electromagnetic interference on the thermocouple wires is related to these factors. The $(x_2 - x_1)$ value is also ambiguous, since there is a difficulty in setting the hot junction of the upper thermocouple at a predetermined distance from the hot junction of the lower thermocouple.

This is especially true for wet samples due to their plasticity, and can be confirmed by calculations of the sample standard deviation. The deviation values were found to be $0.041 \cdot 10^{-7}$ and $0.021 \cdot 10^{-7} \text{ m}^2/\text{s}$ for the 70 and 0 % humidity, respectively. Thus, for the wet sample, the deviation is almost twice as high. To reduce the mobility of the hot junction, the upper thermocouple was placed on a copper plate (dimensions $5 \times 5 \text{ mm}$, and thickness 0.5 mm).

The measurement results also show that reducing the moisture content increases the effective thermal diffusivity. In view of the fact that the density of the tested material is close to bulk, it may be concluded that increasing the pressure on the material will also increase the thermal diffusivity. In this regard, our further research will aim at determining the pressure dependence of the thermal diffusivity.

5. Conclusion

The capability of the temperature wave method (the third kind regular mode) is quite wide. Experiments can be performed even with small-sized samples. The periodic heating technique appears to be reliable, low cost and effective. Nowadays, it is actively developed. However, some issues with its use require looking for further solutions such as the development of advanced measuring devices and methods for determining the thermophysical properties. Employing the same mode parameters to determine the thermophysical properties of different materials may lead to the occurrence of significant errors in output data. The proposed methodology can help in optimizing experiment processes and increasing the measurement accuracy by choosing the appropriate parameters.

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References

1. Gonzalez-Mendizaba D., Bortot D.P., Lopez de Ramos A.L. (1998). A thermal conductivity experimental method based on the Peltier Effect. *International Journal of Thermophysics*, 4, 1229-1238.
2. Ivliev A.D. (2009). Metod temperaturnykh voln v teplofizicheskikh issledovaniyakh (analiz sovetskogo i rossiyskogo opyta [Temperature wave method in thermo-physical investigations (analysis of Soviet and Russian experience)], *Teplofizika vysokikh temperature* [Thermophysics of High Temperatures], 47 (5), 771-792. (Rus).
3. Artyukhina E.L. (2013). *Amplitudnyy poligarmonicheskiy metod temperaturnykh voln dlya kontrolya temperaturoprovodnosti tverdykh izotropnykh materialov* [Amplitude polyharmonic method of temperature waves for thermal diffusivity control of solid isotropic materials]. Transactions of TSTU, 19 (2), 278-28. (Rus).
4. Kazmierczak-Balata A., Bodzenta J., Trefon-Radziejewska D. (2010). Determination of thermal-diffusivity dependence on temperature of transparent samples by thermal wave method, *International Journal of Thermophysics*, 1, 180-186.
5. Lyubimova D., Divin A., Ponomarev S. (2014). Increasing the precision of thermal properties measurement by the periodic heating method. *Chemical Engineering Transactions*, 39, 1315-1320.
6. Graphene nanoplatelets [Electronic Resource]. – Access mode: <http://nanotc.ru/index.php/productions/85-gnp>, free. (The request date: 03/06/2015).
7. Tkachev A.G., Melezchik A.V., Dyachkova T.P., Blokhin A.N., Burakova E.A., Pasko T.V. (2013). *Uglerodnyye nanomaterialy serii «Taunit»: proizvodstvo i primeneniye* [The series of carbon nanomaterials "Taunit": production and application]. *Izvestiya vysshikh uchebnykh zavedeniy. Khimiya i khimicheskaya tekhnologiya* [News of Higher Schools. Chemistry and chemical technology], 56 (4), 55-59. (Rus).
8. Ponomarev S.V., Divina D.A., Shchekochikhin A.S. (2012). The choice of the optimal operating parameters when measuring the thermal diffusivity of thermal insulation materials by the regular mode method of the third kind. *Measurement Techniques*, 1, 68-72.
9. Ponomarev S.V., Mishchenko S.V., Divin A.G., Vertogradskiy V.A., Churikov A.A. (2008). *Teoreticheskiye i prakticheskiye osnovy teplofizicheskikh izmereniy* [The theoretical and practical fundamentals of thermophysical measurements]. Moscow: Fizmatlit, 81-199. (Rus).
10. Gurov A.V., Ponomarev S.V. (2013). *Izmereniye teplofizicheskikh svoystv teploizolyatsionnykh materialov metodom ploskogo «mgnovennogo» istochnika teploty* [Measuring thermophysical properties of thermal insulation materials by a flat "moment" heat source method]. Tambov: Tambov State Technical University. (Rus).
11. Godovsky Yu.K. (1976). *Teplofizicheskiye metody issledovaniya polimerov* [Thermophysical methods of polymers study]. Moscow: Khimiya [Chemistry]. (Rus).
12. Kharitonov V.V. (1983). *Teplofizika polimerov i polimernykh kompozitsiy* [Thermal physics of polymers and polymeric compositions]. Minsk: Vysshaya shkola, [High school]. (Rus).