The object of this study is the thermophysical properties of polymer micro- and nanocomposites, as well as the dependence of their heat conductivity with structural characteristics when using different types of fillers. A set of experimental studies of heat conductivity and specific heat capacity of polymer micro- and nanocomposite materials for polyamide 6 and carbon nanotubes, copper and aluminum as matrix and fillers was carried out. When obtaining composites, a method was used that is based on the mixing of components in the polymer melt. The content of fillers varied from 0.3 to 10 %, and the temperature of composite materials – from 305 to 500 K.

Experimental dependences of heat conductivity coefficients of the studied composites on the content of the filler were derived. It was established that according to the value of these coefficients in order of their reduction, these composite materials are ranked as follows: composites with fillers with carbon nanotubes, copper, and aluminum. It was found that only one percolation threshold is observed, when using a polyamide 6 matrix.

The regularities of changes in the specific heat capacity of the composites under consideration on their temperature when varying within the above limits of the filler content were investigated.

The analysis of the influence of the content of fillers on the degree of crystallinity of the polymer matrix of the investigated composite materials was carried out. It is shown that with an increase in the content of fillers, the degree of crystallinity decreases. The relationship between the thermally conductive properties of the composites under consideration and the specified degree of crystallinity has been established. Higher values of heat conductivity of composites correspond to lower values of the degree of crystallinity.

The reported results can be widely used in the development of highly heat conductive composites for various engineering applications

Keywords: polymer micro- and nanocomposites, carbon nanotubes, heat conductivity coefficient, specific heat capacity

UDC 538.9:536.6
DOI: 10.15587/1729-4061.2022.263417

ESTABLISHING THE INFLUENCE OF THE TYPE OF MICRO- AND NANOFILLERS ON THE THERMOPHYSICAL PROPERTIES OF HIGHLY HEAT CONDUCTIVE POLYMER COMPOSITES BASED ON POLYAMIDE 6

Nataliia Fialko
Corresponding author
Doctor of Technical Sciences, Professor,
Corresponding Member of the National Academy of Sciences of Ukraine*
E-mail: nmfialko@ukr.net
Roman Dinzhos
Doctor of Technical Sciences, Professor**
Julii Sherenkovskii
PhD, Senior Researcher, Leading Researcher*
Nataliia Meranova
PhD, Senior Researcher, Leading Researcher*
Vitalii Babak
Doctor of Technical Sciences, Professor, Head of Department,
Corresponding Member of the National Academy of Sciences of Ukraine
Department of Monitoring And Diagnostics of Energy Facilities
Institute of General Energy of the National Academy of Sciences of Ukraine
Antonovycha str., 172, Kyiv, Ukraine, 03150
Volodymyr Korzhyk
Doctor of Technical Sciences, Professor, Head of Department,
Corresponding Member of the National Academy of Sciences of Ukraine
Department of Electrothermal Processing Material
E. O. Paton Electric Welding Institute of the National Academy of Sciences of Ukraine
Kazymyra Malevycha str., 11, Kyiv, Ukraine, 03150
Maxim Lazarenko
Doctor of Physical and Mathematical Sciences
Department of Molecular Physics
Taras Shevchenko National University of Kyiv
Volodymyrska str., 60, Kyiv, Ukraine, 01033
Nina Polozenko
PhD, Senior Researcher*
Olexander Parkhomenko
PhD**
Volodymyr Makhrovskyi
PhD, Associate Professor**
*Department of Thermophysics of Energy Efficient Heat Technologies
Institute of Engineering Thermophysics of the National Academy of Sciences of Ukraine
Maria Kapnist (Zhelyabova) str., 2a, Kyiv, Ukraine, 03057
**Department of Physics and Mathematics
V. O. Sukhomlynskyi National University of Mykolaiv
Nikolska str., 24, Mykolayiv, Ukraine, 54006

Received date 21.06.2022
Accepted date 12.08.2022
Published date 31.08.2022

How to Cite: *Fialko, N., Dinzhos, R., Sherenkovskii, J., Meranova, N., Babak, V., Korzhyk, V., Lazarenko, M., Polozenko, N., Parkhomenko, O., Makhrovskyi, V. (2022). Establishing the influence of the type of micro- and nanofillers on the thermophysical properties of highly heat conductive polymer composites based on polyamide 6. Eastern-European Journal of Enterprise Technologies, 4 (5 (118)), 15–20. doi: https://doi.org/10.15587/1729-4061.2022.263417*

1. Introduction

Highly heat conductive modifications of polymer micro- and nanocomposites are increasingly used in engineering practice. This determines the relevance of the study of their most important thermophysical properties – the coefficient of heat conductivity and specific heat capacity.

The creation of this modification of polymer composites is associated with the use of various fillers with high heat conductivity. In this regard, it is interesting to obtain detailed information on the thermophysical properties of such composites in a wide range of changes in the content of the fillers used.

Also important are the studies aimed at establishing a connection between the properties of composites and their structure. In particular, it is a relevant task to find such a relation between the thermally conductive properties of composites and the degree of crystallinity of their polymer matrix.

2. Literature review and problem statement

Various aspects of the study of the most important thermophysical properties of polymer micro- and nanocomposites – their heat conductivity and specific heat capacity are reported in many works [1–12]. As for the thermally conductive properties of composite materials, special attention is paid to their study. Thus, [1] provides an overview of the main results of studies of conductive composite materials performed recently. Paper [2] reports data on the theoretical analysis of the thermally conductive properties of polymer composites based on epoxy resin filled with carbon nanotubes. There, in particular, the results of studying the dependence of the heat conductivity of these materials on the geometric characteristics of nanotubes, temperature, degree of solidification of the polymer matrix, etc., are considered. The cited work is mainly of a methodological nature and is aimed at devising a theoretical approach to determining the heat conductivity of polymer nanocomposite materials. In it, the thermally conductive properties of only one nanocomposite were subject to study. In the future, the proposed approach can be used to study a wide range of highly heat conductive micro- and nanocomposites.

Paper [3] considers the possibilities of increasing the heat conductivity coefficient of flexible polymer nanocomposites, in the production of which the electrospinning method is used to combine boron nitride nanosheets with carbon nanotubes (CNTs). The work is focused on the construction of effective thermal interface networks from advanced composite materials for modern electronic devices. However, the composite film obtained during the research showed a relatively low planar heat conductivity – 6.3 W/(m·K). This requires the creation of new flexible highly heat conductive polymer composites based on various matrices and fillers.

Work [4] establishes the dependence of the heat conductivity of nanocomposites based on polypropylene filled with CNT on the main parameter of the temperature regime of their production – the level of overheating of the polymer melt relative to its melting point. Here, it is important to conclude that it is possible to control the thermally conductive properties of composites by changing the parameters of the technological mode of their production. The study, however, is limited to considering one type of matrix and filler.

Paper [5] analyzes the features of the influence on the thermally conductive properties of epoxy resins of the reinforcing material nanofibers when they are filled with various types of carbon nanostructures. The cited paper is an example of the study of the thermally conductive properties of epoxy composites with a relatively wide variation of nanofillers (CNTs, graphite flakes, graphene nanoplatelets, and carbon black).

Work [6] reports the results of a comparison of the heat conductivity coefficients of polymer micro- and nanocomposites based on polyethylene, obtained by various methods. The cited work, like most of those discussed above, is characterized by a limited range of polymer composites under study. There, only two types of composite materials based on polyethylene were subject to research. And although papers on the thermally conductive properties of polymer micro- and nanocomposites are very numerous, and they address many topical issues of nanoscience and nanotechnology, for example [1–6], further development of these studies is necessary. In particular, it is required to obtain detailed information about these properties for highly heat conductive polymer composite materials that have prospects for widespread use in various engineering applications. In addition, it is important to study the characteristics of such materials when using a wide range of micro- and nanofillers and polymer matrices.

As for the studies of the specific heat capacity of polymer micro- and nanocomposites, they are significantly less numerous than the corresponding studies of their thermally conductive properties. In addition, these studies are often of an auxiliary nature or are accompanied by the study of other physical characteristics of the composites in question.

Thus, in [7], along with the specific heat capacity of polymer composites, the temperature dependences of their yield strength, modulus of elasticity, density, etc. are subject to study.

In [8], the results of studies into the heat capacity of polymer composites play a service role in the investigation of their thermal stability. Work [9] mainly focused on the development of a new method designed to separate the components of various amorphous fractions of polymer nanocomposites. There, the results of studies of the heat capacity of composites serve the purpose of substantiating this method.

Paper [10] reports the results of experimental studies to explore the effect of the dispersion of the filler on the heat capacity of nanocomposites "low-density polyethylene – regenerated clay". These results are used to analyze the thermal characteristics of composites related to their morphology.

Nevertheless, in some studies, for example, [11], the study of the specific heat capacity of polymer composites covers various aspects relating to the establishment of its temperature, concentration, and technological dependences. In the cited work, a series of experimental studies are carried out to establish the regularities of the complex effect on the specific heat of polymer nanocomposites of such factors as the temperature regime of their production, the value of the mass fraction of filler, and temperature of the composite material.

The results of studying the temperature dependence of the heat capacity of polymer~~ic~~ micro- and nanocomposites are used to determine the degree of crystallinity of their matrix. For example, in [12], for various methods of obtaining composites, the degree of crystallinity of a polymer matrix made of polypropylene was assessed when it was filled with CNTs or aluminum microparticles. However, attention is not paid to such an important issue as the establishment of a relationship between the degree of crystallinity of the polymer matrix and the thermally conductive properties of composite materials.

Thus, our review reveals that the available papers do not exhaust the need for a comprehensive study of the thermo-

physical properties of high-thermally conductive micro- and nanocomposites. It is important to further investigate these properties toward expanding the range of composite materials both in terms of polymer matrices and fillers. It is of interest to conduct experiments to establish the dependence of the thermophysical properties of composite materials on a set of determining parameters. In particular, studies of the relationship between the structural characteristics and heat conductivity of polymer micro- and nanocomposite materials are promising.

3. The aim and objectives of the study

The purpose of this work is to establish the regularities of changes in the heat conductivity and heat capacity of polymeric micro- and nanocomposites based on polyamide 6 on the content of fillers and analyze the relationship between the heat conductivity of composites and the degree of crystallinity of their polymer matrix. The results obtained could be widely used in the development of highly heat conductive polymer composite materials.

To accomplish the aim, the following tasks have been set:

– to perform a set of experimental studies to establish the dependence of heat conductivity and heat capacity of the resulting composites on the mass fraction of fillers;

– based on the results of experimental studies of temperature dependences of the heat capacity of the materials under consideration, to identify the relationship between the thermally conductive properties of composites and the degree of crystallinity of their matrix.

4. The study materials and methods

Polymer micro- and nanocomposite materials based on polyamide 6 were subject to consideration when it was filled with CNT or microparticles of copper or aluminum. These composites were obtained using a method based on mixing the components in the polymer melt by applying a special disc extruder [4].

The thermophysical properties of the studied composite materials were determined on the basis of standard methodological approaches. To determine their heat conductivity, an improved device IT-λ-400 (Moldova) was used, and the method of differential scanning calorimetry at the DSC-2 unit (Germany) with modified software was used to determine their heat conductivity.

During the studies, the mass fraction of fillers, ω, ranged from 0.3 to 10 %. This range of variation in ω corresponds to the presence of a significant dependence of the heat conductivity of composites on the content of fillers. A detailed description of the methods for obtaining fillers, their sizes, and manufacturers are given in [6]. The main characteristics of the used fillers are as follows. The method of obtaining CNTs is chemical vapor deposition. The outer diameter of CNT is 20 nm, the length is 1.5 μm, and the wall thickness is 5 nm. The method of obtaining microparticles is grinding in a ball mill to form particles with a size of 0.5...1.0 μm.

The degree of polyamide 6 crystallinity χ in the resulting composite materials was determined on the basis of experimental dependences of the heat capacity of composites on temperature according to the formula from [12]

$$\chi = \frac{\Delta H_m}{\Delta H_{mc}} \cdot 100\,\%, \qquad (1)$$

where ΔH_m, ΔH_{mc} is the melting enthalpy of the composite and the fully crystalline polymer.

5. Results of studying the thermophysical properties of polymer micro- and nanocomposites and the degree of crystallinity of the polymer matrix

5. 1. Results of determining heat conductivity and specific heat capacity of polymer micro- and nanocomposites

Fig. 1 illustrates the results of experimental studies to determine the heat conductivity of the composites in question as a function of the mass fraction of fillers (CNTs and microparticles of copper or aluminum).

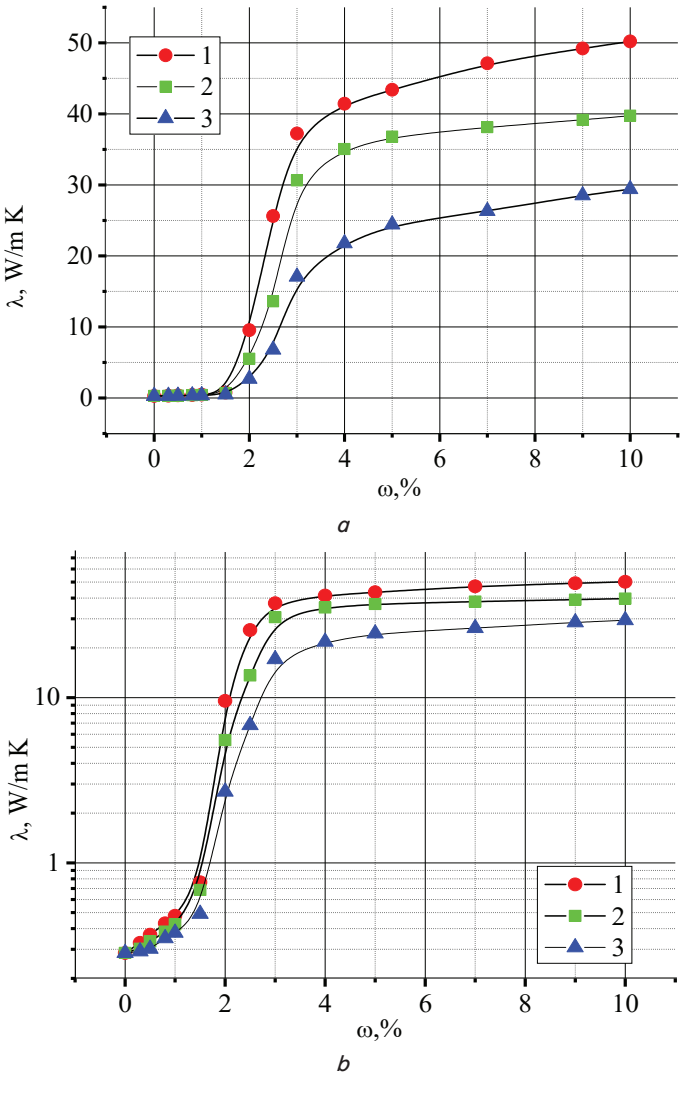

Fig. 1. Dependence on the mass fraction of fillers ω of the coefficient of heat conductivity λ of micro- and nanocomposite materials based on polyamide 6 filled with different fillers:
1 — carbon nanotubes; 2 — microparticles Cu; 3 — microparticles Al;
a — linear ordinate scale; *b* — logarithmic

Table 1 gives the experimental temperature dependences of the specific heat capacity of the studied composites for different values of the mass fraction of fillers ω.

Table 1

Temperature dependence of specific heat capacity c_p of polymer composites based on polyamide 6 filled with CNT, copper and aluminum microparticles for different values of the mass fraction of filler ω

Filler content, %	Temperature, K					
	450	460	470	480	490	500
CNT filler						
0.3	1.96	2.06	2.38	4.37	9.20	1.86
2.0	2.00	2.10	2.43	4.46	9.38	1.90
3.0	2.02	2.12	2.50	4.50	9.66	1.96
10.0	2.16	2.27	2.62	4.81	10.12	2.05
Copper filler						
0.3	2.07	2.17	2.54	4.74	10.11	1.96
2.0	2.11	2.21	2.59	4.83	10.31	2.00
3.0	2.21	2.31	2.63	4.97	10.59	2.08
10.0	2.28	2.39	2.80	5.21	11.12	2.16
Aluminum filler						
0.3	2.08	2.18	2.55	4.77	10.16	1.97
2.0	2.12	2.22	2.60	4.87	10.36	2.01
3.0	2.14	2.26	2.69	4.93	10.63	2.08
10.0	2.29	2.40	2.81	5.25	11.18	2.17

The test temperature range includes the melting point of the polymer matrix in the composite material (490 K).

5. 2. Results of studying the interconnection of the degree of crystallinity of the polymer matrix and the heat conductivity of polymer micro- and nanocomposites

Fig. 2 shows the results of calculations according to formula (1) of the degree of crystallinity of polyamide 6 in the studied range of changes in the mass fraction of fillers.

Table 2 gives the coefficients of the equation describing the relationship between the degree of crystallinity of the polymer matrix of the studied composites and their coefficient of heat conductivity

$$\lambda = a - b\chi. \qquad (2)$$

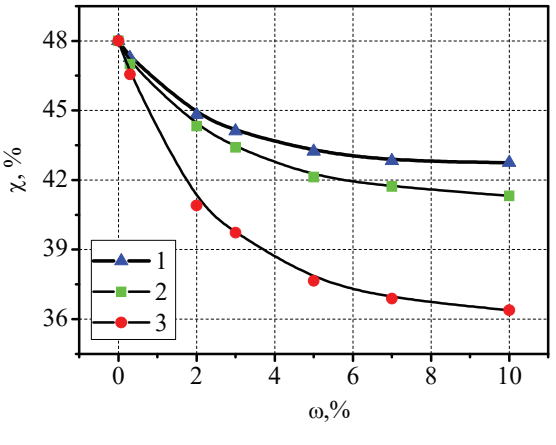

Fig. 2. Dependence of the degree of crystallinity of polyamide 6 on the mass fraction of the filler ω (%) for composites filled with different fillers: 1 – microparticles Al; 2 – microparticles Cu; 3 – carbon nanotubes

Table 2

Values of parameters *a* and *b* depending on (2) for different fillers and different zones of change in their mass fraction ω in polymeric micro- and nanocomposites

Change zone number ω	Parameter	Filler		
		CNT	Cu	Al
2	*a*	951.65	1241.5	923.02
	b	23.03	27.90	20.54
3	*a*	193.3	218.9	435.0
	b	3.930	4.338	9.477

Here, zone 2 corresponds to the range of change ω: 2<ω≤3.0 % and zone 3 corresponds to 3.0<ω≤10.0 %.

6. Discussion of results of studying the thermophysical properties of polymer micro- and nanocomposites based on polyamide 6

According to the data obtained, for the considered composite materials there is a tendency to increase their coefficient of thermal conductivity with an increase in the mass fraction ω of the filler (Fig. 1). Particularly noteworthy is the fact that with relatively small values of ω, different for different composites, there is a very sharp increase in their coefficient of thermal conductivity. This increase corresponds to a mass fraction of ω of 1.77 % when filling polyamide 6 with carbon nanotubes, 2.19 % with Cu microparticles, and 2.67 % with Al microparticles.

The presence of heat conductivity coefficient jumps on concentration dependences $\lambda=f(\omega)$, the so-called percolation thresholds, is explained within the framework of percolation theory. In general, on the curves $\lambda=f(\omega)$ there are two jumps in the heat conductivity coefficient of the composite material. The first jump (the first percolation threshold) is associated with the formation of continuous percolation clusters from filler particles, which play the role of thermally conductive channels. The second jump (the second percolation threshold) observed at higher values of ω is due to the formation of a percolation mesh from aggregates of filler particles, which leads to a further sharp increase in the thermally conductive properties of composites.

In this situation, only one percolation threshold is clearly expressed. This indicates the practical absence of the stage of formation of percolation clusters. That is, under the analyzed conditions, we are talking about the fact that from the filler particles evenly distributed in the polymer matrix with an increase in their mass fraction, a percolation grid is formed, bypassing the intermediate stage of the formation of percolation clusters.

As evidenced by the data obtained (Fig. 1, *b*), the lower the thermally conductive properties of the filler, the greater the value of the percolation threshold and, accordingly, the lower the rate of increase in the heat conductivity coefficient of the composite with an increase in the mass fraction of the filler. Indeed, according to the level of thermally conductive properties, the considered fillers are ranked in descending order as follows – CNT, Cu, and Al. The longitudinal heat conductivity of single nanotubes at room temperature is within 2800...6000 W/(m·K) [13]. The heat conductivity of Cu and Al is 384 W/(m·K) and 209 W/(m·K), respectively [14].

And the values of the corresponding percolation thresholds increase from polymers filled with CNTs to polymers filled with Cu and further Al.

As for the nature of the concentration dependence $\lambda=f(\omega)$, it is qualitatively similar when using all the fillers under consideration. Namely, after reaching the percolation threshold, a very sharp increase in the λ of composites is replaced by a significantly smaller increase in their thermal conductive properties with an increase in the mass fraction of the filler. This change in the behavior of the heat conductivity coefficient of composites corresponds to the value of the mass fraction of the filler equal to about 3 % when using all of the fillers in question.

From the data in Fig. 1, *b* it follows that the values of the coefficient of heat conductivity of composites are highest when filling polyamide 6 with carbon nanotubes, less high – with microparticles Cu, and lowest – with microparticles Al.

At the same time, the values of λ for the composites in the case of the use of these three fillers can differ very significantly. Thus, at ω=10 %, the coefficient of heat conductivity of composites is 50.2 W/(m·K), 39.7 W/(m·K), and 29.4 W/(m·K), respectively, for polyamide 6 filled with CNT, Cu, Al.

Our experimental studies of the temperature dependence of the specific heat capacity c_p of composites indicate that this dependence has a pronounced extreme character (Table 1). At the same time, the maximum c_p corresponds to the melting point T_m of the composite material. This temperature, according to the data obtained in the considered physical situations, is almost equal to the melting point of the polymer matrix. The described nature of the dependence $c_p=f(\omega)$ is observed with all the considered values of ω.

Table 1 demonstrates that the c_p value of the composite materials increases slightly with the increase in the mass fraction of the filler ω. In the studied temperature range at a fixed value of ω, the highest values of heat capacity occur when filling the polymer matrix Al, somewhat smaller – Cu, and smallest – CNT.

The results of our studies of the dependence of the heat capacity of composites on temperature served as the basis for determining the degree of crystallinity of the polymer matrix χ of the studied composites. Fig. 2 shows that with an increase in the mass fraction of the filler ω, the degree of crystallinity χ of the polymer decreases. Moreover, this decrease is the most significant when filling polyamide 6 with CNTs, less significant for Cu, and smallest for Al. The described regularity is due to the fact that with an increase in the mass fraction of the filler, the degree of branching of percolation meshes increases, which are steric obstacles to the formation of crystal structures in the polymer matrix. And since the efficiency of the formation of percolation structures is higher when filling the polymer with CNT than Cu and Al, the decrease in the degree of crystallinity of the matrix with an increase in ω occurs more sharply for composites filled with CNTs.

As for the features of the dependence $\chi=f(\omega)$, here we can distinguish three zones that differ in the intensity of decreasing the degree of crystallinity with an increase in ω. The first zone, corresponding to low values of ω (ω is less than about 2 %), is characterized by a very sharp decrease in the degree of crystallinity. That is, the presence of a relatively small number of filler particles that have not formed into pronounced percolation structures leads to a sharp decrease in the degree of crystallinity χ.

In the second zone, corresponding to the range of change in the mass fraction of the filler from about 2 % to 3 %, the change in the degree of crystallinity χ with an increase in ω occurs less intensively. And then in the third zone (ω>3 %), the intensity of the decrease χ with an increase in ω decreases. This seems to be due to the fact that increasing the degree of branching of the percolation grid has less and less effect on the formation of crystal structures in the third of the considered zones.

Attention is drawn to the fact that similar zones can also be distinguished on curves $\lambda=f(\omega)$. Although, unlike the descending dependence $\chi=f(\omega)$, the dependence $\lambda=f(\omega)$ is ascending. Of particular interest is the comparison of these dependences in the second and third of these zones. Here, in both cases, there is a noticeable drop in the intensity of change in the studied values (λ and χ) during the transition from the second zone to the third one. The relationship between the heat conductivity coefficient λ of the composite and the degree of crystallinity of the polymer matrix χ in this composite in the second and third zones can be expressed by ratio (2). Table 2 gives the values of the coefficients of equation (2) for the two considered zones of change ω when CNT, Cu, and Al are used as a filler.

The existence of a relationship between the values λ and χ for the composites under consideration is probably due to the fact that the value of both these quantities is largely determined by the regularities of the formation of percolation structures from filler particles.

The results of our studies represent a certain contribution to solving the problem of obtaining comprehensive information on the thermophysical properties of highly heat conductive polymer micro- and nanocomposite materials.

The thermophysical properties of highly heat conductive composites based on only one polymer matrix and several highly heat conductive micro- and nanofillers are investigated in the current work.

Further studies into the thermophysical properties of highly heat conductive polymer composites can be aimed at expanding the range of both polymer matrices and fillers. In addition, it is interesting to advance research on the establishment of the relationship between the thermophysical properties and the structure of polymer composites.

7. Conclusions

1. For polymer micro- and nanocomposites based on polyamide 6 filled with CNTs or particles of copper or aluminum, a set of experimental studies into their thermophysical properties – the coefficient of heat conductivity and specific heat capacity – was performed. The dependences of the heat conductivity of the polymer composites under consideration on the mass fraction of the filler in the range of its change from 0.3 % to 10 % were derived. It is shown that the coefficients of heat conductivity of composites when filling the polymer matrix with CNT can significantly exceed the corresponding values when it is filled with copper and aluminum. The possibility of obtaining polymer composites with heat conductivity coefficients of 50.2 has been established; 39.7 and 29.4 W/(m·K) when CNTs, copper and aluminum are used as fillers, respectively, with a content of 10 percent. The fact of presence of only one percolation threshold for the considered composites was discovered. It is shown that smaller values of the percolation threshold correspond to large values of the thermally conductive properties of the filler.

For the designed polymer composite materials, experimental studies of the dependence of their specific heat capacity on the temperature in a wide range of changes in the mass

fraction of the filler were carried out. It is established that the highest values of heat capacity occur when filling the polymer matrix with aluminum, somewhat smaller – when it is filled with copper and the smallest – with carbon nanotubes. It is shown that in the entire considered temperature range there is an increase in the heat capacity of composite materials with an increase in the mass fraction of fillers.

2. Based on the results of experimental studies into the specific heat capacity of composites, data on the degree of crystallinity χ of their polymer matrix were obtained. It was found that the decrease in the degree of crystallinity χ with an increase in the mass fraction of the filler is the most significant when using CNT as a filler, the least significant – copper, and the least – aluminum. It is noted that the nature of the dependence $\chi=f(\omega)$ is due to the fact that with an increase in the mass fraction of the filler, the degree of branching of percolation meshes increases, which are steric obstacles to the formation of crystal structures in the polymer matrix. It is shown that there is a certain correlation between the degree of crystallinity χ of the considered micro- and nanocomposites and their coefficient of heat conductivity λ. The presence of this dependence is explained by the fact that the values of χ and λ are largely determined by the patterns of formation of percolation structures from filler particles.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

Acknowledgments

This study was partially funded within the framework of DR project No. 012U110115 (Order No. 257 as of 25.02.2021) of fundamental and applied research and scientific and technical developments of higher education institutions and scientific institutions for 2021, which are managed by the Ministry of Education and Science of Ukraine.

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