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Structural Features of Carbon Nanomaterials and Determination of Their Thermophysical Properties Using Pulsed Radiation

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The thermophysical properties of carbon nanomaterials (CNMs), such as thermally expanded graphite, carbon nanotubes, and globular multilayer graphene, are investigated. A series of samples is produced by the technique of one-sided static pressing. By means of the impulse method, the parameters

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of the thermal conductivity of the samples are determined depending on the conditions of their production. As found out, the thermal conductivity of the CNMs increases with increasing density and pressure. As assumed, the mechanisms of heat transfer in these systems can be related to the electron–lattice interaction. Heat transfer can occur by various transport mechanisms.

Key words: thermal conductivity, pulsed radiation, thermally expanded graphite, globular multilayer graphene, multi-walled carbon nanotubes.

Досліджено теплофізичні властивості вуглецевих наноматеріялів (ВНМ), таких як терморозширений графіт, вуглецеві нанотрубки та ґлобулярний багатошаровий графен. Серію зразків було виготовлено методою одностороннього статичного пресування. За допомогою імпульсного методу було визначено параметри теплопровідности зразків залежно від умов їхнього виробництва. Встановлено, що теплопровідність ВНМ зростає зі збільшенням густини та тиску. Припускається, що механізми теплопередачі в цих системах можуть бути пов'язані з електрон-ґратницевою взаємодією. Теплопередача може відбуватися за допомогою різних механізмів перенесення.

Ключові слова: теплопровідність, імпульсне випромінення, термічно розширений графіт, ґлобулярний багатошаровий графен, багатостінні вуглецеві нанотрубки.

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1. INTRODUCTION

Modern science connects our future with nanomaterials and nanotechnologies. The widespread implementation of new technologies, including nanotechnologies, into industrial production is achieved by a high level of standardization, which is not possible without the preliminary development of research methods and devices for evaluating new materials. The methods for investigation of carbon nanomaterials and new techniques and tools that allow accurate determination of their structural and thermophysical parameters are developed in many scientific centres and laboratories around the world [1]. In particular, experimental work on the characterization of nanostructured materials is important, along with their manufacture and application. The study of the properties of carbon nanotubes (CNTs), thermally expanded graphite (TEG) and globular multilayer graphene (GMLG), and the development of new materials based on them is a topical task which also includes the development of new hardware for measurements, data processing and representation.

It should be noted that the real experimental characteristics of carbon nanomaterials are much worse than those predicted by theoretical calculations. First of all, this is explained by the structural imperfections in carbon nanomaterials; however, the problem to a large extent is related to the nature of the tests which are used in research works [1–3]. It is very difficult to prepare test samples without damaging the nanotubes. Another problem is the practical difficulties in representing the experimental results with sufficient resolution.

A sufficiently large amount of material is required, when developing materials with given heat insulation properties, which cannot be provided by the modern technologies of graphene production. At the same time, the requirements for graphene-like modifiers are significantly less strict than those for graphene samples that are designed for use in electronics. In particular, one can expect that graphene fragments with large area are not needed for thermal insulation; the use of carbon monolayers is also not necessary. Therefore, increased attention is paid to the development and investigation of high-tech carbon material of a new generation based on thermally expanded graphite. Thermally expanded graphite (it is often called graphite foam or vermicular graphite) is a low-density carbon material that has unique physical and chemical properties: high specific surface area, sufficiently high thermal and chemical resistance, low thermal conductivity, high porosity. The present work represents the results of investigations of the technology developed for producing carbon nanomaterials; the structural features of these materials are studied, and their thermophysical properties are analysed.

Currently, there are two approaches to determining the thermal conductivity of materials: stationary and non-stationary ones. Each of these approaches has its own sources of uncertainty that can affect the accuracy of the measurements. The most reliable method is stationary one, but it can take a lot of time to measure thermal conductivity of one sample. Therefore, the non-stationary method, or the thermal pulse method, is increasingly used for faster measurements. The following factors are essential during the process of impulse heating of material. Since light penetrates into the sample, the sources of optical heating should be volumetric. The energy is released non-uniformly throughout the volume of interaction, as the intensity of light decreases as it penetrates into the sample. This leads to spatially inhomogeneous heating of the material, and causes heat and mass transfer processes between different zones of the sample [4–7].

The aim of the present work was to study the structural features and thermophysical properties of thermally expanded graphite, carbon nanotubes, and globular multilayer graphene.

2. EXPERIMENTAL TECHNIQUES AND DISCUSSION

A carbon-containing gaseous source, natural gas, was used as a raw material for the synthesis of multi-walled carbon nanotubes (MWCNTs). The gas was previously converted on a nickel catalyst, and then, the conversion products were used for the synthesis of MWCNTs. It was found out that the MWCNTs formed at moderate temperatures due to the treatment of freshly reduced iron with products of air conversion of natural gas [8]. As a result of further crystallization of carbon, nanotubes were formed, and iron particles were separated from the main material, *i.e.*, the catalyst material was fragmented. It was found out that carbonisation–decarbonisation cycles in the γ-Fe, α-Fe, and Fe_3C system play a decisive role in the formation of MWCNTs below 700°C [8, 9].

In order to determine the dependence of the density of carbon nanomaterials on the pressing force and thermal conductivity, a series of samples was produced by one-sided static pressing in a cylindrical steel matrix with a diameter of 12 mm. The pressing was carried out at a hydraulic press at a pressure in the range of 170–450 MPa. The mass, volume and density of the samples after pressing were determined. Four samples were produced for each carbon material.

The morphology and microstructure of the synthesized carbon nanomaterials (CNMs) were examined using scanning electron microscopy (SEM) technique at a JSM-6490LV microscope. The microstructure of carbon nanomaterials produced by various methods [10] and synthesis modes is shown in Fig. 1.

The CNT material has a so-called ‘sponge’ morphology (Fig. 1, *a*, *b*), *i.e.*, it is comprised of intertwined tubes of different diameters ranging

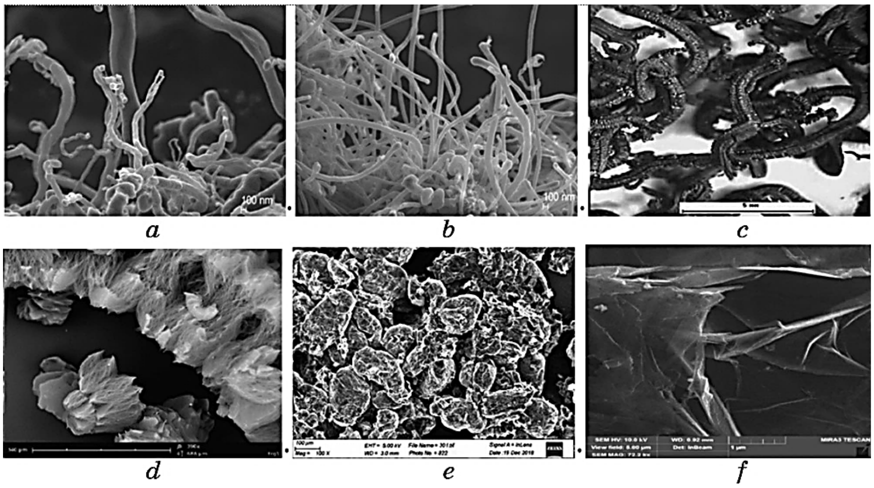
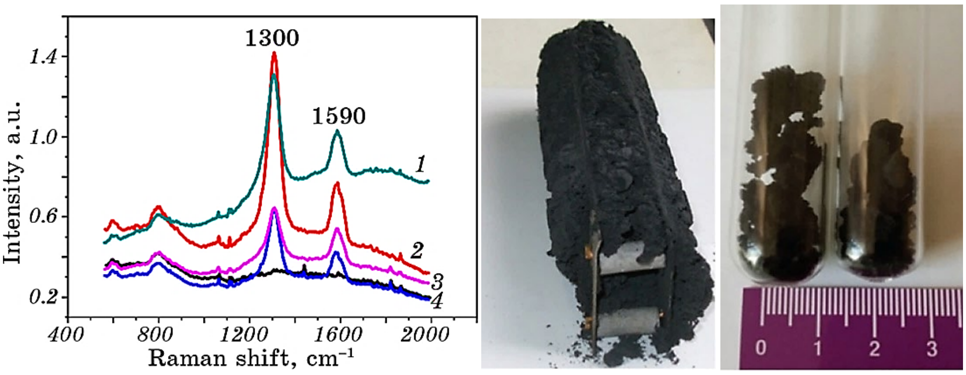


Fig. 1. Microstructure of carbon nanomaterials: multi-walled carbon nanotubes (*a*, *b*), thermally expanded graphite (*c*, *d*), globular multilayer graphene (*e*, *f*).



a *b* *c*

Fig. 2. Raman spectra of carbon nanomaterials and images of synthesized carbon nanotubes: Raman spectra of CNTs (*a*), CNTs on an iron catalyst (*b*), CNT samples in glass tubes for determination of Raman spectra (*c*).

from 20 to 300 nm. The various diameters are explained by the sizes of the iron grains which detached from the freshly reduced metal plate in a hydrogen atmosphere during the disproportionation of carbon monoxide.

Raman scattering spectra of the samples (see Fig. 2, *a*–*c*) were studied using a mini-RamanPro Raman spectrometer Lightnovo (Denmark). A laser with a wavelength of $\lambda = 785$ nm for the spectral range of 600–2000 cm^{-1} was used. The accuracy of determining the wave number was in the range of 0.5–2 cm^{-1}. The exposure time was 100 ms, 50 spectra were averaged. In each case, the Raman spectra of all samples were recorded using the same parameters. The Raman spectra confirmed the presence of carbon nanotubes with two characteristic peaks at 1300 and 1590 cm^{-1}, which correspond in Fig. 2 to the D and G modes, respectively. The first, second, and fourth spectra (Fig. 2) are typical spectra of nanotubes with characteristic D and G modes. In all three samples, the D mode had a higher intensity than the second one, which is not typical for intact single-walled nanotubes. Therefore, one can conclude that the nanotubes deposited by the CVD method were multi-walled and most likely had a curved shape. In all three samples, a shoulder in the G mode was clearly observed (which may be an overtone of the D mode), that indicated the high density of defects in the samples.

In order to determine the dependence of density and thermal conductivity on pressing pressure in the range of 170–450 MPa, sets of samples from carbon nanotubes, thermally expanded graphite, and globular multilayer graphene were produced (Fig. 3).

Thermally expanded graphite is a product of multi-stage technological processing [11] of natural crystalline graphite powder. Figure 1, *c*

a *b* *c*

Fig. 3. Appearance and dimensions of: carbon nanotubes (*a*), thermally expanded graphite (*b*), globular multilayer graphene (*c*).

shows the appearance of TEG particles at the macro-level, which consist of individual worm-shaped 5–10 mm long particles. The microstructure of TEG at the microlevel is shown in Fig. 1, *d*: the structure of a single particle is seen, which consists of separate graphene clusters comprised of 100–500 elements. According to Ref. [12], the main stages of TEG production include oxidation of flaky graphite, its drying, thermal expansion and subsequent processing aimed at obtaining the final product or part. The mechanism of transformation of oxidized graphite into TEG was also analysed; it is related to a sharp increase in pressure between the layers due to rapid heating (thermal shock). The pressure that occurs during the thermal shock causes foaming and intermolecular explosion, which are accompanied by the formation of a peculiar foam-like structure [12].

In Refs. [13, 14], a method of producing globular multilayer graphene was described. The technique includes mechanical processing of TEG with the aim of destruction it into separate graphene layers (from several to dozens of layers) and forming globules with approximately the same size ($\cong$ 100 mm). The microstructure of individual globules is shown in Fig. 1, *e*, and the morphology of globules comprised of crumpled graphene layers is seen in Fig. 1, *f*.

We have developed a method for producing globular graphite, which consists of several stages. The first stage is processing of TEG in a cavitation apparatus with the aim of its temporal transformation from a hydrophobic material into a hydrophilic one. The second stage is the rapid ultrasound processing of TEG soaked in a liquid—the destruction of TEG into individual graphene layers mainly occurs at this stage. The third stage is drying followed by finishing in a mill with the aim to form the structure of the material (in particular, to crush coarse globules and form spherical globules of the same size). A detailed description of the equipment and techniques for producing TEG is given in Ref. [15].

The thermophysical parameters of thermally expanded graphite, carbon nanotubes and globular multilayer graphene were determined by the pulse method for measuring thermal conductivity (PMMTC). This is a non-destructive and non-contact method. In this method, the front surface of the sample is irradiated with a pulse of light in the visible range ($\tau = 100$ ms). This is the 'primary' radiation flow. The ab-

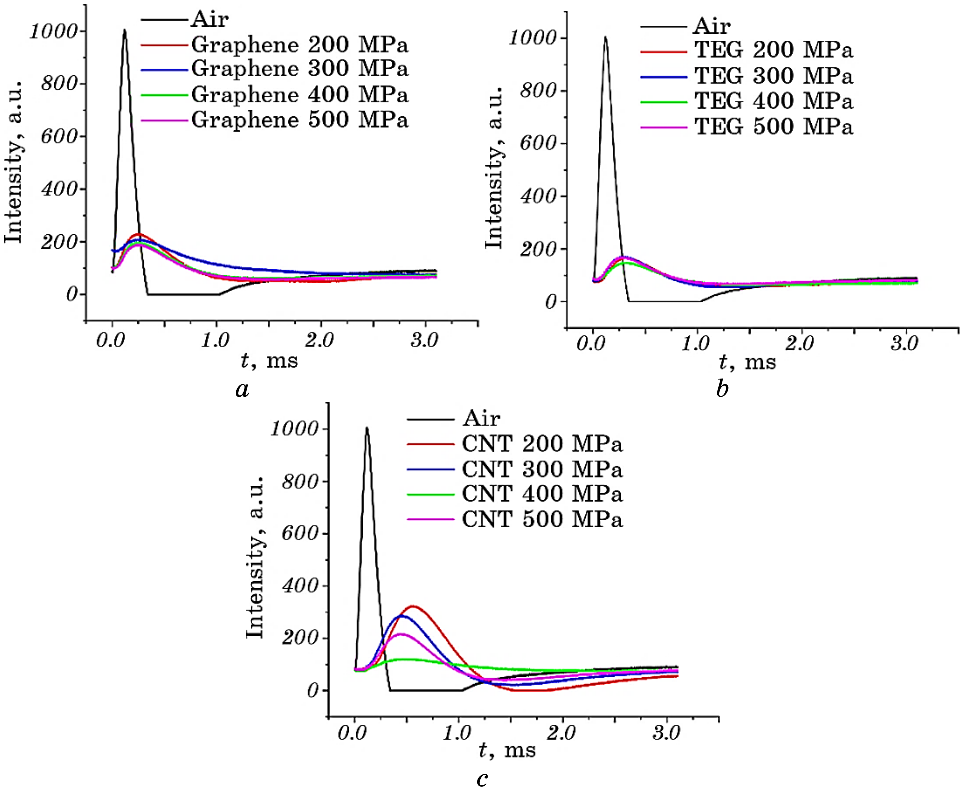


Fig. 4. Measuring thermal conductivity delay time between the 'primary' and 'secondary' heat flows in: globular multilayer graphene (*a*), thermally expanded graphite (*b*), carbon nanotubes (*c*).

sorbed part of this 'primary' flow is converted into thermal energy, which causes an increase in temperature and gives rise to the propagation of temperature waves into the sample. A certain part of not dissipated heat flow causes an increase in temperature on the opposite back surface of the sample, where it converts into a 'secondary' flow of infrared (IR) radiation. This 'secondary' flow of IR radiation is delayed relative to the incident 'primary' one due to the finite rate of the heat diffusion process; it is registered by an IR detector. When a pyroelectric sensor [16] is used as the IR detector, there is no need to know the values of the incident and absorbed energy, the absorption coefficient of the front surface, and the emission coefficient of the back surface of the sample, as well as the profile of the temperature increase over time and the pyroelectric parameters of the sensor [17, 18].

A light-emitting diode (LED) as a radiation heater and a pyroelectric detector as a thermal sensor (PyES) were used. The delay time between

TABLE 1. Calculation of thermal conductivity.

Sample number	Sample mass, g	Volume, cm^3	Density, g/cm^3	Sample thickness L, $\times 10^{-6}$ m	Air τ, $\times 10^{-3}$ s	Sample τ, $\times 10^{-3}$ s	Diffusivity χ, $\times 10^{-6}$ m^2/s
CNT							
1	0.240	0.258	0.99	2280	120	557	1.2
2	0.230	0.171	1.34	1510	120	457	6.76
3	0.240	0.181	1.32	1600	120	469	7.3
4	0.250	0.184	1.36	1630	120	445	8.17
Graphene							
1	0.110	0.062	1.77	550	120	250	2.32
2	0.130	0.068	1.90	680	120	250	2.36
3	0.150	0.071	2.11	710	120	256	2.92
4	0.160	0.078	2.05	780	120	256	3.50
TEG							
1	0.170	0.188	0.90	1660	120	315	1.41
2	0.150	0.115	1.30	1020	120	280	6.50
3	0.160	0.107	1.50	1030	120	280	6.65
4	0.190	0.118	1.61	1040	120	280	6.7

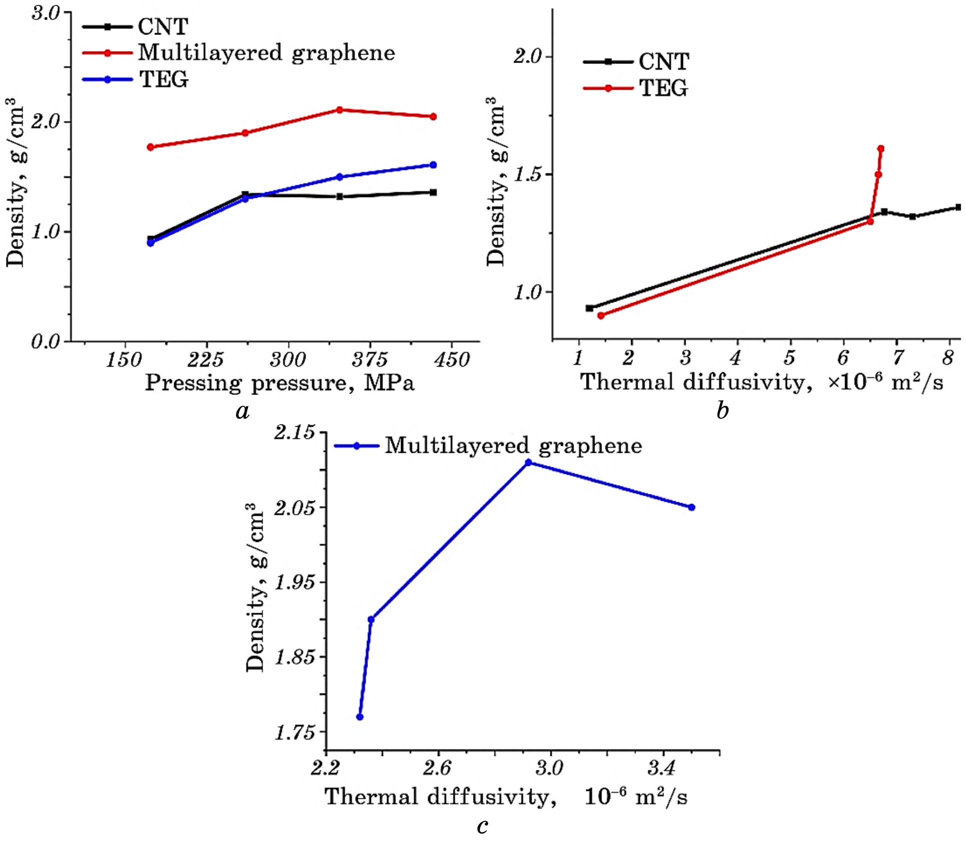
‘primary’ heat flow (without a sample) at PyES and ‘secondary’ heat flow (with a sample) is illustrated in Fig. 4.

The densities and thicknesses of the samples for calculating the thermal conductivity values of carbon nanotubes, thermally expanded graphite and globular multilayer graphene are listed in Table 1.

All studied materials (CNT, TEG, GMLG) consisted of the same substance, carbon; however, they had different methods of synthesis and structures at the macro- and microlevels. The dependences of the density of CNMs on the pressing pressure are shown in Fig. 5. As can be seen, increasing the pressing pressure in the range of 170–450 MPa leads to a slight increase in the density of CNMs. Under the given pressing conditions, samples had density 0.9–2.05 g/cm^3. The dependence of the density of TEG on the pressing pressure shows that this material can be compacted without deformation and change of shape. Nanotubes and globular multilayer graphene partially recovered their initial shape after the load was removed due to elastic aftereffect.

The thermophysical properties of carbon nanomaterials of the same brand can vary significantly. This is explained by different porosity of the materials, which has a significant effect on their thermal conductivity. The porosity of carbon materials of the same brand is uniquely related to the macroscopic density of the samples. It is found out that the density of GMLG, CNT and TEG increases with pressing pressure increasing (see Fig. 5, *a*). In the process of TEG compaction, it was found that TEG powders had an extended zone of structural compaction; *i.e.*, the material formed in a wide range (Fig. 5, *a*) of densities at pressures 170–450 MPa. It was found out that, at the first stage of the formation process, at low loads, the powder particles structurally repacked, then compaction occurred due to the extended surface of the particles (which had a popcorn-like shape), and finally they deformed.

However, the compaction of CNT and GMLG occurred slowly in a small range without significant changes. It is shown that the CNTs and GMLGs during formation at pressing pressures 200–500 MPa without binding additives begin to delaminate and lose strength. It is found out that the thermal conductivity of GMLG, CNT and TEG increases with



a *b*

c

Fig. 5. Dependences of density of carbon nanomaterials on pressing pressure at room temperature 293 K: carbon nanotubes, thermally expanded graphite, globular multilayer graphene (*a*), on thermal conductivity (*b*, *c*).

increasing density and pressure (Fig. 5, *b*, *c*).

The mechanism of changes in thermal conductivity of these systems is likely related to electron-lattice interaction. Heat transfer can occur by various transport mechanisms (collisions, diffusion). However, exactly the electronic mechanism can be responsible for the increase in thermal conductivity. The relationship between the thermal conductivity of carbon nanomaterials and their macroscopic density is determined.

3. CONCLUSIONS

The structural features of thermally expanded graphite, carbon nanotubes and globular multilayer graphene, which were produced at the equipment of the Gas Institute, N.A.S. of Ukraine, have been studied.

A device for the pulse method of measuring thermal conductivity of diverse materials with registration of information on a PC has been developed. This system was developed at the Technical Centre, N.A.S. of Ukraine. Thermal conductivity of carbon nanomaterial samples produced at different pressing pressures was measured. It is determined that the thermal conductivity of CNMs increases with increasing density and pressure. It is shown that the mechanisms of heat transfer in these systems are related to the electron–phonon interaction. Heat transfer can occur by an electronic or diffusion mechanism; nevertheless, exactly the electronic mechanism causes the increase in thermal conductivity of the samples of thermally expanded graphite, carbon nanotubes and globular multilayer graphene.

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AUTHORS’ CONTRIBUTIONS

A. I. Khovavko, E. V. Strativnov, D. S. Filonenko, and A. A. Nebesnyi provided experiment on carbon nanomaterials synthesis; A. A. Nebesnyi made theoretical calculations. A. I. Khovavko and E. V. Strativnov contributed to the statement of the problem and the way to solve it, discussed the results and helped in preparing and approving the final manuscript. A. V. Minitskyi, R. V. Lytvyn, D. S. Leonov, A. A. Kolesnichenko, and M. Yu. Barabash took part in one-sided isostatic pressing and measuring the geometric dimensions of samples, calculating the density, contributed to the statement of the problem and discussion of the results. A. I. Khovavko and M. Yu. Barabash contributed to the statement of the problem and discussion of the results, took part in the formulation of problems and ways to solve it, M. Yu. Barabash analysed the Raman spectra, took part in thermal diffusivity measurements and theoretical calculations. D. S. Leonov and R. V. Lytvyn participated in the task and designed the text composition according to the requirements of article publication. All authors read and approved the final manuscript.

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