

Electrical and thermal diffusivity of highly porous multigraphene structures

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The dependence of electrical conductivity and thermal diffusivity on the density of multigraphene structures at the initial stage of the compaction has been studied. It was shown that at the density of $\sim 0.05 \text{ g/cm}^3$ (ρ_{cr1}), the mechanism of electrical conductivity changes and an inflection point is observed on the curve, showing the dependence of material density on the compaction force. A change in the thermal diffusivity mechanism was detected by the laser flash method at the density of $\sim 0.4 \text{ g/cm}^3$ (ρ_{cr2}). The interval $[\rho_{cr1}, \rho_{cr2}]$ is considered to be the range in which a highly porous multigraphene structure with unique thermal resistance properties exists. When the first critical point is approached, the contact bridges of conductivity are formed, while the second point indicates the transition to continuously layered multigraphene material.

Keywords: multigraphene material, multigraphene structures, pressing, electrical conductivity, thermal diffusivity, porosity.

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Multigraphene material (MGM) is a sheet material obtained by physico-chemical treatment of natural flake graphite. From a chemical point of view, MGM essentially retains the structure of graphite, which allows it to maintain high chemical and thermal stability [1]. However, such an effect makes it possible to give MGM flexibility and enhance its characteristics by pressing independent particles into a homogeneous mechanically durable compact [2,3]. The characteristics and applications of MGM largely depend on the bulk density of the compact. For example, materials with a density of $0.7\text{--}1.1 \text{ g/cm}^3$ have found application as seals, with a further increase in density to 1.8 g/cm^3 the material acquires high thermal conductivity up to $500 \text{ W/(m} \cdot \text{K)}$ [4], which determined its use as an efficient and lightweight heat conductor [5,6].

The MGM production process is based on the chemical introduction of a foaming agent into flake graphite, which, upon rapid heating, generates a gas pressure within the graphite matrix and significantly change its morphology, transforming solid graphite particles into a lightweight foam composed of multigraphene structures (MGS) [7,8]. A distinctive feature of MGS is its ability to compact without a binder, which is facilitated by its low hardness and developed surface. As a rule, the bulk density of MGS varies in the range of $0.001\text{--}0.010 \text{ g/cm}^3$ [9]. Such a significant difference in the densities of the raw materials and the final material creates a significant area of uncertainty. As a rule, researchers pay attention to the final materials with a high density [10], whereas the compaction process and the intermediate highly porous state of the material are poorly described.

A separate advantage of MGS and MGM is the high emissivity inherited from graphite [11]. It is likely that some highly porous form of MGM could potentially be used as a thermal radiator [12]. In this case, it is necessary to address the gaps in the understanding of transitional processes in the formation of MGM from MGS.

The purpose of this study is to determine the critical points in the transition from MGS to MGM by determining the conductivity parameters.

In this paper, we investigated the MGM compact obtained from +899 mesh flake graphite. The powder was exposed to 98% fuming nitric acid in a mass ratio of 1:0.8, after which it was washed with water, dried, and heated to a temperature of 1000°C for 3 s. The formed MGS was compacted in the density range of $0.01\text{--}0.70 \text{ g/cm}^3$. The MGS was indented in a cylindrical mold with a diameter of 12.7 mm in the load range of $10\text{--}700 \text{ N}$. The MGS was compacted directly into the measuring cell to study the electrical conductivity.

The microstructure of the MGS and MGM was studied using scanning electron microscopy. The phase composition of the samples was determined by X-ray diffraction using CuK_α -radiation. Electrical conductivity was measured by recording current-voltage characteristics using a four-probe method. Thermal diffusivity was measured by the laser flash method. The samples were compacted using a universal testing machine under controlled displacement and load.

The electrical conductivity of highly porous MGMs was measured as a function of the density of the samples to investigate the first critical point (ρ_{cr1}). Fig. 1, *a* shows the dependence of electrical conductivity on density.

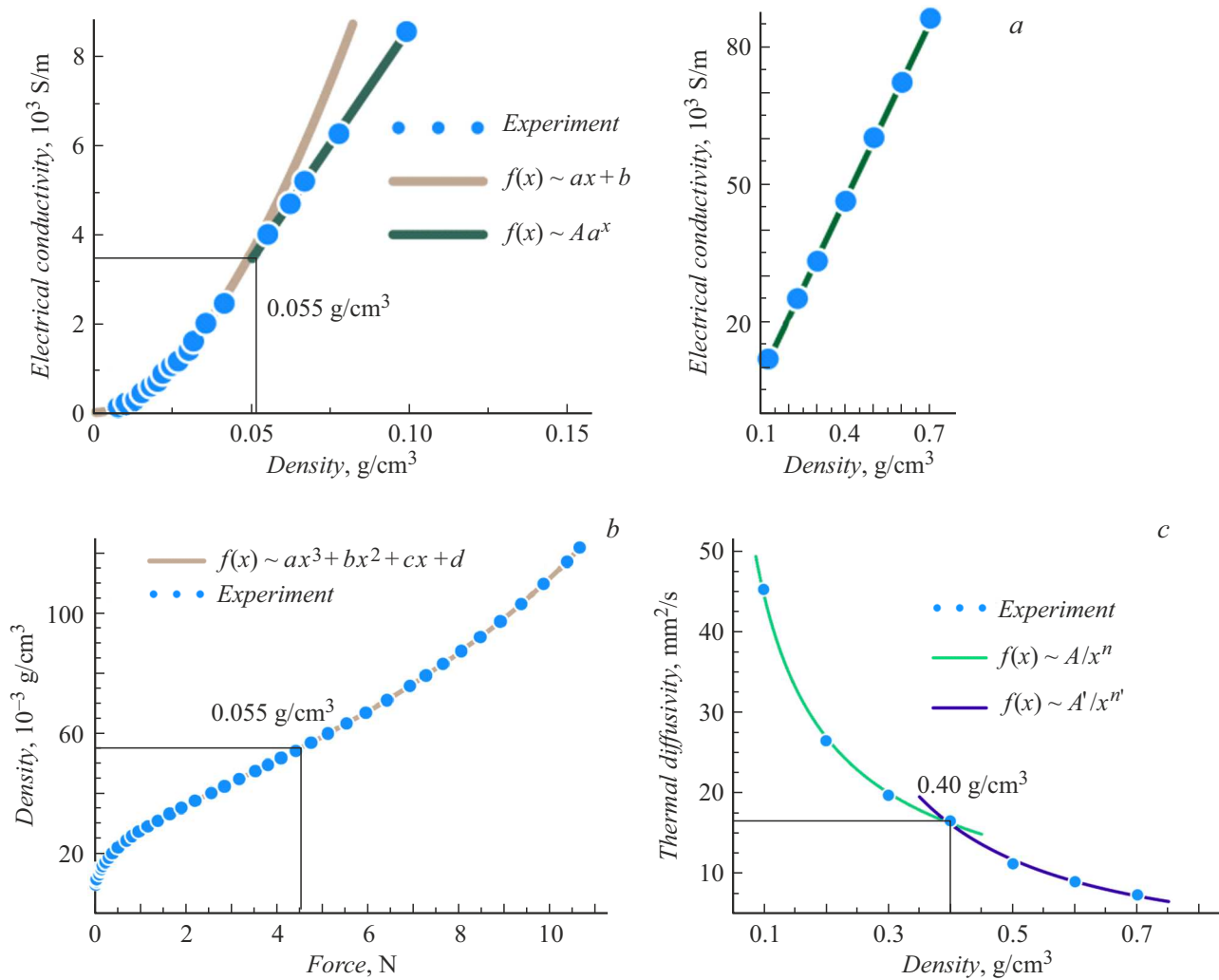


Figure 1. *a* — dependence of MGM electrical conductivity on density; *b* — dependence of MGM density on applied sealing pressure; *c* — dependence of MGM thermal diffusivity on density.

Unlike high-density MGM ($\rho > 0.7 \text{ g/cm}^3$), which demonstrate a linear increase in electrical conductivity with increasing density [13], these samples are characterized by an exponential function, which changes to a characteristic linear dependence after overcoming the first critical point at a density of $\sim 0.055 \text{ g/cm}^3$. This behavior is due to various types of conductivity. At a density of less than 0.055 g/cm^3 , electron transport through the sample occurs due to the percolation effect [14], i.e., randomly formed conductive bridges between independent MGS particles (Fig. 2, *a*).

When the first critical point is overcome, stable conduction channels are formed, and further linear growth is associated with an increase in their number and in the contact area, i.e. with a decrease in the interface resistance (Fig. 2, *b*) between the MGS particles.

A characteristic change in the behavior of the material during compaction can also be observed in compression curve shown in Fig. 1, *b*. The general appearance of the curve appears to be similar to a hyperbolic sine, but describing it using separate functions separated by the

first critical point is not a trivial task. As the sample density increases from the minimum value of 0.005 g/cm^3 to 0.055 g/cm^3 , an increase in the number of incidental contacts is observed, which increases the necessary compression pressure, however, a further increase in density is associated with plastic deformation of a single highly porous compact, and an increase in density is accompanied by the increase in friction forces between deformable interpenetrating particles (Fig. 2, *b*). This behavior confirms the existence of the first critical point.

Another important aspect of the existence of an intermediate highly porous MGM form is the presence of a second critical point (ρ_{cr2}), which can be detected by measuring the thermal diffusivity of MGM parallel to the compression axis (Fig. 1, *c*).

It can be seen that as the density increases from the minimum value of 0.1 g/cm^3 to 0.4 g/cm^3 , the function has a hyperbolic form. The decrease in the conductivity parameter is caused by the organization of the MGS particles into an ordered structure by compressing them:

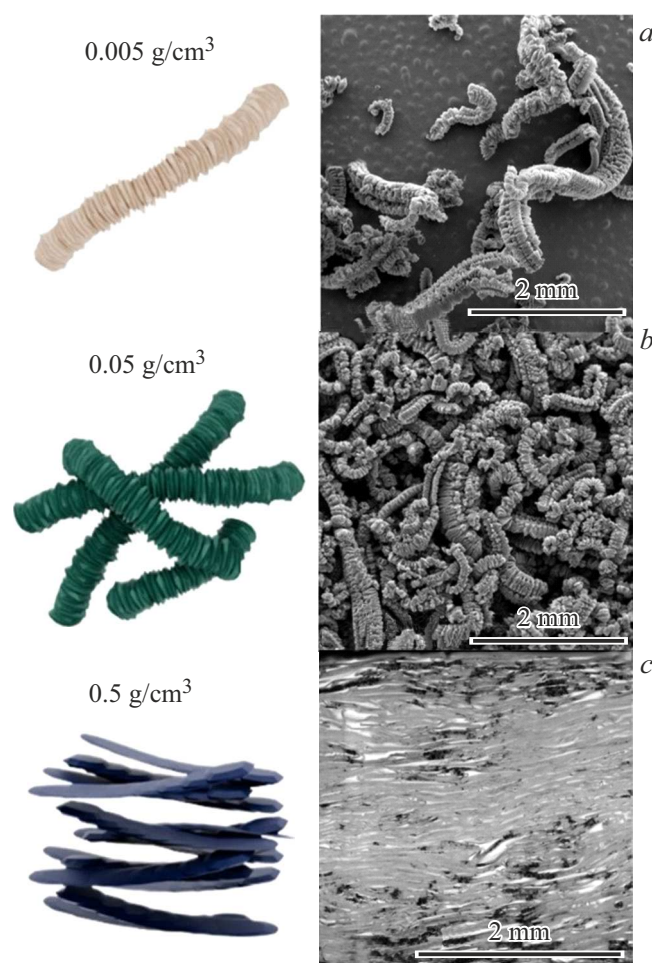


Figure 2. MGM particles (a), highly porous MGM compact (b) and high-density MGM compact (c).

the particles are oriented perpendicular to the axis of compression. When the critical point is overcome, the nature of the curve does not change, but the function values change.

An important difference between the presented thermal diffusivity and electrical conductivity is the direction of measurement and the anisotropic crystal structure of graphite. As is known, graphite exhibits more pronounced conductive properties along the basal planes, which indicates a significant difference in the electrical conductivity and thermal diffusivity of a quasi-single crystalline graphite: 2.26 MS/m and 1250 mm²/s, respectively, along the basal plane, and 590 S/m and 3.55 mm²/s, respectively, perpendicular to it [15]. However, as the MGS particles are compacted, they tend to orient perpendicular to the compression axis, which increases thermal resistance and reduces electrical resistance. In addition, plastic deformations occur, contributing to the transformation of complex-shaped MGS particles into a layered structure. The diffraction patterns shown in Fig. 3 confirm this thesis. The low-density compact (0.005 g/cm³) is characterized by a low

intensity of the main basal (00.2) reflection and the presence of weak (10.1) and (11.0) reflections, which indicates a high degree of misorientation of the MGS particles.

As the density (0.05 g/cm³) increases to the second critical point, the intensity of (00.2) reflex increases significantly and the (10.1) and (11.0) intensities decrease, which confirms the thesis about the reorientation of particles and characterizes an increase in electrical conductivity and a decrease in thermal diffusivity. With a further increase in density (0.5 g/cm³), the (10.1) and (11.0) reflection intensities become indistinguishable at the background level, which indicates a change in the pressing process: the role of reorientation in the compaction process increases (Fig. 2, c).

Thus, by measuring the conductivity parameters of a multi-graphene material, transients in the formation of a continuous layered compact from a mass of independent particles of multi-graphene structures have been identified. The results demonstrate that as the density increases, the material undergoes changes in the nature of its conductivity due to the formation of an oriented highly porous structure. Ideas about the mechanism of formation of an integral compact allow us to conclude about the practical value of developing new low-density carbon-based materials: density and degree of orientation control can be used as a tool for changing the functional properties of a highly porous multigraphene material.

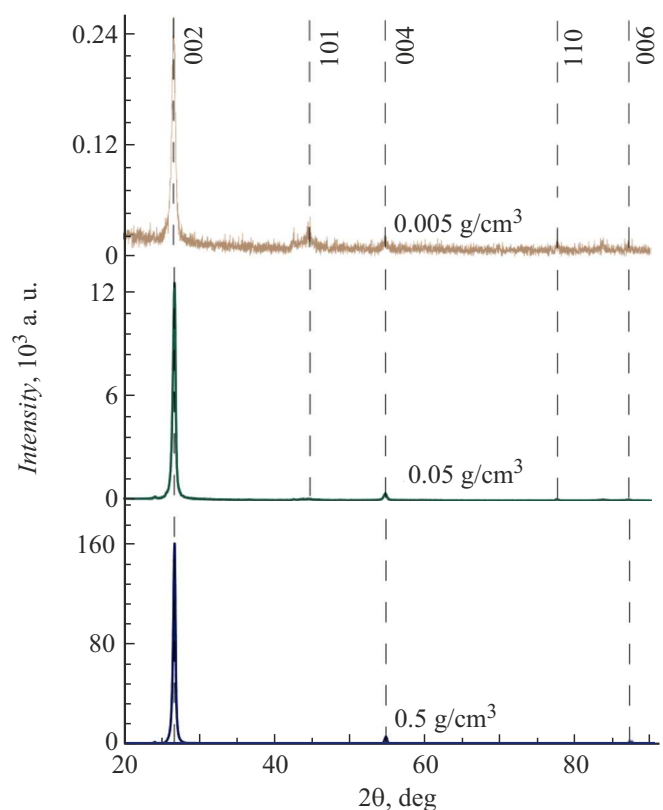


Figure 3. Diffraction patterns of samples with densities of 0.005, 0.05 and 0.5 g/cm³.

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Conflict of interest

The authors declare that they have no conflict of interest.

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