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Study of mechanical characteristics of layered structures based on carbon nanomaterials for creation of bioelectronic components

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The paper presents the technology of formation of layered structures based on carbon nanomaterials intended for the creation of bioelectronic components. The structures were formed by spray deposition and ordering of layers of single-walled carbon nanotubes and reduced graphene oxide. Vertically ordered complex tree-like structures of single-walled carbon nanotubes and reduced graphene oxide were formed using laser radiation of nanosecond ytterbium fiber laser with a wavelength of 1064 nm and energy density of 0.12 J/cm^2. It is shown that the hardness values of the samples of SWCNTs, rGO and hybrid compounds of SWCNTs and rGO increased in the range of 1.5−2 times and amounted to 38.56 ± 4.91 GPa, 34.34 ± 1.56 GPa and 37.05 ± 8.30 GPa, respectively. Also, when exposed to laser radiation with energy density of 0.12 J/cm^2, the values of elastic modulus of CNT, rGO and hybrid compounds of CNT and rGO samples increased by 1.2−1.5 times and amounted to 233.12 ± 18.70 GPa, 235.89 ± 3.85 GPa and 281.69 ± 3.74 GPa, respectively. Also, when exposed to laser radiation with energy density of 0.12 J/cm^2, the values of elastic modulus of the samples of SWNTs, rGO and hybrid compounds of SWNTs and rGO increased by 1.2–1.5 times and amounted to 233.12 ± 18.70 GPa, 235.89 ± 3.85 GPa and 281.69 ± 3.74 GPa, respectively. The adhesion of samples of CNT-based layered structures and the hybrid composition of CNTs and rGOs was determined. By evaluating the scratches, it was obtained that the samples ordered by laser irradiation of 0.12 J/cm^2 are able to withstand loads up to 40 mN. The formed ordered layered structures based on carbon nanomaterials are promising for application as bioelectronic components, for example, neural interfaces for diagnostics or therapy, implanted in the body.

Keywords: carbon nanotubes, reduced graphene oxide, bioelectronics, hardness, elastic modulus, adhesion.

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Introduction

Today, the development of bioelectronic components is of high relevance due to a skyrocketing growth of new technologies and the associated necessity of tackling new tasks in biomedicine. One of the applications of bioelectronic components is creation of neural interfaces. In development of neural interfaces, it is pivotal to allow for a number of key aspects, such as biocompatibility, mechanical strength and flexibility, size and volume, and electrical conductivity of the material. Thus, at the development stage, it is important to select such materials that can meet the conditions related to the key aspects of the bioelectronic components being developed.

Carbon nanomaterials are one of the most promising materials for creating bioelectronic components, including for use as neural interfaces [1]. Carbon nanomaterials, such as carbon nanotubes and reduced graphene oxide, attract much interest due to their high electrical conductivity [2,3], biocompatibility [4,5], and mechanical characteristics [6,7] and modification options, as well [8,9]. One of the key features of carbon nanomaterials is their mechanical strength and flexibility. Due to such properties, a long-term operation of these materials can be ensured if used inside the human body. When placed inside the human body, bioelectronic components will be continuously subjected to mechanical stresses from surrounding tissues and due to the body movements. In this regard, it is critical to make sure that the material has sufficient strength to withstand continuous loads caused by deformations, pressure and friction, and at the same time have sufficient flexibility so as not to cause excessive stress on surrounding cells and tissues. Therefore, it is important to take into account such mechanical properties as hardness, elasticity and adhesion. By studying hardness and modulus of elasticity we may assess the degree of mechanical stability and durability at which bioelectronic components are capable of withstanding the applied loads [10,11]. Moreover, hardness and elasticity are able to influence the growth and adhesion of the cells of the device without exerting excessive pressure on them [12]. By examining the material's adhesion properties we may assess the degree of contact during interaction with cells and tissues of the body [13]. Compliance with adhesion requirements ensures the durability of the device, efficient signal transmission via „material–tissue“ interface, and simplifies the fabrication process, as materials with high adhesion are easily integrated into complex structures, thus, facilitating ultimately the assembly process. The use of laser radiation is known to improve the mechanical properties of carbon nanomaterials due to formation of defects in the lattice and formation of new bonds between the carbon atoms. This ensures formation of the layered structure based on carbon nanomaterials used in creation of bioelectronic components.

This paper gives an insight into the technology of forming samples of layered structures based on carbon nanomaterials using methods of spray deposition and laser ordering. The results of morphology and mechanical characteristics examination were obtained. The adhesion properties were studied by making scratches and assessing the defects of the layered structures made of carbon nanomaterials.

1. Materials and research methods

1.1. Formation of layered structures based on carbon nanomaterials used in creation of bioelectronic components

Samples of layered structures based on carbon nanomaterials used to create bioelectronic components were formed on *n*-doped $4.5\,\Omega \cdot$ cm, (100) oriented silicon four-inch wafers (Si-Mat, Landsberg am Lech, Germany). Other wafers were obtained as cut rectangular plates with dimensions 10×5 mm. Next, the plates were processed in Piranha solution, after which a catalytic pair of Ti (10 nm) and Ni (2 nm) was applied to them by electron beam vapor deposition. For the formation of layered structures the homogenous dispersed media were prepared that contained single-walled carbon nanotubes (SWCNT) (glqq Universalnie Dobavki“, Novosibirsk, Russia), reduced graphene oxide (RGO) („Grafenoks“, Chernogolovka, Russia) and a hybrid compound of SWCNT and RGO structures. The components concentration was 0.1 mg/ml for individual dispersion media with SWCNT and RGO and 0.05/0.05 mg/ml for the hybrid compound. Distilled water was used as a solvent. The prepared dispersed media were applied to the surface of the plates by spray deposition. To do this, the plates were placed on a heating table in order to evaporate the liquid during spraying. The temperature of the heating table was 70 °C. The dispersion medium was applied layer-by-layer in the amount of 50 layers.

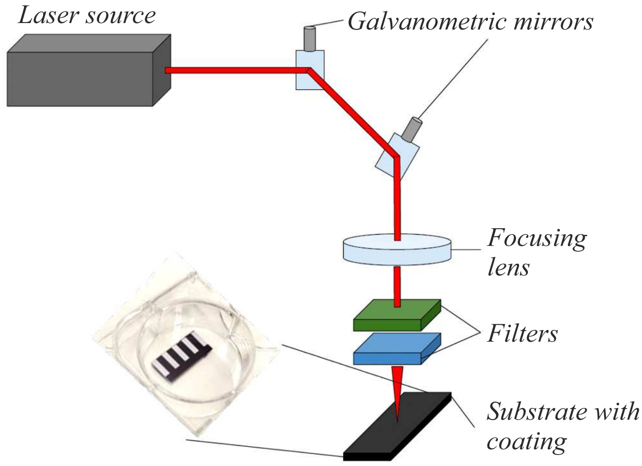

Figure 1. Ordering of layered structures based on carbon nanomaterials with the specified topology of bioelectronic components.

1.2. Laser ordering of layered structures based on carbon nanomaterials used in creation of bioelectronic components

In order to improve mechanical characteristics, the layers formed on the substrate were arranged by means of laser action. Ordering by means of laser irradiation was performed using an ytterbium optical fiber laser with a wavelength of 1064 nm, pulse duration of 100 ns, frequency of 30 kHz and laser radiation within 0.12–0.46 J/cm^2. The laser beam was positioned on a plane using a galvanometric scanner with two mirrors. The positioning accuracy was $(1.0 \pm 0.2)\,\mu$m. To focus the laser radiation, a lens with a focal length of 210 mm was used up to a laser beam with a diameter of 35 mm,μm. The spatial profile of linearly polarized radiation had a shape of the Gaussian distribution. To ensure uniform radiation distribution throughout the exposed area, the system includes a proximity sensor.

To provide ordering of the layered structures based on carbon nanomaterials, the trajectory of laser radiation was set in the software. The trajectory of the layered structures ordering on the substrate was of a square shape and covered the entire sample area. To form the topology of a bioelectronic component, a different trajectory was set, as shown in Fig. 1. Both trajectories were a set of parallel lines, the distance between which was $17\,\mu$m. The distance between the lines was selected so that they partially overlapped each other. At the same time, each line also consisted of laser pulses that overlapped each other. Beam motion rate along the specified trajectory was 240 mm/s. The length of lines along the laser pulses motion trajectory was in the range 5–10 mm. A schematic representation of formation of layered structures based on carbon nanomaterials for creating bioelectronic components is shown in Fig. 1.

As a result, the following samples of ordered layered structures on the substrate were prepared for the study: 1 — SWCNT; 2 — SWCNT ordered by laser radiation; 3 — RGO; 4 — RGO ordered by laser radiation; 5 — hybrid structures of SWCNT and RGO; 6 — hybrid structures of SWCNT and RGO ordered by laser radiation. Silicon wafer substrate served as a test sample.

1.3. Scanning electron microscopy

Structural features of samples on silicon wafers were studied by means of scanning electron microscopy (SEM) using the FEI Helios NanoLab 650 microscope (FEI Ltd., Hillsboro, OR, USA). Accelerating voltage of the electron column was equal to 5 kV, electronic probe current was equal to 86 pA for samples with SWCNT, RGO and SWCNT+RGO. Vacuum chamber pressure was $7.04 \cdot 10^{-4}$ Pa. Samples were attached to a conducting substrate using carbon adhesive tape.

1.4. Measurement of mechanical characteristics

Mechanical characteristics were studied using a scanning nanohardness tester „NanoScan-4D Compact" (NanoScan, Troitsk). To determine the hardness and modulus of elasticity, the nano-indentation method was used, which was used to conduct a series of tests for each of the samples. The essence of the method consists in pressing a tip of a given shape into the surface of the material while simultaneously registering the dependencies of the applied load on the depth of the tip indentation. A Berkovich-shaped indenter was used as the tip, which was itself a triangular pyramid. The geometric dimensions of the indenter were as follows: the radius of the rounded tip is less than 100 nm; the angle between the axis of the pyramid and the face is — $65.3°$; the equivalent angle of the cone is — $70.32°$. Measurements of hardness and modulus of elasticity by the nano-indentation method were performed with measurement across the depth. For each of the samples, 10 measurements were performed, which were then averaged. During the measurement, the indenter was smoothly immersed in the sample to a depth of 200 nm, while simultaneously registering the change curves $F-h$. The time of load applying and removing was 30 s, the time of holding the maximum load — 30 s. The distance between the measurement points was $100\,\mu$m in order to avoid the imprints overlapping. The time of temperature drift recording was 15 s, percentage of the temperature drift taken into account — 10 %. Due to the fact that silicon wafers were used as substrates, the Poisson's ratio was assumed to be 0.265 for calculating the modulus of elasticity.

1.5. Adhesion analysis

Adhesion was analyzed using scratch-method using the scanning nano-hardness tester „NanoScan-4D Compact". For this purpose the surface of samples was scratched with the load within 20–60 mN and scratch length of $500\,\mu$m. Maximal load was applied and held for 30 s. The distance between the scratches was $100\,\mu$m. After scratching the nature of damage to the coating was assessed the width and depth of the scratches.

2. Experiment

2.1. Morphology of layered structures based on carbon nanomaterials used in creation of bioelectronic component

The appearance of layered structures made of carbon nanomaterials obtained by spraying onto substrates and ordering by laser action is shown in Fig. 2.

It can be seen from the above images that SWCNT sample before laser ordering consists of its own bundles of nanotubes, which are unevenly arranged on the surface of the substrate and layered on top of each other (Fig. 2, *a*). The layer-by-layer deposition is explained by method of a sample formation, namely, spray deposition. The outer diameter of nanotubes is (1.6 ± 0.4) nm, and their length is over $5\,\mu$m. In case of RGO sample we may see that its surface is a heterogeneous monolayer consisting of RGO particles partially overlapping each other (Fig. 2, *b*). The particles themselves were uneven sheets with sizes ranging from 500– to 1000 nm. The hybrid structure of SWCNT and RGO (Fig. 2, *c*) was itself a densely applied layered structure where both, individual bundles of nanotubes and RGO sheets were distinguished. At the same time, it can be seen that the resulting structure occupies the surface of the substrate almost uniformly in the studied area.

It is known that covalent bonds C–C are formed between carbon nanotubes under the influence of laser radiation [14]. The formation of these bonds is possible in the areas of carbon nanotube defects due to weak thermal conductivity and radiation intensity. Thus, carbon nanotubes form interconnected networks that lead to improved mechanical properties compared to the original samples [15]. Based on this, it can be concluded that morphology of the studied samples may affect the mechanical characteristics of the samples, depending on the radiation energy density used. The morphology of layered structures based on carbon nanomaterials was analyzed depending on laser radiation energy density used.

For ordering of layered structures based on carbon nanomaterials, the value of laser radiation energy density was experimentally selected, at which it is possible to arrange SWCNT and RGO in their defect areas, due to which an ordered layered structure is formed. To achieve this, the energy density of laser radiation should be in the range 0.12–0.46 J/cm^2. The lower limit of this range was experimentally confirmed, since no ordering was observed in the samples below this value. The ordering and binding of SWCNT and RGO occurred in case when the energy density of laser radiation was ≥ 0.12 J/cm^2. At the same

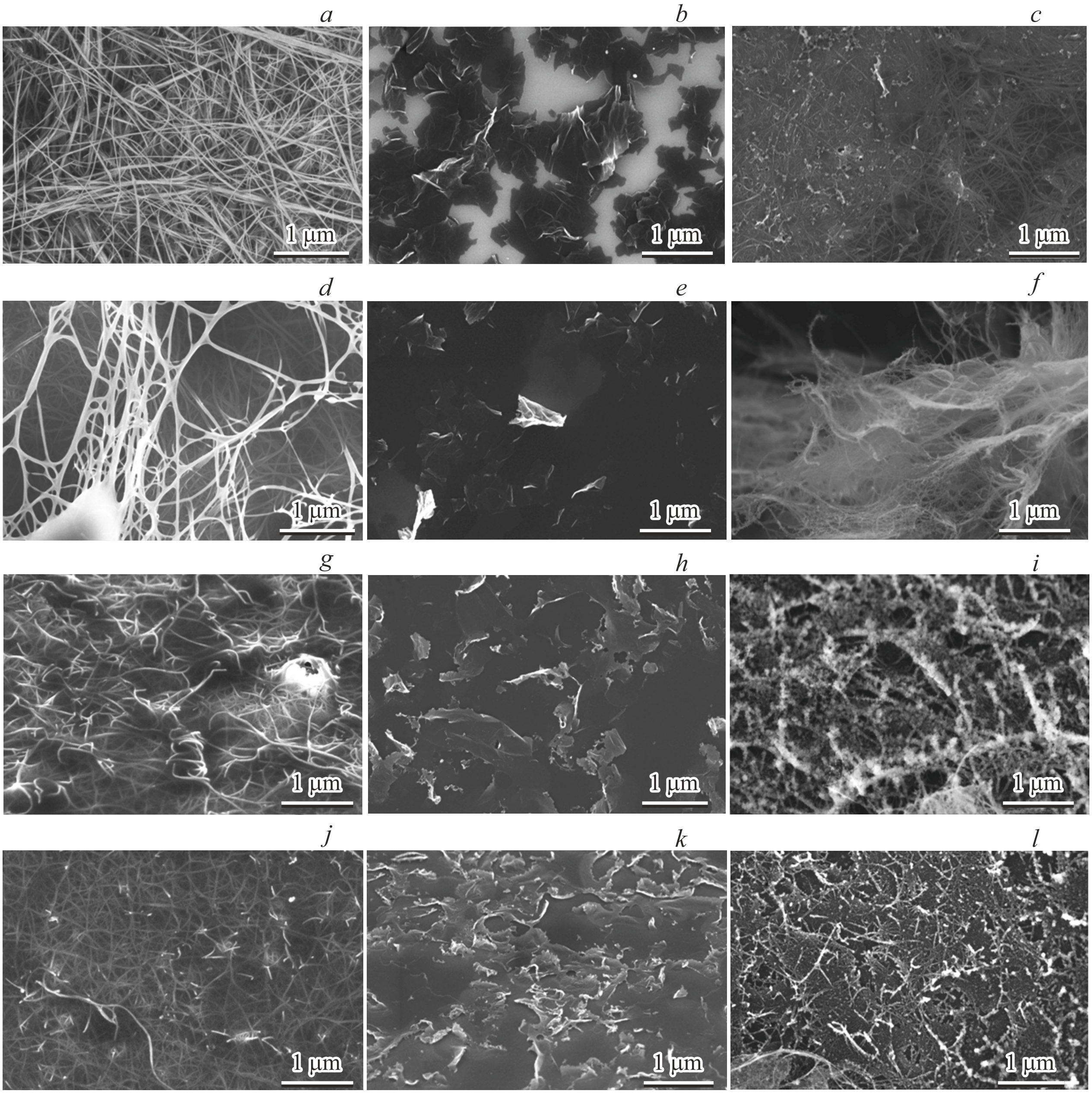

Figure 2. SEM images of layered structure samples based on SWCNT (*a, d, g, j*), RGO (*b, e, h, k*) and hybrid structure of SWCNT and RGO(*c, f, i, l*) at different levels of laser irradiation: without laser exposure (*a–c*); with exposure to laser with an energy density of 0.12 J/cm^2 (*d–f*); with exposure to laser radiation of 0.24 J/cm^2 (*g–i*); with exposure to laser with an energy density of 0.46 J/cm^2 (*j–l*).

time, laser radiation energy is absorbed, which leads to the formation of single- or polyatomic vacancies on their surface in the hexagonal structure of carbon nanotubes [16,17]. As a result, RGO and SWCNT form compounds of pairs of heptagons and pentagons of a carbon atom [18].

When examining the images of SWCNT samples ordered by laser radiation with an energy density of 0.12 J/cm^2 (Fig. 2, *d*), we may see that SWCNT have formed multiple inter-couplings. These couplings, in turn, form extensive networks based on the lower layers of the layered structure. As a result, the near-surface layer of the layered structure was itself a skeleton of standing nets of SWCNT. In case of RGO sample (Fig. 2, *e*), it can be seen that graphene particles partially ignited above the surface of the substrate under the influence of laser radiation. At the same time, another part of these particles was either pressed or fused to the surface of the substrate. In case of the hybrid compound of SWCNT and RGO (Fig. 2, *f*), it can be seen that as a result of exposure to laser radiation, vertically ordered complex tree-like structures were formed, including networks of SWCNT connected by bridge couplings with RGO particles. The height of the structures was about

Table 1. Hardness values of samples of layered structures based on carbon nanomaterials in their original form and after ordering by laser action

Samples	Laser radiation energy density, J/cm^2	
	0	0.12
	Hardness, GPa	
Silicon wafer	0.56 ± 0.29	
SWCNT	22.59 ± 4.01	38.56 ± 4.91
RGO	21.5 ± 8.29	34.34 ± 1.56
Hybrid structure of SWCNT and RGO	20.38 ± 4.57	37.05 ± 8.30

30 μm. It also can be seen that SWCNT couplings cover a significant part of the RGO particle surface.

When examining SWCNT samples ordered by laser radiation with an energy density of 0.24 J/cm^2 (Fig. 2, g), it can be seen that standing nets, by analogy with the previous sample, have not been formed. It can also be observed that SWCNT themselves have a twisted shape and form small couplings. At the same time, it was observed that a significant part of carbon nanotubes have structural breaks. In RGO sample (Fig. 2, h) partial damages of graphene oxide particles are observed. In this case, it is possible to detect the presence of particles, some of which, as in the previous case, were deposited above the substrate. When considering a sample of hybrid structure (Fig. 2, i), there is a noticeably smaller number of ordered complex tree-like structures. Significant amounts of amorphous carbon are observed to be formed on the surfaces of SWCNT and structures made of SWCNT and rGO. From the examined images, it can be concluded that when exposed to laser radiation with an energy density of 0.24 J/cm^2 the threshold is exceeded at which the ordering of layered structures based on carbon nanomaterials begins.

When examining SWCNT samples ordered by laser radiation of 0.46 J/cm^2 (Fig. 2, g), it can be seen that significant portion of the near-surface layer was damaged. It is also possible to notice a significant number of breaks at the ends of SWCNT, while no any pair of mutual couplings between SWCNT has been revealed. For the RGO sample (Fig. 2, k) serious surface damages of RGO particles are observed. High-intensity exposure to laser radiation did not lead to the ordering of complex tree-like structures formed by the hybrid compound of SWCNT and RGO (Fig. 2, l). Significant amount of amorphous carbon formed by laser radiation on the nanotube surface was found on SWCNT surfaces. No couplings between SWCNT and RGO are found. From the above images, it can be concluded that when exposed to laser radiation with an energy density of 0.46,J/,cm^2, the threshold at which the layered structures of carbon nanomaterials are ordered is greatly exceeded, which leads to significant damage of the samples.

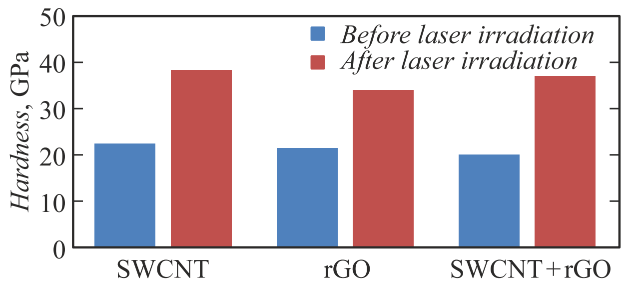

Figure 3. Hardness of samples of layered structures based on carbon nanomaterials.

2.2. Results of measurement of mechanical characteristics

The analyzed morphology of samples of carbon nanomaterials layered structures has shown that for the study of mechanical characteristics, it is most appropriate to measure the samples prior to their exposure to laser radiation and then the samples ordered by laser radiation of 0.12,J/cm^2.. A silicon wafer served as a reference sample. The hardness measurement results are provided in Table 1.

For clarity the obtained results are presented in Fig. 3.

It can be seen that under the influence of structural changes occurring during ordering of layered structures by laser radiation, the hardness increased to 1.5–2 times. These changes in hardness values indicate an effective ordering of carbon nanomaterials in layered nanostructures, which is an important condition for the formation of bioelectronic components. The presence of high hardness values can ensure that samples retain their shape and structure under the influence of mechanical loads inside the body, which will positively affect the stability and durability of the bioelectronic components being developed.

The modulus of elasticity measurement results are provided in Table 2.

For clarity the obtained results are presented in Fig. 4.

It can be seen from the obtained values that, similar to hardness, when exposed to laser radiation, the ordering of layered structures also impacted the modulus of elasticity.

Table 2. Modulus values of samples of layered structures based on carbon nanomaterials in their original form and after ordering by laser action

Samples	Laser radiation energy density, J/cm^2	
	0	0.12
	Modulus of elasticity, GPa	
Silicon wafer	31.89 ± 13.91	
SWCNT	204.80 ± 20.85	233.12 ± 18.70
RGO	197.66 ± 38.62	235.89 ± 3.85
Hybrid structure of SWCNT and RGO	195.10 ± 18.49	281.69 ± 3.74

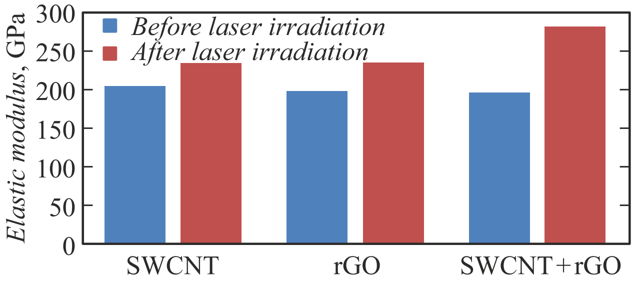

Figure 4. Modulus of elasticity of samples of layered structures based on carbon nanomaterials.

Compared to the initial values of samples before exposure to laser radiation, the modulus of elasticity increased by 1.2–1.5 times. The corresponding values of the modulus of elasticity can provide sufficient resistance to deformation inside the body, which is also an important condition for creating bioelectronic components.

2.3. Adhesion analysis

As a result of the study of morphology of the samples, it was found that the sample of a RGO-based layered structure is itself a heterogeneous monolayer. In this regard, it was decided to study the adhesion of layered structures samples having a denser and more uniform coating on the substrate, i.e. samples of SWCNT-based layered structures and a hybrid structure of SWCNT and RGO. The appearance of scratches on the SWCNT-based layered structure samples is shown in Fig. 5.

When evaluating the SWCNT sample that hasn't been exposed to laser radiation, it is seen that coating of the layered structure begins to separate from the substrate under the action of a force of 40 mN (Fig. 5, *a*). Similarly, this coating starts to be separated from the substrate when impacted by a force of 40 mN on a sample ordered due to exposure to laser radiation of 0.12 J/cm^2 (Fig. 5, *b*). However, when assessing the samples ordered by laser radiation of 0.24 and 0.46 J/cm^2, we may see (Fig. 5, *c, d*) that coating starts to be separated from the substrate under the action of a 20 mN force already. This effect may be related to the morphology of layered structures that have been significantly damaged by laser radiation during ordering. At the same time, despite the formation of a skeleton of standing nets in the subsurface layer, no decrease in adhesion was observed on the sample ordered at a laser radiation energy density of 0.12 J/cm^2 when its mechanical characteristics were enhanced. Presumably, additional studies should be conducted to evaluate scratches caused by force in the range from 30 to 40 mN.

The appearance of scratches on the samples of SWCNT and RGO hybrid structure is shown in Fig. 6.

When evaluating a sample of layered structures of the hybrid compound of SWCNT and RGO, which was not subjected to laser ordering, it can be seen that the coating begins to separate from the substrate when exposed to a force of 40 mN. Similarly, when exposed to a force of 40 mN, the layered structure based on a hybrid compound of SWCNT and RGO started to separate from the substrate when exposed to laser radiation with an energy density of 0.12 J/cm^2. In case of samples of SWCNT-RGO layered structures exposed to laser radiation with an energy density of 0.24 and 0.46,J/cm^2, it can be seen that the coating was separated under the action of force of 20 mN. This trend corresponds to changes in the morphology of layered structures when exposed to laser radiation with an energy density of 0.24 and 0.46,J/cm^2, at which significant damage occurred in the near-surface layers. In case of a sample irradiated by laser with an energy density of 0.12 J/cm^2, additional studies are required to find forces under the action of which the coating starts separating within 30–40 mN.

Conclusion

The results of studies of layered structures based on carbon nanomaterials intended for fabrication of components of bioelectronic devices are presented. A technology is proposed for forming the topology of layered structures based on carbon nanomaterials using methods of layer-by-layer spray deposition of SWCNT, RGO and hybrid compounds of SWCNT and RGO, followed by

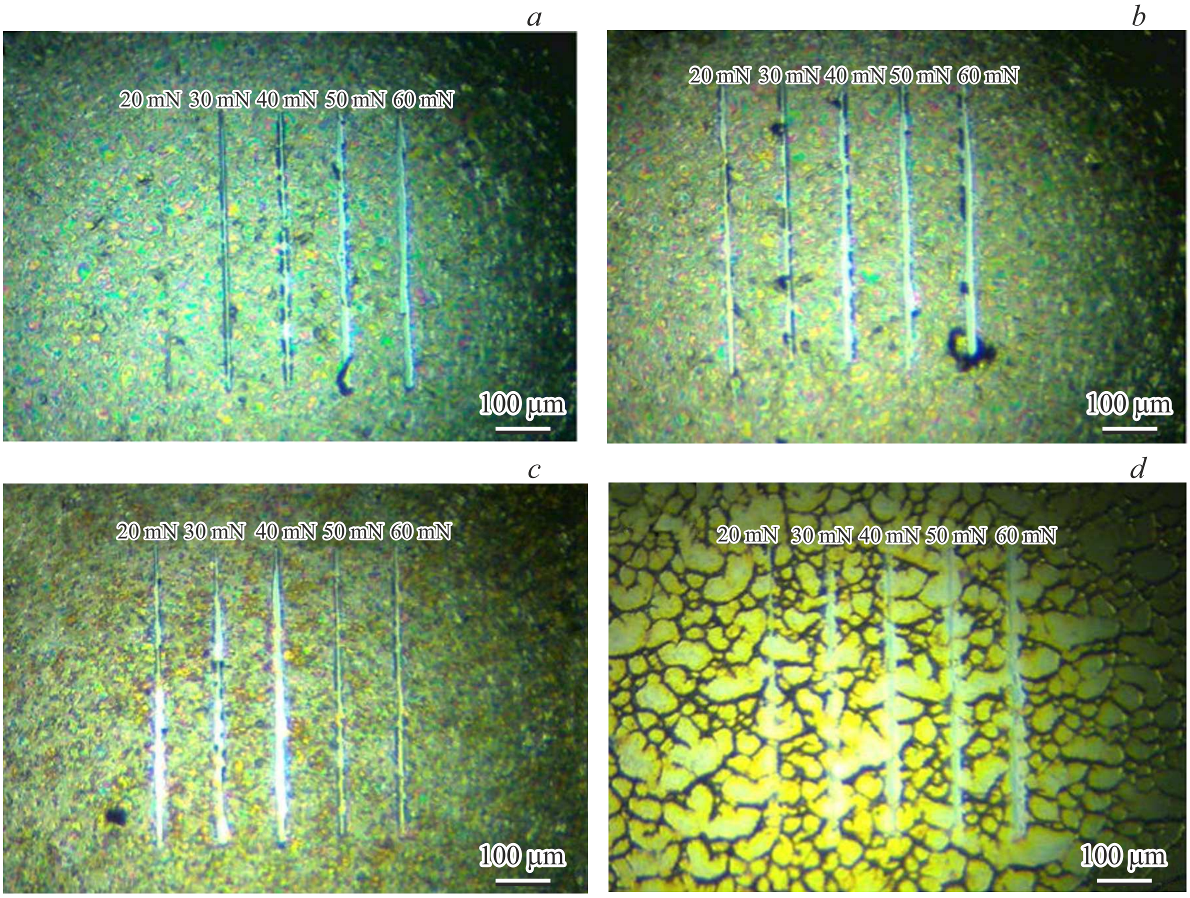

Figure 5. The appearance of scratches on the SWCNT-based layered structure samples without laser exposure (*a*) and on the samples ordered by laser radiation with an energy density of: *b* — 0.12 J/cm^2; *c* — 0.24 J/cm^2; *d* — 0.46 J/cm^2.

their ordering using laser radiation with different energy density levels. It was found that under the action of laser irradiation with an energy density of ≥ 0.12 J/cm^2 the multiple couplings between SWCNT and RGO are formed in the upper layers of the layered structures. Due to the formed couplings the vertically ordered complex tree-like structures were formed, including SWCNT nets connected by bridge couplings with RGO particles. Such ordered layered structures have enhanced mechanical properties in comparison with disordered layered structures. Under the action of laser radiation with an energy density of 0.12 J/cm^2 the hardness of samples of SWCNT, RGO and hybrid compound of SWCNT and RGO increased by 1.5–2 times compared to the initial samples and made (38.56 ± 4.91), (34.34 ± 1.56) and (37.05 ± 8.30) GPa, respectively. Under the action of laser radiation of 0.12 J/cm^2 the modulus of elasticity of samples of SWCNT, RGO and hybrid compound of SWCNT and RGO increased by 1.2–1.5 times and made (233.12 ± 18.70), (235.89 ± 3.85) and (281.69 ± 3.74) GPa, respectively. The obtained values of hardness and modulus of elasticity demonstrate that the shape and structure of samples under the impact of mechanical loads inside the body may be preserved, and also resistance to deformation inside the human body may be provided, which is consistent with the stability and durability of bioelectronic components that shall be ensured. This is especially true for the neural interfaces, which involve a long-term implantation in the spinal cord for a period of up to 10 years. Also the adhesion of samples of SWCNT-based and SWCNT-RGO hybrid compounds layered structures have been defined. From evaluating the scratches it may be seen that the samples ordered by laser radiation of 0.12 J/cm^2 may withstand the loads of up to 40 mN. Thus, studies of the ordered layered structures based on carbon nanomaterials indicate that their use as bioelectronic components, e.g., diagnostics or therapy neural interfaces implanted in the human body, is quite promising.

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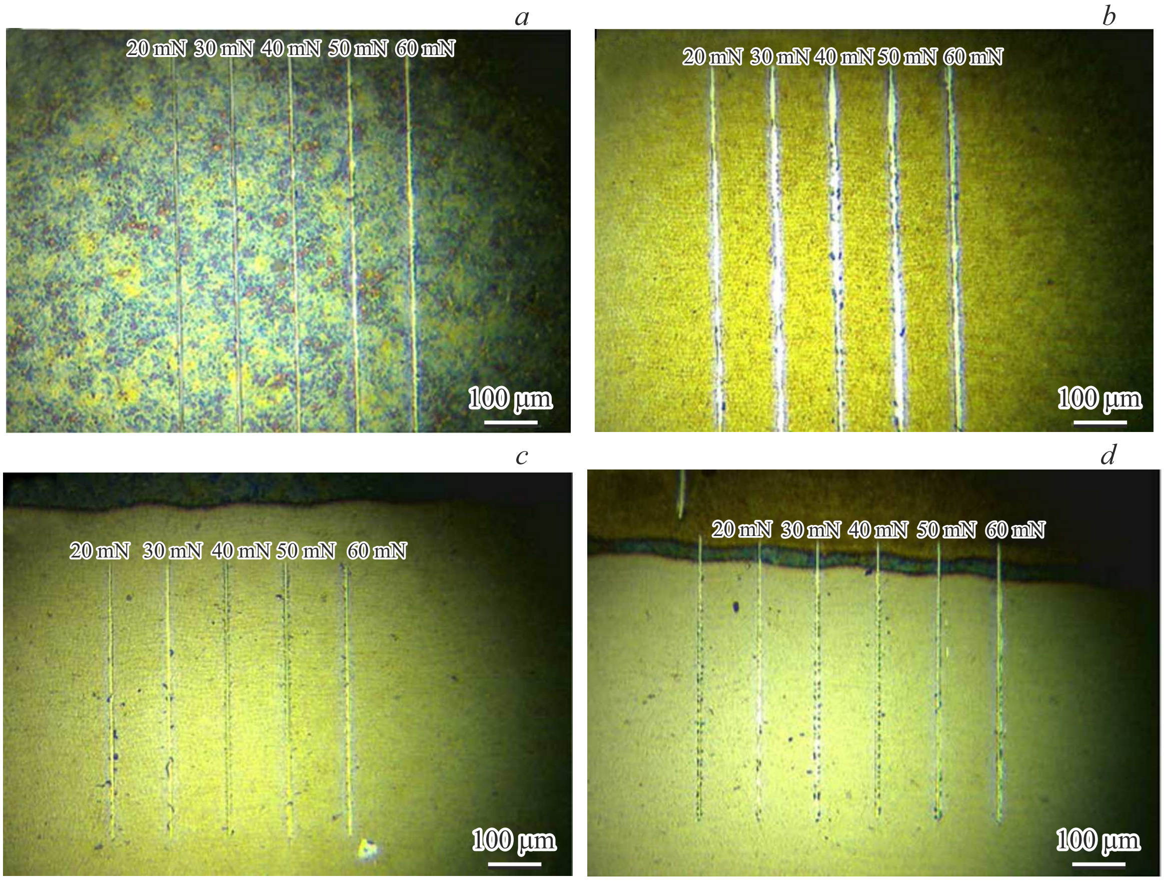

Figure 6. The appearance of scratches on the surface of SWCNT-RGO based layered hybrid structure samples without laser exposure (*a*) and on the surface of samples ordered by laser radiation with an energy density of: *b* — 0.12 J/cm^2; *c* — 0.24 J/cm^2; *d* — 0.46 J/cm^2.

Conflict of interest

The authors declare that they have no conflict of interest.

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