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Plasma-synthesized graphene flakes for highly sensitive monitoring systems

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Abstract: A composite based on the conductive polymer PEDOT:PSS and unique graphene flakes has been developed, which can be used in various health monitoring technologies. Graphene's unique properties are due to the dry synthesis method. Using plasma generated by a direct current plasma torch at 350 Torr, the flakes were produced in uniform sizes. Modes for producing nanostructures with a minimum lateral size of 100 nm have been developed. A technique has been suggested for preparing the nanostructure suspension for two-dimensional (2D) printing of active films on substrates. The printed structures were tested as wearable sensors for human sweat glucose, and exhaled air analysis, as well as humidity and touch sensors. The possibility of analysis of various solutions using such sensor substrates as non-woven fabric (spunlace) and polycarbonate fishing line has been demonstrated. As a result, simple and accessible diagnostics of the human nervous system and detection of enhanced reactions to various external influences are offered. In the systems presented, the sensor conductivity surges in response to the chemical reactions occurring between the graphene surface and the liquid components. In general, due to the use of plasma-synthesized flakes, sensors that combine the simplicity and low production cost technology with high sensitivity have been manufactured.

Keywords: graphene, PEDOT:PSS, non-invasive sensors, 2D printing, low-temperature plasma

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1. Introduction

Modern technologies for nanomaterials synthesis confront two problems: the need to create nanostructures that are highly reactive to chemical compounds in the environment and the need to ensure reproducibility of the method during large-scale synthesis. Usually, achieving both necessitates new extreme conditions, beyond conventional approaches. Low-temperature plasma has an extremely high energy effect on the substances processed, acting as both a universal heat carrier and a reagent. Unlike the other heat carriers, plasma enables creation of non-equilibrium conditions in the chemical reaction zone and thus achieves above-equilibrium yields of the reaction products. The simplest way to produce a low-temperature plasma is the thermal ionization of gas in electrical discharges. Owing to the development of the theoretical foundations of plasma technologies, and elaboration of special equipment, a number of technologies are now in industrial use: pyrolysis of hydrocarbons, production of simple and complex oxides, various composite materials, and highly dispersed flakes of graphene and other two-dimensional materials [1–4]. The

variety of plasma approaches enables synthesis of graphene materials in various arc discharge reactive systems [5]. The advantage of the arc discharge method is the high rate of graphite ablation, in which pure carbon vapor is produced; its further condensation of which results in industrial-scale formation of highly crystalline graphene with no structural defects [6]. It should be noted that, a standard arc discharge cannot be used in continuous production of perfect carbon nanostructures. Because of the motion of the arc and the low rate of graphite evaporation, the nanoparticles flows are mixed, which reduces the selectivity of the method [7].

As regards continuity of the synthesis, using a plasma jet allows the most flexible process optimization, which enables controlled growth of nanostructures with predictable parameters and fewer defects in comparison with chemical methods [8]. Compared with the CVD and the arc discharge method, plasma jets synthesis is a single-stage fast continuous method that can go at reduced and atmospheric pressure, without the use of substrates, catalysts, solvents, or acids. A number of studies of synthesis using plasma torches investigated the influence of the process parameters on the yield and properties of graphene. The authors of [9,10]

have proven the possibility of single-stage synthesis of few-layer graphene by means of a microwave discharge. A high-frequency plasma system for the synthesis of graphene flakes during methane decomposition has also been developed [11]. It has been found that a high reaction temperature and addition of H_2 can facilitate the transformation of the products from spherical particles into graphene flakes. The oxygen to carbon ratio was found to be a key parameter that controls the graphene yield. In [12], graphene synthesis was performed with application of a DC plasma torch at atmospheric pressure. The abovementioned studies have also described the influence of the reaction temperature and precursors on the graphene formation.

The aim of the present study was to synthesize graphene with a narrow size distribution for its subsequent use for an inkjet printer ink and testing of the printed films.

2. Materials and methods.

Experimental setup. Synthesis of graphene flakes in plasma jets generated by DC plasma torch

Electric arc plasma torches with vortex stabilization of the plasma jet are widely applied in practice. A plasma-chemical reactor based on a DC plasma torch for synthesis of carbon nanomaterials is described in detail in [13]. With helium as the plasma-forming gas and propane-butane mixture as the carbon precursor at a pressure of 350 Torr, the resulting graphene flakes had a distinctive feature of narrow thickness distribution (2–5 monolayers) and lateral particle sizes (100–200 nm) [14]. Figure 1 shows the transmission electron microscopy (TEM) image of these flakes, and the sheet resistance of ≈ 200 nm thick graphene flake films. Examination of the graphene edges has revealed occurrence of reconstruction of the edges with the formation of bonds between the upper and lower layers (see Fig. 1a). This statement is supported by the absence of oxygen in the layers of synthesized particles, which should have been present in the case of broken bonds and their functionalization with oxygen related groups. Some quantity of the hydrogen in the plasma-chemical reactor at the time of the flake synthesis is trapped in between the planes of the flakes and is sealed there because of the edge reconstruction. The sheet resistance of

a ≈ 100 –200 nm thick film was 300–400 kOhm/ $\square$ at the indicated helium pressure in the chamber.

We have developed a conductive composite that allows printing ultra-thin (several nanometers) conductive layers from plasma-synthesized graphene and the conductive polymer PEDOT:PSS (poly(3,4-ethylenedioxythiophene)). PEDOT:PSS has the highest conductivity among organic materials (4–10 kOhm/sq) [15]. Moreover, when solvents such as ethylene glycol (EG), glycerol, and dimethyl sulfoxide are added, the conductivity of PEDOT:PSS increases by two to three orders of magnitude due to the doping and partial removal of PSS.

3. Results and discussion

3.1. Multifunctional biosensors based on composite of graphene and conductive polymer PEDOT:PSS

Wearable biosensors for personalized health monitoring have become an alternative to traditional medical diagnostics, improving quality of life through real-time health monitoring. However, a number of critical technological challenges (selectivity, stability of (bio) recognition, efficient sensor designs, non-invasiveness, mechanical strength, and flexibility) should be overcome for better user comfort and progress towards commercial application. We present the integration of a sensor made of the specially designed composite material based on plasma-synthesized graphene flakes into a wearable sweat diagnostics platform for the analysis of components of interest. The use of 2D inkjet printing technology for sensor fabrication simplifies the process and reduces the manufacturing cost. Some of the most common substrate materials used in biosensor development are paper or fabric. Focusing primarily on high response of our composite sensors to glucose in sweat, we also investigated the possibilities of using the sensors to analyze the air humidity, human skin, and state of the human nervous system, the sensor response to breathing or touch, and the possibility of analyzing chemical solutions. In almost all cases, the response of plasma-synthesized graphene sensors was very high compared to the literature data, which makes them very promising.

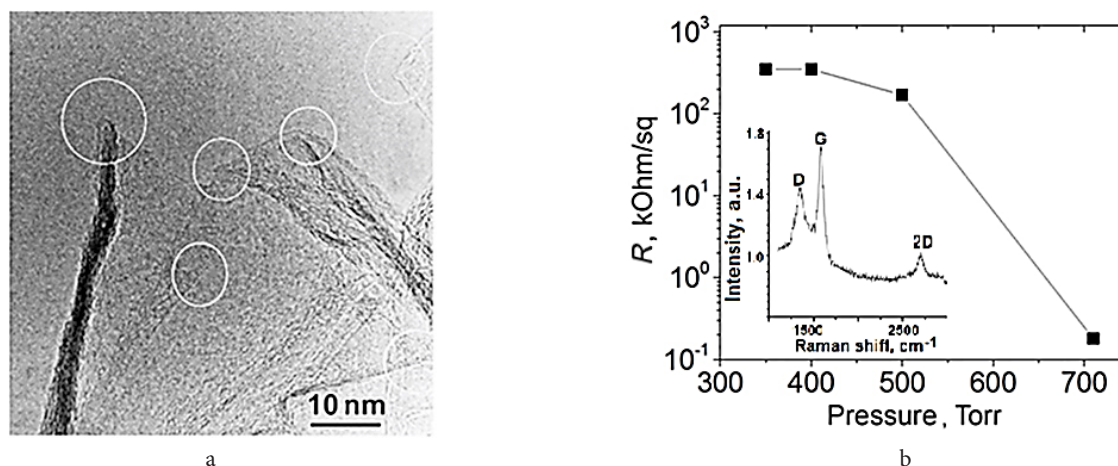


Fig. 1. TEM image of few-layer graphene flakes synthesized in plasma. Circles indicate edges of graphene flakes (a). Sheet resistance of ≈ 100 nm thick vs. pressure in chamber during flake synthesis. Inset: Raman spectrum of graphene flake in printed film (b).

3.2. Non-invasive glucose sensors

Diabetes is one of the most worldwide common chronic diseases requiring regular blood sugar testing. The progress in wearable medical systems for human health monitoring creates a great potential for testing and thus improving the diabetes treatment [16]. Modern wearable biosensors as a convenient measurement tool have become a rapidly growing area of interest because of their potential for integration with the conventional medical diagnostics and the use of miniature analytical devices ("lab-on-the-body") [17–19]. Analysis of sweat, among the various human fluids, has attracted the greatest interest. Considering wearable glucose sensors based on sweat analysis in more detail, it is necessary to note the complex multilayer structure of the sensitive element of sensor. Relatively complex devices with the corresponding price are present on the market of non-invasive glucose sensors.

Using plasma-synthesized flakes allows creating high-sensitivity sensors combining ease of manufacturing and low cost and low cost. Figure 2 shows the general scheme for creating sensors by 2D printing on paper or nonwoven fabric spunlace using the composite ink made of graphene with the addition of the conductive polymer PEDOT:PSS. This sensor design yields a combination of high sensitivity and manufacturing simplicity. The sensors have been shown to have good stability and flexibility. The sensor operation is based on analysis of sweat secreted by the skin. Sensors on ordinary paper turned out to be the most promising. Among the fabrics used, the best results were obtained for spunlace (viscose and polyester). The thickness of the active film of sensor varied from one to six printed layers (1L–6L). The 3L film on the SiO_2/Si substrate had a thickness of ≈ 10 nm. Because of the pronounced roughness of the flexible substrates, it was impossible to determine the thickness of the similar film on paper (see Fig. S1, Supplementary material). So, the thickness will be further described in terms of printed layers. The size of one sensor was 2×6 mm.

Let us consider the sensors on paper. The sensor was fastened by a patch to the wrist and worn for some time. After

wearing the sensor on the skin, the conductivity increased several times, depending on the blood sugar level (Fig. 3). In the experiments, the sensor response was tested for blood glucose levels in the range of 3.5–12 mM. The glucose level was also checked with a commercial glucometer.

Testing with a picoammeter the change in the conductivity of a 2L-thick sensor on paper has shown a current variation by five to six orders of magnitude during measurement of the blood glucose levels in the range of 3.5–10 mM; at higher glucose levels, current saturation is observed, which is associated with the small thickness of the active film of the sensor [20]. The selectivity of the sensor was tested by comparing its responses to water, artificial sweat, ions, lactate, sucrose, individual salts, saline solution, Ringer's solution, and other. The formation of hydrogen peroxide upon interaction of sweat with the sensor was demonstrated, as expected during glucose oxidation [21, 22].

Comparison of the responses of sensors on paper and spunlace shows that with the same thickness of the active film of sensors, the response of sensors on fabric was slightly lower. 3L on spunlace gives practically the same response as 2L on paper. This was caused by the higher roughness of the fabric surface and, accordingly, the higher initial resistance of the sensor.

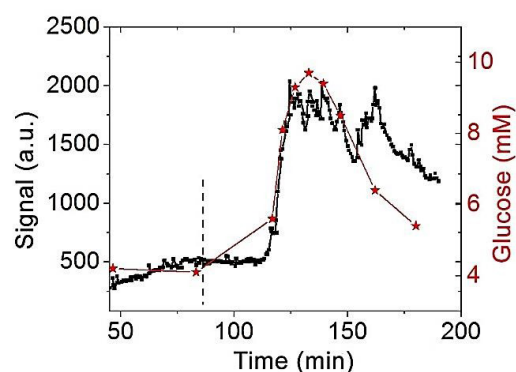


Fig. 3. (Color online) Reader response (black line) and blood glucose (red line) vs. time for two test subjects. Dashed lines: time of meal intake, after which peak blood glucose and peak sensor signal are observed.

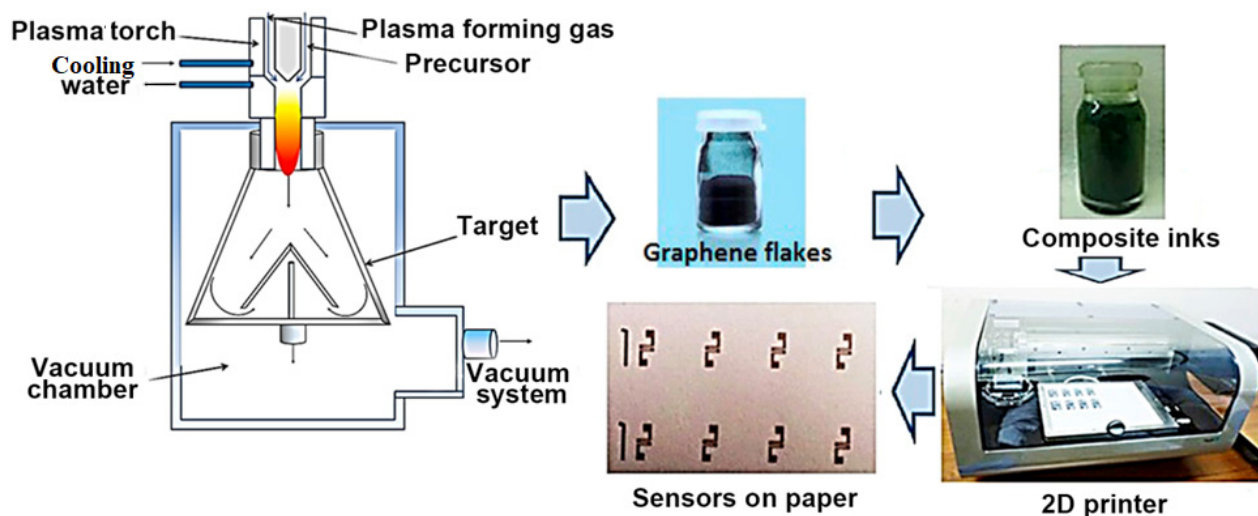


Fig. 2. (Color online) Scheme of sensor fabrication: synthesis of graphene flakes in plasma, creation of composite graphene ink with PEDOT:PSS, printing by Dimatix inkjet printer Fujifilm, and photograph of batch of sensors on paper.

3.3. Humidity sensors

The current response of sensors printed on different papers (two printing passes) is shown in Fig. 4. The maximum relative humidity of skin typically does not exceed 80%. Sensors on different types of paper (bleached office paper with a density of 80 mg/m² and 160 mg/m², as well as unbleached paper with a density of 80 mg/m²) demonstrate approximately the same response. The maximum response is observed with sensor 2L on unbleached paper with a density of 80 mg/m². The sensor on the denser paper had a longer recovery time compared with that on the ordinary paper, which is associated with the wetting of the paper itself and the subsequent release of water from it, see Fig. 4b. The response of the sensor on the ordinary paper for a humidity of 80% is 10⁻⁷ A, which is one to two orders of magnitude lower than the response of the same sensor to glucose in sweat analysis.

A study of electrically active centers in sensor layers by the method of charge deep-level transient spectroscopy (Q-DLTS) has shown that as the humidity grew, there appeared electrically active centers with activation energy of 0.36–0.39 eV and density of up to (6–8)×10¹⁰ cm⁻² [20].

3.4. Breathing sensors

Analysis of exhaled air using non-invasive sensors is relatively simple because no chemical reagents, test tubes, disposable needles, etc. are needed. Just an exhale over the sensor, and the analysis result will be ready in a few seconds or minutes. The prospects of non-invasive diagnostics of diseases are obvious: no necessity to experience pain and other unpleasant sensations and complete absence of risk of infection. Breath analysis with various spectrometers enables detection of cardiovascular, oncological, gastrointestinal, and respiratory diseases in humans.

We have shown that for graphene-based breathing sensors to be highly sensitive, a structure is required in which few-layer graphene (two to three printed layers) is arranged in the form of vertical flakes. Figure 5 shows the response of graphene-based sensors to human-exhaled air. Two or three exhaled components that define the shape of the current pulse of a healthy person have the following time characteristics. The first component leads to a fast response with a rise time of less than 1–4 seconds. This peak can presumably be interpreted as a response to the humidity of the exhaled

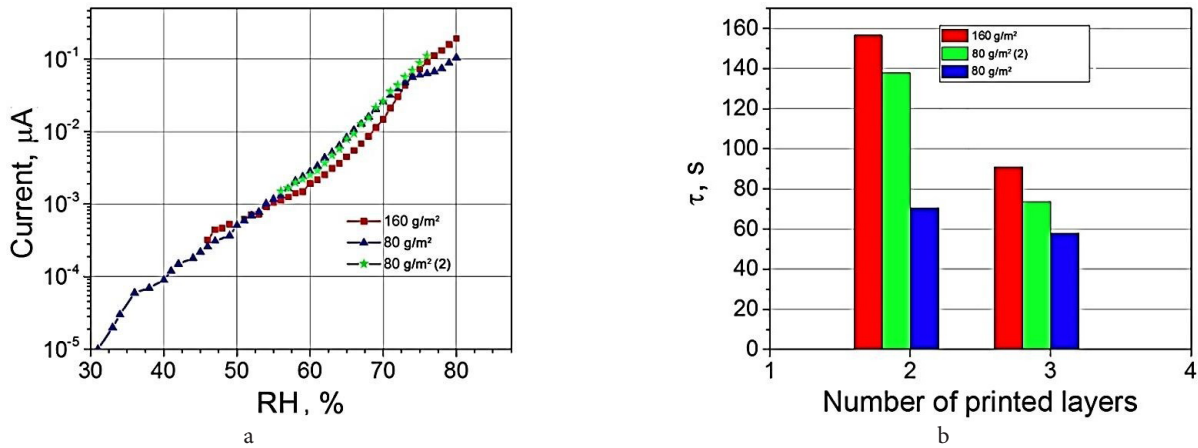


Fig. 4. (Color online) Picoammeter-measured response of 2L graphene: PEDOT:PSS sensor depending on paper type: bleached paper the densities of 80 mg/m² and 160 mg/m² and unbleached paper with density of 80 mg/m² (curve 2) (a). Recovery time for sensors of various thicknesses on paper of various densities (b).

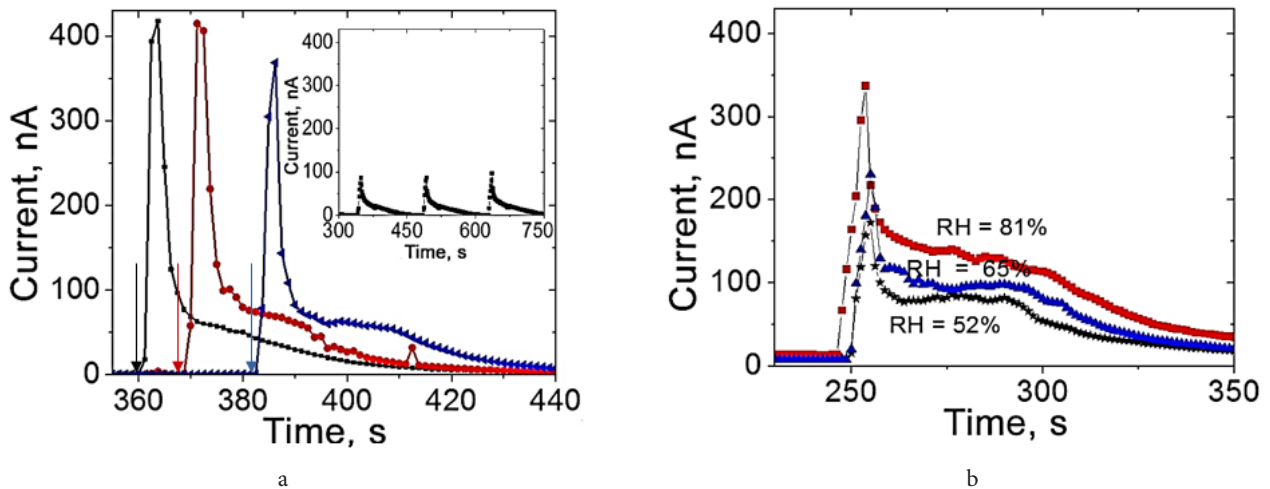


Fig. 5. (Color online) Response of 2L-thick sensor to exhalation for three different human test subjects. Inset: response of control sample consisting of two silver contacts on paper. The moments when the persons started breath mark with arrows (a). Response of sensor to exhalation for different moisture contents in vapors (the humidity was measured with specialized sensor) (b).

air (see Fig. S3). The second and third peaks are most likely associated with the presence of CO₂ and nitrogen compounds in the exhaled air. In middle-aged people, the magnitude of additional peaks is higher than that of the main one (28% and 18%, respectively). The sensor response for people with a number of diseases can be more complex.

3.5. Touch sensitive sensors

A current response in the form of pulses with amplitude of up to two to three orders of magnitude was also observed when the sensor was pressed by a finger. Figures 6 and S2 show the strong current increase as the sensor response to a finger touch or pressure. This combination of responses to pressure and humidity makes it possible to use the sensors, for example, for a keyboard or an artificial skin.

3.6. Sensors sensitive to state of human nervous system

Investigations show a significant increase in stress and anxiety levels worldwide. Common sources of stress include economic issues, political events, social media use, and a general feeling of being overwhelmed by daily life, as highlighted by the National Institute of Mental Health

(NIMH) and The Skill Collective. This does not pose a specific threat, but significantly reduces the quality of life, especially in the long term. Diagnosing nervous system diseases is sometimes difficult because of the variability of symptoms. In modern neurology, highly informative but complicate diagnostic methods are used for examination of the nervous system state. Diagnostics of a person's condition using a system of invasive sensors is developing in parallel with the use of special electrodes or needles to activate a response of the nervous system. Non-invasive testing approaches have clear advantages over invasive ones, both in terms of their immediate applicability and in terms of prospects. Currently, non-invasive methods for analyzing the nervous system are developing too slowly.

Examination of the effect of sweat on the conductivity of the sensor structure under conditions of continuous signal reading has revealed a number 2 to 12 min long peaks on the response curve associated with the excitation of the nervous system in response to certain external factors. The excitations were caused by moderate physical activity, telephone conversations, anticipation of sweets for a person with diabetes, or finger pricking during measurement of the blood glucose levels [21]. The sensor response to the emotional state of a person driving a car and that associated with finger pricking during measurement of the blood glucose are shown in Fig. 7a. The sensor response to solving two simple tasks are given in Fig. 7b. The tasks had varying levels of difficulty, and the amplitude of the sensor response was proportional to the difficulty of the tasks.

Functioning of the nervous system is usually associated with a number of biologically active substances (adrenaline or cortisol) secreted by sweat glands in response to psychoemotional stimuli. Cortisol and adrenaline are biologically active substances (hormones), which are released into the blood in a state of stress with subsequent increase in their concentration in sweat [21]. For sensors on paper, we have previously shown that the presence of cortisol or adrenaline in an aqueous solution leads to a significant increase in the sensor conductivity. It is also known that the dynamics of changes in the cortisol level in sweat are tens of times faster than the dynamics of changes in the glucose level in the blood and thus in sweat.

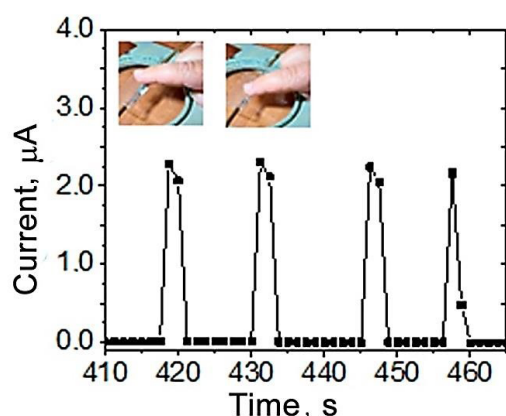


Fig. 6. (Color online) Using sensor of graphene:PEDOT:PSS on paper as touch sensor: response of sensor to finger touch.

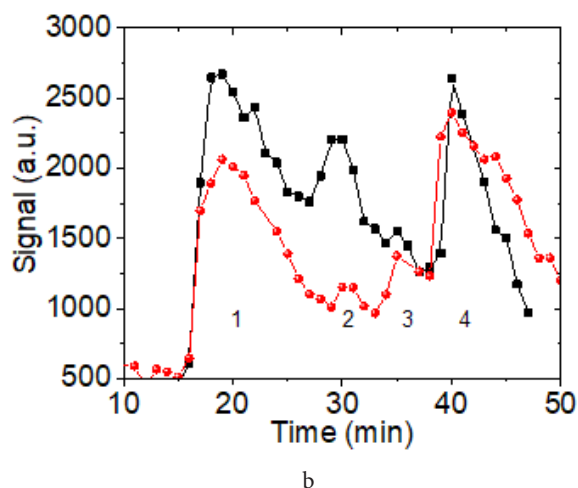
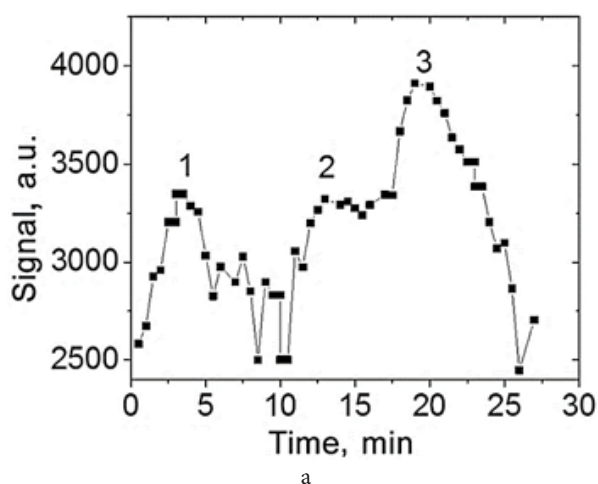


Fig. 7. (Color online) Response of sensor to emotional state of person driving car: 1 — talking on phone intensely, 2 — facing obstacle on road, 3 — driving at high speed on empty road (a). Sensor response on 4 tasks for two persons (b).

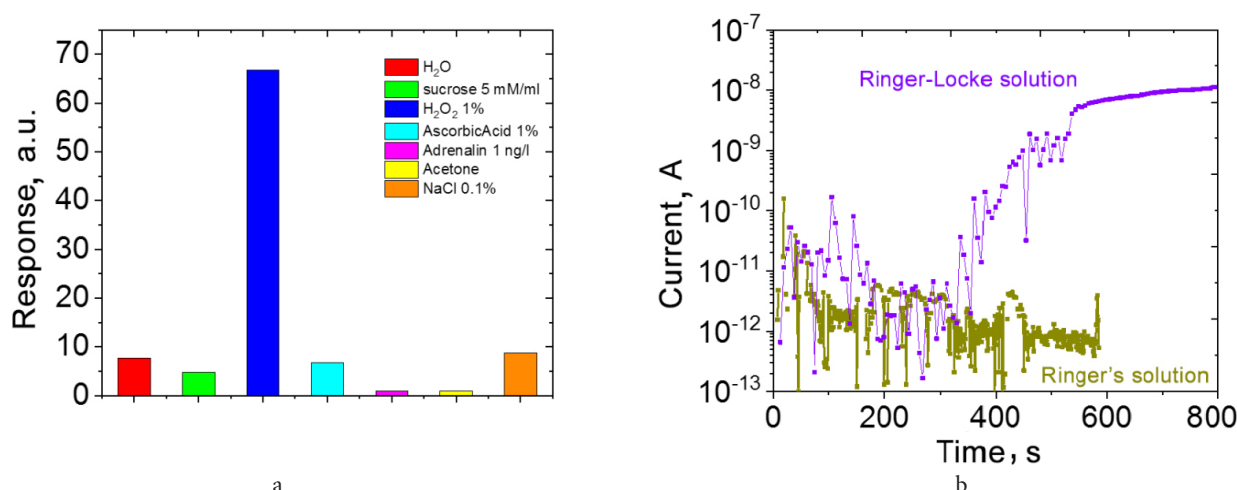


Fig. 8. (Color online) Comparative diagram of sensor responses for various solutions (a). Testing response of sensors on fishing line (2L) with droplets of complex Ringer and Ringer-Locke solutions (b).

3.7. Flexible chemical sensors made of graphene:PEDOT:PSS composite

Sensors on paper have limitations when tested with various solutions because the paper just got soaked during such treatments. For this reason, the sensors used for analysis of chemical solutions were printed on the nonwoven spunlace fabric (viscose and polyester) or applied as a thin active film of the same composite to polycarbonate fishing line (47 μm in diameter) [22]. As a result, designs and sensor structures were developed that had high response when tested with various chemical solutions (Fig. 8).

The testing was carried out with solutions containing glucose, hydrogen peroxide, and the NaCl salt, as well as with complex solutions. Ringer's solution is a multi-component physiological solution containing a large number of components that occur in human sweat. Those are mainly the salts NaCl, KCl, and $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$. Ringer-Locke solution includes the same salts as well as glucose (5.5 mM). It has been found (Fig. 8) that when the solution contains the NaCl salt or Ringer's salt complex, which is partially similar in composition to sweat, no increase in the sensor conductivity is observed despite the presence of water. In the case of Ringer-Locke solution, the sensor conductivity initially decreases slightly, as with Ringer's solution. The current increase begins with a time delay of ≈ 5 min, and the response is weaker than that to simple glucose solutions. The high sensor response to hydrogen peroxide is shown in Fig. S4. Solutions of acetone, sucrose, ascorbic acid, cortisol, and adrenaline were also tested. In general, it can be concluded that sensors on spunlace or fishing line are promising as chemical sensors for a wide range of solutions.

4. Conclusion

Inks made of composites based on plasma-synthesized graphene flakes with the addition of the conductive polymer PEDOT:PSS enable production of highly sensitive sensors for a wide range of applications. Testing the printed structures as wearable glucose sensors for human sweat analysis has shown a decrease in the sensor resistance by

four to six orders of magnitude. During determination of relative humidity, the sensor resistance declined by two to three orders of magnitude (to $\approx 80\%$). It has been shown that various solutions can be tested with sensors with substrates of the nonwoven fabric spunlace or polycarbonate fishing line. Real-time exhaled air analysis is a promising noninvasive method for diagnostics of a number of diseases. Of particular interest is the possibility of simple and affordable diagnostics of the human nervous system and detection of excessive reactions to various external factors.

In general, the use of plasma-synthesized graphene flakes has been shown to be promising for creation of highly sensitive sensors for a wide range of applications, including multifunctional sensor matrices. Of particular note is the combination of ease of manufacture and low cost of the high-sensitivity sensors.

Informed consent of people to participate in the study.

All human subjects voluntarily took part in the experiments, informed of the non-invasiveness of the method and low risks to life and health. The studies were conducted in accordance with the ethical and legal norms of the Russian Federation.

Supplementary material: The online version of this paper contains supplementary material available free of charge on the journal's website <https://lettersonmaterials.com>.

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