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Thin reduced graphene oxide based films for nanoelectronics and sensors

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Abstract. Formation of new devices of flexible and organic electronics requires new materials. One of the perspective classes of materials is graphene derivatives. To enhance wettability of polymer substrates we used multicomponent graphene oxide suspension with further reduction of obtained films with laser irradiation. We used UV and IR lasers for local reduction of these films to form transistors and biosensors. Graphene oxide film from 0.93 mg/ml suspension with lacquer thinner was successfully deposited and reduced for BGTE transistor formation. Reduced graphene oxide film acted as an n-type semiconductor with $2\text{--}8 \times 10^{-3} \text{ cm}^2/\text{V}\cdot\text{s}^{-1}$ mobility.

Keywords: contact angle, dispersion medium, graphene oxide, reduced graphene oxide, thin films, transistor

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Материалы конференции

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Тонкие пленки на основе восстановленного оксида графена для наноэлектроники и сенсоров

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Аннотация. Создание новых устройств гибкой и органической электроники требует новых материалов. Одним из перспективных классов материалов являются производные графена. Для повышения смачиваемости полимерных подложек использовалась многокомпонентная суспензия оксида графена с последующим восстановлением полученных пленок лазерным облучением. Для локального восстановления этих пленок с целью формирования транзисторов и биосенсоров использовались УФ- и ИК-лазеры. Пленка оксида графена из суспензии 0,93 мг/мл с растворителем для лака была успешно осаждена и восстановлена для формирования транзистора BGTE. Восстановленная пленка оксида графена вела себя как полупроводник n-типа с подвижностью $2\text{--}8 \times 10^{-3} \text{ см}^2/\text{В} \cdot \text{с}^{-1}$.

Ключевые слова: угол смачивания, дисперсионная среда, оксид графена, восстановленный оксид графена, тонкие пленки, транзистор

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Introduction

Overcoming limitations of classic microelectronic technology has been one of the main points of interest of scientists during last 30 years. Since the beginning of the 2000s, researchers have tried to create or adopt new materials for electronics to obtain flexibility and overcome shape limitations of electronic products.

Among the variety of new materials like fullerenes [1], nanotubes [2], organic semiconductors [3] graphene derivatives seem to be the most perspective [4]. Graphene oxide (GO) is a material that on one hand can be relatively easy deposited on the large area substrates and on the other hand by the management of the presence of functional groups on its surface. The last fact allows reduced graphene oxide (rGO) [5] to be either conducting or semiconducting. Such management can be done by different techniques, including chemical and electrochemical [6, 7], thermal [8] or laser [9], reduction. Presence of functional groups is attractive for other rGO applications, especially for chemical and biosensors [10]. The use of laser for GO reduction may be the most prospect due to the possibility of treatment localization and obtain different additional chemical [11] effects in case of pico- and femtosecond lasers [12].

Wet deposition methods seem to be the most suitable for formation of large area graphene oxide films. And such methods as spin- [13] and spray [14] coating are most perspective due to the possibility of deposition of large-area uniform films. Management of suspension properties can be done by varying its composition. Moreover, the effect of dispersion on the quality of GO structure hasn't been studied properly. According to the literature data [15–17] we found a set of possible additional organic solvents based on its Hildebrand parameter (δT) that should be close or higher than that of graphene oxide ($\delta T = 25.4 \text{ MPa}^{1/2}$).

Based on the multicomponent GO suspension with thinner for lacquer paints as additional component, we made macrosized uniform films for further local laser reduction. We showed the possibility to use the cheap microsecond laser for local reduction of spin-coated GO film with transistor channels formation. We obtained n-type rGO transistor with mobility in the range from $2 \times 10^{-3} \text{ cm}^2/\text{V} \cdot \text{s}^{-1}$ to $8 \times 10^{-3} \text{ cm}^2/\text{V} \cdot \text{s}^{-1}$ that can be used for flexible electronics.

Materials and Methods

For device formation we used 125 μm thick polyethylene terephthalate (PET) film as a substrate with 15×15 mm size. All substrates were mechanically precleaned with isopropanol with further drying in a 4 atm air stream.

For GO film formation we used graphene oxide water suspension with 3.11 mg/ml concentration was synthesized by the modified Hummers method (MIP Graphen LLC, Russian Federation).

As additional component for better wettability we used thinner for lacquer paints (LT, Tamiya Ink., Japan) with concentration 1.24 mg/ml. that is in range of 1–1.5 mg/ml that is suitable for uniform film formation [18]. Also, Hildebrand parameter of the main component of this thinner – ethylene glycol is $\delta T = 33 \text{ MPa}^{1/2}$.

GO deposition was obtained with the EZ4 spin-coater (LEBO Science, PRC) with the following deposition profile: 1000 rpm for 60 sec with a further 30 min heat treatment in the low-vacuum chamber VAC-24 (Stegler, PRC) at 110 °C. Gate (Al), Source and Drain (Ag) electrodes deposited by thermal sputtering on a modified VUP-4 thermal evaporator (USSR). The thickness of all electrodes was ~100 nm, measured by a quartz monitor crystal (INFICON AG, Germany). For Gate passivation, we used anodic oxidation of Al on a self-designed laboratory setup under 30 V voltage. Local laser treatment was carried out by the self-designed system with a 445nm solid-state laser facility with a motorized table. Output laser power was about 200 mW at a 30 MHz frequency. Treatment time in a spot ($1.5 \times 10^{-3} \text{ mm}^2$) was constant at 30 ms.

Raman spectra were obtained on an InVia Raman spectrometer (Renishaw, UK). FTIR spectroscopy data for films was collected on the Nicolet iS10 (Thermo Scientific, USA) in attenuated total reflectance mode. I–V curves of reduced graphene oxide transistors were measured on Keithley 4200A (Tektronix, USA). Transfer curve measurements were done with the next parameters: Source-Drain voltage was constant at 5 V in all cases; Gate-Source voltages were measured in a range from –1 V to 10 V depending on the specific transistor. The thickness of the GO film was measured on NTEGRA PRIMA SPM (NT-MDT, Russian Federation) in semicontact mode with an NSG 30 cantilever: 320 kHz resonance frequency, 40 N/m force constant (Tipsnano OÜ, Estonia).

Results and Discussion

Deposition of GO films were done with the use of multicomponent suspension with LT as additional component and 1.24 mg/ml concentration. This suspension allows for better wettability of PET substrate, and its characteristic drying time of about 70 seconds is very close to that of spin coating process. Nevertheless, we observed much better adhesion in case of additional low vacuum thermal treatment at 110 °C for 30 min. After abovementioned operations a thin GO film (thickness about 80–90 nm according to AFM data) that uniformly covers all the substrate was formed.

For investigation of laser modification, we used BGTE transistor configuration (Fig. 1) with GO film (suspension with LT as additional component, 1.24 mg/ml) deposited on the passivated gate electrode with further top source and drain electrode formation by thermal sputtering through the mask. Local laser treatment was carried out by the own-designed system with a 445nm solid-state laser facility with a motorized table. For GO reduction, we used laser parameters that were equal to ~35–37 mJ/cm²; the reduction process was made in a camera with nitrogen flow to remove possible impurities arising in the reduction process. Reduced GO areas are shown in Fig. 1 (black regions). Reduction of GO was confirmed by Raman (Fig. 2) and IR spectroscopy (Fig. 3).

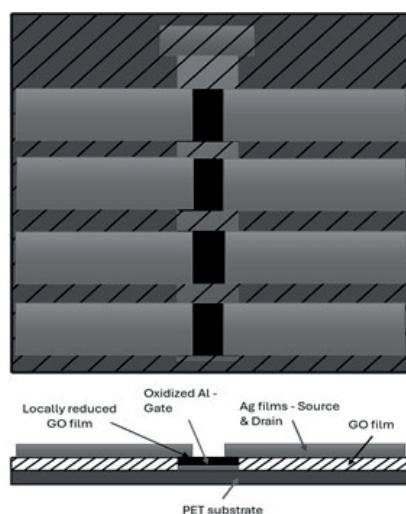


Fig. 1. Scheme of rGO transistor the top view and cross-section

A general view of Raman spectra of rGO correlates with results observed in [19, 20]. From these articles, we can clearly identify high-intensity G, D and 2D bands. We also clearly observe low-intensity D*, G*, D+G, and 2G bands. The relatively sharp and intensive 2D peak in our case tells us that on one hand defects in rGO structure are partially healed, and GO film successfully reduced, on the other the intensity of 2D band is much lower compared to the spectra typical for the fully reduced GO. Thus, we assume that laser irradiation with 35–37 mJ/cm² results in partial reduction of GO film which can be semiconduction due to opening of band gap.

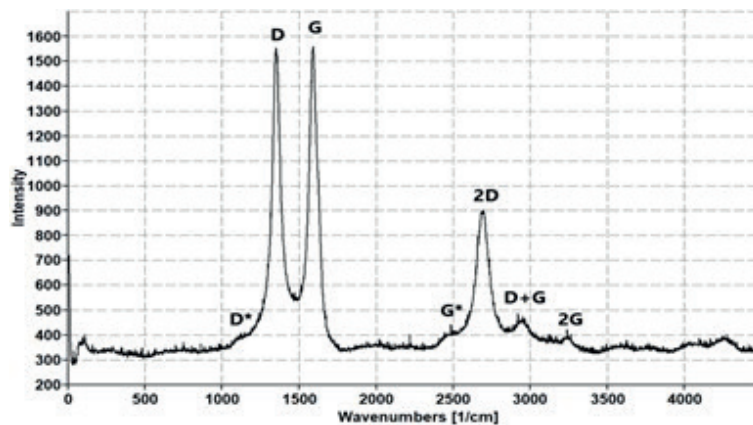


Fig. 2. Raman spectra of locally reduced graphene oxide in transistor channel

In IR spectra (Fig. 3), we clearly observe the presence of different bands, most of which are common for graphene oxide and reduced graphene oxide. This fact is one more confirmation of the partial reduction of the initial graphene oxide film. According to the literature data presented in [21, 22] we can identify the next bands: two small bands at 2927 cm⁻¹ and 2849 cm⁻¹ are asymmetric and symmetric CH₂ stretching of GO. The band at ~1720 cm⁻¹ is attributed to the presence of carbonyl and carboxyl groups; the 1582 cm⁻¹ band comes from aromatic ring stretching; the 1400 cm⁻¹ band comes from -OH bending in phenol etc. Thus, we can clearly see the presence of different functional groups that mean partial reduction of graphene oxide. Moreover, partial reduction can be used for the task of transistor formation due to the presence of carbonyl and carboxyl groups (~1720 cm⁻¹ band).

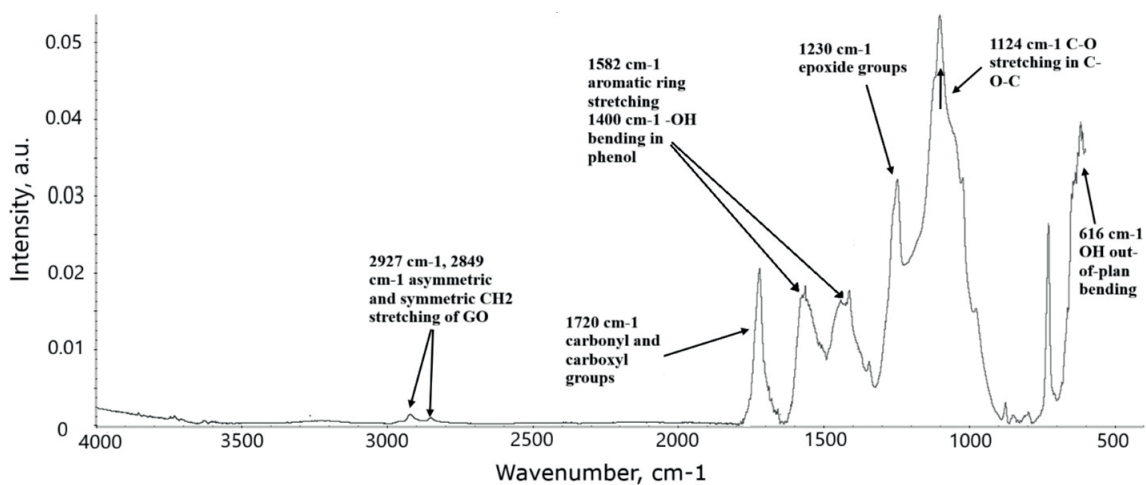


Fig. 3. FTIR spectra of locally reduced graphene oxide in transistor channel

After GO reduction, we made Source and Drain electrodes by thermal evaporation of Ag with a thickness of about 100 nm (measured by a quartz detector in the evaporation camera). The total number of substrates was 5, with four transistors on each substrate, i.e., we made measurements

of 20 individual transistors. Measurement of CV characteristics was made in a nitrogen box with special probes on Keithley 4200A semiconductor measurement station. The typical CV transfer curve of the obtained rGO transistors is shown in Fig. 4.

As can be seen from Fig. 4, the n-type conductivity is observed. This fact correlates with the result of the [23], where the n-type of p-type charge carriers and rGO film depend on reduction temperature. For n-type charge carriers, rGO film should be reduced with temperatures in ranges of 250–450 °C and from 800 to 1000 °C. According to the similarity of FTIR spectra in our article and [23] we can assume the local reduction temperature in the range > 350–450 °C in our case.

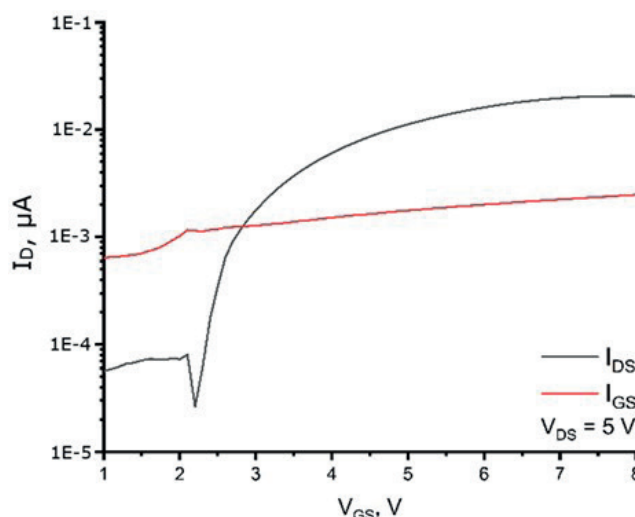


Fig. 4. Transfer CV curve of rGO transistor. I_{DS} – Drain-Source Current, I_{GS} – Gate-Source current, V_{DS} – Drain-Source voltage, V_{GS} – Gate-Source voltage

The mobility of the rGO films was calculated from the CV characteristics of transistors. We found that mobility is in the range from $2 \times 10^{-3} \text{ cm}^2/\text{V}\cdot\text{s}^{-1}$ to $8 \times 10^{-3} \text{ cm}^2/\text{V}\cdot\text{s}^{-1}$. This result is much lower compared with data from [24, 25] and close to the results obtained in [26]. We attribute relatively low mobility to the not full reduction of GO film. Nevertheless, such transistors can be used in biosensor development due to transistor effect on one hand and presence of carboxyl functional groups that is important sensitive biomolecule immobilization through the coupling with carboxyl groups.

Conclusion

The work successfully demonstrated the possibility of formation of uniform GO films ready for further local laser induced reduction by the use of additional organic component in the deposited GO suspension with concentration in range 1–1.5 mg/ml. By the use of relatively cheap laser, it is possible to obtain local reduction of graphene oxide film with formation of BGTE design transistors, where the rGO film behaved as an n-type semiconductor. Despite the relatively low mobility in range about $2\text{--}8 \times 10^{-3} \text{ cm}^2/\text{V}\cdot\text{s}^{-1}$ partially reduced rGO films with the presence of carboxyl functional groups can be used as basis for chemical or biosensor formation due to the possibility of sensitive molecules' immobilization.

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