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# Morphology and electrical properties of modified Langmuir graphene oxide films

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**Abstract:** The work devoted to study of the electrical properties and morphology of Langmuir-Blodgett films of modified graphene oxide (GO). The Langmuir-Blodgett method was used for studying of formation process of Langmuir monolayers formed on the subphase contained dissolved ions of tin chloride salt. The compression isotherms of monolayers formed in the presence of tin ions shifted towards larger occupied areas. Langmuir monolayers of graphene oxide formed under various conditions were transferred on solid substrates and studied by atomic force microscopy and voltammetry. It has been established that the presence of tin ions in the aqueous subphase leads to changes in the morphology of the formed films. Thus, the average roughness and surface area of such films increased from 0.2 nm and 0.04  $\mu\text{m}^2$  to 5.4 nm and 0.46  $\mu\text{m}^2$ , respectively. The presence of tin ions dissolved in the aqueous subphase influenced the type of current-voltage characteristics of the films under study. The maximum reverse current increases from 0.75 nA to -0.3 nA and the resistance increases from 11 GOhm to 700 GOhm. Temperature annealing of the graphene oxide film at 300°C leads to a decrease in the maximum forward current and an increase in the reverse current from 0.0407 nA and -0.071 nA to 0.0065 nA and 0.0616 nA, respectively. For a film formed in the presence of ions in the subphase, annealing does not affect the maximum forward current, and the current of the reverse branch of the current-voltage characteristic increases from -0.1669 nA to 0.0468 nA.

**Keywords:** graphene oxide thin films, Langmuir graphene oxide monolayers, thin films conductivity, graphene oxide hybrid coatings

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## 1. INTRODUCTION

Nowadays, ensuring environmental monitoring of the state of the atmosphere is an urgent task. This is due to the growing level of environmental pollution. This task is of particular relevance in large cities with a large number of road transport. One of the products of car exhaust is carbon monoxide and carbon dioxide. The development of sensor coatings capable of selectively

absorbing carbon monoxide or carbon dioxide is an urgent task in the development of gas sensors. Graphene oxide is a promising material for such sensor coatings. [1-3].

Despite the possibility of creating sensors with high sensitivity [4,5], their main disadvantage is low selectivity with respect to target gases. To solve this problem, films based on graphene oxide are modified with functional groups and nanoparticles [6,7]. In particular, to create coatings selective to carbon dioxide and carbon dioxide, films based on graphene oxide are modified with tin oxide nanoparticles [8], that allows to significantly increase the selectivity and sensitivity of sensors [9-11]. Also, the disadvantages of such sensors include low reproducibility of characteristics from device to device [12,13]. One of the approaches to reduce the number of defects in a graphene oxide film is the Langmuir-Blodgett technology, since it remains possible to orient graphene oxide particles in the film [14].

The Langmuir-Blodgett (LB) Technology is widely used for the formation of thin films of various surfactants, nanoparticles and polymer molecules [15-18]. There are also works devoted to the formation and study of the properties of Langmuir monolayers of graphene oxide [19]. The influence of subphase acidity on the morphology of Langmuir-Blodgett graphene oxide films was studied by authors [20]. It has been shown that changing the acidity makes possible to control the thickness of graphene oxide films. One of the promising tasks in this direction is doping a graphene oxide monolayer with ions of various metals. The study of adsorption process of heavy metal ions to colloidal graphene oxide was performed by authors [21-23]. The presence of similar works allows us to conclude that it is possible to embed charged particles into graphene films. The process of incorporation of  $\text{Cs}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Y}^{3+}$  ions into a graphene film on the surface of water has been studied by authors [25,24]. The authors demonstrated that ion adsorption is determined not only by

electrostatic interaction with graphene oxide, but is more complex in nature. In such works, the authors pay little attention to studying the possibility of incorporating metal ions into Langmuir monolayers of graphene oxide, which could lead to the creation of new types of ordered two-dimensional materials. Also, due attention is not paid to the electrical properties of graphene oxide films with adsorbed ions, as well as the influence of the thermal treatment process on their properties. This problem becomes particularly relevant when attempting to sensitize graphene oxide films with oxide particles.

Therefore, the main goal of the present work was to study the effect of metal ions dissolved in the aqueous subphase on the electrical properties and morphology of Langmuir-Blodgett graphene oxide films. And also, the effect of thermal treatment of films on them.

## 2. EXPERIMENTAL PART

### 2.1. MATERIALS AND METHODS

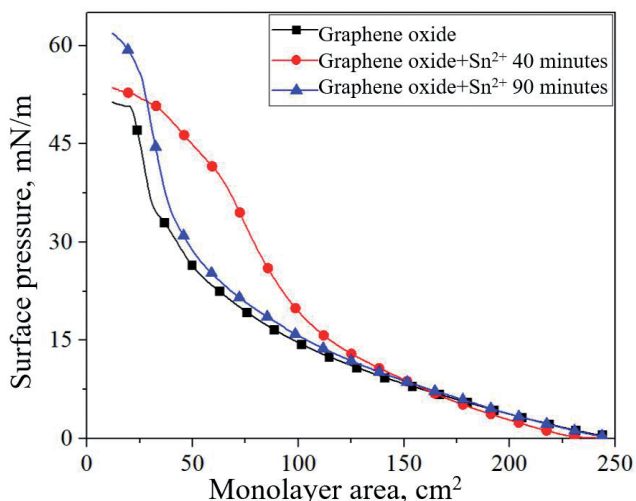
The graphene oxide powder with an average particle diameter of 10 to 100  $\mu\text{m}$  (Rusgrafen LLC, Russia) was used for preparation a solution of graphene oxide. Graphene oxide powder was dissolved in a water:methanol mixture with a volume ratio of 1:5 to obtain a solution with a concentration of 0.5 mg/ml. The resulting solution was sonicated in an ultrasonic bath (Skymen JP-031S, China) with a power of 180 W for 30 minutes. The solution was centrifuged at a rotation speed of 4000 rpm for 30 minutes. After stratification of the solution and precipitation, the supernatant part of the solution was selected, which was sonicated for 40 minutes and precipitated into a centrifuge at a rotation speed of 13,000 rpm. The sediment part of the solution formed during centrifugation was collected. The precipitate was dried in a vacuum chamber at room temperature for 60 minutes and re-dissolved in a water:methanol mixture with a volume ratio of components of 1:5. In this way, a water-methanol solution of graphene

oxide particles with a concentration of 0.1 mg/ml was obtained. Before forming a Langmuir monolayer, the solutions were sonicated for 15 minutes in an ultrasonic bath.

To prepare the subphase with dissolved tin chloride ( $\text{SnCl}_2$ ), dry tin chloride powder was taken and dissolved in deionized water to obtain a concentration of 1 Mol/L. The resulting solution was diluted with deionized water to obtain a concentration of  $10^{-2}$  M, which was used as an aqueous subphase.

**2.2. FORMATION OF LANGMUIR MONOLAYERS AND LANGMUIR-BLODGETT FILMS OF GRAPHENE OXIDE**

All experiments on the formation and transfer of Langmuir monolayers to solid substrates were carried out on a KSV Nima LB Trough KN2002 setup. The temperature of the subphase was maintained constant at 22°C using an A300 circulation cooler (Termex LLC, Russia, Tomsk). Deionized water with a resistivity of 18 M $\Omega$ ×cm and an aqueous solution of tin chloride with a concentration of  $10^{-2}$  M were used as a subphase. The formation of Langmuir monolayers of graphene oxide took place according to the following procedure. An aliquot of graphene oxide solution with a volume of 500  $\mu$ L was applied to the surface of the aqueous subphase. After 20 minutes for deionized water, 40 minutes and 80 minutes for the subphase containing tin ions, the monolayer was compressed by movable barriers with a constant rate of area loss equal to 0.7 cm<sup>2</sup>/min. During the compression process, a  $\pi$ - $A$  diagram was recorded automatically – the dependence of the change in pressure on the area occupied by the monolayer. The compression isotherm of the formed monolayers is shown in Fig. 1. The monolayer was transferred to a solid substrate at a constant surface pressure of 40 mN/m using the Langmuir-Blodgett method – vertical lift. The speed of movement of the substrate through the monolayer was constant and amounted to 1 mm/min. In this way, 5-layer films were formed based on monolayers of graphene oxide (film



**Fig. 1.** Compression isotherms of graphene oxide monolayers formed on deionized water and on deionized water containing dissolved tin ions at 40 minutes and 90 minutes of ion adsorption time by the monolayer.

1), graphene oxide that adsorbed tin ions for 40 minutes (film 2) and adsorbed tin ions for 90 minutes (film 3). The surface properties of monolayers were studied using the compression isotherm method. The linear sections on the compression isotherm correspond to the phase state of the monolayer and are characterized by the compression modulus of the monolayer ( $k$ ) and the specific area of the monolayer in the phase state ( $A_0$ ). The value of  $A_0$  can be obtained by extrapolating the linear portion of the phase state of the monolayer to the  $A$  axis at a value of  $\pi = 0$  mN/m. To calculate the compression modulus of a monolayer, the formula is used:

$$k = -A_0 \frac{d\pi}{dA}, \tag{1}$$

where  $k$ ,  $A$  and  $A_0$  are the compression modulus of the monolayer, the area of the monolayer and the specific area of the monolayer in the phase state under study. Since the values of  $k$  and  $A_0$  change during the interphase transition of the monolayer, analysis of the dependence  $k(A_0)$  allows us to identify areas on the compression isotherm that correspond to the gaseous, inclined-condensed phase and non-inclined-condensed phase of the liquid and condensed phase of the monolayer [26-28].



**Table 1**

Values of the compression modulus ( $k$ ) and specific area of monolayers in the non-obliquely condensed phase ( $A_0$ ), formed under various conditions

Subphase	$A_0$ , cm <sup>22</sup>	$k$ , mN/m
Deionized water	47.5	1.6
40 minutes adsorption	32.8	2.3
90 minutes adsorption	38.8	2

Compression isotherms of graphene oxide monolayers formed on an aqueous subphase in the absence and presence of dissolved tin ions at different times of their adsorption by the monolayer are shown in Fig. 1.

**Table 1** shows the values of  $k$  and  $A_0$  of graphene oxide monolayers formed under various conditions.

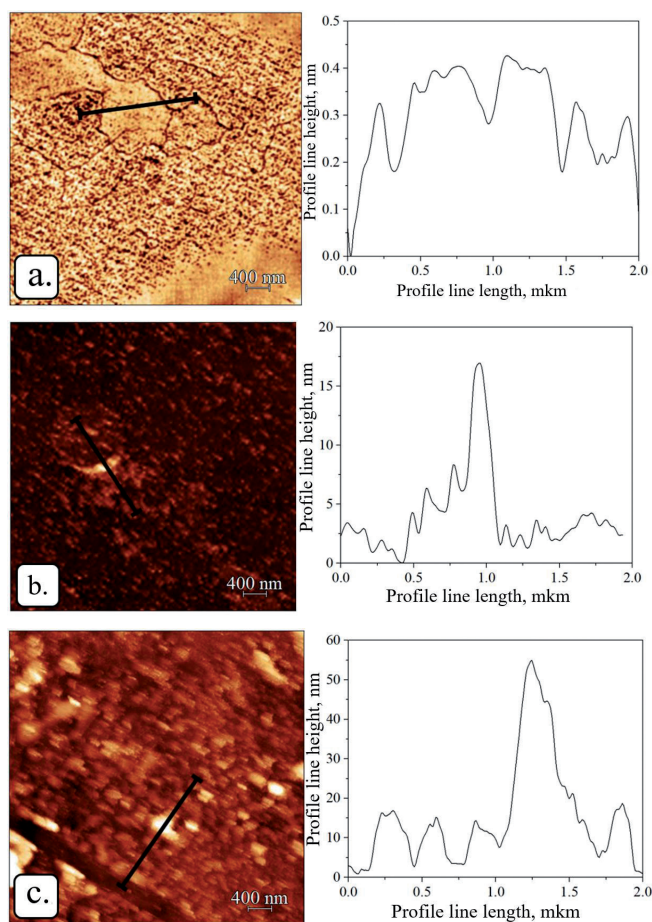
### 2.3. STUDYING THE MORPHOLOGY OF FORMED LANGMUIR-BLODGETT FILMS USING ATOMIC FORCE MICROSCOPY

The formed Langmuir monolayers were transferred to solid substrates made of atomically smooth mica using the Langmuir-Blodgett technology. In this way, monolayer films were formed based on monolayers of graphene oxide, formed in the absence and presence of dissolved tin ions in the subphase. The morphology of the resulting film coatings was studied by atomic force microscopy using an NT-MDT Netgra installation in hybrid mode. **Fig. 2** shows typical images of the surface morphology of the resulting films.

Mathematical processing of the obtained images in the Gwyddion 2.63 program made it possible to determine the average surface area of the film ( $S$ ) and the value of its average roughness ( $R_a$ ). The surface area of the film was calculated by the triangulation method, the average roughness of the film surface was calculated using the formula [29,30]:

$$R_a = \frac{1}{N} \sum_{j=1}^N |r_j|, \quad (2)$$

where  $R_a$  is the arithmetic mean deviation of the profile from the baseline (profile mean line),  $N$  is the number of points at which the roughness parameter  $R_a$  was measured, and  $r_j$  is the absolute



**Fig. 2.** Images of the surface and its profile lines of a graphene oxide film (a), a film based on a monolayer that adsorbed tin ions for 40 minutes (b) and 90 minutes (c).

**Table 2**

Values of the average surface roughness ( $R_a$ ) of the film and its surface area ( $S$ )

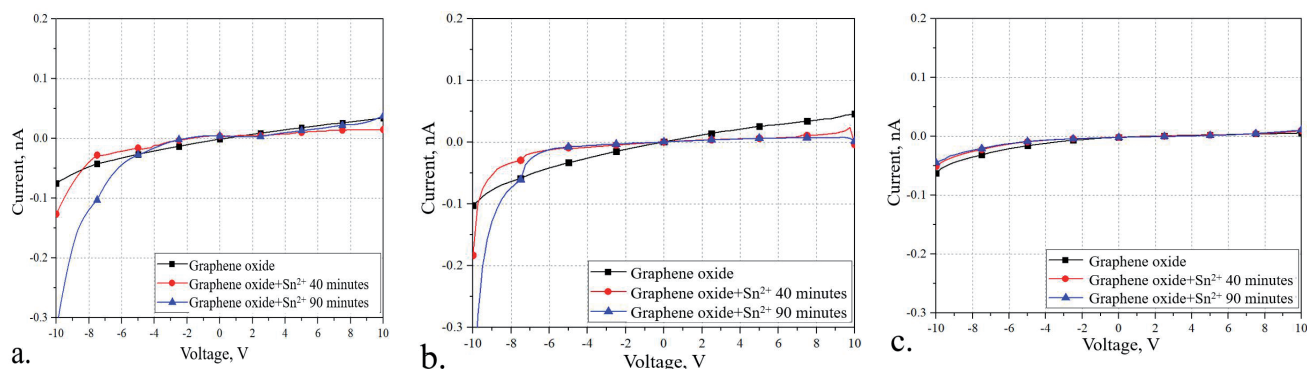
Film	$R_a$ , nm	$S$ , mkm <sup>2</sup>
Graphene oxide	0.2	0.04
Graphene oxide + SnCl <sub>2</sub> 40 minutes	1.4	0.42
Graphene oxide + SnCl <sub>2</sub> 90 minutes	5.4	0.46

deviation of the profile height value from the mean line at each roughness measurement point.

**Table 2** shows the average roughness and surface area of films formed under various conditions.

### 2.4. STUDY OF THE ELECTRICAL CHARACTERISTICS OF THE FORMED FILMS

Langmuir graphene oxide monolayers formed under different conditions were transferred onto solid coverslip substrates with formed interdigitated electrode structures. The distance between the pins was 100  $\mu$ m. The electrical



**Fig. 3.** Current-voltage characteristics of the formed films before annealing (a), after annealing at a temperature of 150°C (b) and 300°C (c).

characteristics of films 1, 2 and 3 were studied by voltammetry. The current-voltage characteristics were measured on a Keythley 6487 picoammeter in the voltage range of +10 V –10 V with a scan step of 0.2 V. The formed film coatings were annealed at temperatures of 150°C and 300°C for 30 minutes in an air atmosphere. The current-voltage characteristics of the films under study are shown in **Fig. 3**.

### 3. DISCUSSION AND CONCLUSIONS

The compression isotherm of graphene oxide monolayers formed under various conditions is shown in Fig. 1. In a graphene oxide monolayer, one can distinguish a gas phase corresponding to section I-II, an inclined condensed phase (section II-III) and a non-obliquely condensed phase (section III-IV). When the surface pressure exceeds 50 mN/m, monolayer collapse occurs, which characterizes a sharp change in the slope of the compression isotherm. The addition of tin ions to the subphase affects the type of compression isotherm depending on the time of interaction of tin ions with graphene oxide particles. With an interaction time of 40 minutes, the collapse pressure of the monolayer increases from 50 mN/m to 60 mN/m. The compression modulus of the monolayer also increases from 1.6 mN/m to 2.3 mN/m. The  $A_0$  value increases from 49 cm<sup>2</sup> to 51 cm<sup>2</sup>. The compression isotherm of the monolayer formed at a time of interaction of graphene oxide and tin ions of 90 minutes also shifted towards larger areas. It can be concluded that tin ions

have an expanding effect on the graphene oxide monolayer.

Fig. 2 shows images of the surface of graphene oxide films formed in the presence and absence of tin ions in the subphase. The monolayer of used graphene oxide particles has a thickness of about 0.2 to 0.4 nm with individual particle diameters from 1 to 4 μm. Such a scatter in heights allows us to conclude that individual graphene particles are non-monolayer. The interaction of a monolayer of graphene oxide with tin ions leads to a change in the morphology of the film. The average film roughness increases from 0.2 to 1.4 nm, the average surface area increases by 0.38 μm<sup>2</sup>. On the surface of the film, aggregates with heights from 3 to 20 nm and areas up to 0.8 μm<sup>2</sup> can be distinguished. An increase in the interaction time of tin ions with a monolayer of graphene oxide leads to an increase in the average roughness to 5.4 nm and the average surface area to 0.46 μm<sup>2</sup>. The average heights of the formed aggregates increased and ranged from 15 nm to 55 nm. The interaction of tin ions and a Langmuir monolayer of graphene oxide leads to the formation of aggregates on the surface of the film. The sizes of the aggregates increased with increasing interaction time between the graphene oxide monolayer and tin ions.

Fig. 3 shows the current-voltage characteristics of multilayer films 1, 2 and 3. The current-voltage characteristic of film 1 has a linear form. Its conductivity was about 0.09 pS.

The presence of tin hydroxide aggregates in the film leads to a change in the type of current-voltage characteristic. The current-voltage characteristics of films 2 and 3 have a nonlinear shape; an increase in the adsorption time of tin ions leads to an increase in the maximum reverse current from 0.125 nA to 0.3 nA. The maximum current of the forward branch of the current-voltage characteristic decreases from 0.025 nA to 0.01 nA with increasing adsorption time of tin ions. Thermal treatment of the formed film 1 for 30 minutes at a temperature of 150°C leads to an increase in the current of the forward and reverse branches of the current-voltage characteristic to -0.1 nA and 0.05 nA, respectively. The maximum current of the reverse branch of the current-voltage characteristics of films 2 and 3 also increases to 0.18 nA and 0.4 nA, respectively. The increase in maximum currents may be associated with the beginning of the reduction process of graphene oxide and an increase in its conductivity. At the same time, the high defectiveness of the films and the loss of conductivity at the edges of reduced graphene oxide particles explain the low conductivity values in the films. Annealing films at a temperature of 300°C leads to an increase in film resistance to 700 GOhm. After annealing at a temperature of 300°C, the current-voltage curves of films 2 and 3 acquire a linear form. A similar effect may be associated with the reduction of graphene oxide and desorption of tin dioxide particles from the film surface.

#### 4. CONCLUSION

Thus, the work studied the electrical properties and morphology of Langmuir-Blodgett films based on graphene oxide during their thermal reduction. The possibility of modifying Langmuir monolayers of graphene oxide with metal ions during the formation of monolayers has been established. The possibility of controlling the electrical conductivity of Langmuir-Blodgett graphene oxide films by thermal treatment has been demonstrated. The recovery temperature regime has been determined, at which the

current-voltage characteristics retain their nonlinear form. The approach used in the work can be used to form sensor coatings sensitive to carbon monoxide and carbon monoxide, to develop gas sensors with greater sensitivity.

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