III. CONCLUSIONS

Fabrication process of sensor based on anodic alumina has been developed. It has been found that increasing number of cantilever beams and oxide thickness decreases sensitivity value. Adding one additional console reduces sensitivity by 75%, and two – by 90%. Increasing the thickness of the anodic oxide film by 10 µm, reduces the sensitivity by 30%. By varying the parameters of the anodizing can change the sensitivity range of the active element sensors in a wide range of values.

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THE REDUCTION OF GRAPHENE FROM GRAPHENE OXIDE

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I. INTRODUCTION

Graphene has a surprising combination of properties: transparency, mechanical strength, elasticity, peak mobility of charge carriers (Dirac massless fermions), and high thermal conductivity; it is elastic and impermeable with respect to molecules of the other substances. It is also more inert than gold when it comes to the effects of external conditions [1, 2].

All of these characteristics, which are apparent in different physicochemical processes, make graphene a highly promising material for practical use in everything from nanoelectronics to composite materials [3].

The best way of obtaining graphene today is to use graphene oxide produced in the form of flakes by laminating graphite with oxidizing reagents. This material can be considered graphite intercalated with oxygen-containing carbon groups that give it the ability to be thoroughly dispersed in water and hydrophilic organic solvents. When interacting with chemical reducing reagents, graphene oxide is converted into thin graphene flakes of low structural quality [4–7] due to the substantial degradation of graphite during the synthesis of graphene oxide. We nevertheless can obtain graphene from this material [6–10]. These data are, however, difficult to compare, since reagents that differed in their chemical properties were used, and the processes involving them were performed under different thermal conditions. In addition, graphite materials of different structural quality and fineness were used to obtain graphene oxide [7, 10].

In this work, we therefore study the effects the reducing agents used in [4, 7–10] had on the structure and chemical composition of graphene oxide in order to assess the prospects for using it to obtain graphene. In all reduction processes, graphene oxide was derived from graphite with the same chemical composition, structure, and fineness. Such research methods as X-ray energy dispersive spectroscopy (XEDS), Raman spectroscopy (Raman spectra), and scanning electron microscopy (SEM) were used to assess the quantitative and qualitative compositions of the initial and final materials, and their structural features.

II. EXPERIMENTAL

Graphene oxide was synthesized according to the Hammers method [8] from pyrolytic graphite crushed with a ball mill to form a fraction of 50–100 µm. Concentrated phosphoric (85 %) and sulfuric acids (95 %), and potassium permanganate (chemically pure), were used for this purpose. The reduction of graphene from graphene oxide was conducted in hydrazine hydrate, ethylene glycol (chemically pure), and hydrogen. The process was conducted with hydrazine hydrate using the original method described in [4].

The reduction of graphene from graphene oxide in ethylene glycol was conducted as described in [10]. Graphene was also reduced from graphene oxide with hydrogen under argon (Ar/H_2; volume ratio 1:1) at 200 and 1000°C [9, 10].

The quantitative and qualitative compositions of the investigated materials were studied via XEDS in three locations on an array. Their Raman spectra were obtained at a laser excitation wavelength of 532 nm

using a Labram spectrometer (Jobin Yvon Technology), while SEM images were recorded with an S-4800 unit (Hitachi). The Raman spectra of the investigated materials are presented in Table 1 and Figure 1.

The data of XEDS method's indicate that graphene oxide contains small amounts of impurities of the reactants used to produce it. In particular, its mass includes ≈0.3% of phosphorus, 1 % of sulfur, and 1 % of manganese. Overall, these data show that graphene oxide and the materials reduced from it had almost no inorganic impurities.

Table 1 – Characteristics of the materials' Raman spectra peaks (1 – crushed graphite; 2 – graphene oxide; 3 – after reduction with hydrazine hydrate; 4 – after reduction in ethylene glycol; 5 – after reduction with hydrogen under argon at 200 °C; and 6 – after reduction with hydrogen under argon at 1000 °C)

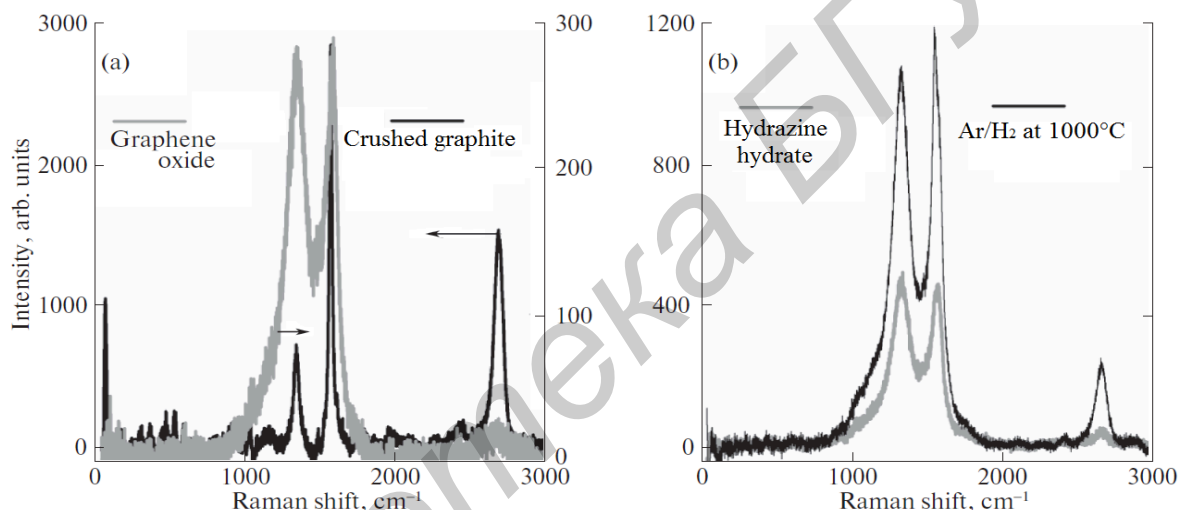| No.	Frequency, cm^{-1}			Relative intensity		
	D	G	2D	ID/IG	I2D/IG	I2D/ID
1	1349	1579	2700	0.24	0.5	2.1
2	1352	1586	2658	1.06	0.03	0.03
3	1350	1583	2685	1.02	0.09	0.08
4	1349	1586	2685	1.09	0.09	0.08
5	1340	1583	2679	1.50	0.08	0.06
6	1344	1580	2675	0.94	0.24	0.26

Figure 1 – Raman spectra of (a) crushed graphite and graphene oxide, and (b) materials obtained from graphene oxide via reduction in hydrazine hydrate and Ar/H2 at 1000°C. Arrows show the axis of intensity

Analysis of these data shows that graphene oxide had substantially higher ID/IG and lower I2D/IG and I2D/ID values than the crushed source graphite. This indicates there were a great many cleaved sp2 carbon bonds in its structure [6–8, 10, 11]. The frequency shift of the G band toward the high-frequency region also indicates considerable defectiveness of the graphene oxide structure. The shapes of the 2D bands in the Raman spectra (Figure 1) also illustrate a different quality of the structure of graphene layers in materials 1–3 and 6 (Table 1). The 2D band in the Raman spectrum of the initial crushed graphite (Figure 1a) is intense and narrow, but does not contain a 2D2 curve in the low frequency region. This indicates initial three-dimensional disorder of the graphene layers in this material [7], due probably to its grinding. The 2D band in the Raman spectrum of graphene oxide (Figure 1b) is not only of low intensity; it broadens due to the high structural defectiveness of carbon rings in flakes of this material [7, 9].

Figure 2 shows SEM images of graphene oxide and the material obtained from graphene oxide via reduction in Ar/H$_2$ at 1000°C, respectively. They indicate that morphology of conglomerates in graphene oxide has great relief in which flakes are tightly grouped (Figure 2a). The structure of conglomerates in the material obtained from graphene oxide via with reduction in Ar/H$_2$ at 1000°C are much looser. In contrast to graphene oxide, the flakes in this material are somewhat affected by strong Van der Waals bonds during aggregation, due to the low number of oxygen-containing structural groups.
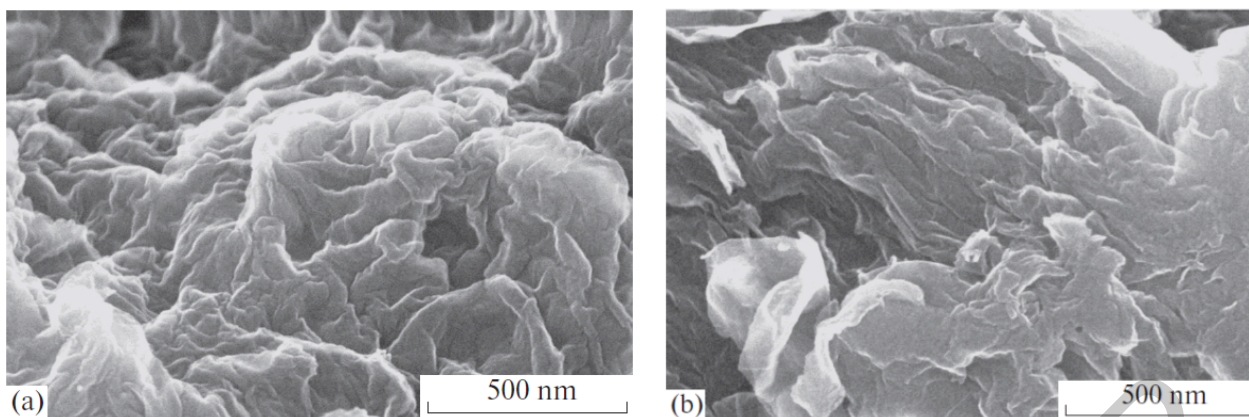
Figure 2 – SEM images of (a) graphene oxide, (b) and material obtained
from graphene oxide via reduction in Ar/H$_2$ at 1000°C, respectively

III. CONCLUSIONS

Our study of features of the reduction of graphene oxide from graphene at different temperatures and in various media indicates that this process is proceeds only upon high-temperature annealing in an inert atmosphere containing hydrogen. This results in [12, 13] complete removal of physically adsorbed water from the micropores in a graphene oxide array and contributes to the destruction of mobile oxygen-containing carbon groups in the structure of flakes, generally enhancing the effect of hydrogen reduction on them. This leads to dramatic changes in the chemical composition and structure of this material, producing single-layer graphene flakes.

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SOLUTION-BASED HOT-CASTING TECHNIQUE TO RECEIVE A MICROMETER-SCALE CRYSTALLINE GRAINS OF PEROVSKITE

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I. INTRODUCTION

The use of organometallic halide perovskites as an absorbing layer of photovoltaic cells (PC) made it possible to increase the efficiency from 7% to 20%, which corresponds to the level of the traditional cells on silicon. At present, the main requirements for the development and fabrication of a FW based on perovskites are an increase in the efficiency of solar cells, reduction in their cost, as well as an increase in their service life and stability in severe atmospheric conditions (high humidity, temperature extremes, UV radiation). One of the solutions of these problems is the development, investigation and modification of methods for the formation of perovskite films. The purpose of this work is to research perovskite films obtained by applying a precursor solution to a hot substrate at different substrate temperatures.