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Using Low-Energy Etching in a Beam-Plasma Discharge to Create Nanoelectronic Structures and Materials

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Abstract: To guarantee the necessary beam-plasma discharge modes are reliably reproduced, a specially designed electronic injector has been devised. The injector's design may be used as a prototype for an injector that uses beam-plasma discharge to operate in technical plasma-chemical reactors. Based on a beam-plasma discharge, a model has been developed to compute the dynamics of currents and voltages of a non-conducting object processed in a plasma-chemical reactor. The Raton program was created in the Matlabtm environment based on the model. By contrasting the computation results with the experiment, the model's suitability is verified.

I. INTRODUCTION

Optimization of the process of etching and cleaning the surface of semiconductors and dielectrics in a plasma-chemical reactor based on a beam-plasma discharge for modern nanoelectronics problems. This goal is achieved by conducting computer and physical experiments aimed at development of methods and technical means for controlling the energy characteristics of ion flows onto the surface of various materials.

The main objectives of the dissertation work:

1. Development of an electronic injector that provides the beam-plasma discharge modes necessary for various tasks for a modernized plasma-chemical reactor.
2. Development of a model that allows, without carrying out labor-intensive experiments, to calculate the dynamics of currents and voltages of a non-conducting structure when a low-frequency voltage of arbitrary shape is applied to it or to the discharge collector.
3. Analysis and optimization using the developed model of technological etching modes.
4. The use of low-energy etching in a plasma-chemical reactor based on a beam-plasma discharge to obtain monoatomic layers of graphite (graphene) and other electrically conductive layered materials of large area.
5. Determination of the main electrical characteristics of the obtained graphene samples.

The scientific novelty of the work is as follows:

1. A model was built to calculate the dynamics of currents and voltages of the non-conducting structure being processed. The adequacy of calculations using the developed model has been confirmed experimentally.
2. Based on the constructed model, it is shown that the method of plasma potential modulation can significantly overcome the problem of surface charge and facilitate the solution of the problem of creating an ion flow of a given energy both for etching semiconductors and dielectrics, and for deposition of semiconductor and dielectric films on the surface of semiconductor structures.
3. The possibility of obtaining monoatomic layers of graphite (graphene) of a large area using a set of mechanical methods has been demonstrated.
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   peeling and low-energy etching of thin graphite single crystals in a plasma-chemical reactor based on a beam-plasma discharge.
4. Unparalleled samples of single-layer graphene and bigraphene single crystals with characteristic sizes exceeding 105×105 μm2, as well as samples of FLG (several-layer graphene) single crystals with dimensions exceeding 500×500 μm2, were obtained.
5. Using the methods of X-ray diffractometry, optical microscopy, atomic force microscopy, Raman spectroscopy, and magneto transport measurements, the high quality of the films obtained from the point of view of nanoelectronics problems and their uniformity in area were shown.

The practical value of the work is as follows:

1. The developed electronic injector of a special design can serve as a prototype of an injector for use in technological plasma-chemical reactors based on beam-plasma discharge.
2. The developed Raton program allows you to calculate the dynamics of currents and voltages of a non-conducting structure, as well as optimize the process of surface treatment of dielectrics and semiconductors in various types of plasma-chemical reactors.
3. The plasma potential modulation method can be used in industrial installations to effectively control the energy characteristics of ions.
4. The possibility of using a plasma-chemical reactor based on a beam-plasma discharge as a module for the production of graphenes and other electrically conductive layered materials of large area has been demonstrated.
5. The obtained graphene samples are used for prototyping and studying the electronic properties of nanoelectronic structures and devices.

Plasma-Chemical Reactors

This section contains an overview of plasma-chemical reactors, which are the most promising for modern problems of micro- and nanoelectronics.

To create plasma, most technological installations use RF or microwave sources. HF sources, as a rule, use a frequency of 13.56 MHz agreed upon for industrial applications [3], microwave sources based on electron cyclotron resonance (ECR) operate at a frequency of 2.45 GHz [4]. An essential part of such sources are matching devices that ensure optimal power transfer to the plasma.

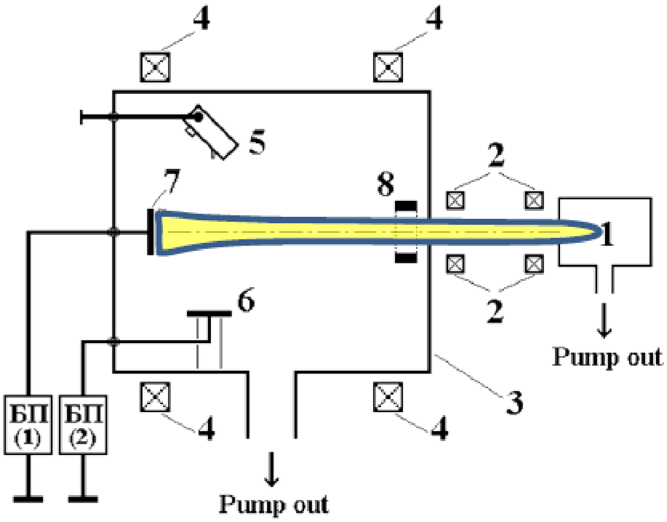

Figure 1. essential part of such sources are matching devices that ensure optimal power transfer

A relatively new type of high-density plasma (HDP) source is also considered - a discharge collector is placed on the opposite wall of the plasma chamber. The potential of the paraxial region can be controlled by changing the spectrum of excited oscillations or by changing the potential of the discharge collector.

The plasma generation system includes a specially designed Pierce-type electron injector; magnetic system with an adjustable field from 3 to 6 mT in the chamber area and from 10 to 50 mT in the electron injector area.

The design of a specially developed Pierce-type electron injector meets the following requirements: the possibility of use in an installation with plasma in the working chamber; ease of replacement of basic elements; the possibility of using a technological plasma-chemical reactor in a prototype for nanoelectronics problems with a small or absent magnetic field in the working chamber. Characteristics of the developed injector: accelerating voltage up to 3 kV; filament power up to 105 W; emission current is up to 0.49 A. The beam diameter at the entrance to the chamber is 1–1.5 cm; pass rate of at least 90%.

The reactor power system consists of a high voltage source for the electronic injector, a filament power supply for the injector cathode, a power supply for the coils, and a power supply for the vacuum equipment. The injector's power source ensures its operation in both continuous and pulsed modes with a pulse duration of τb = 10 - 200 ms.

An electrostatic analyzer with a flat deflecting mirror, moved along the axis at the side wall of the plasma chamber, is used as an ion flow receiver. Analyzer parameters: range of analyzed energies - 0÷105 eV, sensitivity ~0.49•10-9 A/cm2, energy resolution - ΔW/W0=0.12. The signal from the energy analyzer is recorded using a Tektronix 1051B digital oscilloscope.

II. THE DEVELOPMENT AND APPLICATION

This section is devoted to the development and application of a model for calculating the dynamics of currents and voltages of a non-conducting structure when pulsating voltages of various shapes are applied to it or to the discharge collector.

To combat the effect of charging an isolated surface in a beam-plasma discharge to a positive floating potential, it was proposed to apply an alternating voltage to the substrate holder [7].

To optimize the surface treatment process, it is necessary to have information about currents, voltages and, as a consequence, the energies of ions incident on the surface being treated. Due to the great complexity, and in some cases the impossibility of direct measurements, a model was built allowing, without carrying out labor-intensive experiments, to obtain information about currents and energies of ions incident on a non-conducting surface in a beam-plasma discharge when an alternating signal is applied to the substrate holder.

Electrical circuit "substrate holder - non-conducting sample -plasma layer" is modeled by an R-C circuit (Figure 2).

Capacitance Cd is formed by a non-conducting medium between the substrate holder, to which alternating voltage is supplied, and the surface in contact with plasma. Capacitance Cp is formed by a space charge layer between the unperturbed plasma and sample surface.

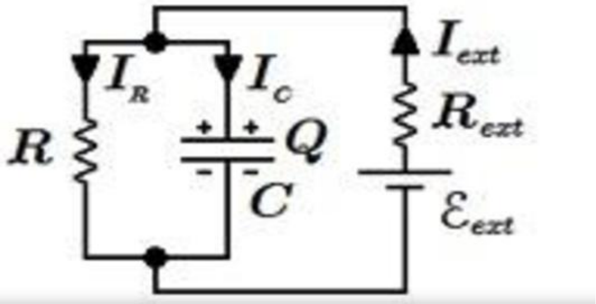

Figure 2. Equivalent electrical circuit underlying the model

A diode through which current I(Up) flows is a symbol nonlinear resistance of the space charge layer. Volt-ampere characteristics(volt-voltage characteristic) of this layer is measured experimentally when replacing the sample with an electrode equal area. Experimental curves with great accuracy approximated by exponential dependence:

$$I(U_p) = -I_i + I_e \exp(\frac{eU_p}{kT}) \quad 1$$

A 1st order nonlinear differential equation for an equivalent circuit is solved in the Raton computer program developed in the Matlabtm environment using the 4th order Runge-Kutta method. Unlike published models [8]

The developed model allows calculations to be made on the basis of an easily measured characteristic, without the use of difficult-to-verify theoretical assumptions.

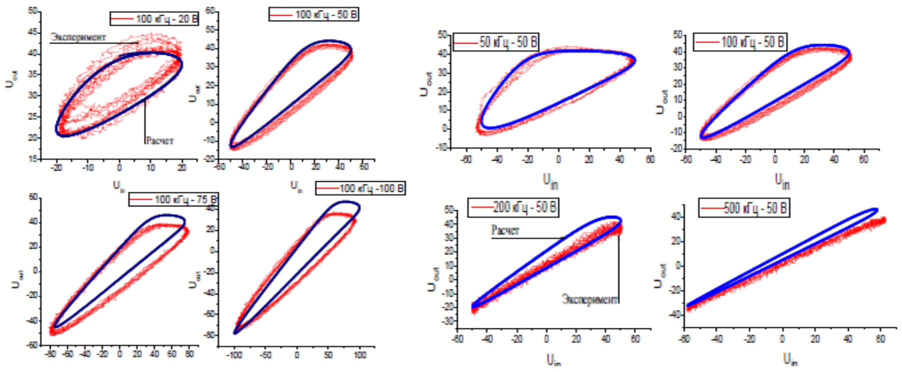

Figure3. Results of comparison of experimental data with simulation results.

To check the adequacy of the model, the voltage on the semiconductor structure simulator probe was measured when an alternating signal with different frequencies and amplitudes from a GZ-33 generator was supplied to the substrate holder. The results of measurements and calculations are presented in Figure 3.

It can be seen from the figure that the calculation results coincide with the experimental results. The deviation of the model from the experiment increases slightly with increasing signal amplitude. This of the space charge layer on the voltage across it. Deviation from the model is also observed with increasing signal frequency. This is due to the use of a quasi-static model, although it is known that at frequencies greater than or close to the plasma frequency of ions ωp ~300 kHz, the inertia of ions distorts the dynamics of the plasma layer.

a change in the potential of the surface in contact with the plasma, and, consequently, the energy of the ions on the surface within a wide range,. To reduce these effects, it is proposed to use a pulsed voltage on the substrate.

To analyze this mode, the calculation program is supplemented with the ability to specify a voltage of an arbitrary shape as a control signal, including that implemented in experiments when working with a high-frequency modulator [9] (Figure 4).

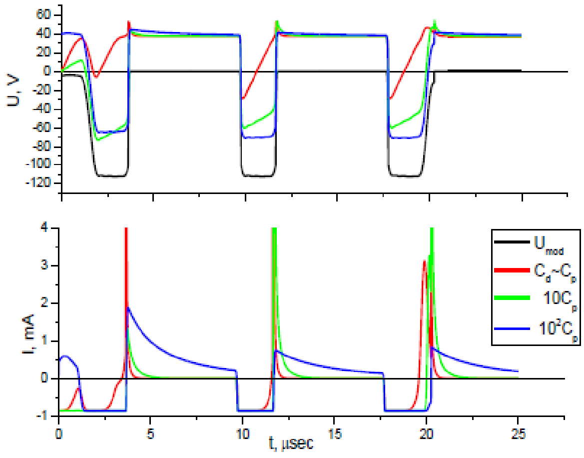

Figure 4. Dependences of the accelerating potential and current on the substrate on time in the etching mode (Ucol = 60 V) for various capacities of the Cd film when a unipolar oscillating potential with an amplitude of 110 V is applied to the substrate holder.

The method described above for compensating the charge of the surface in contact with plasma, limited in application: in the case of large thickness of The substrate's low dielectric constant voltage loss across its capacitance proves to be too large. Modulation plasma potential is an alternate method for controlling the floating surface potential of an electrically isolated object. [10].

To approximate the current-voltage characteristic in modes with modulation

$$I(U_p) = -I_i(U_p) + I_e \exp\left(\frac{eU_p}{kT}\right)$$

$$I_i(U_p) = A + B \cdot U_p$$

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To increase the accuracy of calculations in this mode, accounting was added dependence of the width of the space charge layer on the voltage on it:

$$d = \frac{2}{3}\left(\frac{2V}{kT_e}\right)^{3/4} r_D$$

3

In Figure Figure 5 shows a comparison of the experimental results from [9] with the calculation results. It can be seen that the calculation results correspond to the experimental results.

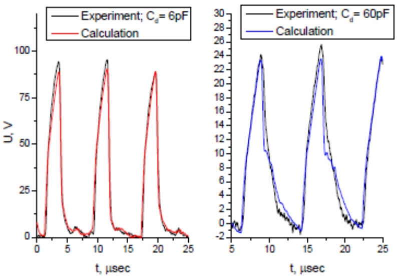

Figure. 5. Experimental time dependences of the floating potential and current on the substrate when the plasma potential is modulated by a unipolar oscillating potential with an amplitude of 110 V in comparison with the calculation results. Discharge parameters – p=0.4 mTorr (Ar), H=20 Oe.

From a comparison of the calculation results for various methods it follows: from the point vision of potentials on the non-conducting structure being processed using the pulse method Plasma potential modulation is identical to the method of pulsed substrate modulation.

The advantage of the plasma potential modulation method in the case whenit is necessary to optimize the etching process in terms of speed, it is possible simultaneous control of both ion energy and ion current on treated surface. In the case when it is necessary to influence for only one parameter, for example, ion energy, the most suitable is substrate potential modulation method.

Thus, the presented experimental results, together with the computer analysis data presented above, make it possible to confidently predict the energy characteristics of the ion flow acting on the surface being treated in characteristic etching and deposition modes, and to optimize the modes according to these characteristics.

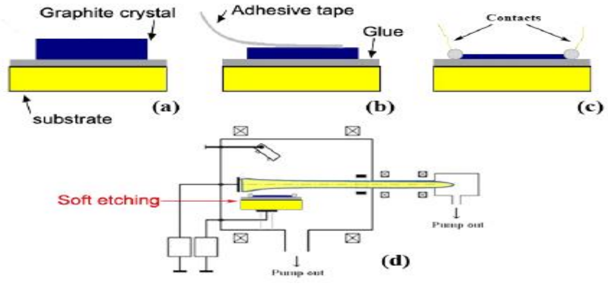

Figure 6. Method for producing thin graphite single crystals.

Thin graphite single crystals with a thickness of ten to hundreds of atomic layers with lateral dimensions from 0.49 mm, serving as blanks for subsequent etching, are obtained by repeated thinning using adhesive tape of natural graphite single crystals glued to a substrate of polycor, sapphire or atomically smooth glass (Figure 6. a-b). Epoxy adhesives White Stycast, PoxiPol, Araldit or NOA61 photopolymer were used as glue. Next, indium electrodes or electrodes made of PP-17 silver paste were pressed to the sample boundaries (c), and then the resulting single crystal was thinned using low-energy plasma etching in a plasma-chemical reactor based on a beam-plasma discharge (d). Etching is carried out in an argon atmosphere.
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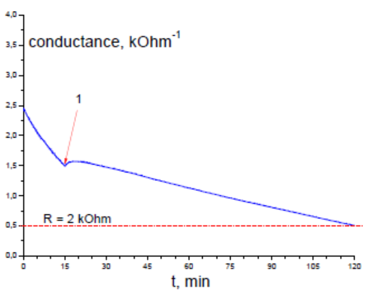
Film thickness is monitored on-line based on their in situ conductivity.

chamber of the plasma reactor, which allows you to accurately select the moment of termination process (the resistance of a square of a homogeneous monatomic layer is estimated as~1.5 – 2 kOhm), as well as control the uniformity of the etching process over time The demonstrates a method for obtaining monoatomic graphite layers of high quality from the point of view of nanoelectronics problems using etching in a plasma-chemical reactor based on a beam-plasma discharge, and also characterizes the resulting samples.



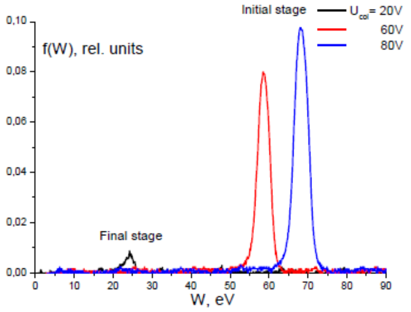


Figure7. Change in conductivity of a graphite sample during etching. Figure8. FREM in etching modes of graphite single crystals depending on the collector potential. 0 10 20 30 40 50 60 70 80 90 0.00 0.02 0.04 0.06 0.08 0.10 f(W), rel. units W, eV Ucol= 20V 60V 80V Initial stage Final stage

III. CONCLUSION

The use of pulsed voltage on the substrate is also effective for controlling the energy characteristics of ion flows in a plasma-chemical reactor based on a beam-plasma discharge in the DLC sputtering The method of modulating the potential difference between the plasma and the substrate is effective when the capacitance of the structure being processed is more than an order of magnitude greater than the capacitance of the plasma layer.

The plasma potential modulation method allows one to largely overcome the problem of surface charge and facilitate the solution of the problem of creating an ion flow of a given energy both for etching semiconductors and dielectrics, and for deposition of semiconductor and dielectric films on the surface of semiconductor structures. It has been shown that the plasma potential modulation method can be effective in mass processing of non-conducting structures, and is also used in conjunction with other types of influence on the structure being processed. The presented experimental results, together with computer analysis data, make it possible to confidently predict the energy characteristics of the ion flow acting on the surface being processed in characteristic etching and sputtering modes, and to optimize the modes according to these characteristics.

The possibility of obtaining single-crystalline nano-sized layers of graphite of high quality from the point of view of nanoelectronics tasks by a combination of methods of mechanical exfoliation and low-energy etching of thin graphite single crystals in a plasma-chemical reactor based on a beam-plasma discharge has been shown. Unparalleled samples of single-layer graphene and bigraphene with characteristic sizes exceeding 105×105 μm2 and FLG samples (graphene from several layers) with characteristic sizes exceeding 500×500 μm2 were obtained. Using the methods of X-ray diffractometry, optical microscopy, atomic force microscopy, Raman spectroscopy, and magneto transport measurements, the high quality of the films obtained from the point of view of nanoelectronics problems and their uniformity in area were shown.

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