Influence of the FIB parameters on the etching of planar nanosized multigraphene/SiC field emitters

I L Jityaev, A M Svetlichnyi, V I Avilov, I N Kots, A S Kolomiytsev, O A Ageev
Southern Federal University, Institute of Nanotechnologies, Electronics and Equipment Engineering, Taganrog, 347922, Russia
jityaev.igor@gmail.com

Abstract. The possibility of fabrication of planar field emission nanostructures based on multigraphene films on semiinsulating silicon carbide using focused ion beam (FIB) technology is considered in this paper. The effect of FIB-parameters on the etching of the structure was determined using a scanning probe microscope. Conductive probes were used for local studies of electrical properties of nanoscale structures. It was found that etching of planar field emission nanostructures at a current of 1 pA did not lead to a change in the depth of the treated area. An increase in current up to 10 pA was sufficient to initiate the etching process of a multigraphene film on the surface of silicon carbide.

1. Introduction
Electron sources based on field emission from nanostructures are of great interest for the development of modern devices. Devices based on field emission unlike those based on photo- and thermionic emission do not require additional heat loss, they allow obtaining a high emission current density, are stable in a wide range of temperatures, have high speed and low noise level. Studies conducted in the 1970s have shown carbon materials to be promising for field emission application [1].

In this paper, we consider multigraphene films obtained by the thermal decomposition of silicon carbide in vacuum [2]. At present, this method is considered as a high-tech, as well as the most promising to produce graphene and multigraphene films with high homogeneity and high quality for electronic applications. In this case, the multigraphene films are characterized by high adhesion to silicon carbide, low work function, and the possibility of depositing on the entire surface of the substrate. The substrate was semiinsulating silicon carbide 6H-SiC. The choice of silicon carbide is due to the high thermal conductivity, the ability to operate the device at high current densities and high-power levels. In addition, an important and determining factor in the choice of silicon carbide as a substrate was its resistance to external corrosive influences. Silicon carbide is considered as one of the most promising materials for high-temperature radiation-resistant electronics. Emission structures based on graphene on silicon carbide are characterized by low threshold voltages [3-6].

Multigraphene films are an excellent material for planar emission nanostructures due to its two-dimensional structure. The nanometer film thickness of the multigraphene contributes to the high form-factor [7]. The planar design of the field emission structure ensures the emission of electrons from the end of the multigraphene film, when a potential difference is applied. Planar field emission structures are considered promising due to the high amplification of the electric field at the sharp ends [8-10].
order to reduce the threshold voltages, it is necessary to reduce the interelectrode gap. Providing a nanosized gap for emission cells with vertical-type emitters is technologically difficult. The use of focused ion beam (FIB) allows nanoscale profiling of substrates. Moreover, the advantages of this method are the ability to process a wide range of materials, the high speed of forming a topological pattern without the use of resists and masks, as well as locality and selectivity of the etching process, achieved by varying the process parameters in a wide range.

Thus, the work is devoted to the investigation of the possibility of manufacturing planar field emission nanostructures based on multigraphene films on silicon carbide using FIB. The etching regimes with the minimum ion beam current (Ii), the minimum time of ion beam exposure at the point (dt), and the number of passes (ne) were determined based on the scanning probe microscopy (SPM).

2. Experiment and results

The electron-ion complex Nova NanoLab 600 (FEI Company, The Netherlands) with electron-beam and ionic columns and a gallium liquid metal ion source (resolution 7 nm) was used to realize the etching of experimental samples by an ion beam and subsequent analysis by the raster electron microscopy method [11]. The possibility of FIB-applying for the etching of multigraphene/SiC experimental samples and subsequent fabricating of planar emission nanostructures was evaluated using a Solver P47 Pro SPM. This is a universal instrument for complex studies with high resolution, both in air and in a controlled gas environment, at temperatures up to 150°C [12-14]. It can be used to control the technological modes of obtaining various coatings, films, and other materials with a thickness of up to 20 mm and a diameter of up to 100 mm. An important feature of SPM Solver P47 Pro is the high measurement accuracy and various measurement techniques.

The experimental samples were oriented in such a way that the flow of accelerated ions hit the surface in the normal direction. Minimum currents (1-10 pA) were used to obtain the minimum possible interelectrode gap for this technology. An increase in the ion current promotes an increase in lateral over-etching. The scanning electron microscope (SEM) image of the experimental sample with 300x300 nm² sections treated by FIB at the ion beam current of 1 pA is shown in Figure 1. The upper row was treated at a fixed beam impact time at the point dt = 1 μs, the number of passes was ne = 100, 200 ... 900. The lower row was treated at a fixed number of passes ne = 100; the time of the beam impact at the point dt = 1.2 ... 9 μs was varied. The resulting SEM-image allows us to qualitatively evaluate the effect of the focused beam on the surface of the experimental sample.

Then, the SPM was used for a more detailed study of the FIB-treated sections. The cross sections of the FIB-treated areas were plotted in Figure 2. Based on the obtained cross sections, it was found that etching did not occur at an ion beam current of 1 pA. The increase in impact time of the beam at the point and the number of passes did not contribute to the etching process of the experimental sample. There was a swelling of the surface, which was most likely due to the introduction of gallium ions into the near-surface zone of the experimental sample.

![Figure 1. SEM-image of the experimental sample with FIB-treated areas.](image-url)
Figure 2. The cross-section of the experimental sample after FIB-treatment at a current of 1 pA: (a) \( dt = 1, 2 \ldots 9 \, \mu s, \, ne = 100 \); (b) \( dt = 1 \, \mu s, \, ne = 100, 200 \ldots 900 \).

Figure 3. SEM-image of planar field emission cell based on multigraphene on SiC.

Figure 4. (a) SPM-image of a planar-type field emission cell, (b) current-voltage characteristics of field emission cell.
The subsequent increase of the ion beams current to 10 pA made it possible to initialize the etching process of the experimental sample. An electronic model in the form of a raster template for FIB etching a planar emission cell with a point emitter was developed on the basis of previous theoretical studies of the design parameters of a planar field emission cell. The electronic model of the emission cell took into account the optimal values of the interelectrode distance, the rounding-off radius of the emitter top, the height and the angle of inclination of the lateral surface relative to the central axis. Thus, a planar field emission structure with a tip emitter and an interelectrode distance determined by the diameter of the ion beam was fabricated. The SEM-image of the fabricated emission cell is shown in Figure 3.

It was found that emission cells with an interelectrode distance of ~ 15-30 nm could be produced reproducibly on the equipment used. The rounding-off radius of the emitter top was 30 nm. The FIB application made it possible to fabricate a cathode and an anode in a single production step. The optimal etching time was 2 min 30 sec. Partial etching of the emitter tip was observed with a longer etching time.

Conductive probes were used to study the fabricated nanoscale structures. The SPM-image of a planar-type field emission cell with a tip-shaped emitter is shown in Figure 4a. The etching depth was ~ 25 nm. The pressure contact was located on the surface of the multigraphene film in the anode region. The conductive probe was brought into contact with the investigated region to measure the local electrical characteristics. It was found that etched areas did not conduct electric current. This confirms that the local sections of the multigraphene film treated with FIB are completely etched to seminsulating silicon carbide. The contact of the conducting probe with the unetched area of the field emission cathode contributed to the appearance of an emission current (Fig. 4b). The emission current was 16.3 V at a potential difference of 10 V. The threshold voltage was ~ 0.2 V.

3. Conclusion

It is shown that the fabrication of the field emission structures with a nanoscale interelectrode distance is possible using the FIB technology. The recommended ion beams current for etching of multigraphene film on silicon carbide is 10 pA. This ensures minimum dimensions of the interelectrode gap and minimizes the processes of over-etching the treated area. The use of SPM made it possible to quantitatively evaluate the FIB effect on the surface of the experimental sample, to check the geometric parameters of the fabricated nanoscale planar field emission structures and local measurements of electrical parameters. The planar design of the tip emitter made it possible to lower the threshold voltages in comparison with emitters of a vertical type based on a multigraphene on silicon carbide [13]. The obtained results make it possible to optimize the technological operations for the fabrication of field emission nanostructures, to reduce the costs of their production, and to improve their emission characteristics. Nanoscale planar field emission structures based on multigraphene films on silicon carbide can form the basis of the element base of energy-efficient nanoelectronics.

Acknowledgments

The equipment of the Research and Educational Centre "Nanotechnologies" of Southern Federal University was used for this study. This work was funded by Internal grant of the Southern Federal University No. VnGr-07/2017-26.

References

[1] Baker F S, Osborn A R and Williams J 1974 J. Phys. D: Appl. Phys. 7 2105-15
[2] Lebedev A A, Kotousova I S, Lavrent’ev A A, Lebedev S P, Makarenko I V, Petrov V N and Titkov A N 2009 Phys. Solid State 51 829-32
[3] Konakova R V, Okhrimenko O B, Svetlichnyi A M, Ageev O A, Volkov E Yu, Kolomiytsev A S, Jityaev I L and Spiridonov O B 2015 Semiconductors 49 1242-5
[4] Konakova R V, Okhrimenko O B, Kolomys A F, Strel’chuk V V, Svetlichnyi A M, Ageev O A, Volkov E Yu, Kolomiitsev A S, Zhityaev I L and Spiridonov O B 2016 J. Superhard Mater. 38 235-40
[5] Eda G, Emrah Unalan H, Rupesinghe N, Amaratunga G A J and Chhowalla M 2008 *Appl. Phys. Lett.* **93** 233502

[6] Lee S W, Lee S S and Yang E-H 2009 *Nanoscale Res. Lett.* **4** 1218-21

[7] Jityaev I L and Svetlichnyi A M 2018 *J. Phys.: Conf. Ser.* **1038** 012055

[8] Gavrilov S A, Il’ichev É A, Poltoratskii É A, Rychkov G S, Dvorkin V V, Dzbanovsky N N and Suetin N V 2004 *Tech. Phys. Lett.* **30** 609-11

[9] Rouhi J, Mahmud S, Hutagalung S D and Naderi N 2012 *Electron. Lett.* **48** 712-4

[10] Aban’shin N P, Avetisyan Yu A, Akchurin G G, Loginov A P, Morev S P, Mosiyash D S and Yakunin A N 2016 *Tech. Phys. Lett.* **42** 509-12

[11] Orloff J, Swanson L W and Utlaut M 1996 *J. Vac. Sci. Tech. B* **14** 3759-63

[12] Avilov V I, Ageev O A, Smirnov V A, Solodovnik M S and Tsukanova O G 2015 *Nanotechnologies in Russia* **10** 214-9

[13] Jityaev I L, Svetlichnyi A M, Kolomiytsev A S, Volkov Y E, Polyakova V V and Ageev O A 2017 *IOP Conf. Ser.: Mat. Sci. Eng.* **256** 012021

[14] Avilov V I, Polupanov N V, Tominov R V, Smirnov V A and Ageev O A 2017 *IOP Conf. Ser.: Mat. Sci. Eng.* **256** 012001