

Conference materials

UDC 621.38

DOI: <https://doi.org/10.18721/JPM.161.233>

Fabrication of nanoscale structures by FIB-induced deposition of materials and study of their electrical properties

A.V. Kotosonova[✉], A.S. Kolomiytsev, O.I. Soboleva

Southern Federal University, Taganrog, Russia

[✉] alena.kotosonova@gmail.com

Abstract. Methods of local formation of nanoscale structures based on the application of focused ion beam open up new possibilities in terms of providing the necessary geometric parameters and ensuring the reproducibility of micro- and nanostructures, which contributes to the development of devices with previously unattainable characteristics. This paper presents the technological modes of formation of nanostructures with a height of 1 μm and diameters from 100 to 500 nm by the FIB method. The structures were formed by ion-induced deposition of carbon and tungsten, as well as electron-induced deposition of tungsten. A method for measuring the electrical parameters of high-aspect ratio structures based on an atomic force microscope (AFM) was proposed. The current value of 25 nA was obtained at 50 V. Threshold voltages for various nanostructures ranged from 7 to 32 V. The stability of structures to the electric field at voltages up to 50 V was investigated. _

Keywords: nanotechnology, focused ion beam, beam-induced deposition, nanopatterning

Funding: The study was carried out with the financial support of the Russian Science Foundation Grant 22-29-01239 in Southern Federal University.

Citation: Kotosonova A.V., Kolomiytsev A.S., Soboleva O.I., Fabrication of nanoscale structures by FIB-induced deposition of materials and study of their electrical properties, St. Petersburg State Polytechnical University Journal. Physics and Mathematics. 16 (1.2) 2023 218–223. DOI: <https://doi.org/10.18721/JPM.161.233>

This is an open access article under the CC BY-NC 4.0 license (<https://creativecommons.org/licenses/by-nc/4.0/>)

Материалы конференции

УДК 621.38

DOI: <https://doi.org/10.18721/JPM.161.233>

Изготовление наноразмерных структур методом ионно-стимулированного осаждения материалов и изучение их электрических свойств

А.В. Котосонова[✉], А.С. Коломийцев, О.И. Соболева

Южный федеральный университет, Таганрог, Россия

[✉] alena.kotosonova@gmail.com

Аннотация. Методы локального формирования наноразмерных структур, основанные на применении фокусированного ионного пучка (ФИП), открывают новые возможности с точки зрения обеспечения необходимых геометрических параметров и обеспечения воспроизводимости микро- и наноструктур, что способствует разработке устройств с недостижимыми ранее характеристиками. В данной работе представлены технологические режимы формирования наноструктур путем ионно-стимулированного осаждения углерода и вольфрама, а также электронно-стимулированного осаждения вольфрама. Предложен метод измерения электрических параметров структур с высоким соотношением сторон, основанный на атомно-силовой микроскопии (АСМ). При приложении смещения 50 В достигнуто значение тока 25 нА. Для различных наноструктур пороговые значения напряжения варьировались от 7 до 32 В. Исследована устойчивость структур к воздействию электрического поля при напряжениях до 50 В.



Ключевые слова: нанотехнологии, фокусированный ионный пучок, ионно-стимулированное осаждение, нанопрофилирование.

Финансирование: Исследование выполнено за счет гранта Российского научного фонда № 22-29-01239 в Южном федеральном университете

Ссылка при цитировании: Котосонова А.В., Коломийцев А.С., Соболева О.И. Изготовление наноразмерных структур методом ионно-стимулированного осаждения материалов и изучение их электрических свойств // Научно-технические ведомости СПбГПУ. Физико-математические науки. 2023. Т. 1.2 № .16. С. 218–223. DOI: <https://doi.org/10.18721/JPM.161.233>

Статья открытого доступа, распространяемая по лицензии CC BY-NC 4.0 (<https://creativecommons.org/licenses/by-nc/4.0/>)

Introduction

The development of elements of nanoelectronics requires new nanostructuring methods that ensure the achievement of the specified geometric parameters and electrical characteristics of the created devices. One of the promising methods for creating nanoscale structures on the surface of solids is the technology of local processing of materials with a focused ion beam (FIB). The FIB method allows one to perform operations of local milling and local ion-induced deposition of materials based on various gases. Despite the insufficiently high productivity of the process, the FIB method is effectively used to solve specialized problems in the creation of electronics and micromechanics elements, e.g. for the formation of the tips of atomic force microscopy (AFM) probes [1] and scanning near-field microscopy probes [2], for the formation of micromechanics structures and nanoscale conductors [3]. In a number of studies, the FIB method was used for the manufacturing of field emission cathodes with nanoscale interelectrode gaps [4–6]. An important advantage of the FIB method is its compatibility with the technological processes of traditional microelectronics, which makes it possible to significantly expand the scope of its application. When forming structures for micro- and nanoelectronics, it is often critically important not only to ensure the specified sizes of structures, but also provide their electrical parameters that determine the characteristics of the created devices.

Experimental details

In this paper, experimental studies were carried out on the formation of nanoscale structures with specified parameters for elements of nanoelectronics by the FIB method.

The objective of this study was to evaluate the effect of the technological parameters of the ion beam on the parameters of structures formed by ion-induced deposition of carbon (i-C) and tungsten (i-W). To increase the objectivity when comparing the characteristics of nanostructures made by the FIB method from various materials, it is necessary to minimize the influence of dimensional factors. In accordance with this, one of the tasks of this experiment was to form structures of identical height and width. The process parameters were selected in order to form 5 structures with a height of 1 μm and diameters of 100, 200, 300, 400 and 500 nm. With ion-induced deposition from the gas phase, the composition of the deposited material will include not only the initial components of the gas, but also gallium atoms from the ion beam [7]. To assess the effect of implanted gallium ions on the electrical properties and durability of the cathode, an additional electron-induced (e-W) deposition of tungsten was carried out, thus excluding the presence of gallium atoms in the deposited material.

The test structures were formed using an electron microscope with the Nova NanoLab 600 FIB system equipped with a gallium ion source. To implement ion-induced deposition of materials, carrier gases of the deposited material, WCO_6 and C_6H_{14} , were locally supplied into the microscope chamber. The formed structures were studied using Ntegra AFM. After the measurements, the structures were examined by scanning electron microscopy (SEM).

Ion-induced deposition of tungsten and carbon. During the preliminary stage of experimental studies, characteristic irregularities were found on the surface of the i-W structures. Similar irregularities, but much less pronounced, were also present on the i-C structures. It was found that reducing the exposure time at the point and increasing the value of the “Refresh time” parameter makes it possible to reduce this effect. However, just selecting the optimal exposure time at the point and ‘Refresh time’ is not enough to obtain a smooth surface of the i-W structure. The appearance of this effect may also be conditioned by other factors, for example, the defocusing of the beam at different heights, the influence of residual vacuum level or the scanning trajectory of the beam during the deposition process.

The control i-W and i-C structures (Fig. 1) were formed by ion-induced deposition of tungsten and carbon at the beam energy of 30 keV and a beam current of 10 pA on a Ti/SiO₂/Si structure with a titanium film thickness of 50 nm. The formation of structures with a minimum diameter was carried out by deposition at one point with a single exposure time of 5 μs, the number of passes and ‘Refresh time’ were 1.4×10⁶ and 15 μs for the i-C structure and 1.8×10⁶ and 150 μs for the i-W. The final diameters were 156 nm for the i-C and 115 nm for the i-W structure.

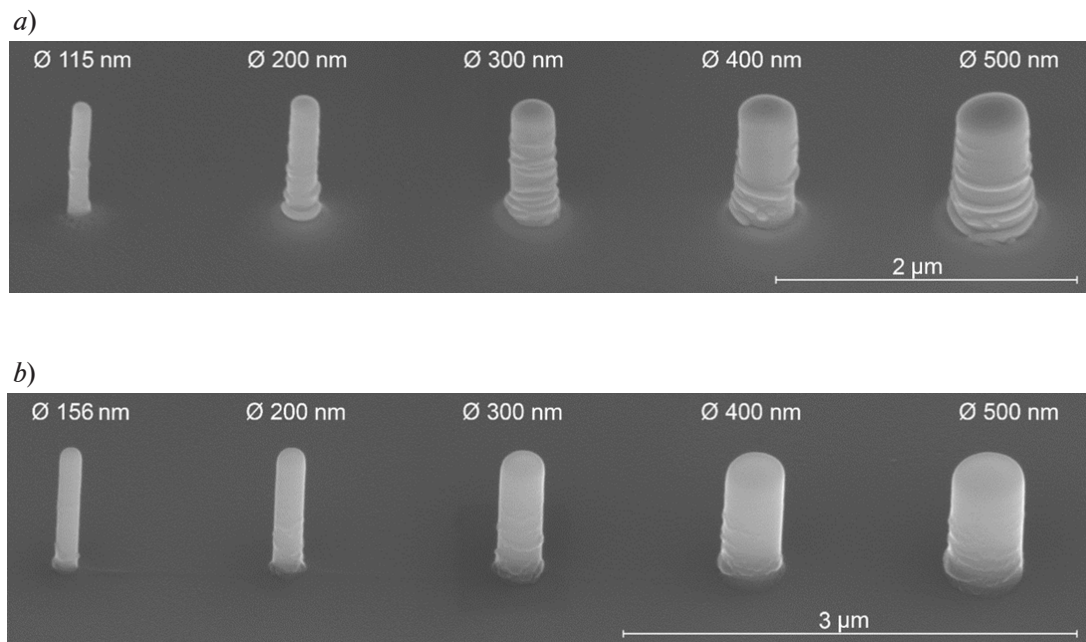


Fig. 1. SEM images of high-aspect ratio nanoscale (a) i-W and (b) i-C structures formed by the FIB-induced deposition. The figures indicate the value of the structures diameter

For diameter values in the range from 200 to 500 nm, the exposure time at the point was 1 μs. When tungsten was deposited by an ion beam, the ‘Refresh time’ value for each structure increased proportionally with increasing diameter (600 μs for 200 nm, 900 μs for 300, etc.). The number of points that the beam passes through in one pass depends on the diameter value, which means that the total scanning time increases with increasing diameter, hence it takes more time to replenish the number of gas molecules in the area of interaction with the ion beam. During the carbon deposition, the ‘Refresh time’ for each structure was equal to 15 μs; this parameter value was optimal for a structure with a diameter of 500 nm, and it was impractical to reduce it for structures with a smaller diameter due to the fact that the total deposition time would change by only a few seconds.

Electron-induced deposition of tungsten. The electron-induced deposition of structures was complicated by the presence of electron beam drift, which is manifested in the appearance of a deviation of the structure from the normal with an increase in its height. To eliminate this effect, it is necessary to reduce the total deposition time, which was done by increasing the beam current. e-W structures with a diameter of 200 to 500 nm were formed at an electron beam current of 2.1 nA, the accelerating voltage was 10 keV, the beam exposure time at a point was 1 μs. The



electron beam has a higher resolution compared to the ion beam (due to the nature of electrons, whose diameter is orders of magnitude smaller than those of ions), however, the beam drift present during the experiments did not allow to achieve the best values of the diameter of the structures, and at a current of 2.1 nA the minimum obtained diameter was 200 nm. Based on this, in order to form a structure with a diameter of 100 nm, the current was reduced to 0.54 nA. The resulting array of e-W structures is shown in Fig. 2.

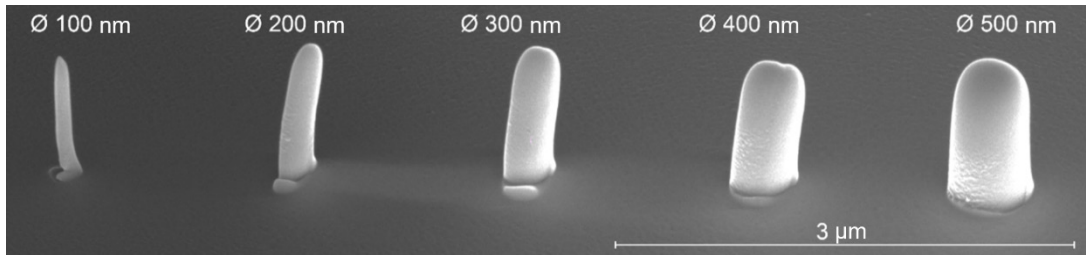


Fig. 2. SEM image of high-aspect ratio nanoscale e-W structures formed by electron-induced deposition. The figures indicate the value of the structures diameter

Method of measuring electrical parameters. According to the research methodology, first, a $10 \times 10 \mu\text{m}$ area was scanned in a semi-contact mode, in which the structures under study were located. The probe was brought into contact with the top of the structure, after which it was withdrawn to a distance of 30 nm. The voltage between the probe and the substrate was manually changed from 0 to 50 V with a small step. At the same time, two characteristics of the process were measured – the dependence of voltage on time and the dependence of current on time. If there was no characteristic increase in current with increasing voltage, the distance was reduced to 10 nm. After the measurements, the obtained dependences were digitized and numerically reconstructed into a CVC.

Results and Discussion

Fig. 3,*a* shows an example of the obtained CVC for the i-W structure with a diameter of 400 nm, measured at a distance probe-surface of 10 nm. The threshold voltage (the voltage at which the current began to exceed the noise level of tens of pA) was 17 V. Fig. 3,*b* shows the time dependence of the current at a constant voltage of 50 V for the same structure.

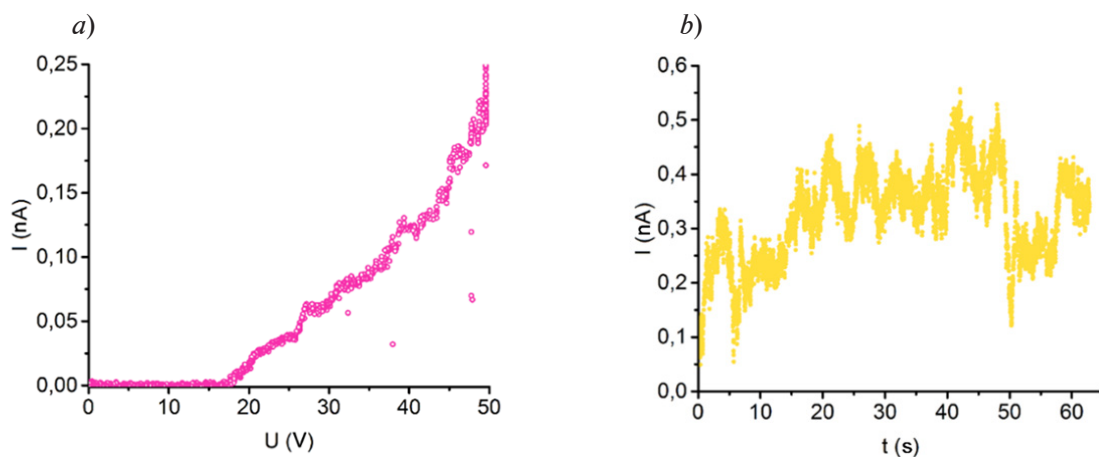


Fig. 3. Results of measuring the electrical parameters of the 400 nm diameter i-W structure at a distance of 10 nm: CVC (*a*) and time dependence (*b*) of the current

The analysis of the obtained dependences showed that, for an i-W structure with a diameter of 500 nm, with a decrease in the distance to the probe from 30 to 10 nm, the threshold voltage decreased from 12 to 8 V; the average value of the current at 50 V increased from 2.5 to 5.1 nA. A comparison of the characteristics of i-W and i-C structures with a diameter of 300 nm showed that the current value on carbon structures was an order of magnitude higher than on tungsten structures, with voltage thresholds equal to 20 and 32 V, respectively. When comparing the parameters of i-W and e-W structures with a diameter of 500 nm, it was found that higher current values were achieved on the e-W structure, while the threshold voltage differed slightly (7 V for e-W and 8 V for i-W). Fluctuation of current values during measurements was observed for all structures.

After the measurements the studied structures were re-examined by SEM. The obtained images are shown in Fig. 4. Image analysis shows that the surfaces of i-C structures with diameters of 100 and 200 nm changed significantly during measurements. The height of the structure with a diameter of 100 nm decreased from 1 μm to 690 nm (Fig. 4,*a*); that of the structure with a diameter of 200 nm decreased from 1 μm to 950 nm (Fig. 4,*b*). Small protrusions appeared on the tops of these and the other structures. The largest number of protrusions is observed on tungsten structures deposited by an ion beam (Fig. 4, *d*, *e*, *f*); for structures formed by electron-induced deposition, the number of protrusions is minimal (Fig. 4, *g*, *h*).

The protrusions that appeared during the measurements could provoke the instability of the current observed during the measurements: some protrusions appeared and others disappeared, which made different contributions to the current.

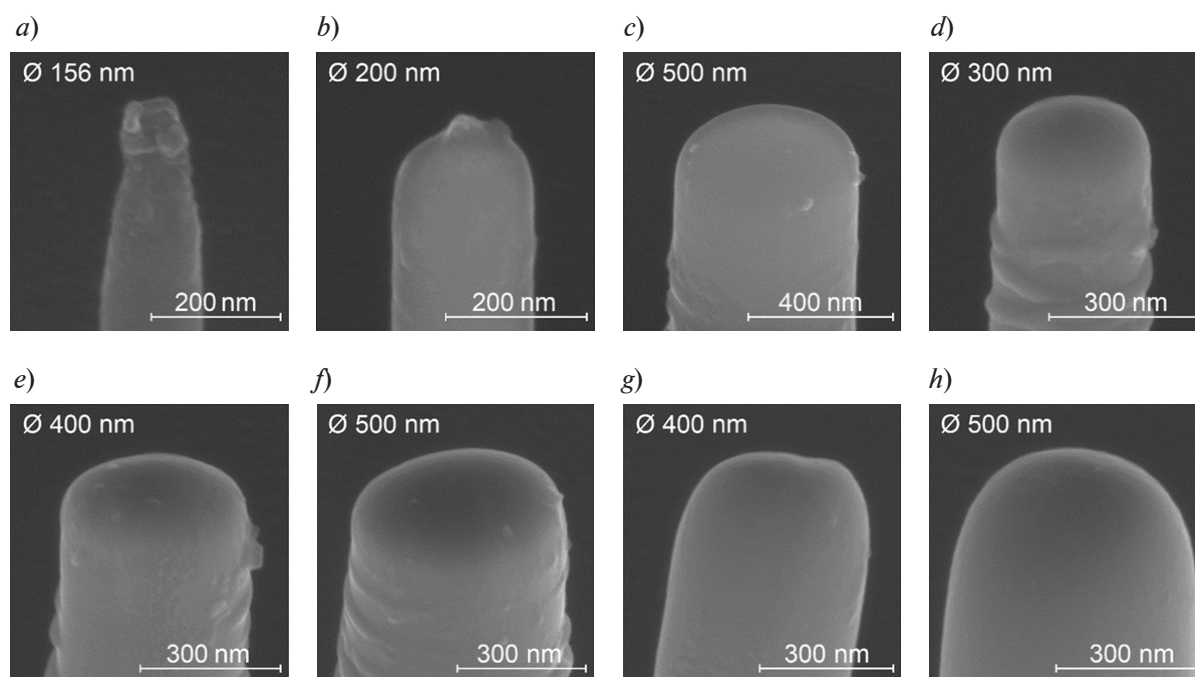


Fig. 4. SEM images of surfaces of (*a*, *b*, *c*) i-C, (*d*, *e*, *f*) i-W and (*g*, *h*) e-W structures after measurements of electrical parameters. The diameters of the structures are given in the panels

Conclusion

Thus, high-aspect ratio nanoscale structures were formed using electron- and ion-induced deposition of tungsten and carbon. The modes of the process were determined, allowing the formation of structures with a diameter of 100–500 nm and a height of up to 1 μm , which can be used in the formation of various elements of nanoelectronics. The study and comparison of electrical parameters and stability under electric field for structures of various sizes and consisting of various materials was carried out. A strong instability of the current observed during probe measurements was detected, which could be caused by the appearance and disappearance of small protrusions on the surface of structures with a bias voltage of up to 50 V.



We proposed a new method for measuring the electrical parameters using an AFM probe, which can be used to study the electrical properties of high-aspect ratio structures, in particular, field emission properties. When measuring the CVC of the formed structures in the range up to 50 V, the values of the measured currents reached 25 nA, the threshold voltages for various structures ranged from 7 to 32 V.

In the course of the research, it was possible to optimize the shape of nanoscale carbon and tungsten structures by selecting the values of the beam exposure time at the point and the 'Refresh time' parameter, which made it possible to increase the smoothness and uniformity of the structures.

It was shown that the application of the method of local ion-induced deposition of carbon and tungsten from the gas phase allowed to form structures with specified geometric and electrical parameters with high reproducibility for solving problems of creating new components of nano-electronics and nanosystem technology.

REFERENCES

1. **Panchenko I.V., Shandyba N.A., Ballouk A., et al.**, Investigation of the influence of the geometric parameters of AFM cantilevers on the resonant frequency of their oscillations, *J. Phys. Conf. Ser. IOP Publishing*. 1695 (2020) 12182.
2. **Kolomiytsev A.S., Panchenko I.V., Shandyba N. A., et al.**, Fabrication of probes for scanning near-field optical microscopy using focused ion beam, *J. Phys. Conf. Ser.: Materials Science and Engineering. IOP Publishing*. 443 (1) (2018) 012015.
3. **Aloysius R.P., Husale S., Kumar A., et al.**, Superconducting properties of tungsten nanowires fabricated using focused ion beam technique, *Nanotechnology. IOP Publishing*. 30 (2019) 405001.
4. **Liu M., Li T., Wang Y.**, Low voltage field emission of Cu nanowire with nanogap in air, *IEEE 12th International Conference on Nano/Micro Engineered and Molecular Systems. Institute of Electrical and Electronics Engineers Inc.* (2017) 333–336.
5. **Xu J., Wang Q., Tao Z., et al.**, High-Quality and Stable Electron Emission Device with Sub-30-nm Aligned Nanogap Arrays, *IEEE Trans. Electron Devices. Institute of Electrical and Electronics Engineers Inc.* 64 (5) (2017) 2364-2368.
6. **Liu M., Fu W., Li T., et al.**, Excellent field emission properties of vanadium oxide nanoemitters in air, *IEEE 17th International Conference on Nanotechnology. Institute of Electrical and Electronics Engineers Inc.* (2017) 351–354.
7. **Gnaser H., Kallmayer C., Oechsner H.**, Focused-ion-beam implantation of Ga in elemental and compound semiconductors, *J. Vac. Sci. Technol. B Microelectron. Nanom. Struct. American Inst of Physics*. 13 (1) (1995) 19–26.

THE AUTHORS

KOTOSONOVA Alena V.
alena.kotosonova@gmail.com
ORCID: 0000-0002-4382-8698

SOBOLEVA Olga I.
osotova@sfedu.ru
ORCID: 0000-0002-0487-7542

KOLOMIYTSEV Alexey S.
askolomiytsev@sfedu.ru
ORCID: 0000-0001-7483-0240

Received 26.10.2022. Approved after reviewing 10.11.2022. Accepted 11.11.2022.