Plasma surface treatment of local modify silicon plates

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Abstract. This paper presents a study of the use of silicon Si for element base manufacture of micro- and nanoelectronics by using combined methods of focused ion beams and atomic layer plasma chemical etching. This technology makes it possible to modify surface of Si substrates in the required topology and geometry, followed by removal of atoms to obtain nanoscale elements. The influence of parameters of method of focused ion beams and plasma chemical etching on parameters of the formed structures is analyzed. So, for example, for formation of structures with maximum roughness, it is necessary to increase values of parameters responsible for reactive ion etching, these are such parameters as: the power of capacitive plasma source, the mixing voltage, and the flow rate of an inert gas (argon).

1. Introduction

Silicon nanotechnology is the foundation of the entire world micro- and nanoelectronics. Silicon (Si) in its various forms is used as a base-substrate for further formation of working surfaces of nanoscale devices and an element base based on it. Silicon nanostructures have become so common due to the distinctive physicochemical parameters and the relative ease of obtaining, forming and processing the material, as well as an ability to apply various types of materials and substances on its surface. However, the main principle of nanotechnology is miniaturization of element base in order to increase efficiency, reduce energy consumption and structural dimensions, which at this stage of development on silicon is becoming an increasingly difficult and expensive task, and soon it will become completely impossible, because of it is impossible to create a workable on the basis of silicon transistor with a gate length less than a few nanometers.

After transition to the 28 nm process technology in the industry, for the first time, a situation arose when the next generation transistor was more expensive, not cheaper, than the previous generation transistor. To date, there are only three companies that continue to miniaturize below the 28 nm norm - Intel, TSMC and Samsung. The entire world community is busy solving this problem, actively looking for new materials and modern manufacturing technologies.

This paper presents a study of the use silicon Si for element base manufacture of micro- and nanoelectronics by using combined methods of focused ion beams and atomic layer plasma chemical etching. This technology makes it possible to modify the surface of substrates in the required topology and geometry, followed by removal of atoms to obtain nanoscale elements. The essence of the technique is that embedded ions are used as positive photoresist masks for plasma treatment in the
sense that this area, due to the specificity of interatomic interaction, will interfere with chemical processes of material removal.

2. Experiment
On the surface of silicon substrates, local modification of 5x5 μm regions was carried out with a focused Ga⁺ ion beam at an accelerating voltage $U_{acc} = 30$ keV and an ion beam current $I_{ib} = 10$ pA with a different number of beam passes (10 - 100) and different doses (2.5 - 25 pC/μm²), respectively. For this, samples were placed in the vacuum chamber of the FIB Nova NanoLab 600 module and oriented in such a way that the flow of accelerated ions hit the substrate in the normal direction. The working vacuum during the exposure to the beams was maintained at a level of $1 \div 2 \times 10^{-4}$ Pa.

Plasma treatment of samples with modified regions was carried out in fluorine-containing plasma with the following parameters: fluorine-containing gas flow $N_{SF6} = 15$ cm$^3$/min, argon flow $N_{Ar} = 100$ cm$^3$/min, pressure in the reactor $P = 2$ Pa, power of the inductively coupled plasma source $W_{ICP} = 200$ W, the power of the capacitive plasma source $W_{RIE} = 35$ W, the bias voltage $U_{bias} = 24$ V, the processing time is 30 - 120 seconds.

An analysis of the obtained results shows that, depending on the combination of modes of focused ion beams and plasma chemical etching, two types of action can be realized: masking of substrate surface and activation of substrate etching during etching.

3. Results and Discussion
Based on the results of experimental studies, the dependences of the height of obtained structures on number of passes of ion beam and different etching times in fluorine-containing plasma have been obtained.

![Figure 1](image-url) (a, b). SEM - image of the structure of silicon surface after local modification by focused ion beam method at 100 passes of ion beam: a) side face, b) top view

The analysis shows that with the specified combination of modes, the locally modified region is characterized by a lower etching rate ($V_{moSi} = 0.019$ nm/sec) than the unmodified region of the substrate surface ($V_{Si} = 2.21$ nm/sec). The masking effect can be explained by the formation of a layer saturated with embedded Ga⁺ ions in the near-surface, locally modified region of the silicon substrate, which are characterized by a greater inertness with respect to fluorine-containing plasma ions than silicon atoms.
The obtained dependences show that with an increase in the number of ion beam passes from 10 to 100 at an etching time in plasma of 120 seconds, height of resulting structures varied from 7 to 266 nm. This is explained by the higher exposure dose to the sample surface and, as a consequence, by the different etching rates of the modified regions. Also, with an increase in etching time in plasma from 30 to 120 seconds with the same number of ion beam passes, height of structures after plasma-chemical etching increases from 10 to 266 nm.

**Figure 2.** AFM - image of the structure of silicon surface after plasma chemical treatment in a fluorine-containing plasma for 120 seconds

Thus, with plasma chemical etching for 120 seconds with an increase in the number of passes from 10 to 100, the height of structures increases from 7 to 266 nm, respectively. With plasma chemical treatment for 30 seconds with 10 passes of ion beam, the etching of modified region is stimulated and a deep structure of 6 nm is formed. This is explained by the appearance of defects due to implantation of gallium ions into the near-surface layers of locally modified region and formation of a damaged layer, which is removed faster during plasma chemical etching than unmodified surface. However, at 100 passes of ion beam with plasma etching for 30 seconds, the modified regions show a masking effect.

AFM images of structures were obtained using the Ntegra probe nanolaboratory (NT-MDT, Russia), and ion processing and acquisition of SEM images of the high-tech complex Nova NanoLab 600. The etching process was carried out on a device for plasma-chemical processing of materials in a combined capacitive and inductive discharge plasma STE ICPe68 (Scientific and Technological Equipment, Russia).

**4. Conclusions**

The experimental studies carried out made it possible to establish the dependences of parameters influence and modes of the process of focused ion beams and plasma chemical etching on parameters of the formed nanoscale structures, made it possible to establish dependence of rate and time of plasma chemical treatment on the morphology of structures on silicon surface. So, for example, for formation of structures with maximum roughness, it is necessary to increase values of parameters responsible for reactive ion etching, these are such parameters as: power of the capacitive plasma source, mixing voltage, and flow rate of an inert gas (argon). To obtain structures with minimal roughness, it is necessary to increase values of parameters responsible for plasma chemical etching, these are such parameters as: power of the inductively coupled plasma source, flow rate of a fluorine-containing gas (sulfur hexafluoride). Also, an increase in plasma chemical etching speed leads to an increase in the roughness parameter and an increase in the angle of inclination of the structure.
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References
[1] Tok E S, Neave J H, Zhang J, Joyce B A and Jones T S 1997 Surf. Sci. 374 397
[2] Shiraiishi K and Ito T 1998 Phys. Rev. B 57 6301
[3] Kley A, Ruggerone P and Scheffler M 1997 Phys. Rev. Lett. 79 5278
[4] Huffaker D L, Park G, Zou Z, Shchekin O B and Deppe D G 1998 Appl Phys. Lett. 73 2564
[5] Vakulov Z E, Klimin V S, Rezvan A A, Tominov R V, Korzun K, Kots I N, Polyakova V V and Ageev O A. 2019 J. Phys.: Conf. Ser. 1410 012042
[6] Klimin V S, Rezvan A A and Morozova J V 2019 J. Phys.: Conf. Ser. 1410 012035
[7] Wong P S, Liang B and Huffaker D L 2010 J. Nanosci. Nanotechnol. 10 1537–1550
[8] Grundmann M, Stier O and Bimberg D 1995 Phys. Rev. B 52 11969
[9] Yamaguchi K, Yujobo K and Kaizu T 2000 Jpn. J. Appl. Phys. 39 L1245–L1248
[10] Gusev E Y, Jityaeva J Y, Avdeev S P and Ageev O A 2018 J. Phys.: Conf. Ser. 1124 0220345.
[11] Romashko R V, Kulchin Y N, Bezruk M N and Ermolaev S A 2016 Quantum Electronics 46(3) 277
[12] Ergun S, Yaralioglu G and Khuri-Yakub T 2003 J. Aerosp. Eng. 16 76.
[13] Klimin V S, Kots I N, Tominov R V, Rezvan A A, Varzarev Y N and Ageev O A. 2019 J. Phys.: Conf. Ser. 1410 012041
[14] Qiu Y, Wang H, Demore C E M, Hughes D A, Glynne-Jones P, Gebhardt S, Bolhovitins A, Poltarjonoks R, Weiher K, Schönecker A, Hill M and Cochran S 2014 Sensors 14 14806.
[15] Judy J W 2001 Smart Mater. Struct. 10 1115.
[16] Michael De Volder F L, Tawfick S H, Baughman R H and Hart A J 2013 Science 339 535—539
[17] Franklin A D et al 2012 Nano Lett 12 758—762
[18] Atul Dhale, Fahim Khan 2013 International Journal of Engineering Research and Development 7 80.
[19] Seymour J P, Wu F, Wise K D and Yoon E 2017 Microsystems & Nanoengineering 3 16066
[20] HajiHassan M, Chodavarapu V and Musallam S 2008 Sensors 8 10 6704-6762
[21] Avdeev S P and Gusev E Yu 2019 Proc. of SPIE 11022 11022-69
[22] Klimin V S, Tominov R V, Avilov V I, Dukhan D D, Rezvan A A, Zamburg E G, Smirnov V A and Ageev O A 2019 Proc. of SPIE 11022 110220E-1
[23] Klimin V S, Rezvan A A, Abramovich T S, Zubova T A, Tominov R V, Vakulov Z E, Kots I N and Ageev O A 2020 J. Phys.: Conf. Ser 1695 012199
[24] Balakirev S V, Eremenko M M, Mikhailin I A and Solodovnik M S 2018 J. Phys.: Conf. Ser. 1124 022025
[25] Filipov S V, Popov E O, Kolosko A G and Romanov P A 2015 J. of Phys.: Conf. Ser. 643 012101