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Effect of FIB-modification of Si(111) surface on GaAs nanowire growth

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Abstract. The paper presents the results of experimental studies of GaAs nanowire growth on Si(111) substrate with Ga focused ion beam modified areas with different treatment doses. We observed a significant difference between the parameters of nanowires arrays formed on modified and unmodified areas. It is shown that changing the dose of Ga ions from 52 fC/µm^2 to 1×10^4 fC/µm^2 allows to form nanowire arrays with a different set of parameters in a single technological cycle with a high selectivity. The possibility of regulating of the NW length in the range of 1–6 µm, the density in the range of 0–7.8 µm^{-2}, the diameter in the range of 28–95 nm and the normally oriented NWs in the range of 5–70 % by focused ion beam have been experimentally demonstrated. The change of modes and mechanisms of the catalytic centers formation and the initial stage of GaAs NWs growth were revealed.

Keywords: nanowires, gallium arsenide, focused ion beam, molecular beam epitaxy, silicon

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Исследование влияния ФИП-обработки поверхности Si(111) на процессы роста нитевидных нанокристаллов GaAs

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Аннотация. В работе представлены результаты экспериментальных исследований роста нитевлияния ионно-лучевой обработки поверхности Si(111) на процессы роста нитевидных нанокристаллов GaAs. Выявлено резкое различие между нитевидными нанокристаллами, сформированными на модифицированных и немодифицированных участках подложки Si. Показано, что изменение дозы имплантации ионов Ga с 52 фКл/мкм^2 до 1×10^4 фКл/мкм^2 позволяет варьировать параметры (плотность, диаметр, длину и ориентацию относительно подложки) массива нитевидных нанокристаллов в широком диапазоне условий в едином технологическом цикле. Представлены основные закономерности, которые отображают нелинейную зависимость параметров сформированных структур от дозы имплантации ионов.

Ключевые слова: нитевидные нанокристаллы, фокусированный ионный пучок, молекулярно-лучевая эпитаксия, кремний, арсенид галлия

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Introduction

III–V nanowires (NWs) are promising objects for creating various elements and devices in the fields of photonics, micro- and nanoelectronics, micromechanics and sensors. This is possible due to the combination of unique electronic, optical and mechanical properties of this nanostructure type [1]. The creation of devices based on NWs requires the development of technologies for controlling their main characteristics, such as: length, diameter, shape, chemical composition, doping type, doping level, and array density [2]. The fabrication of NWs is usually carried out using the “vapor-liquid-solid” (VLS) mechanism, which makes it possible to control the main NWs parameters by changing the size and surface arrangement of metal nanodroplets which act as catalytic centers for further NWs growth.

These changes can be performed using various combinations of epitaxial and lithographic methods [3]. So, depending on the type of VLS mechanism, electron beam lithography [4], nanoimprint lithography [5] and the so-called nanospheric lithography [6] are most widely used for NWs formation with different main characteristics.

Recently, the local ion-beam surface treatment using a Ga focused ion beam (FIB) has been actively studied as an alternative method for controlling NWs parameters [7–10]. This method makes it possible minimizing the main drawbacks of the traditional technological approaches based on the optical lithography operations, chemical wet and plasma etching [11–12].

On the one hand, the focused ion beam method can be used for the formation of holes in the masking oxide layer (in SiO_2/Si structures) with subsequent localization of catalyst droplets in them [7]. On the other hand, the Ga ion beam and self-catalytic VLS growth can be used for direct local formation of catalytic centers [8]. In this case, Ga ions implanted into the substrate by FIB form Ga droplets on the surface during further annealing which promote GaAs NW catalytic growth [9]. So, this technology makes it possible to effectively control the size, density and position of the formed metal droplets by changing various technological parameters of the FIB treatment and pre-growth annealing that thereby largely predetermining the characteristics of subsequently growing GaAs NWs [10]. At the same time, issues related to the mutual influence of the main parameters of ion-beam processing and epitaxial synthesis on the key characteristics of GaAs NWs remain poorly understood. The mechanisms underlying on the FIB-induced NW growth are also have many questions.

The aim of this work is to study the effect of the Si(111) surface treatment with different Ga ion doses on the GaAs nanowire growth.

Experiment

FIB treatment of the Si(111) surface was carried out using a Nova NanoLab 600 scanning electron microscope (SEM) equipped an ion beam system with a Ga ion source. We modified square areas with a size of 5×5 μm by Ga ion beam at accelerating voltage of 30 kV and beam current of 30 pA. The dose of Ga ions varied from 52 fC/μm^2 to 1×10^4 fC/μm^2 by changing the number of FIB passes. Ga ions are predominantly implanted into the substrate surface under the used FIB treatment modes. So, surface etching was supressed.

Self-catalytic GaAs NWs were grown by molecular beam epitaxy (MBE) on a SemiTEq STE 35. Si(111) samples with FIB-modified surface areas were preliminarily annealed under ultrahigh vacuum conditions at a temperature of 600 °C. At this stage, the processes of implanted Ga ions segregation were initiated to form Ga catalytic centres. In this condition, native Si oxide masking layer was not removed. It allowed us to simultaneously form GaAs NWs outside the modified regions. Then, GaAs NWs were grown at the same substrate temperature (600 °C) with the equivalent GaAs deposition rate and thickness equal to 0.25 ML/s and 200 nm, respectively. The Ga and As fluxes were preliminarily calibrated by the GaAs growth rate on GaAs(001) substrates.

The control of the obtained structures morphology was carried out by SEM methods. Analysis of the geometric parameters of GaAs NWs (length, diameter, density and orientation) based on SEM images was performed using specialized software SIS Software Scandium.

Results and discussion

An analysis of the obtained SEM images of the Si(111) surface with FIB-modified areas shows a significant effect of the Ga ion dose on the formation processes and geometric parameters of GaAs NW arrays (Fig. 1).

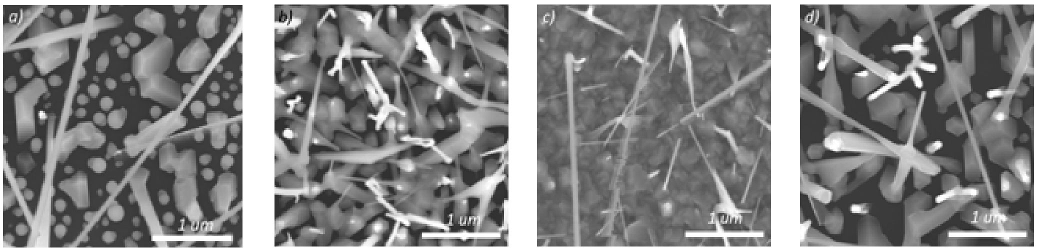

Fig. 1. SEM images of modified areas after GaAs nanowires growth for Ga ion dose of 52 fC/μm² (*a*), 260 fC/μm² (*b*), 1×10⁴ fC/μm² (*c*) and unmodified area (*d*)

Quantitative analysis of the GaAs NW arrays geometric parameters based on SEM images and subsequent statistical processing of the obtained data were performed (Fig. 2).

The NWs growth is significantly suppressed at the area with an ion dose of 52 fC/μm^2 (Fig. 1, *a*) and their density equal to 0.36 μm^{-2} (Fig. 2, *a*). The NWs density value in this area is almost an order of magnitude lower than the array density formed on the unmodified surface (2.56 μm^{-2}). At the same time, Ga droplets array is formed in this area with density of 13.6 μm^{-2}.

Increasing the FIB treatment dose leads to a sharp raising the GaAs NWs density (Fig. 1, *b*). The peak value of 7.8 μm^{-2} is reached at the dose of 5.2×10^3 fC/μm^2, which then gradually decreases to 5.76 μm^{-2} (Fig. 1, *c*). It is also worth noting that the NWs density is more than 2 times higher than their density on the unmodified surface, excluding the data point of minimal dose value (Fig. 2, *a*).

The dependences of the GaAs NWs length and diameter on the FIB treatment dose (Figs. 2, *b* and 2, *c*, respectively) are quite similar. Both graphs show a sharp decrease in the NWs length and diameter with an increase in the ion dose. Starting from a dose of 1.56×10^3 fC/μm^2, the values reach saturation. The NWs density in this range has a pronounced tendency to decrease (Fig. 2, *a*). We assume that this behaviour may be related to the intensification of the parasitic growth in the region of high doses (Fig. 1). The maximum value of normally oriented GaAs NWs is 70 % at a dose of 520 fC/μm^2 (Fig. 2, *d*). This dependence first increases, and then sharply decreases (at the dose of 5.2×10^3 fC/μm^2) with stabilization at a value in the range 6–10 % (the reference value is about 50 %).

We assumed that the use of low ion doses should suppress the formation of Ga droplets and GaAs crystallites on modify area by the following reasons. Ions are predominantly embedded into the crystal structure of the near-surface substrate layer at low doses regime. At the same time, the lattice is enriched with ions and the defects generated by them but retains its structure [13]. The concentration of defects in these areas is relatively low, so crystal structure relaxation is complicated during annealing. The presence of defects also makes it difficult for the implanted Ga ions (atoms) to reach the surface. This, in turn, affects the suppression of the catalyst

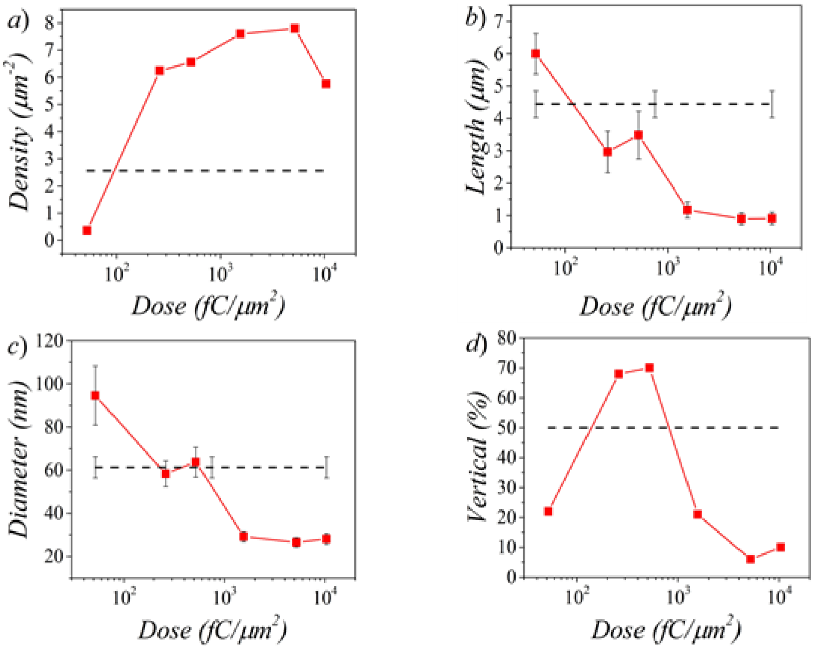

Fig. 2. Dependences of density (*a*), length (*b*), diameter (*c*) and vertical orientation of NWs (*d*) on Ga ion dose (dash lines correspond to values for unmodified areas).

droplets formation from the implanted material. While the expected result should have confirmed this assumption, this study showed that a high-density array of Ga droplets is formed in the area with a dose of 52 fC/μm^2 (Fig. 1, *a*). We believe that the droplets formed at the initial growth stage from supplied growth components. This is confirmed by the fact that there is almost complete suppression of directly catalytic growth with the participation of these droplets (Fig. 1, *a*; 2, *a*). We suppose that the reason of this behavior may be related with the NW nucleation stage. This stage was, apparently, kinetically retarded or extended in time in these areas. This led to the outflow of material to neighbouring regions, where the initial stage of NW formation proceeded much faster.

Changing the ion dose from 52 to 260 fC/μm^2 is accompanied by an increase in defects on the modified region. We think that this leads to enhanced of the crystal lattice relaxation processes at the annealing stage. This causes segregation of embedded Ga ions on the Si substrate surface and, as consequently, the formation of catalyst droplets [14]. The creation of the metal component excess on this area leads to an expected increase in the self-catalytic NWs density (more than 2 times) and GaAs crystallites, respectively (Figs. 1, *b*; 2, *a*). The values of NWs length and diameter decrease to 2.97 μm and 58 nm, respectively (Figs. 2, *b*; 2, *c*). It is also interesting to note that this area has a maximum fraction of GaAs NWs oriented normally to the substrate and equal to 70 %.

With a further increase in the dose up to 1×10^4 fC/μm^2, the nanowire density changes insignificantly, while the diameter, length, and number of vertically oriented structures sharply decrease. We associate this system behaviour with a change in the GaAs NW nucleation mechanism. The formation of the metal component excess for the catalytic centres creation under conditions of 100 % coverage of surface with GaAs crystallites is possible only in the following case. We believe that in the process of epitaxial growth there is an additional uncompensated flow of Ga atoms due to continued segregation of embedded Ga ions. This assumption is in good agreement with the results of experimental studies. First, the NW diameter (Fig. 2, *c*) in the studied ion dose range is more than 2 times below reference values. We attribute this to the fact that the segregation flow of excess Ga to the surface is quite small. Consequently, the size of the formed catalytic centres and the diameter of the growing NWs is relatively small. Second, this flow should decrease with time due to the finite number of embedded Ga ions as can be seen from Fig. 1, *c*. GaAs NWs have a pronounced conical shape and a relatively small length (0.91 μm). Third, in this area the Ga droplets formation happen mainly not on the Si(111) surface, but on various faces of randomly oriented GaAs crystallites. So, the fraction of GaAs NWs oriented normally to the substrate decreases sharply and at doses above 1.56×10^3 fC/μm^2 does not exceed 20 %.

Conclusion

Thus, the performed experimental studies have shown that the control of different NWs parameters by the FIB method is possible by varying the implantation dose of Ga+ ions into the Si(111) substrate. A sharp difference between NWs arrays formed on modified and unmodified regions of the Si substrate was revealed. It is shown that changing the dose of Ga ions from 52 fC/μm^2 to 1×10^4 fC/μm^2 makes it possible to control the main NW parameters, such as: length (1–6 μm), density (0–7.8 μm^{-2}), diameter (28–95 nm) and orientation (5–70 %). The change of modes and mechanisms of the catalytic centres formation and the initial stage of GaAs NWs growth has been experimentally demonstrated. The possibility of forming GaAs NWs arrays with significantly different geometric parameters on one sample in a single technological cycle is experimentally shown.

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