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Effect of ion dose and accelerating voltage during focused ion beam Si(111) surface treatment on GaAs nanowires growth

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Abstract. Experimental studies of the effect of dose and accelerating voltage during ion beam treatment of the Si(111) surface on the substrate structure and growth processes of GaAs nanowires have been carried out. For this purpose, arrays of areas were created on the Si(111) surface by ion beam treatment using an all-over template with variation of accelerating voltage in the range of 10—30 kV and dose in the range of 0.01—10.4 pC/μm^2. Based on the results of the modified surface study after GaAs nanowire growth, the dependences of the main nanowire characteristics (density, length and diameter) on the ion beam dose were obtained. It is shown that the main influence on the formed nanowire characteristics is exerted by the dose of embedded Ga-ions. By changing the value of this ion beam parameter together with the high-temperature annealing, the chemical composition and morphology of the surface silicon oxide layer can be locally controlled, thereby predetermining the parameters of the growing nanowire array. In this case, the accelerating voltage, and, hence, the distribution of ions in the near-surface layer, is of secondary importance during all-over template processing. This is confirmed by the formation of identical nanowire arrays at different accelerating voltages since the growth of nanowires occurs under the same conditions on the Si surface after the annealing stage (as confirmed by Raman spectroscopy results).

Keywords: nanowires, gallium arsenide, focused ion beam, molecular beam epitaxy, A3B5

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Влияние дозы и ускоряющего напряжения при обработке поверхности Si(111) фокусированным ионным пучком на рост нанопроволок GaAs

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Аннотация. Проведены экспериментальные исследования влияния дозы и ускоряющего напряжения при ионно-лучевой обработке поверхности Si(111) на структуру подложки и процессы роста нанопроволок GaAs. По результатам исследований модифицированных областей, сформированных при ускоряющем напряжении ионного пучка 10 – 30 кВ и дозе 0,01 – 10,4 пКл/мкм^2, после роста нанопроволок GaAs, были построены зависимости основных характеристик ННК (плотность, длина и диаметр) от дозы ионного пучка. Показано, что основное влияние на характеристики сформированных нанопроволок оказывает доза ионов Ga, при этом ускоряющее напряжение, а, следовательно, и распределение ионов в приповерхностном слое при сплошной обработке поверхности имеют второстепенное значение.

Ключевые слова: нанопроволоки, арсенид галлия, фокусированный ионный пучок, молекулярно-лучевая эпитаксия, A3B5

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Introduction

Semiconductor nanowires (NWs) have unique properties that make them promising for the fabrication of opto- and nanoelectronics devices [1]. However, this requires the development of effective approaches to manage NW properties: geometrical, structural, optical, etc. Technologies based on standard lithographic processes don`t allow a wide variation of the different NW characteristics [2], differ in the high cost [3] and complexity of the manufacturing templates [4]. An approach based on pre-growth surface treatment with a Ga focused ion beam (FIB) is one of the most promising and frequently discussed methods to overcome the above disadvantages and improve the efficiency of NW parameter control [5–10]. This method is mainly used in two variants. In the first, pre-growth FIB treatment of the Si surface is performed at defined locations with further annealing and formation of metal catalyst droplets in the processing area due to segregation of embedded Ga atoms which allows them to be used for self-catalytic NW growth with certain parameters [10]. The second is based on the FIB formation of nanoscale holes in the silicon oxide layer for the subsequent localization of catalyst droplets in them and the NW growth [8]. In this work, we investigate the effect of dose and accelerating voltage during ion beam treatment of the Si(111) surface on the substrate structure and the GaAs nanowire growth.

Materials and Methods

Experimental studies were carried out on epi-ready Si(111) p-type substrates with a layer of native silicon oxide about 1 – 1.5 nm thick [11]. The ion beam treatment was performed using a Nova NanoLab 600 electron microscope equipped with a FIB system with a Ga ion source. Arrays with dimensions of 5×5 μm were formed on the substrate surface by FIB at accelerating voltages of 10, 20 and 30 kV and implantation doses of 0.01 – 10.4 pC/μm^2. After a preliminary ultra-high vacuum (UHV) annealing of the treated samples at a temperature of 600 °C for 60 minutes under high vacuum conditions, epitaxial GaAs NW growth by molecular beam epitaxy (MBE) method was performed at the same temperature with a nominal deposition rate of 0.25 ML/s for 48 minutes. Annealing and growth were carried out on a SemiTEq STE 35 MBE system. Samples were characterized by SEM and Raman spectroscopy. The Raman spectra were measured at room temperature using a Horiba LabRam HR800 setup equipped with a 532 nm laser.

Results and Discussion

Quantitative analysis of GaAs NW arrays geometric parameters based on SEM images and subsequent statistical processing of the obtained data made it possible to plot dependences of the main NW arrays parameters for 10, 20 and 30 kV (Fig. 1). It is clearly seen that the dependencies for various accelerating voltage correlate well with each other. At the same time, the nonlinear dependence of the main NW parameters on the treatment dose is visible. We associate this behavior of the system with the interaction peculiarities of embedded Ga ions with the SiO_x surface layer during the annealing process. Thus, we distinguish at least three regions with different effects of the ion beam dose on the resulting NW GaAs arrays.

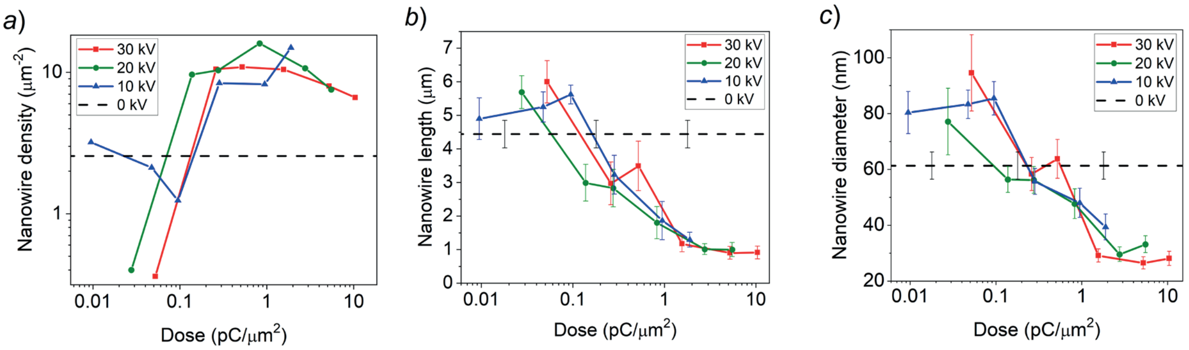

Fig. 1. Dependences of the density (*a*), length (*b*) and diameter (*c*) on Ga ion dose. Dash lines correspond to values for unmodified areas

At low doses (from 0.01 to ~ 0.1 pC/μm^{2}), NW growth is significantly suppressed at all accelerating voltages (Fig. 1, *a*). We suggest that this is due to the interaction of the embedded Ga ions with the SiO_x surface layer during annealing, leading to the formation of an additional masking oxide layer in the form of a more thermally stable Ga_xO_y compound. Thus, the growth of NWs occurs in the region where the oxide layer is present even after 1 h of annealing which has a strong effect on the suppression of the NW growth and significantly reduces their density to 1.24 (for ~ 0.1 pC/μm^{2}), 0.4 and 0.36 μm^{-2} for 10, 20 and 30 kV, respectively (2.56 μm^{-2} for unmodified surface). It is also worth noting that in these areas, at all accelerating voltages, along with a small number of NWs, a high-density array of nanosized Ga droplets is also formed (e.g., at 30 kV up to ~ 13.6 μm^{-2}) which is a very unexpected result, since the NW growth was carried out under arsenic-enriched conditions for 48 min. This circumstance also confirms the above hypothesis and may indicate the desorption of growth components from the surface of the masking oxide and/or their migration to unmodified regions, where the nucleation threshold is lower due to the formation of pores in SiO_x at the annealing stage [12]. At the same time, the NW length and diameter reach maximum values (higher than the control values) (Fig. 1, *b*, *c*).

Increasing the ion dose (from 0.1 to ~ 1 pC/μm^{2}) causes to etching of the oxide layer due to an increase in the ion flow from the substrate which forms an array of nanopores, stimulating the NW formation and leading to a sharp increase in their density. This dose range makes it possible to increase the NW density within the area of ion beam treatment up to 16 μm^{-2} for 20 kV (Fig. 1, *a*) and to vary the density in the range of 8.4 – 16 μm^{-2} by changing the ion dose. In addition, the NW length decreases and becomes even smaller than outside the modification area. For example, at a dose of ~ 0.3 pC/μm^{2}, the NWs are on average 1.5 times shorter (Fig. 1, *b*) than the reference values (4.44 ± 0.4 μm outside the treatment area). This is due to an increase in the NW density under conditions of a limited material source. However, the NW diameter (Fig. 1, *c*) is practically equal to the reference value (61 ± 5 nm) which may indicate an approximately identical (in size) ensemble of initial catalyst (Ga) droplets.

Further increase of the dose (from 1 pC/μm^{2} and higher) leads to the complete removal of the native oxide layer and the formation of nanosized droplets at the annealing stage. During the pre-growth treatment of the substrate with an As flux, the droplets crystallize and form a large array of GaAs nanocrystals which is a parasitic phase from the point of view of NW growth. This leads to a change in the mechanisms of NW growth, causing a corresponding change (decrease) in the values of all the main parameters, regardless of the accelerating voltage (Fig. 1).

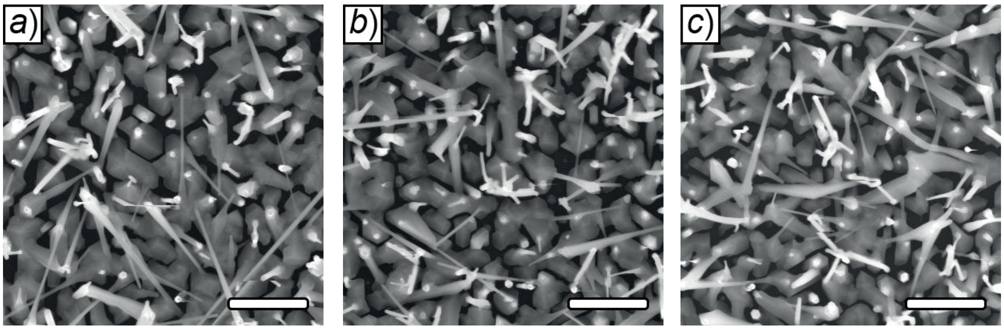

Fig. 2. SEM images of modified areas after GaAs nanowire growth for the same Ga ion dose (~ 0.3 pC/μm²) and accelerating voltage of 10 kV (*a*), 20 kV (*b*) and 30 kV (*c*). Scalebar is 1 μm

Thus, the density values are reduced to a maximum of 6.6 μm⁻² per 30 kV (Fig. 1, *a*). The length and diameter values also decrease and reach the minimum values in the entire dose range considered (Fig. 1, *b*, *c*). With further dose increase, the NWs practically don`t change and are in the range of 0.91 ± 0.2 to 1.29 ± 0.22 μm and 27 ± 2 to 33 ± 3 nm for the NW length and diameter, respectively.

The influence of the accelerating voltage plays a secondary role in this case. This is confirmed by a good correlation between the curves obtained for arrays formed at different accelerating voltages and by a similar value (trend) of the main NW parameters on the ion treatment dose. For example, at a dose of ~ 0.3 pC/μm² (Fig. 2), the NW density values at accelerating voltages of 10, 20, and 30 kV are approximately equal and amount to 8.4, 10, and 10.5 μm⁻², respectively (Fig. 1, *a*). The average values of the length and diameter of the obtained NWs are also approximately equal: 3.23 ± 0.58, 2.83 ± 0.56, 2.97 ± 0.64 μm and 56 ± 5, 56 ± 5, and 58 ± 6 nm for 10, 20 and 30 kV, respectively (Fig. 1, *b*, *c*). The obtained Raman spectroscopy results suggest that such a similarity of the curves, especially in areas with similar doses, for different accelerating voltages can be achieved due to the growth of NW arrays within areas with almost identical conditions on the growth surface after the annealing stage (Fig. 3).

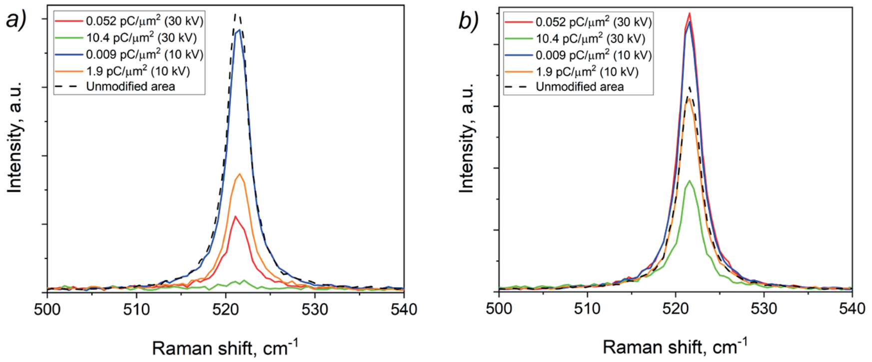

Fig. 3. Raman spectra of areas modified at the minimum and maximum ion beam doses for 10 and 30 kV before (*a*) and after (*b*) UHV annealing

Conclusion

Thus, it can be concluded that the magnitude of the accelerating voltage and, consequently, the distribution of ions in the near-surface layer has virtually no effect on the NW ensemble parameters when an all-over pattern is used. This is confirmed by Raman spectroscopy results which show that the growth of NW arrays after the annealing stage at similar implantation doses occurs under identical surface conditions irrespective of the accelerating voltage which allows NW formation with similar parameters. The characteristics of the obtained NWs are completely determined only by the implantation dose which is achieved by controlling the chemical composition and morphology of the oxide within the treated areas at the annealing stage which makes it possible to predetermine the further NW growth. At the same time, the effect of the dose on the NW parameters cannot be explained by the formation of catalytic centers due to the segregation of embedded Ga atoms upon annealing. Apparently, this is due to the peculiarities of the interaction of ions during ion beam treatment with the near-surface layer of the substrate and requires further investigation.

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