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Localized Ga droplets formation on nanopatterned silicon substrates

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Abstract. In this work, the effect of the modes of processing SiO_2/Si substrates with a focused ion beam on the subsequent epitaxial growth of Ga droplets was investigated. It was shown that an increase in beam passes and ion dose led to a broadening of the pyramidal cavities and also affects the localization of Ga droplets. It was found that the use of additional preliminary processing of substrates in a hydrogen fluoride has a positive effect on the formation of gallium droplets inside the holes. The maximum degree of filling was observed at a hole size of about 350 nm.

Keywords: wet chemical etching, silicon, monolithic integration, focused ion beam, nanopatterning, molecular beam epitaxy, droplet epitaxy

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Локализованное формирование капель Ga на нанопрофилированных подложках кремния

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Аннотация. В работе представлены результаты исследований влияния режимов обработки подложек SiO_2/Si сфокусированным ионным пучком на последующее получение наноструктур Ga методом капельной эпитаксии. Показано, что увеличение проходов пучка и дозы приводит к расширению пирамидальных углублений, а также влияет на локализацию капель Ga. Установлено, что использование дополнительной предварительной обработки подложек в плавиковой кислоте положительным образом влияет на локализацию капельных структур Ga в пирамидальных углублениях. Максимальная степень заполнения наблюдалась при размере углублений приблизительно равных 350 нм.

Ключевые слова: жидкостное травление, кремний, монолитная интеграция, фокусированные ионные пучки, нанопрофилирование, молекулярно-лучевая эпитаксия, капельная эпитаксия

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Introduction

Today, direct monolithic integration of III-V semiconductors on silicon faces problems mainly associated with the difference in lattice constants and thermal expansion coefficients, which in turn significantly affects the defect density of the grown structures [1–3]. In this regard, local integration has proven to be a promising method both for reducing defects and localizing grown structures and for better compatibility with CMOS technology [4]. However, modern substrate nano- or micropatterning technique with traditional lithography to achieve local integration of III-V semiconductors on silicon is not only a labor-intensive but also an expensive process. Therefore, the search for new approaches to creating a pattern on the surface of semiconductor wafers and subsequent epitaxial growth of nanostructure arrays is one of many urgent tasks.

In this paper, we propose to use focused ion beams to form arrays of modified nanoscale holes on the SiO_2/Si surface. Subsequent treatment in KOH allowed us to form pyramidal cavities, the shape of which helps localize the structures and reduce their defect level during epitaxial growth of III-V semiconductors [5–7]. For study the initial stage of the epitaxial growth processes on such FIB-modified substrates, we used droplet epitaxy technique [8] as a first stage of GaAs nano- and microcrystal growth. At the final stage, the effect of pregrowth treatment on the formation and localization of Ga metal droplets was studied.

Materials and Methods

In this work we used Si(001) substrates coated with a SiO_2 layer. The FIB modification of the samples was performed by processing square areas of $l = 0.25 \times 0.25$ and 0.5×0.5 µm with pitches between etched points of (0.5 µm). The number of beam passes N varied from 450 to 1500. After that, the samples were etched in 30% KOH solution to form pyramidal cavities. Then the samples were placed in a growth chamber and annealed for 2 hours at a temperature of about 950 °C. After annealing, the samples were cooled to the growth temperature (T = °750C) and gallium was deposited with a thickness H of 200 ML at a rate of v = 0.1 ML/s (the deposition rate was previously calibrated using the RHEED pattern on the GaAs substrate).

Results and Discussion

Fig. 1 shows SEM images of silicon samples with FIB-modified areas after Ga droplet formation. It is shown that gallium droplet nucleation mostly occurs on the faces of pyramidal cavities (Fig. 1, *a*). This may be due to the fact that the surface after etching is quickly covered with a layer of native oxide, which does not allow Ga droplets to form in the center. Therefore, with other parameters remaining unchanged, for the next samples, additional pre-treatment of the samples was carried out in a 2% hydrogen fluoride solution. As can be seen from the Figures 1, *b* and 1c, this allowed us to improve the localization of droplet structures and ensure their formation in the centers of the pyramidal cavities.

Analysis of the sample morphology showed that an increase in the number of ion beam passes led to a decrease in the localization of the formed gallium droplets in the center and stimulated their formation in the cavity corners. Apparently, such behavior is associated with an increase in the size of the etched cavities during wet chemical etching in KOH due to an increase in the depth of the initial holes during FIB-modification [9]. Thus, an increase in the hole size *a*) *b*) *c*)

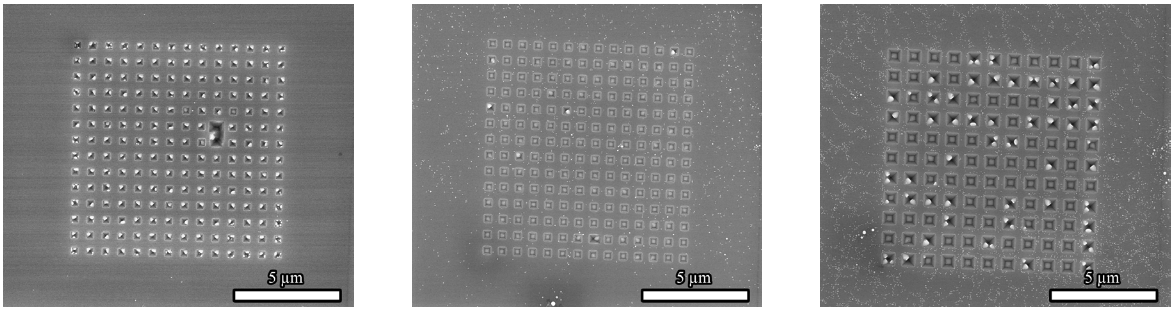

Fig. 1. SEM-images of FIB-modified SiO_2/Si areas with gallium droplets inside etching pits before (*a*) and after (*b*, *c*) growth procedure optimization

affected a decrease in the probability of the gallium droplets nucleation in the center of the cavities (Fig. 1, *c*). Changing the initial size of the FIB-modified areas from 0.25 × 0.25 to 0.5 × 0.5 μm led to a similar effect (Fig. 1, *b*, *c*).

It was found that the maximum filling with Ga droplets (the degree of filling was calculated as the ratio of the number of Ga droplets localized in the center of the cavities to their total number) is observed when the width of the pyramidal cavities is minimal (~350 nm). An increase in the number of passes led to a decrease in the percentage of droplet localization in the center for the initial 0.25 × 0.25 μm areas, while for the initial 0.5 × 0.5 μm areas the results obtained are ambiguous. On the one hand, increasing the number of passes increases the etching depth and, accordingly, the width of the pyramidal cavities, which should reduce the localization of droplets, but, on the other hand, processing in 2% hydrogen fluoride solution led to thinning of the native oxide and the emergence of the possibility of surface diffusion, as well as etching of the overhanging oxide above the cavities, which, in theory, increased the localization of droplets in the center to a certain level. An increase in the degree of filling with an increase in the number of passes for the initial 0.5 × 0.5 μm areas is associated with a decrease in the distance between the cavities.

a) *b*)

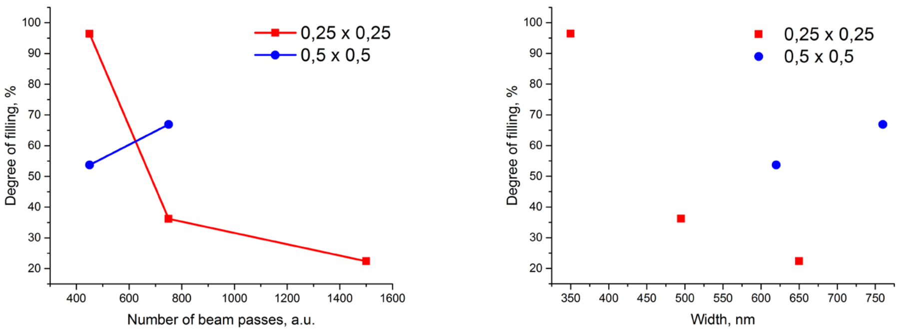

Fig. 2. Influence of the number of beam passes (*a*) and the size of the pyramidal cavities (*b*) on their degree of filling

Conclusion

In this work, the possibility of achieving high selectivity of epitaxial growth on silicon samples with preliminary FIB treatment and wet chemical etching was demonstrated. The results presented also suggest that the gallium ion beam not only has a masking effect when processing silicon, but can also act as a method for creating a template on the surface for epitaxial processes.

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