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Study of FIB-modified silicon areas by AFM and Raman spectroscopy

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Abstract. This paper presents the results of atomic force microscopy and Raman spectroscopy studies of the effect of high-temperature annealing on the height/depth parameters of silicon areas modified by a focused ion beam. It is shown that the focused ion beam treatment with 5 beam passes leads to swelling of the surface of the modified silicon areas. It was found that the depth of the focused ion beam modified area is different after annealing at 600 and 800 °C. An increase in the number of passes in both cases led to an increase in the depth of the focused ion beam modified areas. The results of studies of Raman spectroscopy showed that with an increase in the number of passes, a decrease in the crystallinity of silicon occurs. It is also shown that annealing of such regions leads to the restoration of crystallinity upon annealing at 600 °C and almost complete restoration of crystallinity at 800 °C.

Keywords: silicon, A3B5, molecular beam epitaxy, annealing, atomic force microscopy, focused ion beam, Raman spectroscopy

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Исследование областей кремния, модифицированного ФИП, методами АСМ и рамановской спектроскопии

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Аннотация. В данной работе представлены результаты исследований с помощью атомно-силовой микроскопии (АСМ) и рамановской спектроскопии влияния высокотемпературного отжига на параметры высоты/глубины областей кремния, модифицированных фокусированным ионным пучком (ФИП). Показано, что обработка ФИП с 5 проходами пучка приводит к «вспучиванию» участков модифицированного кремния. Установлено, что глубина модифицированных ФИП участков после отжига при 600 и 800 °С различна. Увеличение числа проходов в обоих случаях приводило к увеличению глубины ФИП-обработанных участков.

Результаты исследований рамановской спектроскопии показали, что с увеличением числа проходов происходит увеличение дефектности кремния. Показано также, что отжиг ФИП-обработанных участков приводит к восстановлению кристаллической структуры при отжиге на 600 °C и практически полному ее восстановлению при 800 °C.

Ключевые слова: кремний, A3B5, молекулярно-лучевая эпитаксия, отжиг, атомно-силовая микроскопия, фокусированный ионный пучок, рамановская спектроскопия

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Introduction

Today, research in the field of searching for new functional materials is gaining more attention. One of these scientific directions is the production of optoelectronic and photonic integrated circuits based on the integration of III-V semiconductors on silicon [1, 2]. Obtaining such structures is possible through monolithic integration which is a promising method that would make it possible to obtain semiconductor structures in a single technological cycle and greatly reduce the cost of their production. However, the direct growth of III-V semiconductors on Si is difficult due to a significant lattice mismatch, which leads to a high level of defectiveness of the grown layers [1, 3]. A different number of methods are used to reduce the number of dislocations: the use of nucleation layers [4, 5], two- or three-step growth of buffer layers [4, 6], as well as the growth of thick buffer layers [7, 9], substrate misorientation [1, 4], the use of dislocation filters [4, 6], aspect ratio trapping (ART) [2, 8] etc. [1, 2, 9], but it has not yet been possible to achieve a minimum dislocation density comparable to the native substrate. In turn, surface modification by a focused ion beam (FIB) is one of the most flexible, accurate, and operational tools for creating a nanoscale relief on a surface [10] similar to ART method. FIB allows not only to control the processes of self-organization of epitaxial nanostructures, but also the positioning and localization of their formation. It is also assumed that partial or complete amorphization of the surface in this way will help to avoid the occurrence of antiphase domains, improve stress relaxation and localize most of the defects in the lower, nucleation layers.

In this work, atomic force microscopy (AFM) and Raman spectroscopy studies of the effect of high-temperature annealing on the height parameter of FIB-modified silicon areas were carried out, as well as to evaluate its defectiveness.

Materials and Methods

The modification of the silicon substrates was carried out by treating the areas of 5×5 μm with a focused Ga^+ ion beam. The processing parameters for AFM studies were: accelerating voltage – 30 kV, the number of beam passes varied from 5 to 200. The processing parameters for Raman spectroscopy studies were: accelerating voltage varied from 5 to 30 kV; the number of beam passes varied from 1 to 200. After FIB processing, the substrates were studied by AFM and Raman spectroscopy. Next, the samples were subjected to thermal annealing in an MBE chamber for 60 minutes. The annealing temperature varied from 600 to 800 °C. Then they were removed from the MBE chamber and examined by AFM and Raman spectroscopy to compare the resulting parameters.

Results and Discussion

AFM studies of the samples without annealing showed that the processing of silicon areas with a small number of beam passes results in swelling of these areas (Fig. 1, *a*). A further increase in the number of beam passes led to etching and a gradual increase in the depth of the modified areas (Fig. 1, *b*). It should be noted that the height of the processed area was compared with the level of the substrate with the number of beam passes equal to 10. After that, the formation of a deepening was observed with an increase in the number of beam passes.

Raman spectroscopy studies have shown that an increase in the number of passes leads to a decrease in silicon crystallinity because the intensity of the main crystalline (c-Si) TO-phonon peak (521 cm^{-1}) decreases (Fig. 1, *c*). This behavior is associated with an increase in the distortion of the Si crystal lattice due to defects introduced during Ga ion implantation. It should be noted that the intensity of the c-Si peak from the area treated with 200 passes is greater than the c-Si peak from 100 passes. We attribute this behavior to the specifics of the FIB method, since simultaneous processes of saturation of the area with the implanted material and etching of the substrate occur. Thus, at 200 passes, most of the amorphous phase was etched off and the signal from crystalline silicon increased.

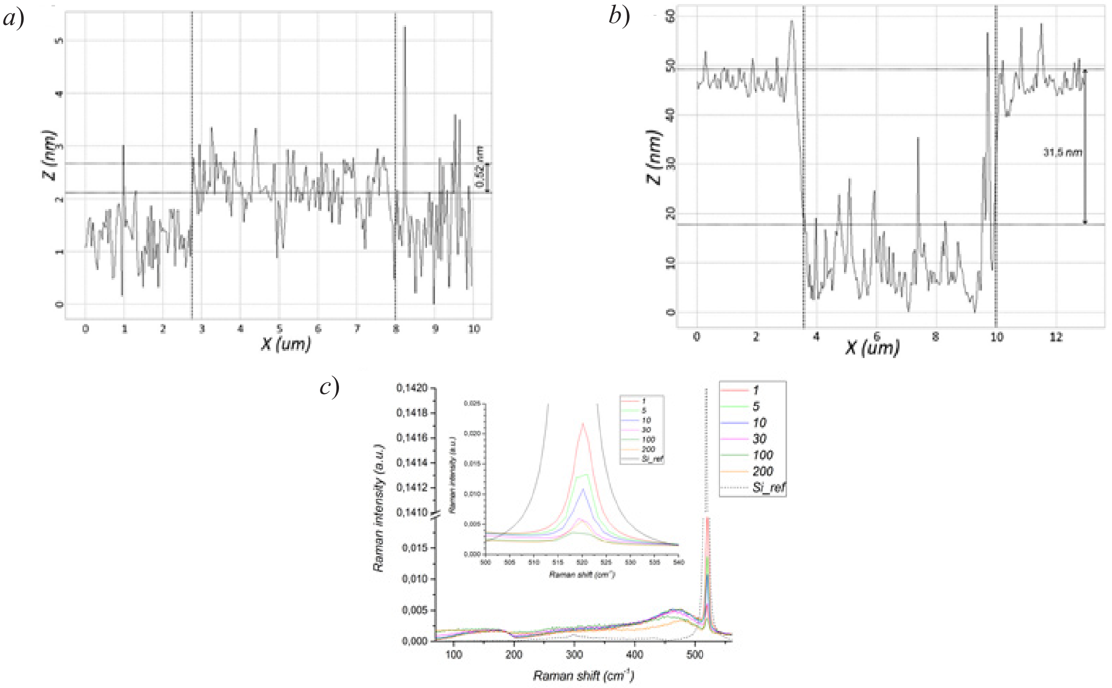

Fig. 1. AFM profiles of FIB-modified silicon areas treated with (*a*) 5 beam passes and (*b*) 200 beam passes. (*c*) Normalized Raman spectra of FIB-modified Si areas with different beam passes.

Next, the silicon samples were annealed at 600 and 800 °C. AFM studies of samples annealed at 600 °C showed that no surface swelling was observed with 1 beam pass and the height of the treated area, as in the case of processing with 10 passes without annealing, was compared with the substrate level (Fig. 2, *a*). An increase in the depth of the modified areas is also observed compared to the samples without annealing (Fig. 2, *b*). This is due to the release of the implanted material to the surface and its partial desorption. It should be noted that a significant change in depth is observed when processing with a large number of passes (100 and 200). Apparently, this is due to the fact that during FIB processing with large number of beam passes, high damage to the modified areas and, accordingly, high defectiveness occur, which contribute to a more intensive release of the implanted material and its evaporation.

The situation slightly changes in the AFM study of samples after high-temperature annealing at 800 °C. With a small number of beam passes, there is no swelling of the surface, and a deepening of the changed areas is immediately observed (Fig. 1, *a*). This indicates that during annealing, the implanted material emerges on the surface, followed by its evaporation and/or etching of the modified area, which leads to the formation of a deepening. A subsequent increase in the number of passes led to an even greater increase in the depth of the modified areas compared to annealing at a lower temperature. Moreover, the change in the height/depth

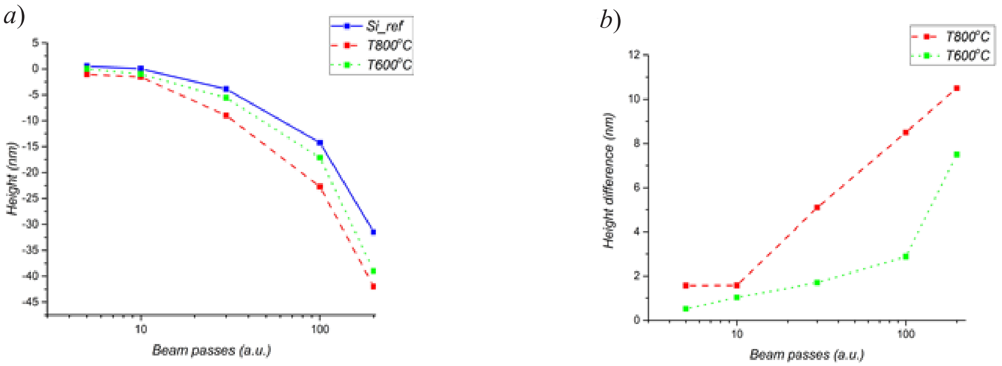

Fig. 2. Height of FIB-modified areas before and after annealing (negative height values indicate etch depth) (*a*); Height difference between annealed and non-annealed substrate at different annealing temperatures (*b*)

of the modified areas is not the same if we compare the cases of samples annealed at 600 and 800 °C (Fig. 2, *b*).

Raman spectroscopy studies of samples annealed at 600 °C showed that, upon annealing, the intensity of the c-Si TO-phonon peak exhibits a nonmonotonic change with increasing number of beam passes (Fig. 3, *a*). This minimum intensity behavior at 30 passes is possibly due to the fact that the implanted material remains embedded in the silicon lattice after annealing. Apparently, with such a set of parameters, there is no critical damage of the silicon crystal lattice, during which an intense release of the implanted material to the surface occurs, as in the cases with 100 and 200 passes. An increase in the annealing temperature to 800 °C leads to an increase in the intensity of the crystalline Si TO-phonon peak (521 cm^{-1}) for almost all beam passes to the spectral values obtained from the untreated substrate (Fig. 3, *b*). In this case, it can be concluded that such annealing is not suitable, since no amorphous silicon phase remains on the surface and ubsequent growth will occur in exactly the same way as on a substrate not treated with FIB.

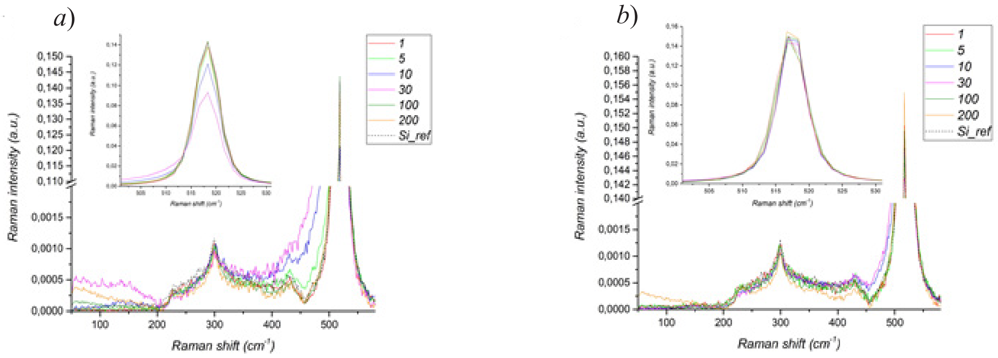

Fig. 3. Normalized Raman spectra of FIB-modified Si areas at different beam passes after annealing at 600 °C (*a*) and 800 °C (*b*)

Conclusion

As can be seen from the above results, the annealing temperature and the amount of implanted material critically determine the depth and crystallinity of the modified silicon regions. An increase in the annealing temperature led to an increase in the depth of the treated areas, as well as an increase in their crystallinity due to the etching of the amorphous layer. We concluded that the appropriate annealing temperature is annealing at 600 °C due to the incomplete removal of the amorphous phase and the presence of an implanted material on the surface, which will act as nucleation centers for subsequent growth. We believe that the growth of III-V structures on such FIB-modified substrates will lead to complete localization of the structure, and potentially such a solution will help achieve high-quality monolithic integration of III-V structures on silicon substrates.

REFERENCES

1. **Park J.-S., Tang M., Chen S., Liu H.,** Heteroepitaxial Growth of III-V Semiconductors on Silicon, Crystals. 10 (2020) 1163.

2. **Kunert B., Mols Y., Baryshniskova M., Waldron N, Schulze A., Langer R.,** How to control defect formation in monolithic III/V hetero-epitaxy on (100) Si? A critical review on current approaches, Semiconductor Science and Technology. 33 (2018) 093002.

3. **Kroemer H.,** Polar-on-nonpolar epitaxy, Journal of Crystal Growth. 81 (1987) 193–204.

4. **Chen S., Li W., Wu J., Jiang Q., Tang M., Shutts S., Elliott S. N., Sobiesierski A., Seeds A. J., Ross I., Smowton P. M., Liu H.,** Electrically pumped continuous-wave III–V quantum dot lasers on silicon, Nature photonics. 10 (2016) 307–311.

5. **Lee A. D., Jiang Q., Tang M., Zhang Y., Seeds A. J., Liu H.,** InAs/GaAs Quantum-Dot Lasers Monolithically Grown on Si, Ge, and Ge-on-Si Substrates, IEEE Journal of Selected Topics in Quantum Electronics. 19 (4) (2013) 1901107.

6. **Li K., Liu Z., Tang M., Liao M., Kim D., Deng H., Sanchez A. M., Beanland R., Martin M., Baron T., Chen S., Wu J., Seeds A., Liu H.,** O-band InAs/GaAs quantum dot laser monolithically integrated on exact (001) Si substrate, Journal of Crystal Growth. 511 (2019) 56–60.

7. **Georgakilas A., Stoemenos J., Tsagaraki K., Komninou Ph., Flevaris N., Panayotatos P., Christou A.,** Generation and annihilation of antiphase domain boundaries in GaAs on Si grown by molecular beam epitaxy, Journal of Materials Research. 8 (8) (1993) 1908–1921.

8. **Alcotte R., Martin M., Moeyaert J., Cipro R., David S., Bassani F., Ducroquet F., Bogumilowicz Y., Sanchez E., Ye Z., Bao X. Y., Pin J. B., Baron T.,** Epitaxial growth of antiphase boundary free GaAs layer on 300 mm Si(001) substrate by metalorganic chemical vapour deposition with high mobility, APL Materials. 4 (2016) 046101.

9. **Bolkhovityanov Yu. B., Pchelyakov O. P.,** GaAs epitaxy on Si substrates: modern status of research and engineering, Physics – Uspekhi. 51 (5) (2008) 437–456.

10. **Detz H., Kriz M., Lancaster S., MacFarland D., Schinner M., Zederbauer T., Andrews A. M., Schrenk W., Strasser G.,** Lithography-free positioned GaAs nanowire growth with focused ion beam implantation of Ga, Journal of Vacuum Science & Technology B. 35 (2017) 011803.

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