Selective area epitaxy of $n^+$-GaN layers on SiO$_2$ patterned GaN/c-$\text{Al}_2\text{O}_3$ templates by PA MBE

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Abstract. The $n^+$-GaN epilayers were synthesised by PA MBE on the SiO$_2$ patterned GaN/c-$\text{Al}_2\text{O}_3$ templates, grown by MOCVD. Formation of the polycrystalline GaN atop of the SiO$_2$ mask during PA MBE was observed. It was found that macroscopic voids at the interface polycrystalline GaN/SiO$_2$/n-GaN template appeared during the PA MBE process. The polycrystalline GaN film was completely removed by etching in hot aqueous KOH solution. Hall measurements have shown that the value of electron concentration in $n^+$-GaN contact layer is about $n_e=4.6\times10^{19}\text{cm}^{-3}$.

1. Introduction
Wide bandgap semiconductors, such as gallium nitride (GaN), play a special role in a progress of modern electronics. Because III-nitrides have a set of unique structural, electrical, optical and mechanical properties, devices, based on GaN are successfully implemented in optoelectronics and have a great perspective for the high-power and high frequency electronic applications [1].

At the same time, there are several serious obstacles for further development of the nitride electronics. The first is a well-known problem, which is connected with the lack of low-cost natural substrates for GaN. So, silicon carbide (SiC), sapphire (c-$\text{Al}_2\text{O}_3$) and silicon (Si (111)) substrates are typically used for epitaxial growth of GaN. Heteroepitaxial growth of the material on the substrates with lattice mismatch leads to the formation of structural defects such as threading dislocations, inversion domains, etc. Another well-known challenge for further development of modern nitride electronics is the formation of the low resistance ohmic contacts [2]. The contacts are usually formed by the rapid thermal annealing of the sputtered contact material, so the morphology of the contact pad may suffer from high temperature annealing [3]. Nowadays a special attention is paid to the study of non-alloyed ohmic contacts. They can be formed on the heavily doped $n^+$-GaN contact layers, synthesized by selective area growth (SAG) [4, 5].

Here we report on the results of the studies of $n^+$-GaN contact layer synthesis by plasma-assisted molecular beam epitaxy (PA MBE) on SiO$_2$ patterned GaN/c-$\text{Al}_2\text{O}_3$ templates.

2. Experimental details
In this work the GaN layers, grown on c-$\text{Al}_2\text{O}_3$ substrate by metalorganic chemical vapour deposition (MOCVD) were used as templates. After the MOCVD growth the template surface was patterned with SiO$_2$ mask about 160 nm thick, formed by plasma-enhanced chemical vapour deposition (PECVD)
and standard photolithography process. Thereafter the preliminary selective MOCVD growth of GaN layer in the window openings of SiO$_2$ mask was carried out.

Prior to the PA MBE growth several preliminary actions were made. The backside of c-Al$_2$O$_3$ substrate was covered with Ti film by e-beam vacuum deposition. Then the template was cleaned with organic solvents CCl$_4$ and isopropyl alcohol and rinsed in deionized water. In order to remove excess water from the template surface, a preliminary annealing at T $\approx$ 200°C was carried out in the UHV load lock chamber of PA MBE setup. After that, in-situ pre-epitaxial cleaning of GaN/c-Al$_2$O$_3$ template surface of atoms adsorbed from the atmosphere (O, C etc.) was performed. The details of pre-epitaxial cleaning of the template surface were previously discussed [6]. Finally, the PA MBE selective regrowth of the upper n$^+$-GaN contact layer was carried out using Veeco Gen 200 setup. The schematic view of the obtained sample is shown in figure 1.

The grown sample was studied using scanning electron microscopy (SEM) and Hall measurements. The polarity of the sample was identified by etching in hot aqueous KOH solution [7].

3. Results and discussion
The study of the synthesized sample by SEM has shown that the n$^+$-GaN contact layer, grown on the template GaN/c-Al$_2$O$_3$ patterned with SiO$_2$ mask, has inhomogeneous morphology and there are 3 different areas (figure 2a). The first consists of n$^+$-GaN layer, grown on the template in SiO$_2$ mask window openings. It demonstrates quite homogeneous surface morphology and smooth transition from MOCVD template to the n$^+$-GaN contact layer, grown by PA MBE (figure 2b). The second area is the edge of the mask window. It was observed, that after the SiO$_2$ mask formation, the GaN layer formation occurred only at a distance of 15-17 µm from the mask (figure 2a). Finally, the third area consists of polycrystalline GaN (poly-GaN) layer, which was formed atop of the SiO$_2$ mask (figure 2c). However, some another works demonstrate, that the using of SiO$_2$ avoids GaN formation in the mask region [8, 9]. Moreover, it was found that the SiO$_2$ mask was destroyed during the PA MBE process, so multiple macroscopic voids were formed at the interface n-GaN/SiO$_2$/Poly-GaN and Poly-GaN layer was partially suspended (see figure 2c). It was suggested, that formation of the voids was caused by etching the mask either with Ga droplets due to high solubility of Si in Ga which takes place at a temperature T>400-650°C in ultra-high vacuum [10] or with high energy activated nitrogen species [11].
Figure 2(a, b, c). (a) cross-sectional SEM image of the grown sample. 1 - n⁺-GaN layer grown directly on the template surface, 2 - the edge of the mask window, 3 - poly-GaN atop of SiO₂ mask; (b) larger SEM image of the region 1; (c) larger SEM image of the region 2.

Then the sample was chemically etched in hot (70°C) aqueous KOH solution for 5 min in order to identify the crystallographic polarity of the GaN layer. During this process the polycrystalline GaN layer was slightly etched (figure 3b), thus it was decided to continue etching. After 10 min etching in hot KOH solution the poly-GaN was completely removed from the sample surface and the remains of SiO₂ mask were found (figure 3c). At the same time, it was found that the n⁺-GaN contact layer, formed by PA MBE, and the MOCVD template morphology remained smooth. Thus, they demonstrate Ga-polarity. Hexagonal etch pits, which were found on the GaN template surface (figure 3b, c), indicate the presence of dislocations. The studies of the grown sample by Hall measurements demonstrated that the value of electron concentration in n⁺-GaN contact layer is about $n_e \approx 4.6 \times 10^{19} \text{ cm}^{-3}$. 
4. Conclusion
In this work the results of the studies of n+-GaN contact layer synthesis by SAG on SiO$_2$ patterned GaN/c-Al$_2$O$_3$ templates are presented. The n’-GaN epilayers were synthesised by PA MBE on the templates, grown by MOCVD. The formation of the polycrystalline GaN atop of the SiO$_2$ mask during PA MBE was observed. It was found that SiO$_2$ mask can be partially destroyed during the PA MBE process with the consequent formation of the multiple macroscopic voids at the interface poly-GaN/SiO$_2$/MOCVD-GaN. The polycrystalline GaN film was completely removed by etching in hot aqueous KOH solution. Hall measurements have shown that the value of electron concentration in n’-GaN contact layer is about $n_e \approx 4.6 \times 10^{19}$ cm$^{-3}$.

Acknowledgments
This work is supported by the grant of the Ministry of Education and Science of the Russian Federation № 16.9789.2017/BCh and Skoltech (agreement no. 3663-MRA, project №4).

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