Optimizing Structured SiN-masks for Self Separation of Full 2"-GaN Wafers by Hydride Vapor Phase Epitaxy

Martin Klein

Using a previously shown method, we prepared 2"-GaN wafers as templates for a self separation process. Self separation is happening during cooldown after growing thick layers of GaN in our hydride vapor phase epitaxy (HVPE) reactor. Our templates consist of GaN grown by metalorganic vapor phase epitaxy (MOVPE) directly on sapphire. These GaN layers are masked with 200 nm of SiN that are structured by means of optical lithography. They are subsequently overgrown with a thin GaN layer by metalorganic vapor phase epitaxy (MOVPE). Previous experiments found that the ideal interlayer for self separation during cooldown in HVPE is created when using a hexagonally shaped pattern as mask. We now tried several possible variations of this pattern to find the optimal geometrical proportions supporting the desired self separation.

1. Introduction

Becoming a more and more common material, galliumnitride (GaN) still suffers from the lack of suitable substrates. This leads to the need for heteroepitaxy in mid and low price applications. Although homoepitaxy is used for producing blue lasers, available GaN substrates are much too costly for growing LEDs and transistors. To achieve affordable homoepitaxial substrates, great efforts have been made in the field of ammonothermal growth [1,2]. Yet thick layers grown by HVPE are still a good candidate to provide GaN wafers for future industrial processes and this technique is under constant research. Nevertheless this is still not a trivial task. Differences in the thermal expansion coefficients of GaN and sapphire result in big layer curvatures and the large mismatch in lattice constants leads to strain and defects in the GaN layer. In order to cope with these problems, several methods have been developed to remove the GaN layer from the substrate such as laser-lift-off (LLO) [3], mechanical polishing [4] or growth on etchable substrates like GaAs [5] and ZnO [6]. Another approach is the usage of interlayers to weaken the material bond at a defined position. This technique can be used to generate a predetermined breaking point at which the layer separates from the template. During process cooldown the difference in thermal expansion coefficients of the sapphire substrate and the GaN layer leads to a large strain at the interface. The forces inherited in this procedure result in the separation of the GaN layer from the substrate at the predetermined position. Our institute has been working on developing this technique for quite some time. This lead to a standardized procedure for creating working breaking point layers [7].

A substantial part of developing such a layer is finding a good compromise between top layer quality and easy separation of the GaN layer from the substrate. SiN masks have proven to be a good starting point to meet these requirements. First, SiN is deposited by means of plasma enhanced chemical vapor deposition (PECVD) and subsequently structured by optical lithography and reactive ion dry etching (RIE) technique. These masks are overgrown by MOVPE to create a starting layer for HVPE growth, where subsequently thick GaN layers are grown on top of these templates. During this last growth-step, the thin SiN layer dissolves. This is a result from the harsh process conditions in HVPE growth. The dissolved mask material etches the underlying GaN which has been deposited in MOVPE. This leaves a porous interlayer that promotes separation at this exact position. Self separation finally occurs during cooldown of this HVPE process. Up to date self separation can be achieved with high repeatability. Nevertheless the goal to create full 2" freestanding wafers has only been reached by chance, most wafers are cracking into pieces during separation. This is the motivation to find new mask designs which specifically address this problem.

2. Experimental

In our latest investigations we created various new hexagonal mask designs in order to find the influence of the masked area and the trench width on material growth and separation. The findings of our previous studies were used as starting values.

| Mask name | Pattern type | Trench width | Period |
|------------|-------------------|--------------------|-------------------|
| $HexFL_v2$ | hexagon | $1.5\mu{ m m}$ | $30\mu\mathrm{m}$ |
| $HexFL_v4$ | hexagon | $2.5\mu{ m m}$ | $30\mu\mathrm{m}$ |
| $HexFL_v1$ | hexagon | $3.0\mu{ m m}$ | $30\mu{ m m}$ |
| $HexFL_v5$ | hexagon | $4.5\mu\mathrm{m}$ | $30\mu{ m m}$ |
| $HexFL_v3$ | hexagon | $1.5\mu{ m m}$ | $40\mu\mathrm{m}$ |
| HexFL_v6 | separated hexagon | $3.0 \mu m$ | $39 \mu m$ |

Table 1: Geometries of separation masks.

Table 1 shows the geometrical details of the used masks. For most of the masks there was no change in the principal structure of the hexagonal pattern compared to earlier studies [7]. This approach is shown on the left side of Fig. 1. In contrary to this approach, the structure shown on the right side of this figure shows a completely new idea. With this pattern, consisting of separated hexagons, we intended to investigate the influence of the connections between the hexagons in our conventional mask.

For overgrowing these templates we used our commercial Aixtron single-wafer HVPEsystem with a horizontal quartz-tube. The system is heated by a five zone furnace which enables to set a temperature of 850 °C in our source zone and 1050 °C in the growth zone. In the source zone, GaCl is formed by streaming HCl over a bath of liquid gallium.



Fig. 1: Structures of connected and separated hexagons.

Ammonia is used as nitrogen precursor. These two gas streams are carefully adjusted to meet optimal growth conditions on the supplied template residing in the growth zone. As carrier gases we use a 1:1 mixture of H_2 and N_2 . The V/III-ratio is in the range of 80 - 90. The reactor pressure is set to 900 hPa.

At first the templates were overgrown by about 70 μ m of GaN with a fairly low growth rate of 35 μ m/h. In this first step we optimized growth conditions for lateral growth, thereby promoting a fast coalescence of the GaN surface. After this first step we investigated the resulting layers to find differences in material quality, depending on the mask. Next we grew another 12 hours with a growth rate of about 85 μ m/h. The goal was to get a freestanding GaN layer of about 1 mm.

These freestanding samples have been investigated by means of x-ray diffraction (XRD) and photoluminescence (PL) to compare the mask designs and to confirm the suspected good quality. Additionally we conducted an etch pit density (EPD) experiment to verify our data gathered by these studies.

In a second experiment, we varied the thickness of the structured SiN layer to see if we can create a weaker bond between the template and the layer. This weaker bond could result in a more homogenous separation of the top layer.

3. Results and Discussion

3.1 Mask overgrowth

As described in the previous section, we stopped growth after a layer thickness of about 75 μ m to investigate the overgrowth of our masked templates. Figure 2 shows microscopic pictures of the surfaces of the resulting layers in Nomarski mode with a magnification of 500. All samples show a flat surface with no significant differences.

| HexFL_v2 (30 μ m, 1.5 μ m) | $\mathbf{HexFL_v4}~(30\mu\mathrm{m},2.5\mu\mathrm{m})$ | HexFL_v1 (30 μm, 3.0 μm) |
|---|--|---------------------------------|
| | | |
| | | |
| | / | |
| HexFL_v5 (30 μm, 4.5 μm) | $HexFL_v3$ (40 µm, 1.5 µm) | HexFL_v6 (Sep. hexagons) |
| HexFL_v5 (30 μm, 4.5 μm) | HexFL_v3 (40 μm, 1.5 μm) | HexFL_v6 (Sep. hexagons) |

Fig. 2: Layer surface after growing $75 \,\mu\text{m}$ of GaN. Pictures from optical Nomarski microscope with a magnification of 500.



Fig. 3: Layer surface with pits after growing $75 \,\mu\text{m}$ of GaN. Pictures from optical Nomarski microscope with a magnification of 25. No such image has been made from mask HexFL_v1.

At a lower magnification of 25 some pits are visible on every sample as seen in Fig. 3. These result from defects in the mask or in lithographic structuring. For structure HexFL_v3 we experience a bad coalescence. Obviously, the mask coverage was too large. On the other hand, the sizes of the mask trenches seem not to affect the mask overgrowth. We noticed that the density of such defects increases with the distance from the wafer center, regardless of the mask pattern.

3.2 Self separation



Fig. 4: Photographs of the final, separated layers. Freestanding GaN with about 1 mm thickness.

After these midgrowth investigations, we did a second HVPE overgrowth of these templates to achieve a total layer thickness of 1 mm. Photographs of the resulting layers are shown in Fig. 4. No separation took place at the intended position for mask design HexFL_v6. With the separation layer not working, forces are still too strong for the toplayer to be stable during cooldown. This results in separation of pieces by cracks in the toplayer. The same behaviour holds true for the HexFL_v5 mask with 4.5 μ m trenches. In case of the separated hexagons, the assumption is that when separation starts at one place of the weak layer it stops before reaching the next weak spot. With the 4.5 μ m trenches the bond between template and layer generally seems to be too strong for separation at this position.

The other extreme can be found in pattern HexFL_v3. With trenches of only $1.5 \,\mu\text{m}$ and a large period of $40 \,\mu\text{m}$, the connection between template and layer is too weak. So separation begins at different places at the same time leading to big strain inhomogenieties in the layer. As a result, the freestanding top layer bursts into tiny pieces during separation. By using a mask coverage lying between those values the separation process seems to be fairly stable. This results in the separation of big chunks, which can include the whole wafer diameter and consist of up to one third of the total top layer surface.

3.3 SiN thickness variation

In addition to investigating different mask patterns we tried to vary the thickness of our structured SiN layer. Whereas our standard process is based on a SiN thickness of 200 nm, we fabricated samples with a SiN thickness of 400 nm and 800 nm for this study. Figure 5 shows microscopic pictures of the surface of the fully processed templates. The structure with 400 nm SiN layer has a perfectly developed MOVPE GaN layer with almost no



Fig. 5: Microscopic images of structured and overgrown SiN with $400 \,\mathrm{nm}$ and $800 \,\mathrm{nm}$ SiN layer thickness.

mask defects. In contrast some GaSi alloy developed on the 800 nm mask during MOVPE growth. With these alloys residing on the mask this template is not suitable for HVPE growth.

When overgrowing the 400 nm mask, we also faced severe problems. The resulting layer showed many pits and although the mask pattern works well with a 200 nm SiN layer this new sample didn't show proper separation. The reason for this behaviour hasn't been found yet.

3.4 Layer quality

For determining the quality of our layers we conducted several measurements on the resulting freestanding samples. In Fig. 6 the results of a full sample x-ray diffraction (XRD) map of the (002)-reflection is shown. The diagonal of the shown piece is spanning the whole wafer diameter. Thus the resulting values for the (002) full width at half maximum (FWHM) seem to be extremely low and homogenous over the whole wafer. We also achieved comparatively low bow values in the range of 160 km^{-1} . These values seem to depend on the size of the investigated piece, so further studies on this subject have to be done in the future. Comparing the various mask patterns no significant differences could be found in XRD measurements. All samples show extremely good layer quality.

PL investigations also indicate an excellent material quality as seen in Fig. 7. Many sharp peaks can be distinguished. Among these the Si and O donor bound exciton transition could be identified with very extremely narrow linewidths in the range of 500 μ eV. Samples grown on different masks show comparable spectra.



Fig. 6: XRD quality map of a big freestanding sample.



Fig. 7: Low temperature bandedge PL of a freestanding sample.

An etch pit density (EPD) investigation carried out on a sample piece, previously etched by molten KOH, shows a value of $5.5 \cdot 10^5 \,\mathrm{cm}^{-2}$ which confirms the outstanding layer quality.

4. Conclusion

Several new mask patterns for self separation processes during cooldown in HVPE have been tested and the geometrical limits of these patterns for a successful self separation have been found. Within fairly large boundaries, the different masks did not lead to significantly different results. It could also be shown that a thicker dielectric layer is ineffective for the self separation process. Finally the homogenous and excellent quality of thick HVPE GaN layers has been proven.

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