Influencing the Bow of Thick Hydride Vapor Phase Epitaxial GaN by Prestraining MOVPE Templates

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A major challenge that arises when growing GaN substrates in a single run process is the remaining curvature of the freestanding material after the removal of the foreign substrate. For a long time the dislocation density gradient has been suspected to be the cause. However, by conducting etching experiments, we have found that this cannot be the only reason. We postulate that the initial strain situation of the MOVPE template can have a big influence on the bowing of HVPE samples and on the remaining curvature of separated GaN layers. By investigating accordingly grown samples, we present proof of this assumption.

1. Introduction

GaN has gained a very significant share in the semiconductor market over the last years. Applications extend from high-power, high-frequency transistors [1] and solid state lighting by GaN-based light emitting diodes (LEDs) [2] to laser diodes in the blue and ultraviolet emission regime [3]. Most of these devices are epitaxially grown on foreign substrates such as sapphire, silicon-carbide and silicon. By this heteroepitaxial approach defect densities in the range of $10^8\,\mathrm{cm}^{-2}$ can be achieved [4]. Although the number of defects is quite high, this method is most commonly used for LEDs and transistors because of its low cost and high availability. In laser diodes, however, high defect densities result in short lifetimes and low efficiencies, so that low defect density GaN substrates are urgently needed [5].

A quite simple approach to produce GaN substrates is using hydride vapor phase epitaxy (HVPE) to grow thick layers. These layers are subsequently separated from the substrate. A typical layer thickness of freestanding GaN by HVPE is 1 mm, which can be reached in about 10 hours of growth. In order to grow such layers in HVPE, foreign substrates are needed, so that many problems connected with heteroepitaxy still arise. During the growth of thick layers, nearby dislocations may annihilate each other. By this effect defect densities on the surface of the thick GaN layer in the range of $10^6\,\mathrm{cm}^{-2}$ can be achieved when growing on templates inheriting defect densities in the $10^8\,\mathrm{cm}^{-2}$ regime [6]. When we reach about 1 mm of layer thickness, we stop the growth and start to cool down the reactor. During this cool-down, great stress forces build up in the wafer due to different thermal expansion coefficients of layer and substrate. By carefule usage of these forces and a specially designed interlayer close to the sapphire substrate, we manage to separate the GaN layer from the substrate [7].

A more severe problem arising with heteroepitaxy, is the curvature of the wafer due to differences in the thermal expansion coefficients of substrate and grown layer. With the GaN layer removed from the substrate one would expect that the layer is now totally relaxed and no bowing remains. In fact photo luminescence measurements confirm that the material is macroscopically relaxed. However these layers still experience some bowing. In [6] F. Lipski and F. Scholz showed that this bowing can't be caused by a gradient of the dislocation density in the material alone. Instead they postulated that the strain situation created at the beginning of the growth of the template could have a great influence on HVPE growth and the bowing of still attached and separated thick GaN layers. During the last year we conducted several growth runs to prove this postulation and to optimise the template conditions to obtain layers with lower curvature values.

2. Prestraining MOVPE Templates

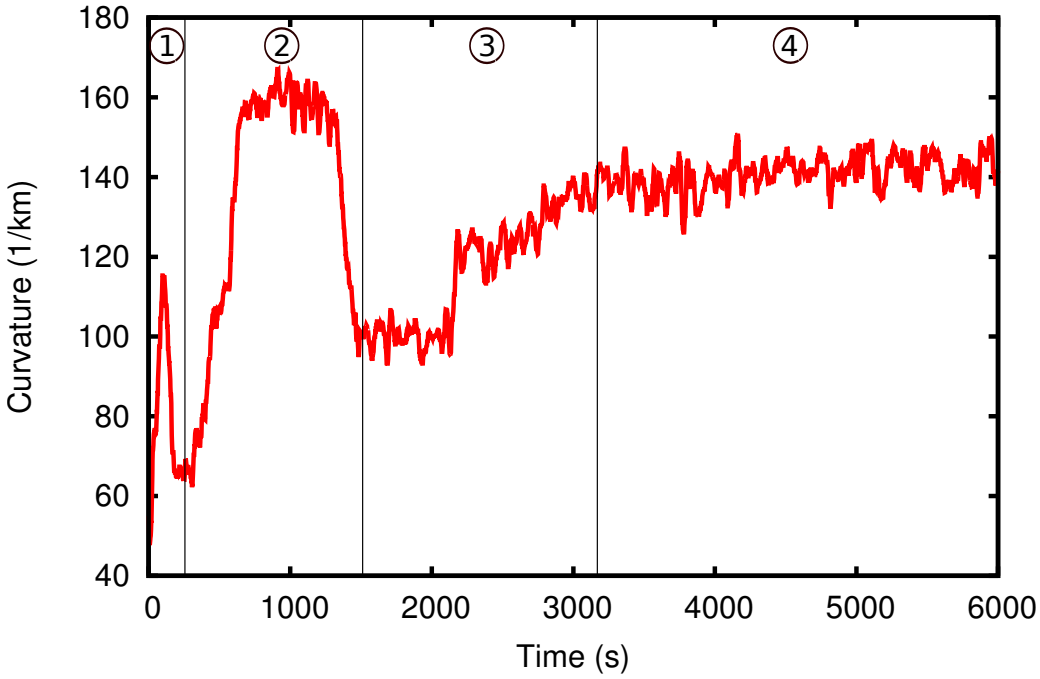

Fig. 1: *In-situ* curvature measurement during MOVPE growth. Sections 1–4 contain different growth steps.

When comparing the remaining bow of freestanding samples with the initial bow of the templates that have been used to grow these layers, no obvious connection can be seen. However, we found some evidence that the nucleation layer and some interlayers can have an influence on the strain of the layer grown on top. To control this strain situation we used a combination of our oxygen-doped aluminium–nitride (AlN) nucleation layer and a submonolayer of SiN_x, normally used for defect reduction. We figured out that by varying the thickness of the GaN buffer between those layers, it is possible to directly influence the strain situation. This could be confirmed by photoluminescence (PL) experiments [4]. The

peak with the highest energetic position could be found when we deposit the SiN_x directly on top of the nucleation layer, marking the compressive end of the possible strain created by this three-layer system. The thicker we grow the GaN buffer between nucleation and SiN_x layer, the lower the PL emission energy, indicating that the strain is being moved towards a more tensile state [4].

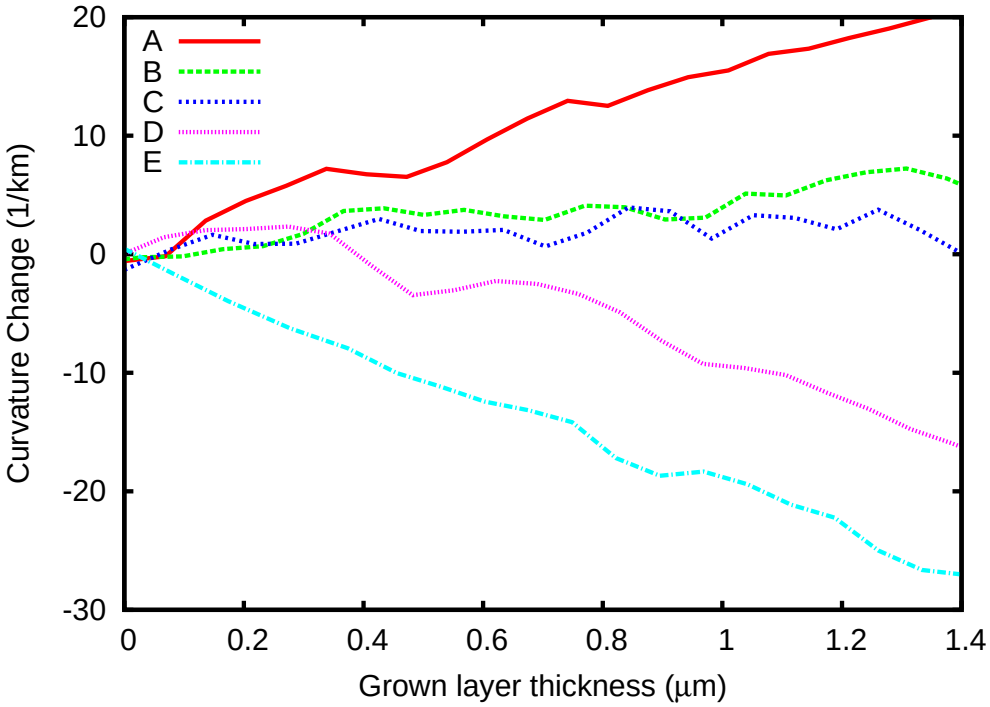

Fig. 2: *In-situ* curvature measurement in MOVPE. Curves A–E have been measured during template growth runs with different growth parameters.

Our metal organic vapor phase epitaxy (MOVPE) machine is equipped with a LayTec Epicurve to monitor curvature during growth. With this tool we can determine the wafer curvature by measuring the distance of two reflected laser spots. Therefore we see the influence of process changes already during growth. Figure 1 shows a typical *in-situ* curvature measurement curve, taken during one of our template runs in our MOVPE. In section **1** we see that the wafer already has some initial curvature. This is a statistical value due to the sapphire fabrication process, and some curvature at this point normally can't be avoided. Subsequently we see a spike in section **1** which is a measurement artifact caused by pumping and purging the reactor. In section **2** the sapphire is heated up in a hydrogen ambient for thermal cleaning. After cooling down to growth temperature we deposit the nucleation layer, a GaN buffer and the SiN_x interlayer in section **3**, before we create a closed and flat layer in section **4**. For more compressively strained layers we observe a downward curve in this section, so the curvature develops towards a convex shape. For more tensile strained layers the curve goes upward, developing a more concave shape. We believe that for freestanding layers the best situation is when the curvature doesn't change at all, therefore our goal was to find the right buffer layer thickness to

achieve this state.

Figure 2 shows a more detailed picture of section **4** from Fig. 1 from some different template runs. Sample A includes no SiN_x interlayer. Therefore this curve marks the most tensile strain state that can be achieved by this method. Even higher tensile strains can be achieved by using a GaN-based nucleation layer instead of our AlN-based nucleation. Sample B has been taken during growth of our standard process, optimised for a low etch pit density (EPD). This sample includes a 300 nm thick buffer layer between nucleation and SiN_x interlayer. Sample C has a thinner GaN buffer and is close to be perfectly optimized for further HVPE growth, almost no change of the curvature value can be seen. If we reduce the buffer layer further, we obtain a curve as shown for sample D. This sample already shows a quite compressive behaviour. Leaving the buffer layer out entirely, we receive the curve named sample E, which marks the most compressive strain state possible with the combination of oxygen-doped aluminium–nitride nucleation layer and a sub-monolayer of SiN_x as an interlayer.

2.1 Influence on thin HVPE layers

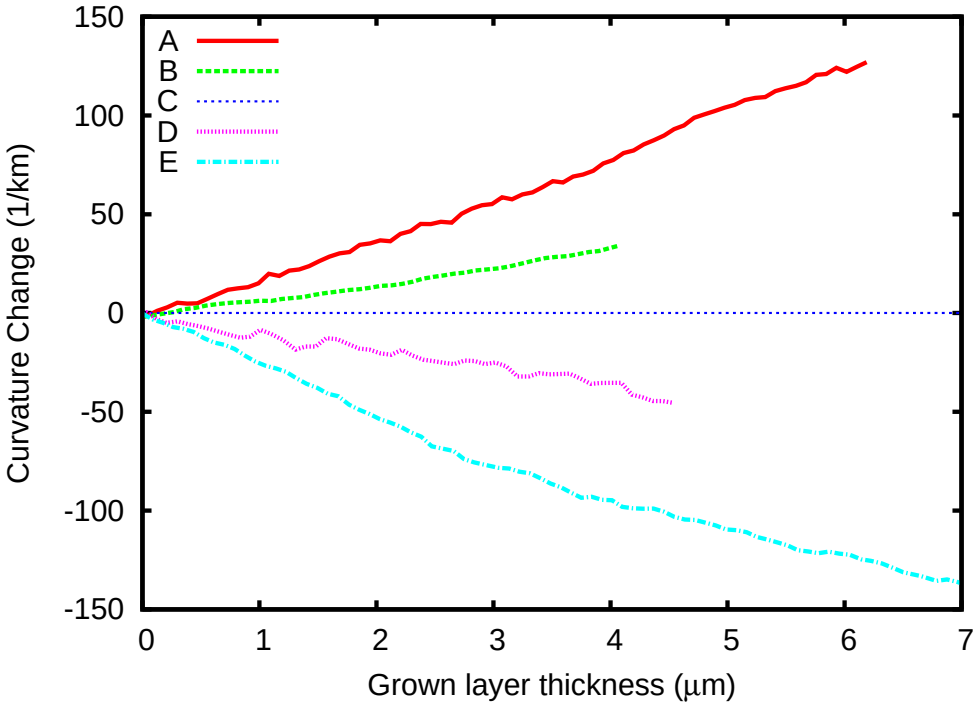

Fig. 3: *In-situ* curvature measurement like in Fig. 2 but during overgrowth in HVPE. Samples A–E correspond to the samples in Fig. 2.

The samples shown in Fig. 2 have been overgrown by 65 µm of GaN in HVPE. The *in-situ* curvature measurements of the first micrometers of growth of these HVPE runs can be seen in Fig. 3. Unfortunately we don't have any real data for curve C as the *in-situ* measurement was not available in this run. For all other samples we can see that the

curvature evolution during MOVPE and HVPE growth is more or less the same for a given sample.

Next we measured the room temperature curvature of the resulting wafers. In order to get these values, x-ray diffraction (XRD) rocking curve scans have been taken at several positions along the wafer diameter. The curvature can then be calculated from the distance between the measurement spots and the difference in the respective rocking curve angles. Our measurement results are shown in Fig. 4. The ordering of the samples is as we would expect from the *in-situ* measurements. The sample with the strongest tensile development shows the smallest curvature, whereas the sample with the strongest compressive development shows the largest curvature. As some edge effects seem to influence the curvature of our wafers we always take center value as figure of merit. In case of sample A the measurement range is significantly shorter than for all other samples. Because of the strong tensile strain in the GaN layer, the overall stress became too strong during cool-down and the wafer cracked at the edge several times.

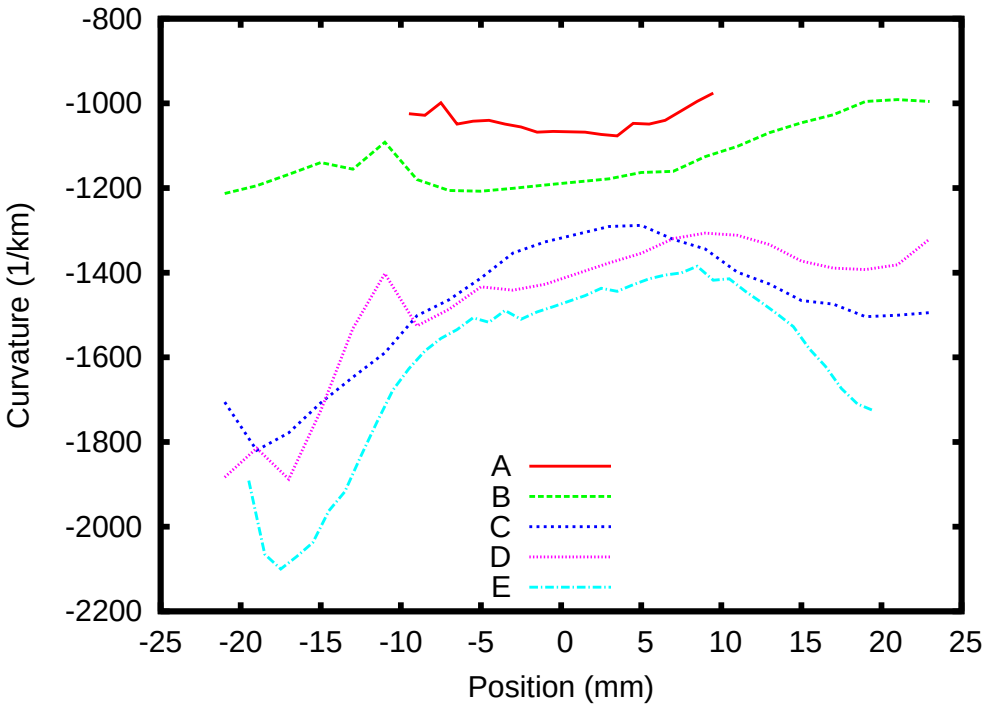

Fig. 4: Room temperature *ex-situ* curvature measured by x-ray diffraction along the wafer diameter of 65 µm thick HVPE samples. Samples A–E correspond to the samples in Fig. 2 and Fig. 3.

2.2 Influence on freestanding GaN layers

In order to investigate the influence of strain-optimized samples on self-separated layers, we grew a set of freestanding samples with old and new templates, stopping growth at different thicknesses for self-separation. The curvature values of former samples with EPD

optimized growth steps are shown as squares in Fig. 5. We can see that for these samples the curvature is highly dependent on the layer thickness. The reason herefore is given in the next paragraph. The triangles are marking the curvature values of the curvature optimised templates at growth temperature. Next to the triangles we find the curvature values of the corresponding freestanding samples, represented by light dots. The small curvature difference of template and freestanding layer confirms that the curvature is not undergoing big changes during HVPE growth.

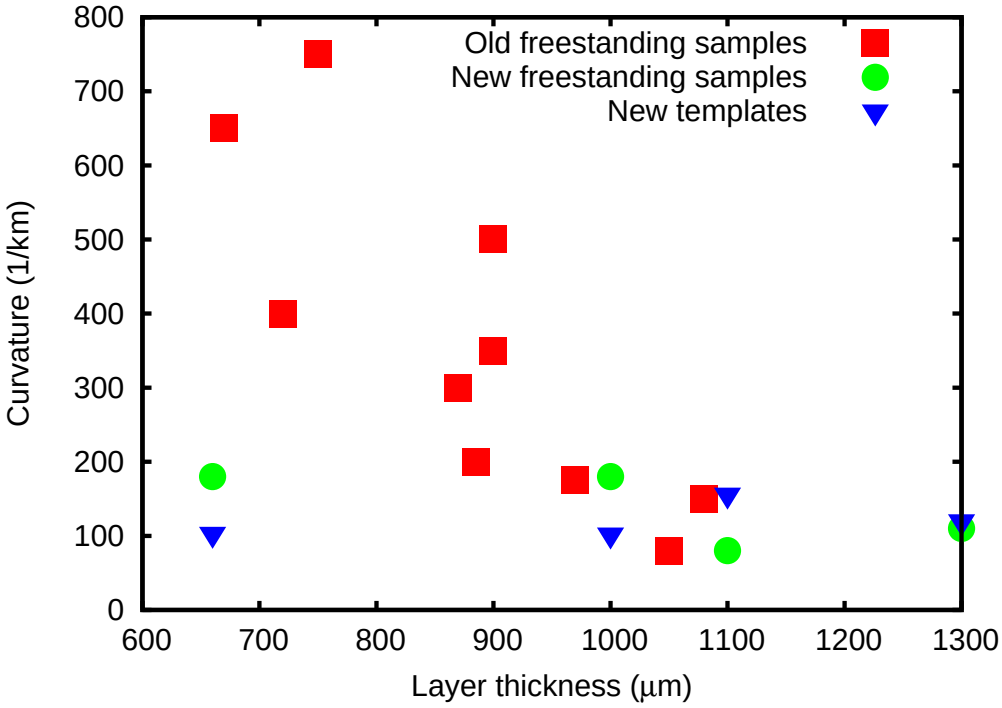

Fig. 5: Remaining curvature of self-separated HVPE, samples separated at different thicknesses.

Templates optimised for low EPD values are generally tensile strained. This leads to a growing curvature in the beginning of HVPE growth. The curvature value increases up until the GaN has about the same thickness as the sapphire substrate underneath. When we grow thicker than this, the curvature gets reduced again. When we grow on a bent surface the material would need to to change its strain state in order to reproduce the same curvature in the next monolayer. Instead the material produces a certain low amount of new dislocations to keep the former strain. By this mechanism some curvature is frozen into the material. When we now separate the layer at a certain thickness, this frozen curvature remains in the material. To reduce the remaining bow one could growth thicker layers before separating. But if we grow significantly thicker than 1 mm, the material is not separating at the intended position. Instead, separation occurs roughly 200 µm above the separation layer. Therefore it is not possible to reduce the curvature to zero with these templates. As for the strain optimized samples, we are able to set the remaining curvature by influencing the curvature of the template at growth temperature, regardless of the thickness during separation. A simple approach to achieve this would be by using

uncurved or intentionally curved sapphire wafers, which inherit a concave curvature rather than the usual convex curvature. Fortunately, a method has been published recently on how to create such sapphire substrates [8] and we are planning to grow samples on such substrates in the near future.

3. Conclusion and Outlook

A big problem that still remains when producing GaN wafers in a single wafer process is the remaining curvature of the separated layers. In the bygone year we were able to establish a template process that holds out the prospect of effectively handling this problem. When using our former standard templates, the curvature during growth is dependent on the layer thickness. By optimising the growth sequence in the first stage of our MOVPE process, we were able to stop the development of the wafer curvature in subsequent growth steps. This also holds true for a subsequent overgrowth in an HVPE machine. After establishing this template process we investigated the HVPE growth on templates with different values for the change of the curvature during the process. In these experiments we got low curvature values for all separated layers grown on our new templates, regardless of the thickness of the separated layer. For all other samples, the remaining curvature depended on the thickness of the separated layer and in most cases significantly larger than the remaining curvature of layers grown on optimised templates.

In the near future we intend to reduce the remaining curvature in separated layers by reducing the initial curvature of our MOVPE templates. A very promising parameter to start with is the initial curvature of the sapphire substrates. Only recently a company has developed a process to influence the initial curvature of sapphire wafers [8], so that corresponding substrates are now available for scientific purposes.

Acknowledgment

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