Freestanding GaN-substrates and devices

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Various physical aspects and potential applications of the laser-induced separation of GaN epilayers from their sapphire substrate are reviewed. The effect of short laser pulses on the thermal decomposition of GaN and possible applications of the laser-induced dissociation of GaN for fast etching of this material is discussed. Particular emphasis is placed on the defect-free delamination of large area GaN films with thicknesses ranging from 3 to 300 µm from sapphire substrates. The use of the resulting freestanding GaN films in device technology and homoepitaxy of III-nitrides are outlined. Specific examples are the flip-chip bonding of freestanding InGaN/GaN LEDs to a silicon submount and the production of pseudosubstrates for the homoepitaxy of high quality GaN epilayers.

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1 Introduction

Compared to well established semiconductor materials systems such as Si/Ge or GaAs/AlGaAs, some of the most challenging aspects of GaN-based thin-film technology come from the fact that the most common substrate materials used today (sapphire and SiC) have very different properties than the device layer itself. In contrast to the majority of modern semiconductors, present commercial GaN–based devices are exclusively prepared by heteroepitaxy onto foreign substrate materials. Surprisingly, the ensuing dislocation densities in the range of $10^7$ to $10^8$ cm$^{-2}$ did not prevent the commercial success of III-nitrides for light-emitting devices in the visible to ultraviolet spectral range. However, it is now widely accepted that the ultimately possible performance of GaN-based devices can only be reached by the use of homoepitaxy onto bulk GaN or AlN substrates. Unfortunately, despite many years of intense research, the high equilibrium pressures and temperatures of the III-nitrides so far have rendered the growth of bulk substrates for homoepitaxy with production grade sizes and quality impossible.

An alternative approach, which has been pursued since a couple of years, is the use of thick GaN layers grown by hydride vapour phase epitaxy (HVPE) on sapphire. These thick layers are subsequently removed from the sapphire substrate by a suitable process and then can serve as homosubstrates for device fabrication. The purpose of this review article is to summarize the present state of research concerning such freestanding GaN homosubstrates, with special emphasis on laser-based lift-off processes for the separation of the thick GaN-layers from their sapphire substrates. The review is organized as follows: after a brief resumé of the various substrates used for heteroepitaxy so far, we will discuss in detail the physical and technological aspects of the laser lift-off (LLO) processing for the production of freestanding GaN substrates and devices. In the final section of this article, the surface preparation of laser-separated HVPE-layers and typical results obtained by subsequent homoepitaxy will be addressed. For a
more detailed discussion of the growth of III-nitrides on heterosubstrates such as sapphire, GaAs, or Si
the reader is refered to other articles in this volume.

2 Substrates for III-nitride heteroepitaxy: a brief survey

In the case of the standard vapor phase epitaxy methods for GaN growth (HVPE, MOCVD), the high
growth temperatures (>1000 °C) and the high concentration of ammonia and hydrogen considerably
reduce the choice of possible substrates. The deposition of one or even multiple low-temperature buffer
layers in the first step of device fabrication is essential to allow the use of foreign substrates such as
sapphire, SiC, Si, GaAs or GaP. Even for molecular beam epitaxy (MBE) with growth temperatures
which are about 250 °C lower than those for HVPE and MOCVD growth, the stability of the substrate
surface at 800 °C and under the influence of nitrogen radicals is a critical issue. For device production on
an industrial scale, the substrate has to fulfill further criteria such as minimum size (2"), atomically flat
surfaces, and availability in large quantities at an acceptable price. Sapphire was and still is the most
common substrate for the deposition of GaN-based light-emitting diodes (LEDs), because of its reasona-
ably low cost and wide availability, and despite the fact that it has a large lattice constant and thermal
expansion coefficient mismatch with respect to GaN.

However, high dislocation densities caused by this mismatch are detrimental to the performance of
more demanding device structures than LEDs, such as laser diodes operating at high power densities [1].
Therefore, the lattice constant mismatch has been viewed as the most important criterion for determining
the suitability of a material as a substrate for GaN growth. In practice, other material’s issues including
crystal structure, composition, chemical reactivity and surface termination, or thermal and electrical
properties also have a strong influence on the final epitaxial layer. In the end, the substrate determines
the crystal orientation, polarity, surface morphology, strain, and the defect concentration of the heter-
epitaxial III-nitride films and, thus, is directly related to an optimal device performance. By now,
many different substrate materials have been tried out GaN epitaxy, including metals, oxides, nitrides,
and semiconductors. A still incomplete list of the more commonly used substrate materials, their crystal
structure, lattice constants and thermal expansion coefficients is given in Table 1. Among many other
issues, the influence of the substrate on the crystal polarity and macroscopic polarization of the group III-
nitrides is particularly important. E.g., the chemical reactivity and the optimum growth conditions re-
quired for high quality epitaxy generally depend on the polarity of the crystal, which in turn is deter-
mined by the surface termination of the substrate. The magnitude and sign (tensile or compressive) of the
strain incorporated into the heteroepitaxial film depends on many details of the nucleation and growth
process, and its optimization still is more a “black art” rather than a well understood phenomenon. The
densities of misfit and threading dislocations in GaN films deposited on the substrates in Table 1 are
typically between \(10^8\) and \(10^{10}\) cm\(^{-2}\). For comparison, the dislocation densities in GaAs or silicon homoepitaxy are as low as \(10^5\) to \(10^7\) cm\(^{-2}\) or almost zero [2]. In addition, inversion domain boundaries and
stacking faults caused by the heteroepitaxy give rise to non-radiative recombination centers in the band
gap and reduce the minority carrier lifetimes [48][49]. Threading dislocations form an undesirable path
for enhanced impurity diffusion, causing an inhomogeneous impurity distribution throughout the het-
eroepitaxial films and giving rise to an overall degradation of electronic and optical properties [50].

Therefore, many efforts have been made to improve the crystal quality of the heteroepitaxial layers,
mainly in the specific case of GaN on sapphire. Successful approaches include an appropriate surface
preparation of the substrate such as nitridation, deposition of low-temperature (LT) AIN or GaN buffer
layers, multiple LT buffer layers [51], epitaxial lateral overgrowth (ELOG) [52], PENDEO-epitaxy [53],
and other techniques [54-56]. Despite of all these efforts, heteroepitaxial GaN layers with dislocation
densities below \(10^7\) cm\(^{-2}\) cannot be obtained in a practical manner.

3 Freestanding GaN-substrates and devices by laser lift-off

As a viable alternative to direct heteroepitaxy of device relevant layer sequences on heterosubstrates,
the use of bulk crystals or bulk-like “pseudo-substrates” consisting of thick (several 100 μm) GaN layers
Table 1 Some characteristic structural properties of substrate materials for GaN heteroepitaxy.

<table>
<thead>
<tr>
<th>Material</th>
<th>Structure</th>
<th>Lattice constants (Å)</th>
<th>Thermal expansion coefficient (10^{-6} \text{K}^{-1})</th>
<th>References</th>
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<tr>
<td>w-GaN</td>
<td>wurtzite</td>
<td>3.1885 5.185 5.45</td>
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<td>5</td>
<td></td>
</tr>
<tr>
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<td>4.04</td>
<td>9</td>
<td></td>
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<tr>
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</tr>
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<td>SiC</td>
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<tr>
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<td>diamond</td>
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<td>MgAl₂O₄</td>
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<td>8.083 7.45</td>
<td>22[23][24]</td>
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<tr>
<td>MgO</td>
<td>rocksalt</td>
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<td>7[25]</td>
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<td>LiGaO₂</td>
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<td>5.4063 6.3786 5.0129 9.0</td>
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<td>NdGaO₃</td>
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<tr>
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<td>3.236 25.15</td>
<td>33[34]</td>
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<tr>
<td>Ca₃La₃(PO₄)₃O₂</td>
<td>apatite</td>
<td>9.446 6.922</td>
<td>35</td>
<td></td>
</tr>
<tr>
<td>MoS₂</td>
<td></td>
<td>36[37]</td>
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<td>39</td>
<td></td>
</tr>
<tr>
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<td>apatite</td>
<td>9.446 6.922</td>
<td>35</td>
<td></td>
</tr>
<tr>
<td>Hf</td>
<td>hcp</td>
<td>3.18 5.19</td>
<td>40[41]</td>
<td></td>
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<tr>
<td>Zr</td>
<td>hcp</td>
<td>3.18 5.19</td>
<td>41</td>
<td></td>
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<tr>
<td>ZrN</td>
<td>rocksalt</td>
<td>4.5776 4.22</td>
<td>42</td>
<td></td>
</tr>
<tr>
<td>Sc</td>
<td>hcp</td>
<td>3.309 5.4</td>
<td>43</td>
<td></td>
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<tr>
<td>ScN</td>
<td>rocksalt</td>
<td>4.502 4.389</td>
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<td>rocksalt</td>
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<td>45</td>
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<tr>
<td>TiN</td>
<td>rocksalt</td>
<td>4.241 4.241</td>
<td>46[47]</td>
<td></td>
</tr>
</tbody>
</table>

grown with a high deposition rate on sapphire substrates by HVPE has attracted considerable attention in recent years. There are various motivations for such an approach. First, the capability to produce material with a superior quality, e.g. a largely reduced density of dislocations and much narrower photoluminescence linewidths compared to the conventional heteroepitaxial material on sapphire or SiC has been demonstrated by different groups [57, 58]. Secondly, as far as the use of such bulk or bulk-like substrates for homoepitaxy is concerned, two-dimensional growth can be achieved without the need for additional steps such as surface nitridation or the growth of additional nucleation or buffer layers. Moreover, in the case of freestanding GaN-substrates, laser cavities can be obtained by simple facet cleaving [59, 60],
and the GaN substrate can be rendered electrically conductive, allowing for a significant simplification in device processing and circuit integration. In particular the latter two issues require a complete removal of the sapphire substrate after HVPE growth of the thick GaN pseudo-substrate.

Recent progress in the HVPE growth of GaN has demonstrated that critical problems such as obtaining large-area crack-free wafers of up to 2" diameter and 300 μm thickness can indeed be overcome [61, 62]. However, the surface roughness of such thick HVPE layers often is in the range of several microns, which is detrimental for any device application. Therefore, surface polishing after the HVPE growth is required to prepare epi-ready GaN pseudo-substrates. The necessary polishing procedures are hampered by the mechanical brittleness of sapphire and the significant bowing of the GaN/Al₂O₃ bilayers, which is caused by the differences in thermal expansion coefficients between GaN and sapphire and leads to a sometimes critical accumulation of thermal stress during the cool down from the high growth temperature (≈ 1050 °C) to room temperature. Again, a complete removal of the Al₂O₃ substrate also would remove most of these problems. Finally, as far as devices are concerned, Nakamura et al. have reported a significant improvement in the lifetime of laser diodes and cleaved facets after polishing the sapphire substrate away [59, 63].

All of the issues mentioned above make it highly desirable to develop a process which enables a fast, reliable, and high-yield detachment of thick HVPE-grown GaN layers from their sapphire hetero-substrates. In practice, such a process will involve a sacrificial layer somewhere between the substrate and the GaN-layer, which is removed by a specific chemical or thermal treatment. One possibility would be to deposit a sacrificial layer which makes use of selective chemical etching, e.g. of AlN versus GaN in KOH. However, in this case the detachment of large area GaN pseudo-substrates would be slowed down by chemical diffusion and would impose additional boundary conditions for the deposition of buffer layers. Just recently, an epitaxial procedure was developed to cause GaN films to self-detach from its sapphire substrate during cool down from growth temperature due to the accumulation of thermal stress and the lattice mismatch between GaN and sapphire [64]. In this case, a rather complex pretreatment of the wafer including lithography is needed, which limits the relevance of such a processing step for future mass production schemes. A third approach is the complete removal of the substrate by etching or polishing. This, however, is quite time-consuming, especially in the case of sapphire or SiC substrates.

A process which is much more flexible and significantly faster than the above mentioned methods is the laser-induced delamination of III-nitrides from a transparent substrate such as sapphire, or laser lift-off (LLO) for short. In this process, the separation of a GaN layer from the substrate is achieved by irradiation of the substrate-film-interface through the substrate with high power laser pulses at a wavelength which is transmitted by the substrate, but is strongly absorbed in the GaN layer. In the case of GaN on sapphire, the most suitable laser systems in this context are the third harmonic of a Nd:YAG laser [65, 66] or excimer lasers [67]. The absorption of such high intensity laser pulses causes a rapid thermal decomposition of the irradiated GaN interfacial layer into metallic Ga and gaseous N₂ (Fig. 1). The intrinsic advantages of such a LLO process compared to the other possibilities mentioned above are:

- Sapphire as a readily available, well developed, and reasonably expensive substrate material can be used. It would be even possible to recycle the separated sapphire substrates after LLO for further deposition runs.
- The LLO process does not require any specific sacrificial layers in the growth sequence. Even better, the usual low-temperature and, thus, low-quality AlN or GaN buffer layers are removed together with the substrate in the same process.
- The LLO method is quite fast and can be scaled up easily. Using standard Nd:YAG or excimer laser systems with pulse repetition rate of 1000 Hz, a 2” wafer in principle could be lifted-off in a few seconds.
- Since LLO does not require any direct mechanical or chemical contact with the wafer, the lift-off process could also occur directly after deposition of the III-nitride film in the deposition reactor before cool-down to room temperature. This would just require a suitable optical window and an external laser scanning arrangement, but at the same time would avoid the build-up of critical thermal strain,
Fig. 1 (online colour at: www.interscience.wiley.com) Schematic view of the laser lift-off (LLO) process. High intensity laser pulses enter the sample via the sapphire substrate and thermally decompose a thin GaN-layer at the substrate interface. The shock waves resulting from the explosive production of nitrogen gas during each laser pulse are damped by placing the GaN-sample into sapphire powder. A hot plate can be used to raise the substrate temperature during the process, in order to relieve some of the accumulated thermal strain.

which occurs in thick GaN films on heterostructures during the cool down from the deposition temperature of 1100 °C to room temperature because of the large mismatch between the thermal lattice expansion coefficients of the two materials.

3.1 Laser-induced decomposition of GaN

At sufficiently high temperatures, the stability of GaN is limited by the decomposition of the crystal into nitrogen gas and liquid gallium: \(2\text{GaN}(s) \rightarrow \text{N}_2(g) + 2\text{Ga}(l)\). The flux of nitrogen molecules, \(\Phi(\text{N}_2)\), leaving the crystal surface in vacuum shows an exponential increase with temperature above 830 °C, which can be parametrized as:

\[
\Phi(\text{N}_2) = 1.2 \times 10^{31} \text{cm}^{-2}\text{s}^{-1} \exp(-3.9 \text{eV}/kT).
\]

The resulting thermal decomposition rate of GaN as a function of temperature is shown in Fig. 2 for a linear temperature ramp of 0.3 K/s [68]. The decomposition rate reaches approximately one monolayer per second at a temperature of 930 °C. Thus, GaN can be removed very efficiently via thermal decomposition by methods which enable a controlled local heating of the sample to temperatures above 900 °C.

As mentioned above, one possibility to locally decompose GaN is by absorption of intense light with photon energies above the bandgap of GaN (3.42 eV), e.g. the 355 nm (3.49 eV) third harmonic of a Nd:YAG pulsed laser [69, 70]. The properties of neodymium-doped yttrium aluminum garnet (Nd:YAG) are the most widely studied and best understood of all solid-state laser media. The active medium is \(\text{Nd}^{3+}\), which is optically pumped by flash lamps, generating pulses at 1064 nm. The resulting pulse length is shorter than 10 ns, the peak of the optical power is tens of megawatts, and the pulse power is very stable, with intensity variation from pulse to pulse of less than 3%. The high peak power of the pulses permits efficient frequency conversion to 532 nm (2nd harmonic) and 355 nm (3rd harmonic) in non-linear potassium dideuterium phosphate crystals (KD*P).

In Fig. 3, the effects of a single shot of a Nd:YAG laser with energy densities in the range between 180 and 410 mJ/cm² on the macroscopic optical appearance of thin GaN layers on sapphire are shown. Below a critical absorbed pulse intensity of approximately 250 mJ/cm², no visible alteration of the GaN-layer occurs. For higher intensities, however, the absorbed photon energy leads to local heating of the layer above the critical sublimation temperature of about 900 °C, causing the destruction of the GaN.

The remaining metallic Ga is responsible for the dark areas appearing after the laser treatment in the optical pictures. As can be seen from Fig. 3, small lateral inhomogeneities of the laser beam have a dras-
tic effect on the macroscopic quality of the transformation process. In this respect, the better coherence properties of Nd:YAG lasers compared to Excimer lasers are somewhat problematic, since it is more difficult to obtain flat intensity profiles over macroscopic areas due to interference pattern formation. On the other hand, the shorter pulse length of Nd:YAG lasers (6-10 ns) compared to Excimer lasers (40 ns) can be of some advantage in the LLO-processing of thin GaN-based device structures, where thermal and mechanical strain is much more critical than for thick HVPE-grown quasi-substrates.

Fig. 2 (online colour at: www.interscience.wiley.com) Thermally induced decomposition of GaN and the corresponding flux of nitrogen from the GaN surface using a heating rate of 0.3 K/s [68].

Fig. 3 (online colour at: www.interscience.wiley.com) Effect of a single shot from a Nd:YAG-laser on the optical appearance of a GaN layer on sapphire. For absorbed energy densities below 200 mJ/cm², no visible effects can be observed. For energy densities above the sublimation threshold of approx. 250 mJ/cm², the entire surface of the GaN epilayer is transformed into metallic Ga, giving rise to the dark colour in the upper half of the specimen pictures. Close to the sublimation threshold, inhomogeneities of the intensity profile of the laser beam are clearly visible in the irradiation patterns.
Fig. 4 (online colour at: www.interscience.wiley.com) Experimentally determined decomposition depth caused by a single pulse of a Nd:YAG laser in GaN at room temperature as a function of the pulse intensity absorbed in the thin GaN layer on sapphire. The change in slope for pulse intensities exceeding 350 mJ/cm$^2$ is probably due to absorption or reflection of the laser light by the metallic Ga layer formed during the process.

The extinction depth of the 355 nm Nd:YAG laser line in undoped GaN at room temperature is about 100 nm, corresponding to an absorption coefficient of $10^5$ cm$^{-1}$. Given the sublimation threshold of 250 mJ/cm$^2$ determined above, and pulse intensities up to 100J/cm$^2$ which can be obtained by beam focusing for typical laboratory ND:YAG laser systems, GaN layers up to a thickness of 500 nm can in principle be removed by a single laser shot.

Fig. 5 (online colour at: www.interscience.wiley.com) Dependence of the laser-induced etch depth of GaN on the number of laser pulses with and without the presence of HCl-vapor. The laser treatment was performed at 300 K with an absorbed pulse intensity of 290 mJ/cm$^2$ and a pulse repetition rate of 10 Hz. From the slope of the straight line an average etch rate of 330 nm/s can be deduced.
For a more quantitative investigation, GaN layers with a thickness of 1 µm were partly covered with a reflective coating and then submitted to single pulses of a Nd:YAG laser with intensities between 100 and 600 mJ/cm². After laser exposure, the metallic Ga layer was removed by wet chemical etching in HCl, and the effective depth of the removed material was determined by depth profiling. The results are summarized in Fig. 4.

The obtained decomposition depth for a single pulse increases monotonically from 0 at the threshold intensity of approx. 250 mJ/cm² to almost 100 nm for an intensity of 600 mJ/cm². Above 320 mJ/cm² a slight saturation sets in, probably due to the self absorption or the high reflectivity of the metallic Ga layer formed on the surface of the irradiated GaN. This effect can be reduced by performing the laser irradiation in an HCl atmosphere, which causes a fast removal of the metallic Ga in the form GaCl₃. This chemical removal of the Ga is particularly important for repeated laser treatments of GaN, e.g. with longer sequences of pulses. An example is shown in Fig. 5, where GaN was irradiated with a pulse repetition rate of 10 Hz in the presence of HCl. As could be expected, a linear dependence of the etch depth on the number of pulses was obtained, with a removal of 33 nm GaN per pulse or, equivalently, an average etch rate of 330 nm/s. Without HCl, the accumulation of metallic Ga at the surface causes a saturation of the etch depth versus the number of pulses at approximately 100 nm.

As shown in Fig. 6, this laser-induced etch rate is much higher than presently employed ion-assisted etch methods, such as electron cyclotron resonance plasma etching (ECR), chemically assisted ion beam etching (CAIBE), high-density magnetron reactive ion etching (RIE-MIE), or inductively coupled plasma etching (ICP). For more details and further references see [71, 72, 73].

Despite of the advantage of having fast etching rates, light diffraction at mask edges and borders will be detrimental for the application of laser-induced etching in the processing and isolation of small scale devices (e.g. mesa-structures). However, a high resolution laser structuring of GaN down to a dimension of 100 nm can be achieved with holographic patterning without the use of masks and therefore avoiding the effect of light scattering at edges and borders [69]. Another issue is the amount of surface damage caused by the various etch methods. In the case of laser-etching, little systematic experience exists so far, but first structural investigations indicate that crystal damage caused by the laser treatment is restricted to a surface layer of about 100 nm [74–78], which can easily be removed by conventional etching methods.

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**Fig. 6** (online colour at: www.interscience.wiley.com)
Comparison between the maximal etch rates for GaN achieved by laser-induced etching and conventional reactive ion etching methods (ICP: inductively coupled plasma, MIE: magnetron ion etching, CAIBE: chemically assisted ion beam etching, ECR: electron cyclotron resonance, RIE: reactive ion etching). Using laser-induced thermal decomposition of GaN, much higher etch rates in the range of 1 µm/s can be realized.
Fig. 7 (online colour at: www.interscience.wiley.com) Temporal and spatial variation of the temperature at the sapphire/GaN interface (depth zero) during and after a Nd:YAG laser pulse (left figure, $\lambda = 355$ nm, $\tau = 6$ ns, $I = 300$ mJ/cm$^2$) and a KrF excimer laser pulse (right figure, $\lambda = 248$ nm, $\tau = 38$ ns, $I = 600$ mJ/cm$^2$). The intensities of the two pulses were chosen such as to obtain the same maximum temperature of 1100 K (sublimation temperature, cf. Fig. 2).

A more detailed understanding of the laser lift-off process requires a quantitative analysis of the temporal and spatial temperature profiles at the substrate/III-nitride interface during and after the short laser pulse. As an example, we discuss here briefly the comparison between the two main laser systems used so far for LLO-processing of GaN: (i) the third harmonic of the Nd:YAG laser at $\lambda = 355$ nm with a pulse length of $\tau = 6$ ns, and (ii) the KrF excimer laser with $\lambda = 248$ nm and $\tau = 38$ ns. Because of the much longer pulse duration in the case of the KrF laser, a higher pulse energy of typically 600 mJ/cm$^2$ is necessary to heat the GaN above the sublimation threshold, whereas pulse energies of 300 mJ/cm$^2$ are sufficient in the case of the Nd:YAG laser [69, 74]. Since optical thermalization and relaxation processes in GaN occur on a much faster timescale of ps or a few ns, the temperature profiles generated by the different laser pulses can be calculated quite easily in a one-dimensional model based on the temporal shape of the laser pulse, the known absorption coefficients of GaN, and the thermal properties (heat capacitance and conductivity) of GaN and sapphire [79–83]. Fig. 7 shows a comparison of the resulting temperature profiles for the two above cases. Both profiles look qualitatively quite similar, but the thermal load on the sapphire/GaN heterosystem is significantly reduced in the case of the Nd:YAG laser compared to KrF irradiation. Because of the shorter pulse length in the former case, high temperatures above 700 K persist only for about 14 ns and are restricted to the first 500 nm next to the interface, whereas in the case of the KrF pulse, the same temperature is present during almost 100 ns and extends over a range of 1 µm into the GaN layer. Thus, Nd:YAG laser systems will have a significant advantage especially for the LLO-processing of thin, temperature sensitive device structures such as InGaN-based light emitting diodes or laser diodes (see Section 3.4).

3.2 Laser-induced lift-off of thick HVPE GaN wafers

A successful separation of thick HVPE-GaN layers from their sapphire substrates depends on several factors, including film thickness and thickness homogeneity across the wafer, growth procedure, and the resulting stresses and stress gradients caused by the large thermal expansion coefficient mismatch. During the past years, we have processed HVPE-GaN wafers prepared by different growth methods, but mainly thick GaN multilayer samples deposited with in-situ growth stops and HVPE-GaN grown on MOCVD-GaN templates structured with the ELOG technique. Due to the potential commercial rele-
vance of GaN pseudosubstrates and the technological difficulty of growing thick, crack-free GaN films, it was not possible to obtain relevant information about further growth details. Nevertheless, some useful correlations between the structural and morphological properties of the GaN samples and the results of their delamination could be obtained.

For samples with thicknesses ranging from 70 to 300 µm (i.e., comparable to the sapphire substrate thickness of 330 µm), the differences of the thermal expansion coefficients result in substantial wafer bowing. Despite this macroscopic bowing, most of the wafers exhibited very little or even no near-surface cracking across the whole wafer area. However, the bowing of samples with thicker GaN films (>100 µm) conflicted with the laser lift-off procedure, because the strain inhomogeneity at the boundary of already released and thus relaxed areas and still attached portions of the wafer often gave rise to extensive fracturing. To reduce the bowing-induced fracturing, we used homogeneous heating of the sample during the laser lift-off. Already at temperatures around 600 °C, the strain in the sample and, consequently, the bowing was visibly reduced and hardly noticeable through simple optical inspection of the GaN surface. This is consistent with temperature-dependent X-ray diffraction measurements performed by Leszczynski et al. [84], who have shown that the lattice constants of a 2 µm thick GaN film on sapphire approach the values of bulk GaN already at about 800 K. Ideally, the performance of in-situ laser lift-off in the deposition reactor close to growth temperature would be the best solution to avoid all bowing or cracking problems caused by the thermal expansion mismatch between GaN and the sapphire substrate.

As exemplified by Fig. 8, extended areas could be routinely released without sample damage by performing the laser treatment at temperatures above 600 °C, but below 830 °C (decomposition temperature of GaN). As shown in Fig. 1, the wafer is positioned in a steel holder filled with a thin layer of sapphire powder. A transparent sapphire window on top avoids unwanted movements of the wafer during laser scanning. The role of the sapphire powder with small grain size is to allow a free changing of the bowing radius with temperature during the heat-up period. The powder is also important as a damping system during the laser processing as well as after the delamination during the cool-down time to support the free-standing GaN film.

After removal of any residual GaN deposit on the backside of the substrate, which sometimes occurs in HVPE growth and would interfere with the laser lift-off, the laser treatment was performed in a computer-controlled scanning system which allowed a specific choice of scanning speed, step size, overlap between subsequent laser shots, and trajectory across the wafer. Even for the laboratory-size laser system used in our studies, the laser scanning of a 2” wafer itself takes only 3 min for a step size of 2 mm. A spiral scan trajectory towards the center of the wafer was found to be best suited for the lift-off of samples with circular shape, because of the symmetrical stress distribution between the free and still attached parts of the wafer. This is particularly important for the laser processing of GaN wafers with strong thickness inhomogeneity.

Fig. 8 Photograph of a 275 µm thick free-standing GaN film, after removal from the 2” sapphire substrate [65]. The missing pieces at the wafer border actually had broken off right after the HVPE growth.
3.3 Laser-induced lift-off of thin GaN films

A much more challenging problem than the lift-off of thick HVPE films as described in the previous section is the delamination of GaN films in the thickness range from a few micrometers up to about 60 µm. These films are mechanically too fragile to be separated from their substrate in the same way as described above. It is necessary to attach a support layer to the GaN layer in order to avoid large area fracturing after laser lift-off. The choice of a proper material which can be used for this support of thin freestanding GaN films is of crucial importance. Such a material should have an elastic modulus comparable to that of GaN and should be easily removable. On the other hand, it should be flexible enough to accommodate changes in the bowing radius during liftoff. Finally, it would be desirable to have a support material which can stand high temperatures up to 650 °C. Unfortunately, to the best of our knowledge, such a material is not yet commercially available. We therefore performed a series of experiments using different types of commercial adhesives and glues like a two-component epoxy resin, and silicone thermoresistant elastomer. Sticking GaN samples to a “blue tape” used in semiconductor production for fixing samples during sawing is also a possible approach. All delamination processes described below were performed at room temperature, due to the relatively low temperature resistance of the applied support materials.

As an example, completely crack-free GaN films were delaminated by fixing them to two-component epoxy resin prior to laser scanning. A glass carrier was used to support the GaN samples, which were pressed onto a thin layer of the transparent resin with a thickness of less than 0.5 mm. The laser liftoff was performed 5 min after curing the glue at room temperature. Figure 9 shows the delaminated GaN layer and the sapphire substrate, which is completely free at the end of the process. The strong adhesion of the used two-component epoxy resin is advantageous for a perfectly crack-free GaN liftoff, but on the other hand it is difficult to separate from the GaN film after sapphire removal due to its high chemical stability. Nevertheless, the epoxy can be dissolved with dichloromethane or in boiling water after several minutes.

An alternative procedure for the laser lift-off of free-standing full size 2” GaN membranes makes use of a silicone elastomer during laser processing and a thermoplastic adhesive to support the film after sapphire removal. A schematic step-by-step view of this process is shown in Fig. 10. The free side of the HVPE-GaN film on sapphire is covered with the thermoresistant (up to 300 °C) silicone elastomer forming a ~3 mm thick support layer. The film is then fixed to a metallic plate with doublesided adhesive tape to avoid moving of the flexible silicone elastomer. After laser scanning at room temperature, the sapphire substrate is removed. Then, the free side of the delaminated GaN film is protected with a ~3 mm thick transparent thermoplastic adhesive, which allows a safe peel-off of the silicone elastomer (Fig. 10 d)).

A 60 µm thick GaN membrane with a diameter of 2” is shown in Fig. 11. The GaN membrane is still protected by the thermoplastic adhesive, which can be easily dissolved in acetone without critical mechanical stress. The flexibility of the silicone elastomer allows the accommodation of the GaN film bowing. After fixing the membrane with the much more viscous thermoplastic adhesive, the residual bowing...
of the GaN film and some cleavage lines can be observed through specular light reflection (left side of Fig. 11). Despite of some cracks appearing over the GaN membrane, the procedure described above is fully reproducible and, thus, applicable to the delamination of large HVPE-GaN films with a thickness of some tens of micrometers. A different approach to produce completely crack-free thin and large area GaN-based heterostructure wafers for device production is based on the wafer bonding technique as discussed in the following section.

3.4 Delamination of thin wafer-bonded GaN heterostructures and devices

The monolithic integration of two dissimilar materials onto one platform with wafer-bonding and laser lift-off is a convenient alternative to heteroepitaxial growth when this is not possible or too problematic. In the case of group III-nitrides, the integration is motivated by combining the superior optoelectronic properties of GaN-based heterostructures with receptor substrates which possess much better thermal and electrical properties than the original sapphire substrate. For devices processed on sapphire, all electrical contacts must be made from the top side. This complicates contact and packaging schemes and results in higher operation voltages as well as a spreading-resistance handicap [67]. Most importantly, the poor thermal conductivity of sapphire is detrimental for high-power device performance of LEDs, laser diodes, and high-power transistors. Hence the transfer of GaN-devices to other substrates with better thermal properties would be of enormous benefit. In addition to the cheap and readily available Si, GaAs, Ge, Mo, and copper are receptor wafers with certain advantages.

A major issue in wafer bonding is the choice of the optimal bonding layer. The bonding material has to adhere to both the epitaxial film and the receptor substrate, providing full surface contact despite of sub-micron surface asperities. A temperature necessary to achieve bonding should be low (< 200 °C) to avoid thermal stress, and without leaving behind any phase with a melting point below the bonding temperature. Moreover, it should have low electrical resistance to allow easy contact formation with the bonded devices. For the specific case of the III-nitride material system, Wong et al. have used a Pd-In metal bonding to connect GaN to Si [85], whereas Jasinski et al. have succeeded in producing GaN/GaAs bond-structures by direct wafer fusion [86]. 2 µm thick GaN films grown on sapphire were fused to a GaAs substrates in nitrogen ambient by applying uniaxial pressure of 2 MPa at bonding temperatures of 550 and 750 °C.

Individual devices on a GaN- wafer can be scribed or isolated via mesa-etching prior to wafer bonding, thus enabling the production of separated devices after the laser lift-off. A possible technological process flow diagram for the production of efficient nitride based LEDs using a metal bonding and laser lift-off procedure is illustrated in Fig. 12. In this way, the individualized devices on the sapphire wafer can be transferred to a receptor substrate like Si, GaAs or Cu.
Fig. 11 (online colour at: www.interscience.wiley.com) The left hand side shows the HVPE-GaN membrane with a diameter of 2" and thickness of 60 µm attached to thermoplastic adhesive after the laser lift-off. The specular light reflection due to the bowing of the film and some cleavage lines are indicated. The silicon elastomer used as a support during laser processing can be completely peeled off, as shown on the right hand side. The separated sapphire substrate is also shown.

Fig. 12 (online colour at: www.interscience.wiley.com) Process flow for bonding and transfer of a GaN-based LED from sapphire onto a receptor wafer using PdIn$_3$ as the bonding material.
The GaN-based heterostructures are typically thinner than 5 µm, so that practically no bowing of the wafers occurs, and laser lift-off can be performed at room temperature. The energy density of the laser shots should be kept as close as possible to the threshold value necessary for GaN decomposition. Any additional energy will lead to unwanted thermal and mechanical stress which can favour film spalling and peel-off after sapphire removal.

Figure 13 shows a successfully delaminated GaN film wafer-bonded to GaAs, where some peeling-off occurred only at the very border of the wafer. The isolation of the devices prior to wafer-bonding can prevent the propagation of cracks and spalling, thus conserving the integrity of the device structures during laser delamination. A yield as high as 90% and above can be reproducibly achieved for 2” wafers. In order to illustrate the potential of the LLO processing for commercial device production, we now have a closer look at the laser lift-off in the case of blue/violet InGaN/GaN light emitting diodes.

For the realization of high-brightness LEDs with efficient current injection and heat sinking, the flip-chip technique used for chip mounting is a promising approach which becomes increasingly important. Advantages are the possibility of higher packaging density, higher frequency operation due to shorter contacts, a minimum of interconnects ("direct die attach"), and a low height of the whole assembly, as no bonding wires are necessary. Basically, in the flip-chip technology the die is mounted onto a substrate (printed circuit board) with the active device contact side down. The electrical connection is made simultaneously for all contacts in a single step. One of the contact partners must have “bumps”, i.e. raised areas composed of electrically conductive material. Different galvanic and mechanical processes with or without melting of the metallic bumps are currently available for this purpose, but one of the
most widely used processes is traditional soldering technology modified for direct chip mounting. In this case, metallic alloy solders for the bumping and connection process are used. A schematic view is given in Fig. 14.

In the specific example described here, InGaN/GaN LED chips provided by the IAF Fraunhofer Institut in Freiburg, Germany were flip-chip bonded to metallized Si-submounts by ball bonding with a wire bonding machine. First, Au-balls were positioned on the interconnecting metallization of the Si-submount, consisting of a Ti/Au/Ti plating base covered by an electroplated Au-layer. Subsequently, the LED chips were mounted upside-down by thermal compression welding of the bump/LED contact interface at a temperature of \( \sim 200 \, ^\circ C \). In Fig. 15, a series of isolated LEDs is shown, where a sapphire stripe with the devices was sawed away from the wafer and the individualized LEDs were finally flip-chip bonded to the Si-submount. Entire device stripes can be connected to the base substrate in a similar way.

The laser-induced delamination method was applied directly to the flip-chip contacted devices grown on sapphire. As the size of the devices is much smaller than the laser-shot area, a single pulse is sufficient to remove the sapphire substrate, which is beneficial to maintain the structural integrity and the performance of the device even after the laser treatment. As in the delamination of GaN wafers described above, an important aspect for a successful laser processing of the fragile GaN devices is to minimize the structural and thermal stress during the short laser / target interaction. The use of a suitable underfill is indispensable to absorb the mechanical impact caused by the high power laser pulse on the thin film supported only by the contact bumps. Wafer stripes of size \( 0.5 \times 25 \, \text{mm}^2 \) containing a series of LEDs in line as well as individually sawed LEDs like the ones shown in Fig. 15 with a device area of \( 250 \times 250 \, \mu\text{m}^2 \) and a thickness of 3.8 \( \mu\text{m} \) were prepared with an optimized underfill for the laser lift-off. After removal of the sapphire substrate, the residual Ga on the exposed backside was etched away by
contact with HCl vapour for 10 sec. After removing the protective adhesive with acetone, the performance of the freestanding flip chip bonded LEDs was compared to that prior to the LLO process. As demonstrated by the electroluminescence spectra in Fig. 16, no degradation of device performance due to the laser processing was observed.

4 GaN homoepitaxy on freestanding substrates

As already mentioned in the introduction, the exploitation of the real potential of GaN-based devices is still hindered by the lack of a suitable bulk III-nitride substrate (GaN and/or AlN). Here, the use of freestanding GaN pseudosubstrates obtained e.g. by laser lift-off from thick HVPE-GaN grown on sapphire [87, 88] may provide a real alternative to the still difficult and time-consuming equilibrium growth of bulk substrates. Although homoepitaxy of GaN on such bulk substrates has resulted in the best material quality so far [2, 89], the available substrate sizes are still incompatible with a commercially viable device production. In this section, we will therefore review recent results obtained on homoepitaxial GaN layers grown on freestanding pseudosubstrates produced by laser lift-off.

4.1 Surface conditioning of freestanding substrates

The surface morphology of the thick HVPE-GaN films used in this work was characterized by optical microscopy and surface profiling. Complementary, the microscopic structure of the surface was determined by atomic force microscopy, which shows a smooth surface morphology with a rms roughness of 0.4 nm. Dislocation densities between $8 \times 10^6$ and $5 \times 10^7$ cm$^{-2}$ can be estimated from the pin-hole concentration at the surface (black spots in the AFM image in Fig. 17), which agrees well with the values published by Vaudo et al. [90]. However, on a larger scale, the Ga-face oriented growth surface of HVPE-GaN exhibits a much rougher morphology, consisting of hexagonal pyramids with diameters of several 10 µm and a height of typically 1 µm, as shown by the optical micrograph on the left hand side of Fig. 17. Some samples also exhibit characteristic large surface voids on the as grown side of the sample shown in Fig 18. These large volume crystal defects are mostly localized at the borders of the 2” HVPE-GaN wafers, and are probably caused by a deviation from the optimal growth conditions together with the fast growth rates of 100 µm/h and more. They are obviously detrimental for homoepitaxial
Fig. 18 Typical large defects found at the Ga-face surface of thick HVPE-GaN samples. The voids can reach a diameter of 100 µm and depth of 50 µm, as shown by the side view on the left. From the top view it is possible to recognize the hexagonal symmetry of the voids, despite of their almost perfect circular shape.

overgrowth and have to be removed by lapping and polishing. On the substrate side of the thick HVPE GaN layer, the laser-induced delamination gives rise to a characteristic surface morphology consisting of small grains with a size of approximately 2 µm, and a similar rms roughness.

In order to produce free-standing GaN pseudo-substrates with adequate surface characteristics for further epitaxial overgrowth, a lapping and fine-polishing procedure was developed for the delaminated GaN films [91]. First a planarization step was introduced to get rid of residual sample bowing after laser lift-off by using a diamond lapping pad with a grain size of 40 µm. Afterwards a polishing sequence under controlled pressure of about 2 kg/cm² was performed with diamond grain sizes of 15, 7, 3 and 0.25 µm for approximately 10 minutes per step. The last step for the preparation of the Ga-face surface was a long mechanical polishing with a special fine polishing solution (grain size of 0.04 µm). For the N-face surface, a chemical polishing step with a 1:10 KOH : H₂O solution was applied to obtain the final surface smoothness. The rms roughness achieved in this way, as measured by AFM, is of the order of 1 nm for N-face and 2.2 nm for Ga-face surfaces. An additional chemo-mechanical polishing step to remove subsurface damage caused by the mechanical polishing was found to be inefficient for Ga-face surfaces. For this purpose, chemically assisted ion beam etching (CAIBE) or reactive ion etching (RIE) were also investigated by other groups [73, 92]. As confirmed by photoluminescence measurements, an optical quality similar to the unpolished original GaN surface after the HVPE growth could be recovered by this treatment.

4.2 Structural and electronic properties of homoepitaxial GaN layers on freestanding GaN pseudosubstrates

After optimization of the surface conditioning of freestanding GaN pseudosubstrates as described in the last section, homoepitaxial overgrowth on such substrates was performed by a conventional MOCVD process [93]. Prior to the growth, the HVPE-GaN pseudosubstrate was annealed at 800 °C under a flow of 2 slm of nitrogen and hydrogen (N₂ / H₂ = 1) for 10 min. Then NH₃ was added to the transport gases and the temperature raised to 1050 °C within a time period of 5 min. A 2 µm thick epitaxial GaN film was grown on the Ga-face side of the substrate, with a V/III ratio of 40,000 and a growth rate of 600 nm/h. As demonstrated by AFM investigations, epitaxial layers with an outstanding surface morphology can be obtained (Fig. 19).

In the cross section of the AFM micrograph shown on the left hand side of Fig. 19 it is possible to distinguish bilayer steps separated by terraces with a width of about 200 nm. Compared to the reference sample on the right hand side of Fig. 19 (an in-situ overgrown MOCVD-GaN sapphire based template) the estimated dislocation density is reduced by almost 3 orders of magnitude for the homoepitaxial layer. The estimated dislocation density of 2 × 10⁷ cm⁻² was obtained based on a series of AFM micrographs and is in the range determined for the original HVPE-GaN substrate.
High resolution X-ray diffraction (HRXRD) measurements were performed to check the structural quality, strain and mismatch between the GaN pseudosubstrate and the homoepitaxial layer. In Fig. 20, \(2\theta-\omega\) scans of the (0002), (0004) and (0006) reflexes are compared, showing a full width at half maximum (FHWM) as low as 17 arcsec. From the high dispersion (0006) diffraction peak and reciprocal space
maps of the (2 0 5) reflex, the maximum lattice mismatch between substrate and the homoepitaxial GaN film can be estimated to $\Delta a/a = 3 \times 10^{-5}$. The FWHM of rocking curves is limited by a small residual curvature of the free-standing GaN substrate, caused by remaining biaxial compressive strain.

To evaluate the optical and electronic quality of the homoepitaxial GaN layers, photoluminescence (PL) measurements were performed at liquid helium temperature. The 333.6 nm line of an Argon ion laser was used for excitation. The photoluminescence was dispersed using a triple-grating spectrometer with a focal length of 800 mm and was detected by an In2 cooled CCD camera. With this setup, a spectral resolution of better than 1 cm$^{-1}$ or 120 µeV can be obtained. The observed PL transitions in Fig. 21 are typical for a nominally undoped GaN films with a small density of residual shallow impurities. The high optical quality of the GaN film is confirmed by the presence of the well resolved free-excitonic lines $X_A$, $X_B$, and the excited state $n=2$ of $X_A$ at 3.480, 3.486, and 3.500 eV, respectively, and the neutral donor bound-exciton transition ($D^0_n - X$) at 3.474 eV with a FWHM of 550 µeV [94]. Other PL lines due to the neutral acceptor bound-exciton ($A^0 - X$) at 3.469 eV, the neutral donor bound-exciton ($D^0 - X$) at 3.478 eV, and the transition involving the excited 2s-like state of the donor ($D^0_2 - X_{2s}$) at 3.453 eV are also seen [95].

On the low energy side of the spectra in Fig. 21, small bands emerge at 3.384 eV and 3.391 eV in the PL spectra of the GaN substrate, which are attributed to phonon replicas of the free-exciton line and the acceptor bound-exciton, respectively. A strong band centered at 3.403 eV (FWHM = 19 meV) in the homoepitaxial GaN film is assigned to overlapping LO-phonon replicas associated with the free and bound exciton transitions.

A comparison of the results presented above with homoepitaxial films grown by MOCVD and MBE on high pressure/high temperature synthesized bulk GaN substrates reveals similar linewidths, but slightly different energy positions of the donor bound-exciton transitions [96, 97]. This shift in energy is most likely due to slight differences in residual strain. In contrast to the films grown on HVPE-GaN substrates, the ten times larger lattice mismatch of $\Delta a/a = 2 \times 10^{-4}$ reported for homoepitaxial GaN films on high pressure/high temperature grown bulk GaN substrates is due to the significantly higher residual carrier concentration of the order of $10^{19}$ cm$^{-3}$ in the latter substrates [98].

Fig. 21 (online colour at: www.interscience.wiley.com) Low temperature PL-spectra of a homoepitaxial GaN film on a free-standing HVPE-GaN substrate. The corresponding PL spectra of the substrate prior to homoepitaxial overgrowth are shown by the lower spectrum.
Figure 22 shows the PL spectra of homoepitaxial GaN on a freestanding GaN substrate for various excitation intensities. The excitonic character of the observed transitions is confirmed by the absence of an energy shift with increasing excitation intensity. The integrated intensities of each transition increases superlinearly with the excitation power. According to Schmidt et al. [99], such a dependence of the luminescence intensity $I$ on excitation power is expected for excitonic transitions.

Figure 23 (online colour at: www.interscience.wiley.com) Temperature dependence of the photoluminescence for a homoepitaxial GaN film on a freestanding HVPE substrate. See text for further details.
Finally, Fig. 23 summarizes the temperature dependence of the various transitions in Figs. 21 and 22. All PL-lines show a significant change with increasing temperature in the range from 5 to 55 K. The higher energy peaks labeled \( X_A \) and \( X_B \) broaden and become relatively stronger, eventually becoming the dominant PL lines above 50 K. This is consistent with their assignment to free-exciton lines. Pronounced quenching due to thermal dissociation is observed for the bound-exciton recombinations \((D_{2u}^0 - X)\), \((D_{2e}^0 - X)\), \((D_{2u}^0 - X)_{n=2}\), and \((A^0 - X)\). The shift in the exciton peak positions is in agreement with the band-gap shrinkage of wurtzite GaN [100], except for a small energy offset due to residual strain.

## 4 Summary and conclusions

The present state-of-the-art concerning the laser-induced lift-off of both thin and thick GaN films and heterostructures from sapphire substrates has been reviewed. The physical background of the laser-induced thermal decomposition of GaN by short intense pulses of KrF excimer lasers and the third harmonic of Nd:YAG lasers is discussed, and potential applications for rapid etching of GaN have been outlined. Of particular interest for future applications is the possibility to produce freestanding GaN films by laser lift-off of heteroepitaxial layers from transparent sapphire substrates. Specific applications include the production of freestanding GaN pseudosubstrates starting from thick HVPE grown epitayers, as well as the delamination of thin GaN device heterostructures for the purpose of wafer bonding onto foreign substrates or for flip-chip bonding in device technology. Using optimized processes, the defect free lift-off of entire 2" wafers can be achieved for GaN film thicknesses ranging from 3 to 300 µm. Separation of thin device heterostructures from their sapphire substrates opens up new possibilities for the formation of electrical contacts, the extraction of photons, and for thermal management. Thick GaN films grown by HVPE can be removed from their sapphire substrates to obtain freestanding GaN pseudosubstrates for the homoepitaxial growth of high quality epitayers. The required processing steps, including surface preparation after laser lift-off, have been described and the structural and optoelectronic properties of homoepitaxial GaN layers deposited on freestanding pseudosubstrates have been investigated. All results suggest that the laser lift-off method may become a major technique in future III-nitride device technology.

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