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Bowing of thick GaN layers grown by HVPE using ELOG

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Intrinsic stress in GaN layers grown by HVPE on sapphire substrates results in wafer breakage for thicknesses exceeding 40 μm. Using ELOG 180 μm GaN can be grown on 2 inch. The bowing of the wafers caused by extrinsic stress due to the different thermal expansion is not affected by ELOG. Using WSiN as mask material a 600 μm thick free-standing 2 inch GaN layer has been obtained by exploiting the shear forces occurring during cooling after growth for separation from the ELOG template.

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1 Introduction

The growth of GaN using hydride vapour phase epitaxy (HVPE) is widely accepted to be the method for fabrication of large area, thick GaN layers which can be used as substrates for further device epitaxy. Usually, the growth has to start on foreign substrates like sapphire. Sapphire substrates offer advantages resulting in the availability of excellent GaN/sapphire templates with dislocation densities of 10^9 cm^{-2} from planar MOVPE growth which can be further reduced to about 10^7 cm^{-2} using epitaxial layer overgrowth (ELOG) [1]. However, bowing of the wafers is a major problem. This bowing is introduced by intrinsic stress at growth temperature and extrinsic stress during cool-down [2]. The intrinsic stress depends on the growth conditions. Bowing can result for example from intrinsic tensile stress due to island coalescence [3] and from other changes of the microstructure like impurity incorporation. The intrinsic stress can lead to the formation of cracks in the growing GaN layer during growth which can be closed again with increasing layer thickness. Such cracks in the layer can induce also microcracks into the sapphire surface [4]. Additionally, bowing occurs due to extrinsic stress induced during post-growth cool-down caused by differences in the thermal expansion coefficients. With increasing GaN layer thickness the extrinsic stress can lead to macrocracks and wafer breakage [5]. However, even if wafer breakage can be avoided [6], the bowing does hardly allow for polishing of the surfaces and further use in epitaxy remains difficult due to inhomogeneous thermal contact across the substrate holder. Therefore, separation of thick GaN layers from sapphire substrates resulting in freestanding GaN substrates is required. Post-growth laser separation [7] is feasible but difficult and unreliable [8]. The use of TiN interlayers [9] is published to be a viable method but probably requires extensive optimization.

In this study, it is tried to exploit the different crack formation we have observed for ELOG in comparison to planar growth to obtain separation of GaN from the substrate. Bowing and crack formation were investigated for different sapphire thicknesses for planar growth and for ELOG with different mask materials to improve the insight into the mechanisms.

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2 Experimental

The HVPE growth of GaN layers was performed in a horizontal AIX-HVPE reactor described in [6] at a total reactor pressure of 800 hPa and a growth temperature of 1040 °C. GaCl formed from metallic Ga and HCl with a flow rate of up to 125 sccm and NH_3 at a V/III ratio of 5 is transported in a H_2/N_2 carrier gas mixture resulting in planar growth rates of 150 µm/h [10]. Templates with 2 µm GaN layers grown by MOVPE on (0001) sapphire substrates with thicknesses of 330 µm and 100 µm were used. For HVPE growth via ELOG, a mask was deposited on such templates. The mask material with a thickness of 100 nm was either SiN_x deposited by PECVD or sputtered WSiN. The mask pattern consisting of hexagonally aligned circular openings with 5 µm radius and a periodicity of 15 µm was fabricated with optical lithography and dry etching. The layer thickness was determined gravimetrically or by micrometer screw. The curvature of the GaN layers was measured at room temperature using a Tencor Alphastep AS-IQ. Selected GaN layers were characterized by high resolution X-ray diffraction.

3 Results and discussion

The growth on templates with 330 µm thick sapphire substrates without masks resulted in mirror-like smooth surfaces with hexagonal pyramidal structures as typically observed for the HVPE growth of GaN on sapphire [10]. Layers grown with masks had similar morphology after coalescence but smoothed further due to additional hexagonal plateaus increasing in diameter with increasing layer thickness. Wafer breakage during cool down was usually observed when the GaN layers grown on the templates without ELOG mask exceeded thicknesses of 40 µm. Under the same conditions GaN layers grown using a SiN_x mask did not break up to a GaN thickness of 180 µm. In Fig. 1 optical micrographs focussed on the GaN/sapphire interface of GaN layers of 35 µm thickness with crack-free surfaces grown without and with a SiN_x mask are shown. Cracks in the GaN which also induce microcracks in the sapphire substrate can be observed for the maskless growth. In contrast, using ELOG no cracks were formed, i.e. the intrinsic tensile stress in the GaN layer occuring during growth was effectively reduced due to ELOG.

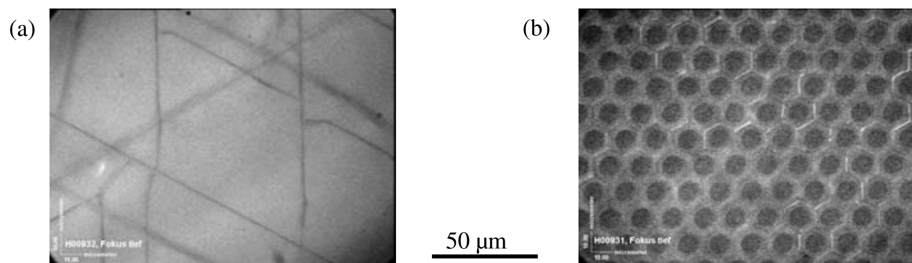

Fig. 1 Optical micrographs with the focus on the interface of GaN layers of 35 µm thickness grown without mask (a) and with SiN_x mask (b).

The thickness and the curvature κ, i.e. the reciprocal bending radius R, of GaN layers on 330 µm thick sapphire for maskless growth and for ELOG with SiN_x mask are shown in Fig. 2. It can be seen that the values of curvature with and without ELOG are similar. Calculations for the curvature and the maximum in-plane stress which occurs at the GaN/sapphire interface were performed using a bilayer approach consistent with [4]. For simplicity, temperature independent biaxial elasticity moduli calculated from stiffness constants and thermal expansion coefficients were assumed for GaN [11, 12] and sapphire [13]. Despite these simplifications and the spread of the available literature data, there is good agreement between the calculated curves and the measured data. The model shows that the bowing due to the thermal mismatch increases with GaN layer thickness up to 180 µm while for higher thicknesses the bowing

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is predicted to decrease again. Additionally, the tensile stress at the sapphire surface increases as the GaN/sapphire thickness ratio increases while the compressive stress of the GaN at the interface decreases. Thus the use of a high GaN/sapphire thickness ratio should result in cracking only in the sapphire substrate which could lead to freestanding GaN layers. To investigate this issue thinner sapphire substrates of 100 µm were used. As can be seen in Fig. 2 the calculated maximum bowing for 100 µm substrates is shifted to lower thicknesses of the GaN layer with in-plane stress values similar to those for the thicker substrates. The maximum of curvature was indeed found to be shifted from GaN thicknesses of about 200 µm with κ of about -2.4 m^{-1} (R = -41 cm) on 330 µm thick sapphire to about 100 µm with κ of about –13.3 m^{-1} (R = –7.5 cm) on 100 µm thick sapphire substrates. The bowing of the GaN layers

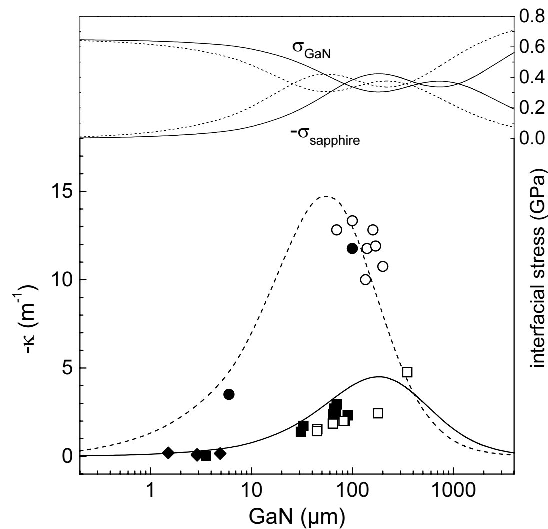

Fig. 2 Calculated curvature and interfacial stress vs. GaN layer thickness for sapphire substrates of 330 µm and 100 µm. Data points from GaN layers grown on 330 µm substrates by MOVPE without ELOG (◆), and by HVPE without ELOG (■), with ELOG (□), and on 100 µm substrates by HVPE without ELOG (●), and with ELOG (○).

grown on these substrates is also shown in Fig. 2. Indeed, for layer thicknesses exceeding the one of maximum curvature the bowing was found to decrease again. However, wafer breakage was always observed. In case of a maskless grown GaN layer of about 800 µm on 100 µm thick sapphire, the wafer already broke during growth due to the intrinsic tensile strain in the GaN. Partial delamination of the cracked sapphire was found on the remaining pieces of the wafer. Additionally, breakage in the GaN layer parallel to the growth plane was found. Therefore, it was concluded that the incorporation of a preferential fracture plane is of advantage. In the ideal case for ELOG the interfacial stress is concentrated on the reduced area of the window regions. Modelling of residual stresses for thermally strained GaN/Al_2O_3 heterostructures supports the assumption of crack formation along the interface in ELOG [14]. SEM micrographs of GaN layers obtained with the SiN_x mask showed that such horizontal cracks occur. But it was also found, that the GaN partially nucleated on the mask material preventing the concentration of the stress into the window region. To improve the ELOG process the nucleation of GaN on the mask material must be prevented. It is known from MOVPE growth [15] that tungsten masks act as a catalyst for decomposition of GaN by hydrogen radicals. Therefore, we used sputtered WSiN as mask material. A 60 µm GaN layer was grown in parallel on templates masked with SiN_x and WSiN for direct comparison. It was found after cool down that the GaN layer grown on the SiN_x mask sticked to the starting substrate whereas the one grown on the WSiN mask was completely peeled off. The growth of 600 µm GaN on a template with 330 µm thick sapphire using the WSiN mask resulted in the self-separated 2 inch freestanding GaN layer shown in Fig. 3. The radius of the curvature is positive, with κ =

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$0.8\ m^{-1}$ (R = 1.25 m), still indicating residual tensile strain built in during growth. The FWHM of ω-scans of the (0002) and (30-32) reflections are 180 and 122 arcsec which leads to an estimated density of dislocations at the surface of $2\times10^{8}\ cm^{-2}$ [16]. While measures for a further reduction of the dislocation density

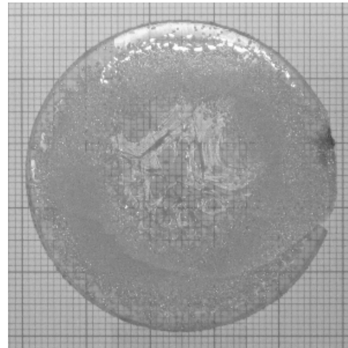

Fig. 3 Self-separated freestanding 2 inch GaN layer grown by HVPE using a WSiN mask for ELOG.

and also improvement of the surface morphology (elimination of v-pits) are necessary, the use of WSiN masks for ELOG in HVPE is a promising route to freestanding GaN substrates without post-growth separation steps.

4 Conclusion

The comparison of GaN layers grown by HVPE on maskless and masked GaN/sapphire templates showed that ELOG is useful to decrease tensile stress during growth and to prevent crack formation in the GaN layer. Using a SiN_x mask no effect of ELOG on the wafer bowing is observed. Partial sticking of GaN to the mask prevents the concentration of the stress into the window regions. Using WSiN masks sticking of GaN to the mask is strongly reduced and a preferential fracture plane parallel to the substrate is formed in the mask region. This resulted in selfseparation and freestanding GaN substrates as demonstrated for a 600 µm thick 2 inch layer.

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References

[1] B. Beaumont, Ph. Vennéguès, and P. Gibart, phys. stat. sol. (b) **227**, 1 (2001)

[2] S. Hearne, E. Chason, J. Han, J. A. Floro, J. Figiel, J. Hunter, and I. S. T. Tsong, Appl. Phys. Lett. **74**, 356 (1999).

[3] T. Böttcher, S. Einfeldt, S. Figge, R. Chierchia, H. Heinke, D. Hommel, and J. S. Speck, Appl. Phys. Lett. **78**, 1976 (2001).

[4] E. V. Etzkorn and D. R. Clarke, J. Appl. Phys. **89**, 1025 (2001).

[5] K. Hiramatsu, T. Detchprohm, and I. Akasaki, Jpn. J. Appl. Phys. **32**, 1528 (1993).

[6] E. Richter, Ch. Hennig, M. Weyers, F. Habel, J.-D. Tsay, W.-Y. Liu, P. Brückner, F. Scholz, Yu. Makarov, A. Segal, and J. Kaeppeler, J. Cryst. Growth **277**, 6 (2005).

[7] M. K. Kelly, R. P.Vaudo, V. M. Phanse, L. Görgens, O. Ambacher, and M. Stutzmann, Jpn. J. Appl. Phys. **38**, L217 (1999).

[8] P. R. Tavernier and D. R. Clarke, J. Appl. Phys. **89**, 1527 (2001).

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[9] Y. Oshima, T. Eri, M. Shibata, H. Sunakawa, and A. Usui, phys. stat. sol. (a) **554**, 194 (2002).
[10] E. Richter, Ch. Hennig, H. Kissel, G. Sonia, U. Zeimer, and M. Weyers, phys. stat. sol. (c) **2**, 2099 (2005).
[11] J.-M. Wagner and F. Bechstedt, Phys. Rev. B **66**, 115202 (2002).
[12] V. Kirchner, H. Heinke, D. Hommel, J. Z. Domagala, and M. Leszczynski, Appl. Phys. Lett. **77**, 1434 (2000).
[13] online information from sapphire suppliers: www.mkt-intl.com, www.tydex.ru
[14] W. M. Ashmawia, M. A. Zikrya, K. Wang, and R. R. Reeber, J. Cryst. Growth **266**, 415 (2004).
[15] H. Miyake , M. Yamaguchi, M. Haino, A. Motogaito, K. Hiramatsu, S. Nambu, Y. Kawaguchi, N. Sawaki, Y. Iyechika, T. Maeda, and I. Akasaki, MIJ-NSR Vol. 5S1, Art. W2.3 (1999).
[16] T. Böttcher, PhD thesis, University of Bremen (2002).

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