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Bending in HVPE GaN free-standing films: effects of laser lift-off, polishing and high-pressure annealing

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We have studied the effects of laser lift-off and polishing processes on the bending of free-standing HVPE grown GaN thick films. Their structural characteristics were accessed by reciprocal space mapping and lattice parameters measurements as well as by Raman scattering and photoluminescence. The in-plane strain difference between the two faces was found to have determining effect on the bending of the free-standing films. Removing the high-defect-density near-interface region either by melting caused by laser lift-off, or by polishing, or by point defects dissociation caused by high-pressure annealing was found to lead to flattening of the strain distribution along the film thickness and a significant reduction of the bending of the free-standing films.

1 Introduction

Free-standing thick GaN films grown by hydride vapor phase epitaxy (HVPE) and separated from the substrate are currently considered as the best substitution of a real bulk substrate material for subsequent device growth. Such free-standing HVPE-GaN 2” wafers are now offered on the market, although still very expensive and having a few critical problems that need to be resolved. Several groups have reported a significant improvement of the quality of the films with increasing thickness, manifested in a strong reduction of dislocations, impurities, native defect densities and strain along the thickness of the HVPE-GaN films [1–4]. However, this inhomogeneous defect distribution is believed to lead to a significant wafer bending [5] and consequently may contribute to the cracking [6], the latter being the most crucial problem, hampering the development of the HVPE free-standing quasi-substrate material and increasing the production cost.

In this work, we study the bending and residual strain distribution in free-standing (FS) HVPE-GaN films separated from the substrate under different conditions. High resolution X-ray diffraction (XRD) measurements of the bending and lattice parameters at both faces, as well as reciprocal space mapping were used to assess the structural characteristics. High-pressure high-temperature annealing was employed to force point defects redistribution, and Raman scattering and photoluminescence spectroscopies were employed to reveal its effect on the bending, aiming to understand the impelling reasons for this phenomenon in FS HVPE-GaN thick films.

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2 Samples and experimental procedure

The HVPE-GaN films with thickness of about 270 μm were grown [7] on sapphire with two-step lateral overgrown metalorganic chemical vapor deposited GaN templates [8]. We studied samples that were either self-separated or separated by using the laser lift-off (LLO) technique [3] followed by polishing of the interface side of the film, as well as as-grown films residing on sapphire for comparison reasons. In addition, we studied self-separated free-standing GaN films annealed at high-pressure (10 kbar) and high-temperature (at 1150-1450 °C) in N_2 atmosphere.

We used high resolution XRD on a Philips X'Pert diffractometer with the $CuK\alpha_1$ radiation to estimate the bending of the samples by performing rocking curve measurements of the 006 reflection. The residual strain in both faces of the free-standing films were determined by using the lattice parameters, following the procedure described in details in Ref. [9]. Reciprocal space maps (RSMs) were recorded as consecutive coupled 2θ-ω scans, each separated by an ω offset. The stress distribution along the thickness was assessed by micro Raman scattering (RS) performed at room temperature with 514 nm line of Ar^+ laser in backscattering geometry in cross-sections of the films. Complementary low temperature (LT) photoluminescence (PL) measurements were performed under excitation with the 266 nm laser line with an excitation power of 5 mW and a spectral resolution better than 0.3 meV at 350 nm.

3 Results and discussion

3.1 Effect of substrate separation

We first evaluate the effect of substrate removal on the bending of the FS films in relation with their structural properties as revealed by RSMs (Fig. 1) and lattice parameters (Table 1). We found a bending with concave shape in all the free-standing films, contrary to the convex-shape bending in films residing on sapphire. Among the free-standing films, the self-separated ones were found to possess the highest bending although smaller than that in the as-grown film, residing on sapphire. It is reflected also in the RSMs which are less elongated in the lateral direction due to the smaller bending contribution to the

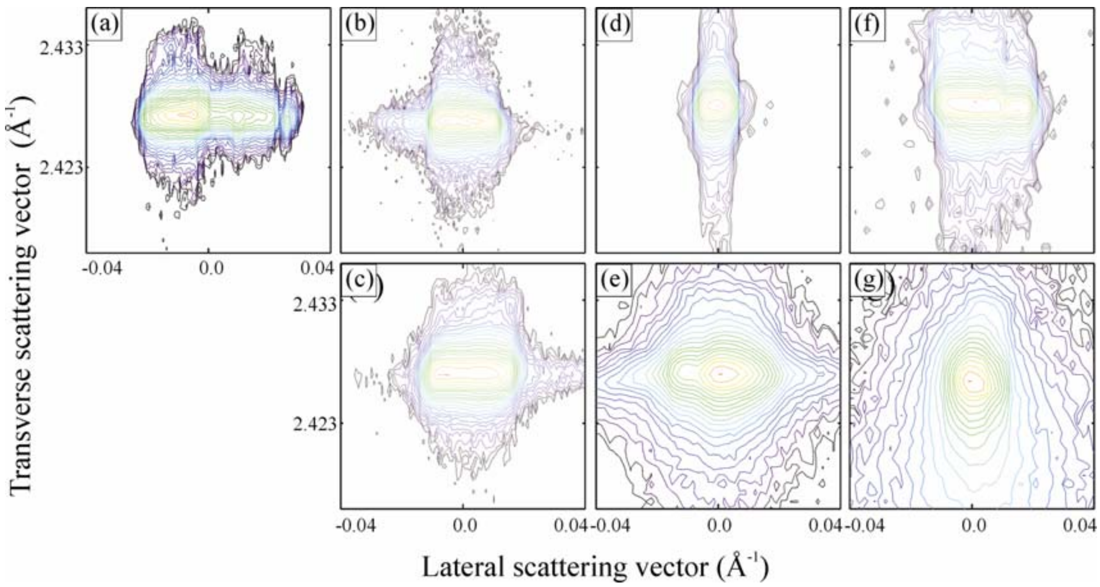


Fig. 1 Reciprocal space maps around the GaN 0002 reciprocal space point of a 270 μm-thick HVPE-GaN film: (a) residing on sapphire; (b,c) FS self-separated; (d,e) FS after laser-lift off separation; (f,g) FS after LLO and polishing taken from the Ga face (a,b,d,f) and N face (c,e,g) of the free-standing films.

Table 1 Bending, lattice parameters *c* and *a* with their standard errors and strain components in growth and in-plane directions for an as-grown thick film residing on sapphire and three differently separated free-standing HVPE-GaN films.

		Ga face		N face	
	R (m)	*c, a* (Å)	$(c-c_0/c_0)(10^{-5})$ $(a-a_0/a_0)(10^{-5})$	*c, a* (Å)	$(c-c_0/c_0)(10^{-5})$ $(a-a_0/a_0)(10^{-5})$
as-grown	-0.30	*c*=5.18516(2)	1.2±0.4		
		a=3.18900(3)	-9.3±0.6		
FS – self-separated	0.43	*c*=5.18514(8)	0.9±0.4	*c*=5.18528(5)	3.4±0.4
		a=3.18903(8)	-8.2±0.6	*a*=3.18923(3)	-2.1±0.6
FS – laser lift-off	1.21	*c*=5.18514(2)	0.8±0.4	*c*=5.18527(3)	3.3±0.4
		a=3.18910(8)	-6.0±0.6	*a*=3.18922(6)	-2.3±0.6
FS – LLO +polishing	4.60	*c*=5.18511(3)	0.2±0.4	*c*=5.18511(5)	0.3±0.4
		a=3.18925(8)	-1.3±0.6	*a*=3.18928(8)	-0.4±0.6

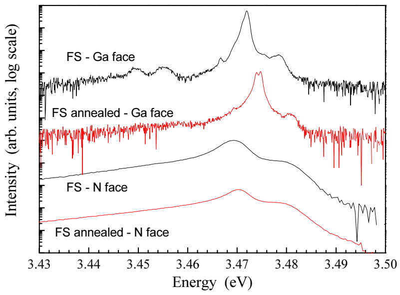
rocking curve linewidth of the main 002 reflection. The residual strain in the Ga face in the self-separated film was slightly reduced with respect to that in the as-grown film due to the release of the biaxial compressive component by substrate removal. The latter was confirmed in several PL studies of the strain in our and similar HVPE-GaN samples. The largest bending in this type of FS films (Table 1) could be explained by the largest difference in the *a* lattice parameters and consequently the largest in-plane strain difference in the two faces.

The bending was reduced by about a factor of 3 in FS films separated from the substrate by laser lift-off technique. The difference between the in-plane residual strains in both faces of such films was much less compared to the respective value in self-separated films. It is visualised in the RSMs (Fig. 1 (d,e)) that single domains are now possible to be recorded due to the reduced effect of bowing, although the main peak is still significantly elongated in the lateral direction due to film mosaicity and the presence of screw and mixed types of dislocations.

The bending has the smallest value (10 times smaller compared to the bending in the self-separated films) in free-standing films separated by laser lift-off, followed by polishing of the interface side. Removing of about 50 µm from the highly defective N-face side of the FS films results in an equalizing of the in-plane lattice parameters being much closer to the values of the reference GaN power constant (a_0 = 3.1893 Å [10]) and thus, leading to a very small difference in the in-plane strains between the two faces. The polishing of the GaN, however, could introduce additional impurities and/or structural defects. One can see in the RSM of the polished N face of the FS film a strong asymmetry with small broadening of the main map in the radial direction, indicating some grain tilting probably due to defect inclination.

3.2 Effect of high pressure annealing

Aiming to reveal which defects have the determining effect on the bending and residual strain in the free-standing HVPE GaN films we studied a series of FS self-separated films annealed at four different temperatures in the range of 1150 to 1450 °C. The HP annealing treatments were found to lead to dissociation of Ga vacancy related defects, both oxygen complexes and cluster defects [11], which is in principle expected, but also to Ga vacancy recovery at very high annealing temperature. The same is confirmed by the LT PL spectra taken from both faces before and after the HP annealing (Fig. 2). Consequently, the complex point defect redistribution influences the lattice parameters, leading to equalizing of the residual stress in the both faces. Such a flattening of the stress distribution was revealed in HP annealed FS HVPE-GaN film, by RS measurements. Figure 3 shows the frequency shift of the E_2 phonon mode, known to be sensitive to the strain, with respect to the strain-free mode position, and the respective stress [12] along the film thickness. Contrary to the well known exponential dependence of the strain distribu-



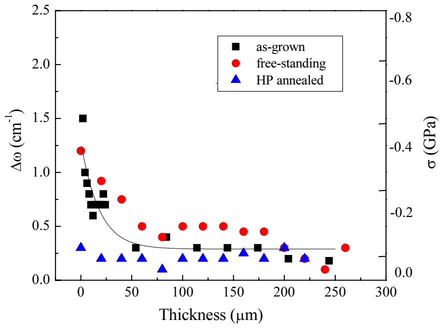


Fig. 2 (left) Low temperature PL spectra of FS self-separated films before and after the HP annealing at 1450 °C taken from both the Ga and N faces.

Fig. 3 (right) Frequency shift of the E_2 mode and residual stress as a function of the distance from the sapphire interface side of three representative films.

tion in thick HVPE-GaN films, both residing on sapphire and FS, the uniform distribution of the strain along the film thickness in the HP annealed samples (Fig. 3) indicates a strong effect of the point defect redistribution on the residual strain. The strain is, however, still nonzero and thus, the bending in FS HVPE-GaN films still persists.

In conclusion, we found that the bending in FS HVPE-GaN films is strongly influenced by the substrate separation conditions. The largest bending is found in self-separated films, while samples separated by the laser lift-off technique and followed by polishing experience the smallest bending. The in-plane strain difference between the two faces was found to dominate the bending of the FS films. Point defect dissociation induced by HP annealing resulted in flattening of the strain distribution along the film thickness and reducing of the bending. Removing the higher-defect-density interface side of the films leads to further decreasing of the bending. A controllable laser lift-off process is preferable and the polishing seems to be the way for reducing the defect density gradients and bending, however, a precise optimisation of both processes is required to avoid additional contaminations and complications.

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