Annealing AlN Capped, MOCVD Grown GaN for Extended Periods of Time at Elevated Temperatures

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GaN films contain large numbers of crystalline defects when they are grown on heterostructure substrates. Additionally, the ion implant process generates additional point defects. Crystalline defect concentration can not be reduced by annealing under normal circumstances because nitrogen preferentially evaporates at temperatures required for diffusion to occur. Many papers have been written on rapid thermal annealing of ion implanted GaN with various caps[1,2,3,4,5,6,7]. Annealing above 1100 ˚C for more than two minutes[8] usually results in GaN decomposition. However, GaN can be capped with an AlN film to prevent decomposition, which has a lower nitrogen partial pressure, and AlN can subsequently be etched off preferentially. The challenge is to make certain the AlN adheres well to, and completely covers, the GaN. We demonstrate good coverage and adhesion to the flat surfaces of (0001) oriented films by first growing a 70 nm AlN adhesion layer at 700 ºC by MOCVD and then sputtering a 900 nm AlN film at 500 ºC for greater mechanical strength. [9,10,11,12] The AlN capped unintentionally doped GaN was annealed for 30 minutes at temperatures ranging from 1150 to 1300 ˚C or at 1150 ˚C for different times ranging from 15 to 120 min. When the proper film thicknesses are used and the surface of the GaN film is flat the cap remains intact. When the film deposition parameters are not optimum, the cap can crack, or polycrystalline discoids can form in the cap when AlN grows over hexagonal flat bottoms with semipolar side walls in the film as seen in Figure 1. Polycrystalline discoids can later be dislodged by the nitrogen pressure, and nanorods nucleate and grow underneath them shown in Figure 1c). Nanorods > 10 µm long and ~ 100 nm in diameter can be created. In figure 2b) & c) AFM scans show no thermally produced etch pits created in the smooth portions of the GaN film, but there is atomic movement as is indicated by the increase in the surface roughness with annealing time and temperature as determined by AFM and optical profilometry. The average RMS surface roughness of the originally smooth portions of the films is 0.395 nm before anneal, 0.371 nm after the 1150 ºC anneal, and 1.313 nm after the 1300 ºC anneal. The variable time series samples roughened with increasing time. Failure from edges inward also increased with time. That there has been atomic movement is further illustrated by the fact that the average peak width for the $(10\underline{1}2)$ asymmetric x-ray rocking curve values in Figure 3 often decrease, sometimes more than 50", while the peak width for the symmetric (0002) peak tends to decrease by smaller amounts. This suggests that there has been a decrease in the rotation of the domains about the *c*-axis possibly through grain growth. Figure 4 shows a representative topograph of the GaN surface before anneal that measured large variation in warpage like concave, saddle point, flat and convex. After anneal all samples were convex with a bow between 1.5 and 2.5 microns from center to edge (1 cm^2). This reproducible matching bow can be attributed to relaxation at annealing temperature, then bowing on cooling of GaN film on a sapphire substrate with different coefficients of thermal expansion. The Hall data also shows that annealing drives the film towards its equilibrium concentration as the net carrier concentration can be reduced by almost a factor of two, and the mobility also decreases by ~1/3 even though the crystalline structure has improved. One possible explanation is that the carbon incorporated during the MOCVD growth of the films has moved from an interstitial site to a substitutional N-site where it is an acceptor. That the point defects configuration is altered by the annealing is also shown in the PL data.

Figure 1: SEM images of a) GaN surface with hexagonal pits and b)&c) AlN cap that cracked during anneal with GaN nanorods nucleating under polycrystalline AlN discoids seen on top of rods

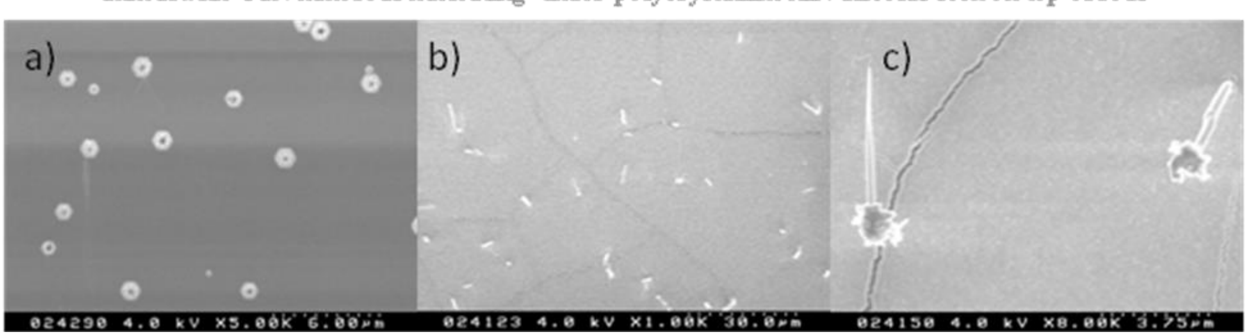


Figure 2: AFM topographs of GaN surface a) before anneal, b) after 1150 °C anneal for 30 minutes and c) after 1300 °C anneal for 30 minutes with the AlN capped removed by 2M KOH solution.

a) Control sample b)1150 °C 30min c)1300 °C 30min

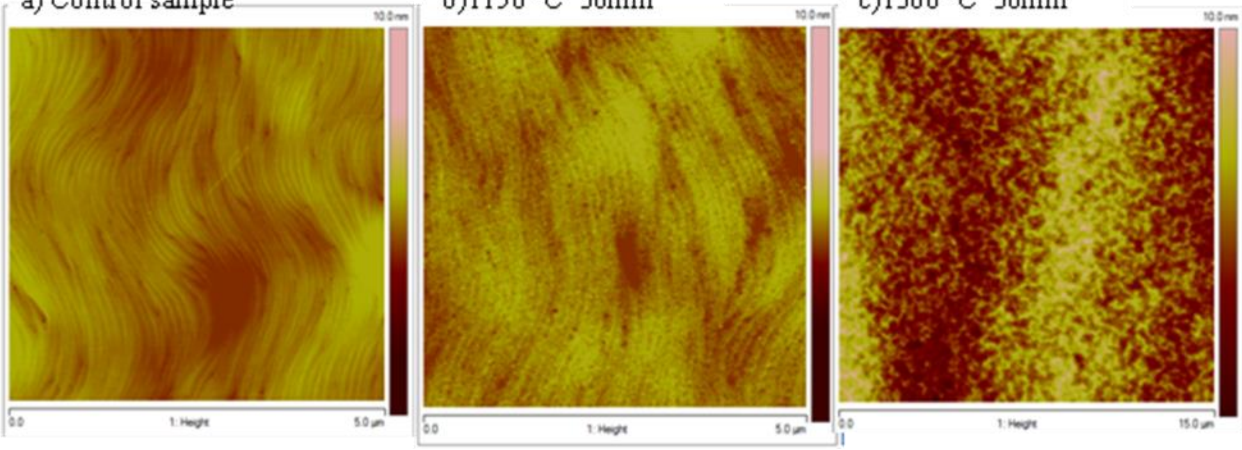


Figure 3: Table of X-ray diffraction rocking curve FWHM values before and after anneal of the (002) and (102) planes and a representative plot of the 1200 °C 30 minute anneal.

Temp °C	Time min.	pre-anneal (002)arcsec	annealed (002)arcsec	pre-anneal (102)arcsec	annealed (102)arcse
1150	30	275 (±1)	251 (±1.7)	416 (±8.2)	389(±12.9
1200	30	271 (0)	248 (±5.6)	410(±10.5)	356 (±9.2)
1250	30	274 (±1)	227 (±2.1)	421 (±9.8)	381(±16.3
1300	30	271 (0)	227 (±4)	398 (±11)	353 (±3)
1150	15	307 (±7.5)	268 (±3.1)	458 (±5.6)	397(±10.7
1150	30	275 (±1)	251 (±1.7)	416 ±(8.2)	389(±12.9
1150	60	244 (±3.2)	232 (±4.7)	409 (±2.6)	362(±11.7
1150	90	282 (±2.2)	250 (±3.2)	427 (±7.5)	415 (±1)
1150	120	276 (±3.8)	240 (±1.2)	433 (±5.4)	430 (±4.6)

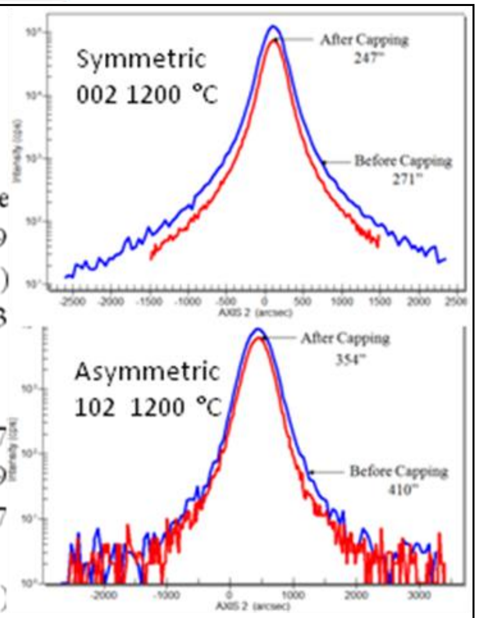
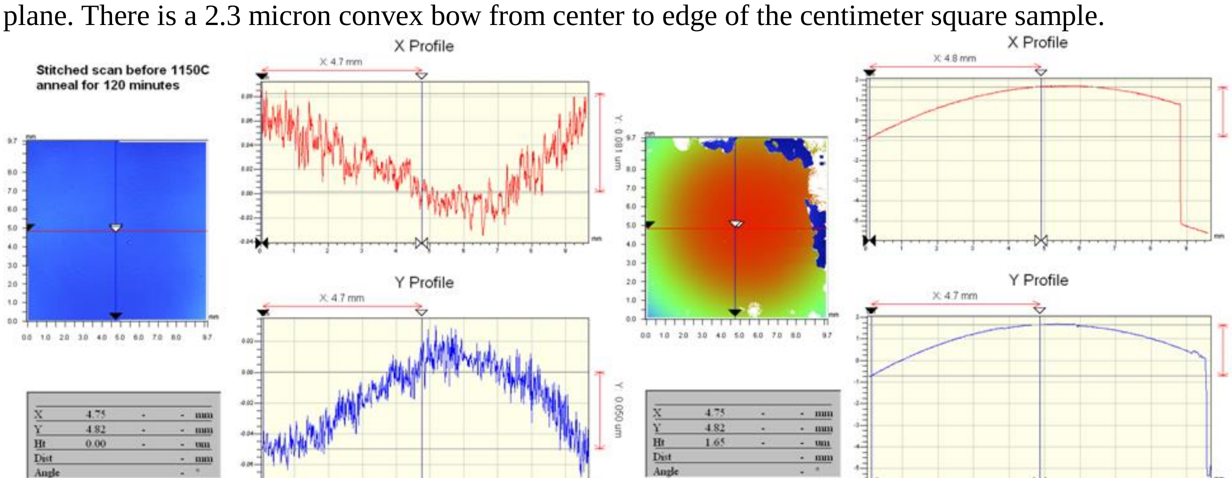


Figure 4: The first topograph is of the GaN surface before capping with AlN and annealing at 1150C for 120 minutes. The sample is fairly flat except for a slight horizontal concave and vertical convex bow. The second topograph is after anneal at 1150 ºC for 120 minutes and AlN cap removal. Most of the GaN survived the anneal process except for the right and top edges. The deep blue areas are where the GaN decomposed down to the sapphire substrate which left a 6 micron vertical side wall along a more stable plane. There is a 2.3 micron convex bow from center to edge of the centimeter square sample.



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