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Citation: J. Appl. Phys. 87, 955 (2000); doi: 10.1063/1.371966
View online: http://dx.doi.org/10.1063/1.371966
View Table of Contents: http://jap.aip.org/resource/1/JAPIAU/v87/i2
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Ion channeling studies on mixed phases formed in metalorganic chemical vapor deposition grown Mg-doped GaN on Al$_2$O$_3$(0001)

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(Received 19 July 1999; accepted for publication 13 October 1999)

Rutherford backscattering spectrometry and ion channeling were used to determine the relative quantities of wurtzite and zinc-blende phases in metalorganic chemical vapor deposition grown Mg-doped GaN(0001) on an Al$_2$O$_3$(0001) substrate with a GaN buffer layer. Off-normal axial channeling scans were used. High-resolution x-ray diffraction measurements also confirmed the presence of mixed phases. The in-plane orientation was found to be GaN[110]∥GaN[1120]∥Al$_2$O$_3$(1120). The effects of rapid thermal annealing on the relative phase content, thickness and crystalline quality of the GaN epilayer were also studied. © 2000 American Institute of Physics. [0001-8979(00)01802-8]

The growth of GaN epilayers has been under extensive investigation because they are attractive for applications in blue light emitting devices, high-temperature and high-power devices at microwave frequencies, etc. It is known that GaN exists in two polymorphs: wurtzite and zinc-blende structures with direct band gaps of 3.4 and 3.2 eV, respectively. The wurtzite (hexagonal-close-packed (hcp)) phase is more stable than the zinc-blende (cubic) phase. There is only a small difference between the energies of formation of these two phases of GaN. In most cases of GaN growth on Al$_2$O$_3$, the zinc-blende phase is found to coexist with the predominant wurtzite phase. The presence of mixed phases in GaN has been identified by many techniques such as x-ray diffraction (XRD), photoluminescence, cathodoluminescence, Raman spectroscopy, x-ray photoelectron spectroscopy, near edge x-ray absorption fine structure spectroscopy, transmission electron microscopy and reflection high energy electron diffraction. In this communication, we report the identification of mixed phases and the effects of rapid thermal annealing (RTA) on p-type, Mg-doped, metalorganic chemical vapor deposition (MOCVD) grown GaN using Rutherford backscattering spectrometry (RBS) and ion channeling. Complementary high-resolution x-ray diffraction measurements were also done to support the presence of cubic and hcp phases in GaN.

The p-type, Mg-doped GaN samples in this study were grown on (0001) sapphire substrates by MOCVD in a horizontal reactor. The substrates were cleaned by organic solvents, etched by H$_2$SO$_4$:H$_3$PO$_4$=3:1 solution at about 200 °C, followed by a de-ionized water rinse. Trimethylgallium and ammonia were used as the sources of Ga and N, respectively. Biscyclopentadienyl magnesium was employed as a p-type dopant. The carrier gas was hydrogen, and the growth pressure was 76 Torr. Prior to growth, the substrates were outgassed at 1120 °C. A GaN buffer layer of 30 nm thickness was grown at 550 °C. The GaN epilayer was grown at a substrate temperature of 1080 °C. The as-grown Mg-doped GaN films were converted to conductive p-type material by RTA. Different samples from the same wafer were annealed at 750, 850, 950, and 1050 °C by RTA in a nitrogen atmosphere for 1 min. Then, a small area in each sample was etched by reactive ion etching (RIE) to the substrate surface for RBS and ion channeling measurements on the substrate without dechanneling from the epilayer. RBS and ion channeling measurements were carried out with a 2 MeV He$^{++}$ beam from a 2 MV tandem accelerator at The Chinese University of Hong Kong. The ion beam size was 1 mm in diameter with a divergence of 0.05°. A surface barrier detector with a resolution of 18 keV was used at a scattering angle of 170°. High-resolution XRD measurements were done with Cu $K_a_1$ ($\lambda = 0.154 05$ nm) x rays monochromated by a four-crystal Bartels monochromator.

RBS and ion channeling measurements have been done on as-grown Mg-doped GaN and annealed samples. Figure 1 shows typical RBS spectra of the as-grown and 950 °C-annealed samples with the incident beam along a random direction and the beam aligned along the [0001] direction of GaN. The width of the Ga signal in the random spectrum of the annealed sample is less than that of the as-grown sample due to a decrease in thickness of the GaN epilayer upon annealing. In the aligned spectra, a large change in the slope of the Ga signal is seen near the lower energy side which is due to the presence of defects at the GaN/Al$_2$O$_3$ interface. The yield in the annealed sample is lower than the yield of the as-grown sample, indicating improvement of crystalline quality upon annealing. The ratio of the yield near the surface in the aligned condition to the yield in the random condition is called $\chi_{\text{min}}$ and is a measure of the crystal-
The in-plane orientation is attributed to the chemical bonding between Al$_2$O$_3$ and GaN.\textsuperscript{8} Coincident site lattice matching favors the in-plane orientation.

We speculate that the incorporation of the Mg dopant during MOCVD growth can alter the surface energies such that coincident site lattice matching is preferred. It is also known that MOCVD grown GaN films contain small inversion domains.\textsuperscript{10} If the growth conditions had favored the formation of inversion domains in the nucleation layer, coincident site lattice matching would be preferred.

In addition to channeling in the [0001] direction which is near the surface normal, channeling was also done at tilt angles of 35.26\(^\circ\) and 43.22\(^\circ\) in the \{112\} plane which corresponds to the [110] or [114] axis of the cubic phase (whose [111] orientation is parallel to [0001]) and the [1102] axis of the hcp phase, respectively. Figure 3 shows the angular dips for the 850 °C-annealed sample. In spite of a very low \(\chi_{\text{min}}\) for the [0001] axis, the \(\chi_{\text{min}}\) for the [1102] axis is very large (0.7) compared to the reported value of 0.3.\textsuperscript{11} The higher value of \(\chi_{\text{min}}\) can be attributed to the presence of both hcp and cubic phases of GaN which is also obvious from the dips observed around 35.26° from the [0001] axis. The dip due to channeling along the [110] axis should be much deeper than the channeling along the [114] axis. But both the dips are of comparable depth in Fig. 4. This implies that the zinc-blende structure exists in twins corresponding to ABCABC and CBACBA packing of equal proportion which is in agreement with previous reports.\textsuperscript{5} The presence of the [110] and [114] axes of the cubic phase in the \{112\} plane indicates that one of the \{110\} planes of the cubic phase is parallel to \{112\} planes of the hcp phase and that the in-plane orientation is GaN[110]|GaN[1120]. Similar in-plane orientation between the cubic and hcp phases was observed in the GaN growth on Al$_2$O$_3$.

### Table I. Ion channeling and high-resolution XRD results of GaN/Al$_2$O$_3$ (0001) annealed at various temperatures by RTA.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness (nm)</th>
<th>(\chi_{\text{min}}) of GaN along [1102]</th>
<th>(\chi_{\text{min}}) of GaN along [114]</th>
<th>(\chi_{\text{min}}) of GaN along [0001]</th>
<th>(\chi_{\text{min}}) of GaN along [110]</th>
<th>(\chi_{\text{min}}) of GaN along [1102]</th>
</tr>
</thead>
<tbody>
<tr>
<td>As grown</td>
<td>1210</td>
<td>0.69</td>
<td>0.81</td>
<td>0.050</td>
<td>0.30</td>
<td>0.59</td>
</tr>
<tr>
<td>750 °C</td>
<td>1200</td>
<td>0.59</td>
<td>0.73</td>
<td>0.048</td>
<td>0.44</td>
<td>0.47</td>
</tr>
<tr>
<td>850 °C</td>
<td>1100</td>
<td>0.68</td>
<td>0.57</td>
<td>0.026</td>
<td>0.56</td>
<td>0.69</td>
</tr>
<tr>
<td>950 °C</td>
<td>1140</td>
<td>0.72</td>
<td>0.68</td>
<td>0.019</td>
<td>0.26</td>
<td>0.45</td>
</tr>
<tr>
<td>1050 °C</td>
<td>1010</td>
<td>0.79</td>
<td>0.79</td>
<td>0.019</td>
<td>0.50</td>
<td>0.53</td>
</tr>
</tbody>
</table>

FIG. 1. RBS and ion channeling spectra for the as-grown and 950 °C-annealed GaN/Al$_2$O$_3$ samples taken with a 2 MeV He$^+$ beam incident along the [0001] direction and at a random direction.

FIG. 2. Azimuthal angular scan from GaN and Al$_2$O$_3$ with a 2 MeV He$^+$ beam at a tilt angle of 5° for the 850 °C-annealed sample.
a Si(111) substrate with a GaN buffer layer.

Off-normal axial channeling measurements were also done in the other samples and $\chi_{\text{min}}$ values are given in Table I. Unlike the case of the 850 °C annealed sample, the $\chi_{\text{min}}$ of the [110] and [114] axes in other samples are different, implying that the twins are not of equal proportion. This might have given rise to the unequal $\chi_{\text{min}}$ for the [1102] and [1102] axes. But if we look at the $\chi_{\text{min}}$ values of the [1102] axis of all the samples, we can see that it becomes smaller in all the annealed samples except in the case of 850 °C compared to the as-grown sample. This is due to the partial conversion of cubic to hexagonal phase which is in agreement with the results of Wu et al. So the presence of mixed phases can be identified by RBS and ion channeling from the off-normal axial scans. Although we can get qualitative information on the presence of mixed phases from the dips in the tilt angular scan in the [1120] plane, quantification of the phase content from the $\chi_{\text{min}}$ values is complicated due to the twin structure in the cubic phase.

To confirm the RBS and ion channeling results, complementary high-resolution x-ray diffraction was done in the [1120] plane for the 850 °C-annealed sample. The $\chi_{\text{min}}$ values of GaN along the [1102] and [110] axes can give information on the relative amount of cubic and hcp phases. But the results are affected by the presence of twins in the cubic phase and quantification becomes complicated. The in-plane orientation is found to be GaN(110)|GaN(1120)|Al$_2$O$_3$ (1120) which is favored by co-incident site lattice matching. Upon rapid thermal annealing above 750 °C in a nitrogen atmosphere, there is a partial conversion of the cubic phase to the hcp phase, an improvement in crystal quality due to a decrease in the mosaic spread and lattice defects and a decrease in thickness of the GaN layer due to desorption of GaN. High-resolution XRD measurements also confirm the presence of mixed phases.

This work was partly supported by Hong Kong Research Grants Council Grant No. PolyU107/96E.

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