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Ion channeling studies on mixed phases formed in metalorganic chemical vapor deposition grown Mg-doped GaN on Al₂O₃(0001)

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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 Al₂O₃(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]||Al₂O₃[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. [S0021-8979(00)01802-8]

The growth of GaN epilayers has been under extensive investigation because they are attractive for applications in blue light emitting devices,^{1,2} high-temperature and high-power devices at microwave frequencies,³ 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 Al₂O₃, the zinc-blende phase is found to coexist with the predominant wurtzite phase.⁴ The presence of mixed phases in GaN has been identified by many techniques such as x-ray diffraction (XRD),^{5,6} photoluminescence, cathodoluminescence,⁴ 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 H₂SO₄:H₃PO₄=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 $K\alpha_1$ ($\lambda=0.154\text{ }05\text{ nm}$) 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/Al₂O₃ 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 χ_{\min} and is a measure of the crystal-

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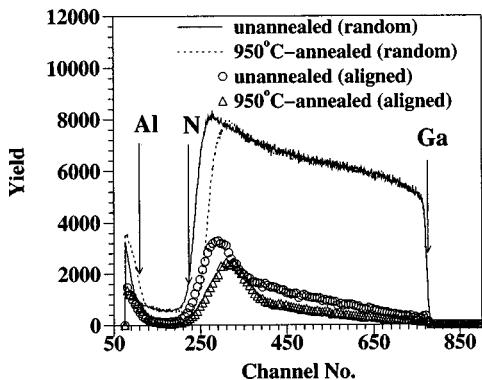


FIG. 1. RBS and ion channeling spectra for the as-grown and 950 °C-annealed GaN/Al₂O₃ samples taken with a 2 MeV He⁺ beam incident along the [0001] direction and at a random direction.

line quality and the values for the GaN[0001] axis in all the samples given in Table I. Lower χ_{\min} values upon annealing are due to a decrease in the mosaic spread of grains and lattice defects in the crystal. Thicknesses of the film measured from the width of the Ga signal in the random spectra are also given in Table I. The decrease in thickness of the film with an increase in annealing temperature can be attributed to desorption of GaN.⁷

There is no angular misorientation between the [0001] Al₂O₃ and [0001] GaN directions as is evident from the tilt angular scan about the [0001] axis which is not shown here. Figure 2 shows the azimuthal angular scan of the RBS yield from the GaN epilayer and the Al₂O₃ substrate taken at a tilt angle of 5° for the 850 °C-annealed sample. The Ga signal plotted is the normalized yield integrated in the window of channel numbers 690–740 (corresponding to a depth of 70–182 nm) of the RBS spectrum. The Al yield was taken from the substrate area exposed by RIE etching. The deepest dip corresponds to channeling in {1120} planes and we can see these dips for GaN as well as for Al₂O₃ at the same angular positions. This implies that the in-plane orientation is GaN(1120)||Al₂O₃(1120).

Our result is in contrast to the reported in-plane orientation of GaN(1010)||Al₂O₃(1120) in the growth of GaN(0001) on Al₂O₃(0001) with GaN or AlN buffer layers.^{8,9} Coincident site lattice matching favors GaN(1120)||Al₂O₃(1120) while GaN(1010)||Al₂O₃(1120) is attributed to the chemical bonding between Al₂O₃ and GaN.⁸

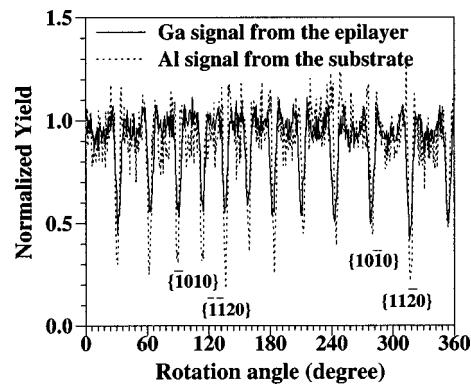


FIG. 2. Azimuthal angular scan from GaN and Al₂O₃ with a 2 MeV He⁺ beam at a tilt angle of 5° for the 850 °C-annealed sample.

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.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° and 43.22° in the {1120} 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 χ_{\min} for the [0001] axis, the χ_{\min} for the [1102] axis is very large (0.7) compared to the reported value of 0.3.¹¹ The higher value of χ_{\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.⁶ The presence of the [110] and [114] axes of the cubic phase in the {1120} plane indicates that one of the {110} planes of the cubic phase is parallel to {1120} 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

TABLE I. Ion channeling and high-resolution XRD results of GaN/Al₂O₃(0001) annealed at various temperatures by RTA.

Sample	Thickness (nm)	χ_{\min} of GaN along					% of GaN(222) x-ray intensity
		[1102]	[114]	[0001]	[110]	[1102]	
As grown	1210	0.69	0.81	0.050	0.30	0.59	37.6±10
750 °C	1200	0.59	0.73	0.048	0.44	0.47	44.6±8
850 °C	1100	0.68	0.57	0.026	0.56	0.69	35.9±8
950 °C	1140	0.72	0.68	0.019	0.26	0.45	38.2±6
1050 °C	1010	0.79	0.79	0.019	0.50	0.53	40.2±8

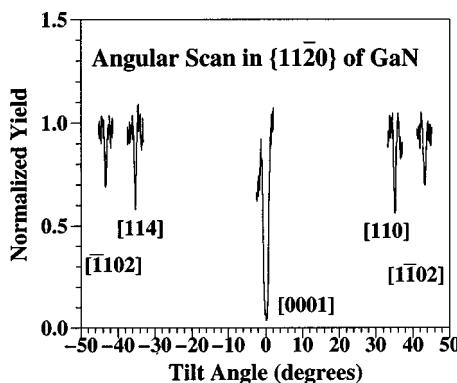


FIG. 3. Tilt angular scans from GaN with a 2 MeV He^{++} beam in the $\{11\bar{2}0\}$ plane for the 850°C -annealed sample.

a Si(111) substrate with a GaN buffer layer.¹²

Off-normal axial channeling measurements were also done in the other samples and χ_{\min} values are given in Table I. Unlike the case of the 850°C annealed sample, the χ_{\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 χ_{\min} for the $[1102]$ and $[1\bar{1}02]$ axes. But if we look at the χ_{\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.*¹³ 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 $\{11\bar{2}0\}$ plane, quantification of the phase content from the χ_{\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 all the samples. Since the wurtzite and zinc-blende structures differ simply in their packing sequences (*ABAB* and *ABCABC*, respectively), the wurtzite (0002) and zinc-blende (111) stack-

ing planes have almost the same lattice spacings. In the XRD peak of GaN(0002), it is difficult to resolve the peaks corresponding to GaN(0002) and GaN(111). But peaks due to GaN(0004) and GaN(222) can be resolved in the high-resolution θ - 2θ scan as was shown by Bel'kov *et al.*⁵ Figure 4 shows the θ - 2θ scan of GaN for the 950°C -annealed sample. The curve is fitted with two Gaussian components with mean values of 2θ at 72.70° and 72.82° which correspond to d -spacings of the cubic phase and hcp phase of GaN. Hence the presence of a mixed phase in GaN is evident and is in agreement with the ion channeling results. Out of the total x-ray intensity in the θ - 2θ scan shown in Fig. 4, the percentage of the intensity from the cubic component of GaN is given in Table I. Within the statistics of the high-resolution XRD data, we cannot tell whether the cubic phase content decreases upon annealing. But, the cubic phase content is likely to decrease upon annealing since it is metastable. This is evident from the lower χ_{\min} of GaN[1102] axis in the annealed samples, as discussed before.

In summary, RBS and ion channeling measurements in off-normal axis directions are used to identify the presence of mixed phases in MOCVD grown GaN on $\text{Al}_2\text{O}_3(0001)$. The χ_{\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 $\text{GaN}(110)\|\text{GaN}(11\bar{2}0)\|\text{Al}_2\text{O}_3(11\bar{2}0)$ which is favored by coincident 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.

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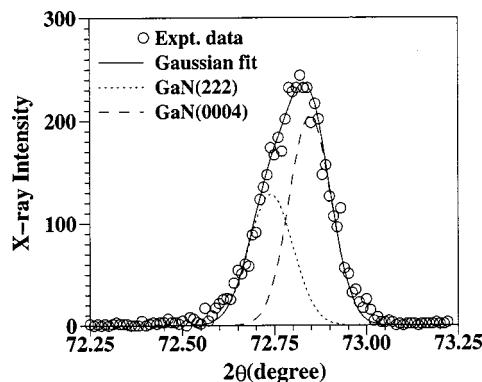


FIG. 4. High-resolution XRD peak from GaN with $\text{Cu K}\alpha_1$ x rays showing the cubic and hcp components for the 950°C -annealed sample.

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