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Channeling contrast microscopy of GaN and InGaN thin films

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Abstract

The recent development of blue and green light emitting diodes (LED) based on single quantum well structures made from GaN and related materials (AlGaN, InGaN) has created many efforts to achieve a complete characterisation of devices grown under various conditions. Here we report on CCM measurements on GaN thin films ($d = 0.7$–3.0 μm) grown by metal organic vapour phase epitaxy (MOVPE) and on 500 Å InGaN films grown epitaxially on top of the GaN thin films. The samples were analysed by broad beam channeling and channeling contrast microscopy (CCM), using 1–2 MeV H^+ and He^+ ions. Generally, very low minimum yields were found ($\chi_{min} = 2$–4%), indicating nearly perfect crystal structures. The susceptibility to ion–beam induced damage was assessed by random and channeled 1 MeV He^+ irradiation and subsequent CCM analysis. CCM also revealed the presence μm-sized regions in the InGaN films with increased In signal strength. The channeling PIXE data for 500 Å thin films are found to be in excellent agreement with the corresponding RBS results, allowing the determination of channeling yields of elements for which RBS data is difficult to obtain.

1. Introduction

The potential for the formation of devices operating from the red to the UV region of the energy spectrum has attracted special attention to GaN and related materials (e.g. InGaN and AlGaN) in recent years. Applications of short wavelength photonic devices range from display technologies and data storage to UV detectors. Therefore many efforts to achieve a complete characterisation of structures grown by various techniques and under various conditions are made in order to optimise the device growth processes [1–3].

Channeling contrast microscopy (CCM) used with backscattering (BS) and proton induced X-ray emission (PIXE) provides laterally resolved information on the crystal quality of single crystal and epitaxially grown samples, and the large reduction in the bulk signal also serves to increase the sensitivity to non-substitutional impurities [4]. We have used CCM to investigate GaN and InGaN/GaN thin films grown by metal organic vapour phase epitaxy (MOVPE) on [0 0 0 1] oriented Al_2O_3 substrates. The relatively large beam fluences used in CCM necessitate an assessment of the beam damage produced. We report on damage accumulation in GaN thin films for channeled and

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random implants of 1 MeV He^+. Furthermore we discuss preliminary results on micron sized regions found in 500 Å InGaN/GaN structures with increased In signal intensity. Finally, channeling BS and channeling PIXE data from these samples are compared.

2. Experimental

The growth process is briefly described here, details can be found in Ref. [5]. GaN thin films were grown on [0 0 0 1] orientated sapphire (Al_2O_3) by MOVPE. Trimethylengallium (TMGa), Trimethylenindium (TMIn) and NH_3 were used as precursors. Prior to the deposition of the 0.7–3.0 μm thick GaN films at 1040°C, a 250–300 Å thick GaN buffer layer was grown at 530°C. A 500 Å InGaN layer was deposited on top of the GaN film for a set of samples with different In mole fractions and dopants (e.g. Zn, Si), at a growth temperature of about 710°C in a N_2 and H_2 mixed ambience.

The measurements were carried out using the nuclear microscope facility at the National University of Singapore [6]. For the micro-channeling measurements a He^+ and H^+ beam of typically 100 pA were used. Channeling RBS spectra were recorded with a 50 mm^2 PIPS detector of 12 keV resolution at 160° scattering angle. PIXE spectra were recorded with a 62 mm^2 Si(Li) detector at a distance of 20 mm from the target. The targets were mounted on a eucentric goniometer with a 24 mm translational range for both the x and the y direction that allows rotations around the x and the y axis with a resolution of 0.1 mrad. The beam spot size was typically 1–2 μm for the CCM measurements. The broad beam data was taken in the same geometry, but the increased beam-spot sizes of 300 μm allowed currents of typically 1 nA.

3. Results and discussion

3.1. Beam damage

Fig. 1 shows the random incidence 1.5 MeV proton backscattering spectrum of a 2.2 μm thick GaN film on a Al_2O_3 substrate, and its deconvolution to the contributions from the different elements present, as calculated using experimental non-Rutherford scattering cross-sections for N and O by means of the BS simulation code SIMNRA [7]. The close agreement indicates that proton backscattering is well suited for the analysis of samples a few μm thick. Fig. 1 also displays the [0 0 0 1] axially channeled spectrum with a χ_{min} of 2% that indicates excellent crystal quality in the surface region. The peak in the channeled spectrum near the GaN/Al_2O_3 interface is due to the amorphous GaN buffer layer mentioned above. The GaN films did not show any contrast in axially or planar aligned CCM measurements.

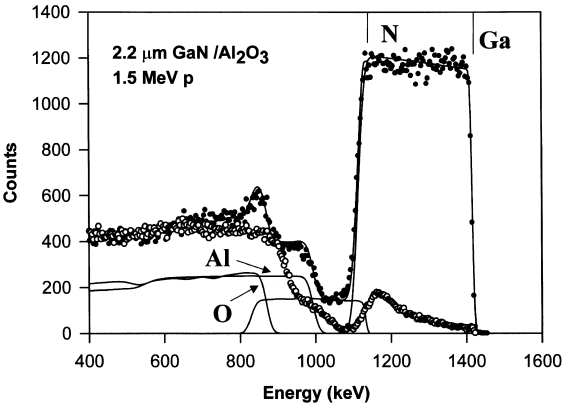

Fig. 1. Random and [0 0 0 1] channeled 1.5 MeV H^+ backscattering spectra from a 2.2 μm GaN/Al_2O_3 sample, together with the deconvolution of the signals from the elements present. For Ga and N the surface energies are indicated.

The beam damage to the GaN crystal lattice, both for random and channeled incidence of a 1 MeV He^+ beam, was assessed in the following way: a 100×100 μm^2 square was irradiated with a fluence of 7.8×10^{16} He^+/cm^2 with random incidence, and a different region was irradiated with a fluence of 7.4×10^{16} He^+/cm^2 in a [0 0 0 1] axially channeled geometry. Fig. 2 shows the CCM map of the randomly irradiated region from a subsequent CCM measurement where PIXE and BS spectra were simultaneously taken. A 1.5 MeV H^+ beam was used in this analysis. Clearly both the Ga-K X-ray yield and the backscattering yield from the Ga (the integral over the Ga signal, see

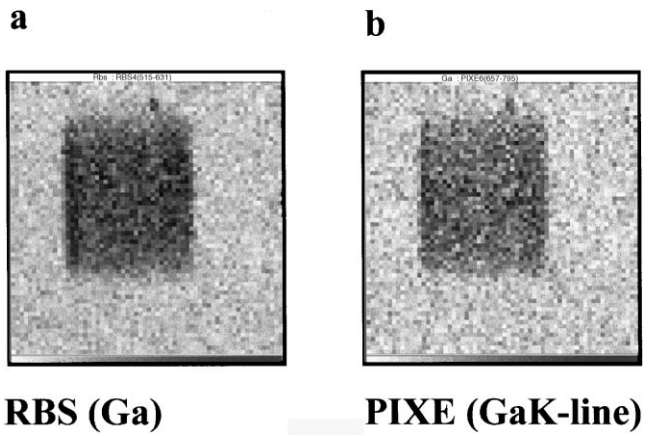

Fig. 2. 1.5 MeV H^+ backscattering CCM maps from the random implant. (a) The integral of the Ga backscattering signal is mapped. (b) The Ga–K intensity is mapped. The gray-scale indicates intensity, darker regions have higher intensities.

Fig. 1, are markedly increased. Fig. 3 shows the Ga-region of the BS spectra from both damaged and virgin regions. TRIM [8] results indicate that the 'end of range' damage peak for 1 MeV He^+ in GaN is located at a depth of 2.3 μm, below the GaN/Al_2O_3 interface, therefore the increased backscattering yield throughout the GaN clearly indicates that disorder was created in the GaN. No channeling contrast was found in the region of the channeled irradiation. Fig. 4 shows the Ga region of the BS spectra taken during the first and the last 20% of the channeled implantation. The spectra are very similar, thereby confirming that only negligible amounts of damage were produced. These results indicate that CCM maps of GaN can be taken without excessive damage to the lattice as long as prolonged irradiation with random incidence is avoided.

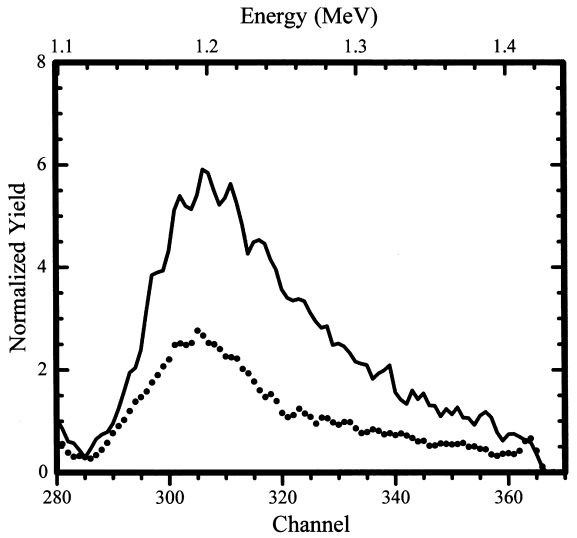

Fig. 3. The Ga-region in the channeled 1.5 MeV H^+ backscattering spectra from damaged (full line) and undamaged (dotted line) regions of the map in Fig. 2(a).

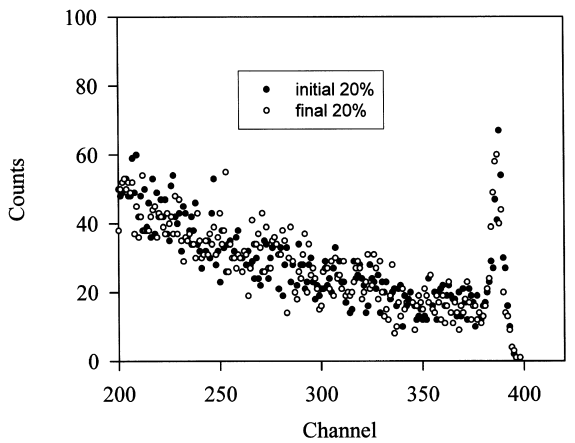

Fig. 4. One MeV He^+ [0 0 0 1] channeled spectra taken during the 7.4×10^{16} He^+/cm^2 channeled implant. Spectra taken during the initial 20% and the final 20% of the irradiation are shown.

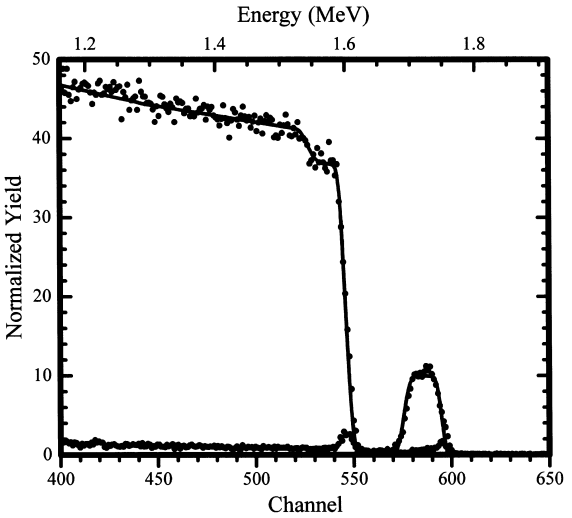

Fig. 5. Random and [0 0 0 1] channeled 2 MeV He^+ backscattering spectra from 500 Å $In_{0.11}Ga_{0.89}N$/GaN.

3.2. InGaN

Thermodynamic calculations predict the existence of a solid phase miscibility gap between InN and GaN [9]. This might cause a phase separation of InGaN into In-rich and In-poor regions, under extreme conditions leading to the formation of In 'islands'. This unstable nature is expected to play an important role in the self-formation of quantum dots of InGaN based structures [10]. It is also of interest to understand possible growth conditions for a controlled phase separation of InGaN. Preliminary evidence for such a phase separation was found in an undoped 500 Å $In_{0.11}Ga_{0.89}N$ sample. Fig. 5 shows the random and [0 0 0 1] axially channeled 2 MeV He^+ RBS spectra of this sample. Again excellent crystal quality is observed, a χ_{min} of 2.2% was measured. Fig. 6 displays 2 MeV He^+ CCM maps from this sample. Both the RBS (Fig. 6(a)) and the PIXE In map (Fig. 6(b)) clearly show correlated regions of increased In intensity. In non-channeled incidence the contrast disappears. Experiments with a larger number of samples are under way to clarify the nature of the observed structures.

3.3. PIXE channeling

The poor depth resolution of PIXE limits the usefulness of the channeling PIXE technique for thick targets because χ_{min} values can be derived only for an average excitation depth. For a thin film however, similar χ_{min} values are expected from PIXE and RBS measurements [11]. This is demonstrated in Fig. 7. Nine 500 Å InGaN films were used, with In mole fractions between 0.01 and 0.16

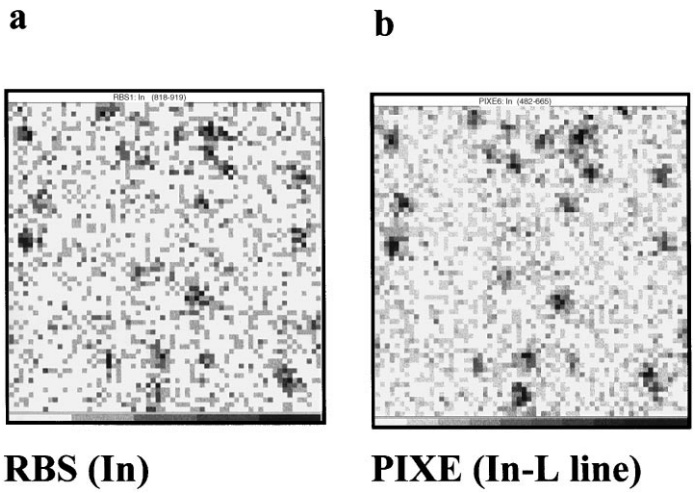

Fig. 6. Two MeV He^+ backscattering CCM maps the sample described in Fig. 5. (a) The integral of the In backscattering signal is mapped. (b) The In–L X-ray intensity is mapped. The gray-scale indicates intensity, darker regions have higher intensities.

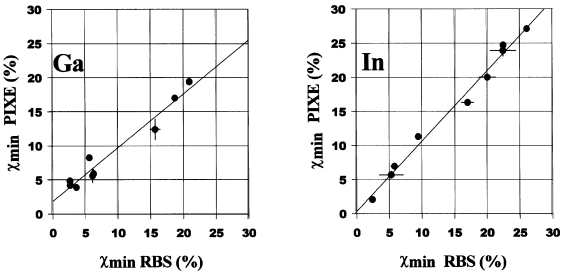

Fig. 7. Comparison of the PIXE and RBS channeling yields measured for Ga and In from nine InGaN/GaN samples.

and other growth conditions varied, leading to widely varying χ_{min} values. Two MeV He^+ broad beam channeling measurements were carried out, simultaneously collecting PIXE and RBS spectra. Fig. 7(a) shows the Ga PIXE χ_{min} values as a function of the Ga RBS χ_{min} values, and Fig. 7(b) shows the equivalent In data. In both cases a 'total' RBS χ_{min} was used, the integral of the region in the spectra associated with the In or Ga in the InGaN film. For the PIXE data, all In-L lines and the sum of the Ga–K lines were used. The Ga data show a linear correlation, but for low RBS and PIXE χ_{min} values the PIXE data deviate from a straight line. This is explained by the contribution to the Ga X-ray intensity from the underlying GaN film. However, the PIXE In χ_{min} values closely follow the RBS data, with a slope close to unity and an offset of 0.33%. PIXE channeling should therefore enable one to obtain quantitative information on the lattice position and crystal order of light elements were RBS data is difficult to obtain, e.g. for Al, Zn, and Si, which are commonly used as dopants in InGaN devices.

4. Conclusion

In conclusion, we have used the CCM measurements to assess the GaN susceptibility to 1 MeV He^+ induced beam damage, and found that negligible damage occurs for channeled irradiations, while for random incidence the channeled backscatter yield increases strongly at a dose of 7.8×10^{16}. CCM was used to image regions of increased In signal intensity, possibly indicating the presence of phase-segregation effects. It was demonstrated that channeling PIXE results correlate closely to channeling RBS results for such films, allowing the analysis of light elements were RBS analyses might prove difficult.

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