Thin Solid Films 424 (2003) 139–142

www.elsevier.com/locate/tsf

2 MeV proton channeling contrast microscopy of LEO GaN thin film structures

T. Osipowicz[a,*], E.J. Teo[a], A.A. Bettiol[a], F. Watt[a], M.S. Hao[b], S.J. Chua[b]

[a]*Research Centre for Nuclear Microscopy, National University of Singapore, Singapore 119260, Singapore*
[b]*Institute of Materials Research and Engineering, 3 Research Link, Singapore 117602, Singapore*

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 large efforts to optimise the growth methods used in their production. Rutherford Backscattering (RBS), in combination with channeling analysis, is a powerful tool for the quantitative characterization of the depth profile and the crystallinity of such structures. New growth modes, e.g. lateral overgrowth processes, are being rapidly developed. Channeling contrast microscopy (CCM), which employs a focused ion beam in order to obtain laterally resolved channeling yield data, is ideally suited to determine micro structural characteristics, (e.g. defect densities, tilts in lattice planes, strain) of such samples. Here we report results from proton channeling contrast measurements of laterally overgrown GaN thin films. Such measurements require a sub-micron ion beam focus size as well as an highly stable accelerator system, both of which are available at the Research Centre for Nuclear Microscopy at the National University of Singapore, where an ultrastable Singletron accelerator was recently installed.

Keywords: Lateral epitaxial overgrowth; Thin film; GaN; CCM

1. Introduction

The lateral epitaxial overgrowth (LEO) process significantly reduces the density of extended defects in GaN films grown by metal organic chemical vapor deposition (MOCVD) and hybrid vapor phase epitaxy (HVPE) on sapphire and SiC substrates [1,2]. A reduced threading dislocation density leads to a number of benefits in device performance, such as longer lifetime cw blue lasers [3], low reverse bias leakage current light emitting diodes [4], p–n junctions [5] and low gate leakage current AlGaN/GaN field-effect transistors. Despite the improved performance of LEO GaN devices, difficulties remain in controlling the structural quality of the overgrown material, such as the tilting of crystal planes in overgrown regions (wings) with respect to those of the window region [6,8,9]. Even though an empirical correlation was found between the wing tilt and the aspect ratio of the overgrown structure, the mechanism behind the wing tilt formation has not been clarified [6,7].

X-Ray diffraction studies are used to measure wing tilt with the scattering plane perpendicular to the stripe direction [6,8,9]. However, this technique is unable to provide laterally resolved information of the crystallographic tilts across the GaN stripes. Recently, we reported the use of channeling contrast microscopy (CCM) to image crystallographic tilts in micron sized overgrown regions of LEO GaN, using a 2-MeV alpha particle beam with a sub-micron spot-size [10]. Rocking curves measured perpendicular to the GaN stripes reveal the wing tilt relative to the window region. Here we report results from the 2-MeV proton beam CCM measurements of the same sample. A proton beam is, in certain cases, the preferable probe, because of its larger range and its smaller critical angle, e.g. for thicker samples with regions inaccessible with alpha beams.

*Corresponding author. National University of Singapore, Research Centre Nuclear Microscopy, Physics Department, 3 Research Link, Singapore 117602, Singapore. Tel.: +65-874-6745; fax: +65-777-6126.

0040-6090/03/$ - see front matter © 2002 Elsevier Science B.V. All rights reserved.
PII: S0040-6090(02)00914-8

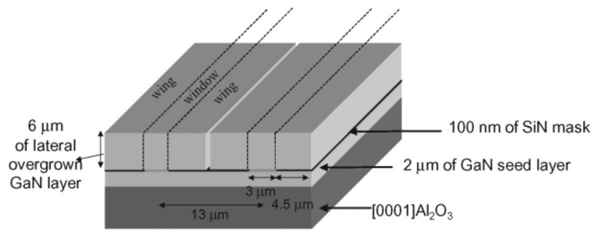

Fig. 1. Structural sketch of the LEO GaN sample.

2. Experimental

The lateral overgrown GaN stripes, patterned by the underlying Si_3N_4 mask, were deposited on a GaN buffer layer/[0001] Al_2O_3 substrate, as sketched in Fig. 1. Initially, a 2-μm thick GaN seed layer was grown by MOCVD (40 min) on a *c*-plane sapphire substrate. A 100-nm SiN mask layer was deposited on GaN by plasma enhanced chemical vapor deposition (PECVD) and then patterned into $[1\bar{1}00]_{GaN}$ oriented stripes which

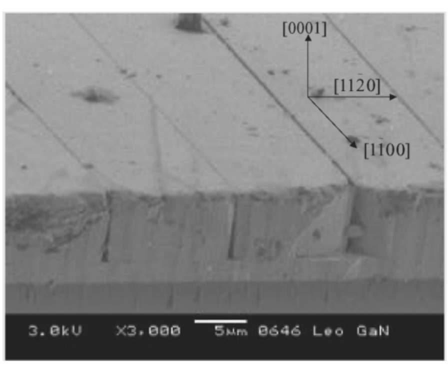

Fig. 2. Cross-section SEM picture of LEO GaN stripes.

define a 3-μm opening (window) at a periodicity of 13 μm. The lateral overgrowth GaN layer was achieved at a pressure of 100 torr, with trimethlygallium (TMG) and NH_3 at a flow rate of 123 μmol/min and 10 000 sccm, respectively, in combination with 6000 sccm of H_2 diluent. After 3.5 h of LEO growth on the mask, GaN stripes of 6-μm thickness propagated vertically through the window, with a width of 12.8 μm over the mask so that the stripes are close to coalescence, as seen in the SEM picture shown in Fig. 2.

The characterisation of LEO GaN was carried out using the new 3.5 MeV Singletron accelerator [11] at the National University of Singapore. Initially, 2 MeV H^+ broad beam channeling was used to find the [0001] axial position and subsequently, the beam was focused down to a 1-μm spot size, as verified by RBS scans of a 2000 mesh Au micro grid. For the microbeam channeling experiments, a beam current of 50–100 pA was typically used. The collimators were set so that the divergence of the beam was less than 0.2°. This condition provides reasonable channeling conditions, because the beam divergence is significantly less than the critical half angle of approximately 0.6° along the [0001] axis of GaN. In order to obtain data comparable with that from previous CCM measurements with a 2 MeV He^+ microbeam on the same LEO GAN sample the angular scan was performed through the axial direction of [0001] perpendicular to the GaN stripes, along the [1120] direction (see Fig. 2). The sample was mounted in such a way that the GaN stripes were aligned with the *y*-axis, and then rotated around the *y*-axis. The tilt angle with the [0001] direction is designated as ϕ in this paper. The channeling RBS data were recorded with a 300-mm^2 PIPS detector of 19 keV energy resolution at a scattering angle of 145°. A large solid angle of 222 msr was necessary in order to reach reasonable statistics in

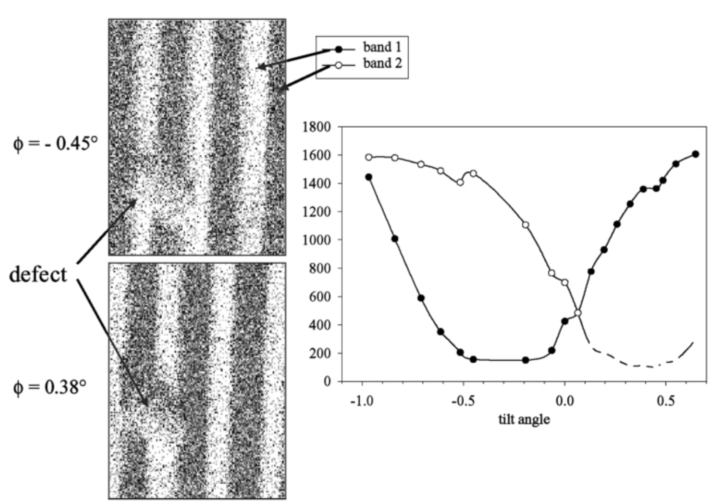

Fig. 3. (a and b) CCM maps for the given tilt angles ϕ, bands 1 and 2 are indicated. The defect used for alignment is also shown. (c) Angular scans extracted from bands 1 and 2, as a function of the tilt angle ϕ.

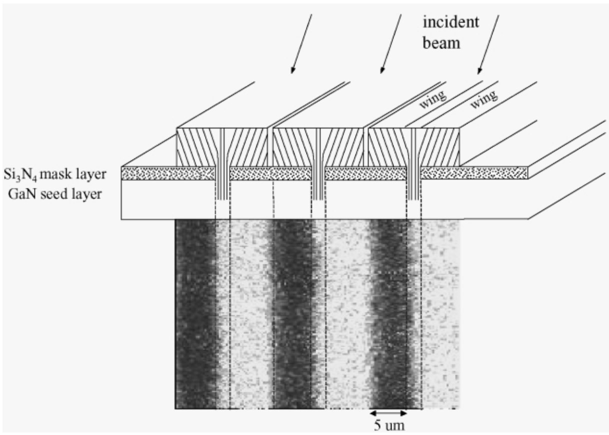

Fig. 4. Origin of the contrast pattern observed in the CCM maps.

spite of the limited beam current available. The target was mounted on an eucentric goniometer with a 24-mm translational range for both the x- and y-direction that allows rotations around the x- and y-axis with a resolution of 0.025°. The beam was scanned over an area of 52×52 μm^2, with an average collection time of 30 min.

3. Results and discussion

Fig. 2 shows a cross-sectional SEM micrograph of the LEO GaN just before coalescence. The GaN stripes have a thickness of approximately 6 μm, with a width of 12.8 μm and a periodicity of 13 μm. The gaps are not visible in the CCM maps due to the limited beam resolution.

As shown in Fig. 3a,b, bands of high and low contrast with a periodicity of 13 μm are found in the CCM maps. This contrast changes with the direction of the incident beam, as shown for two angles ϕ in Fig. 3a,b. The bands correspond to regions of opposite wing tilt direction, and they are designated band 1 and band 2 as shown in Fig. 3.

The goniometer produces lateral shifts of the sample position with sample rotation, typically 2 μm per 0.2°. This is because it is difficult to position the sample precisely at the eucentric point of the goniometer, and possibly because of mechanical backlash as well. Therefore, a defect present in the sample was used to align the CCM maps for different tilt angles ϕ as seen in Fig. 3a,b. As can be seen from Fig. 3, band 1 (defined in Fig. 3) appears dark (large channeling yield) at $\phi=0.38°$, and bright (low channeling yield) as the sample is tilted to $-0.45°$. Similarly, band 2 appears bright at 0.38° and dark at $-0.45°$. From these observations, it can be concluded that the overgrown regions (bands 1 and 2) have opposing plane tilts. Fig. 4 illustrates the situation. In the axial direction, slight dechanneling is observed from both sides of the overgrown region, with low channeling yields from the window region. As the tilt angle moves to positive values, the CCM maps show high yields from band 1 and low yields from band 2. Similarly, band 2 shows high yields for negative tilt angles.

All data were collected in list-mode, therefore, it is possible to extract separate rocking curves from band 1 and band 2 (as defined in Fig. 3a). Fig. 3c shows these angular scans, clearly individual channeling dips are present for both bands, displaced by approximately 0.32 and 0.38° from the [0001] GaN seed layer direction. No wing tilt is observed along the stripe direction when the sample is rotated around the x direction.

Laterally resolved channeling data have been extracted from the RBS spectra in near-surface regions. Fig. 5 shows random and channeled spectra of the band 2 part of the LEO GaN, for $\phi=0.39$ and $\phi=-0.84$. A χ_{min} of 5% is obtained in the $[0001]_{GaN}$ direction, indicating excellent crystal quality.

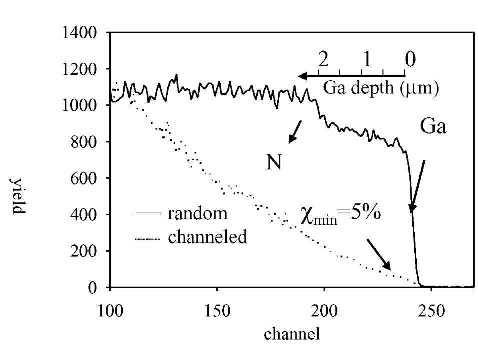

Fig. 5. RBS spectra of random and [0001] channeled GaN.

4. Conclusions

We have demonstrated the possibility of directly imaging lattice plane directions in micron-sized laterally overgrown GaN thin film structures with a sub-micron diameter beam of 2 MeV protons. If list-mode data sets are generated, off-line quantitative channeling analysis of such structures is possible. The results are very similar to the ones obtained with a 2-MeV alpha microbeam. The use of a proton beam is advantageous in cases where it is necessary to assess crystal structure deeper than a few microns below the sample surface, beyond the range of 2-MeV alpha particles.

References

[1] H. Marchand, X.H. Wu, J.P. Ibbetson, P. Fini, P. Kozodoy, S. Keller, J.S. Speck, S.P. DenBaars, U.K. Mishra, Appl. Phys. Lett. 73 (1998) 747.

[2] K.J. Linthicum, T. Gehrke, D.B. Thomson, E.P. Carlson, P. Rajagopal, T.P. Smith, A.D. Batchelor, R.F. Davis, Appl. Phys. Lett. 75 (1999) 196.

[3] S. Nakamura, M. Senoh, S.I. Nagahama, N. Iwasa, T. Yamada, T. Matsushita, H. Kiyoku, Y. Sugimoto, T. Kozaki, H. Ume-

moto, M. Sano, K. Chocho, J. Cryst. Growth 189/190 (1998) 820.
[4] C. Sasaoka, H. Sunakawa, A. Kimura, M. Nido, A. Usui, A. Sakai, J. Cryst. Growth 189/190 (1998) 61.
[5] P. Kozodoy, J.P. Ibbetson, H. Marchand, P. Fini, S. Keller, J.S. Speck, S.P. DenBaars, U.K. Mishra, Appl. Phys. Lett. 73 (1998) 975.
[6] P. Fini, J.P. Ibbetson, H. Marchand, L. Zhao, S.P. DenBaars, J.S. Speck, J. Cryst. Growth 209 (2000) 581.
[7] O.k-H.yun Nam, M.D. Bremser, T. S. Zheleva, R.F. Davis, Appl. Phys. Lett. 71 (18) (2000) 2638.
[8] P. Fini, L. Zhao, B. Noran, M. Hansen, H. Marchand, J.P. Ibbetson, S.P. DenBaars, U.K. Mishra, J.S. Speck, Appl. Phys. Lett. 75 (1999) 1706.
[9] P. Fini, A. Munkholm, C. Thompson, G.B. Stephenson, J.A. Eastman, M.V. Ramana Murty, O. Auciello, L. Zhao, S.P. DenBaars, J.S. Speck, Appl. Phys. Lett. 76 (26) (2000) 3893.
[10] E.J. Teo, T. Osipowicz, A.A. Bettiol, F. Watt, Nucl. Instr. Meth. B 181 (2001) 2312–2317.
[11] D.J.W. Mous, R.G. Haitsma, T. Butz, R.H. Flagmeyer, D. Lehmann, J. Vogt, Nucl. Instr. Meth. B130 (1997) 31.