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Authors

Barabash, R I
Ice, G E
Liu, W
et al.

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Mapping Misorientation and Crystallographic Tilt in GaN Layers Via Polychromatic Microdiffraction

R.I. Barabash^{1*}, G.E. Ice¹, W. Liu¹, C. Roder², S. Figge², S. Einfeldt^{2*}, D. Hommel², T.M. Katona³, J.S. Speck⁴, S.P. DenBaars⁴ and R. F. Davis⁵

¹Materials Science and Technology Div., Oak Ridge National Laboratory, Oak Ridge, TN, 37831, USA;

²Institute of Solid State Physics, University of Bremen, P. O. Box 330 440, 28334 Bremen, Germany;

³Electrical and Computer Engineering Department, College of Engineering, University of California at Santa Barbara, Santa Barbara, CA 93106, USA

⁴Materials Department, College of Engineering, University of California at Santa Barbara, Santa Barbara, CA 93106, USA

⁵Materials Science and Engineering Department, North Carolina State University, Raleigh, USA

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The spatial distribution of strain, dislocations, and crystallographic orientation in uncoalesced and coalesced GaN layers grown on striped Si or SiC substrates was studied by polychromatic x-ray microdiffraction and high resolution monochromatic x-ray diffraction. Tilt boundaries formed at the column/wing interface depending on the growth conditions and geometry of the striped substrate. The measurements revealed that the free-hanging wings are tilted upward at room temperature. A misorientation between the GaN(0001) and the Si(111) or SiC (0001) surface normal is observed. Distinct structural properties of the pendeo and cantilever epitaxially grown samples are discussed.

1 Introduction When GaN films are grown on substrates like sapphire, Si or SiC, they are found to contain a high density of threading dislocations (10^{10}cm^{-2}) due to lattice mismatch. Dislocations impair the performance of GaN-based light emitting devices [1]. Lateral epitaxial overgrowth (LEO) techniques greatly reduce the density of threading dislocations. Pendeo- (PE) [2-4] and cantilever epitaxy (CE) [5 - 8] are the most promising LEO growth techniques.

The crystallographic tilt of the laterally growing wings relative to the seed (column) region usually accompanies all CE and PE GaN layers. For both CE and PE, point and extended defects generated in the GaN during overgrowth play an important role in the formation of strains, misorientations and wing tilt, as does the mismatch in the thermal expansion coefficients between GaN and the substrate. Understanding and controlling the local lattice orientation, direction and the origin of the wing tilt and microstructural evolution processes associated with LEO, differential thermal contraction and formation of dislocation arrays and cracking remain significant challenges in technologically important CE and PE grown GaN based blue light emitting diodes.

2 Experimental In this paper we report results using Polychromatic X-ray Microdiffraction techniques (PXM) to penetrate both the GaN layer and the substrate. Two samples grown on Si (111) by CE and three samples grown on SiC (0001) by PE were analyzed. During CE growth (samples CE-A and CE-B) and during PE growth (samples 1 and 2) the subsequent overgrowth process was stopped before the wings coalesced into a layer. In the third PE grown sample the process continued and the coalesced GaN layer was formed (sample 3). Further details of the growth process for PE grown samples can be found in Ref. [2, 5] and for CE grown samples in the Refs. [8, 9].

In PXM analysis the focused polychromatic synchrotron beam is diffracted from a submicrometre-diameter probing location within each GaN stripe (column/wing) of the layer, producing a complete diffraction pattern from the GaN layer and the substrate on a charge-coupled device (CCD) area detector (Fig. 1). The surface of the sample is inclined at 45° to the incident beam. The CCD detector is placed at 90° relative to the incident beam [5]. The Cartesian reference frame is set such that the Y-axis coincides with the GaN stripe direction. The X-axis is perpendicular to the stripe in the plane of the GaN film in the $\langle 11\bar{2}0 \rangle$ direction. High resolution x-ray diffraction (HRXRD) measurements were performed using a Philips X'Pert

* Contact author e-mail <barabashr@ornl.gov>

*Present address: Ferdinand-Braun-Institut, Gustav-Kirchhoff-Str. 4, 12489 Berlin, Germany

MRD diffractometer equipped with a fourfold Ge (220) or a twofold hybrid monochromator in Ge (220) configuration and a threefold Ge (220) analyzer.

3 Results and discussion

In contrast to conventional monochromatic diffraction, the PXM technique provides local (0.5 μm in diameter) information about the orientation and strain. To check the wing tilt trend the probing spot was moved across the GaN stripes with a step of 0.25 μm . Several line scans across the GaN stripes were performed and several thousand Laue patterns were obtained in total. An example of a complete Laue pattern from the column region of the CE-B sample is shown in Fig. 1a. High energy X-rays (7 - 25 keV) penetrated both the GaN layer and the Si substrate. The position of Si Laue spots on a CCD remained the same for all probed locations and was used as a reference to check the relative orientation between different GaN locations. The trend of the wing tilt was determined to be upward for both CE and PE grown samples. Two CE grown samples were chosen: sample CE-A with a large wing tilt (0.58°) and sample CE-B with the tiny wing tilt (0.036°) [8]. It was a challenge to determine the trend of the wing tilt with PXM for sample B as it was difficult to resolve a shift of the GaN(0006) reflection when the probe was moved over the sample. Since the wing tilt in sample CE-B is much smaller than in sample A, the shift was masked by the broadening of the reflection. Therefore, reflections of higher order such as the GaN ($10\bar{1}9$), ($10\bar{1}11$), ($10\bar{1}13$), and the Si (664) and (775) were measured (Figs. 1b–e). The wing tilt can be most clearly seen in Fig. 1d corresponding to the narrow gap between the wings.

Fig. 1 (a) Complete Laue pattern from the CE grown GaN (001) layer on Si (111) substrate for CE-B sample and blown up regions of interest marked with dash containing several Si and GaN Laue spots obtained from the different location on the stripe: column (b), right wing of the stripe 1 (c), gap between the wings (d), left wing of the stripe 2 (e). (f) The scheme of the stripe geometry.

Here the beam probes both the left wing and the right wing so that a doubled peak is found for every GaN reflection. For the probe positions (c) and (e), each displaced 0.25 μm from location (d) to the left and to the right, respectively, one of the two peaks dominates as the beam is either mainly on the left or on the right wing. The evaluation of the direction of the peak shift reveals an upward wing tilt in sample CE-B by $\sim 0.036^\circ$.

Analysis of the Laue pattern together with the geometry of the stage movement (Fig. 1) showed that wings were tilted upward for both the PE and CE grown samples. The detailed description of this procedure for characterization of the wing tilt in PE and CE samples can be found elsewhere [5, 9]. PXM measurements of the wing tilt coincided with HRXRD results for all specimens.

PXM is probing so called “orientation space” of the sample. Broadening of the GaN Laue reflections along and perpendicular to the stripe direction is sensitive to the misorientation of the GaN layer within the probed region. We characterize the misorientation with a special misorientation vector $\mathbf{m}$. Its projections on the X and Y axes describe the misorientation distribution across the GaN stripe (m_x) and perpendicular to it (m_y). Intensity profiles for the column and wing regions for CE and PE samples across the stripe are shown for the two CE (CE-A and CE-B) samples in Fig. 2 and for two uncoalesced PE samples with different

width-to-height ratio in Fig.3. A comparison of the PXM results from CE-A and CE-B samples shows that the broadening is in general more pronounced for sample CE-A. Along the X direction (perpendicular to the stripe) the broadening of the Laue spots for all PE and CE samples is larger for the column regions than for the wing, as can be seen from Figs. 2 and 3.

Fig. 2 Intensity distribution along the m_x (across the stripe) for CE-A sample (a-c) and CE-B sample (d-f); left wing (a, d), column (b, e) and right wing (c, f). (0006) GaN Laue spots are shown in the inserts.

Fig. 3 Intensity distribution along the m_x (across the stripe) for PE grown sample 1 (a) and sample 2 (b) together with corresponding (109) GaN Laue spots from the left wing (bottom), column/wing interface (central) and center of the column (top). Intensity profile of the Si reflection is shown for comparison (top).

Fig. 4 Intensity distribution along the m_Y (parallel to the stripe) for CE-A sample (a-c) and CE-B sample (d-f) together with (0006) GaN spots from the left wing (a, d), column (b, e) and right wing (c, f).

Comparison of the intensity distribution in m_X direction for PE and CE samples (Figs.2 and 3) shows that wing tilt of the two PE samples is smaller than for CE-A sample but larger than for the CE-B sample. Analysis of the (0006) GaN Laue spot (inserts in Fig. 2) for CE grown samples reveals unusually strong broadening in the direction parallel to the stripe direction (which was not observed for PE grown samples). Therefore the analysis of the Laue spots intensity in the direction parallel to the stripe direction was performed for CE grown samples (Fig. 4). Profiles of intensity distribution along the YY direction (shown by a dashed line in the inserts of Fig. 4) demonstrate distinct behaviour of samples CE-A and CE-B.

For the CE-A sample, the intensity distribution in the Y direction typically consists of several peaks. This may be consistent with the formation of small angle boundaries with the plane perpendicular to Y direction creating lattice rotations around the X axis. Interestingly the CE-A sample wings are tilted parallel to the stripe in opposite directions with respect to the column regions (compare Figs. 4 a - c).

The intensity distribution for the wing regions of CE-B sample in Y direction may be considered to consist of two components: a narrow component at the intensity level higher then $I(m_y) = 0.5I_{\max}$ and a very

broad component observed at the intensity level below $I(m_y) = 0.5I_{\max}$ (Figs. 4 d, f). Both components

have a relatively smooth intensity distribution. In the sample CE-B both wings are well aligned in the direction parallel to stripes. Intensity distribution in the column region of CE-B (Fig.4 e) is highly asymmetric with a strong long tail on the left side pointing towards the Si (111) pole. This behavior may result from a gradient of orientations from the bottom layers of the column to the top. The upper layer of the column region may have exactly same orientation in the Y direction as in the wings (Figs. 4 d -f) (the m_Y position of the maximum for the column region coincides with it's location for wings region). The detailed model responsible for such a distinct behaviour of CE samples will be published elsewhere [9].

For the coalesced PE sample, the interpretation of both PXM and HRXRD data becomes more complicated. Examples of reciprocal space maps (RSM) obtained with HRXRD from coalesced and uncoalesced PE sample are shown in Fig. 5. The RSM from uncoalesced PE or CE GaN layers typically consist of three maxima which can be easily identified as belonging to the column and two wings. In contrast the RSM from the coalesced PE grown GaN layer reveals four intensity maxima which are difficult to interpret (Fig.5 b).

Fig. 5 RSM of the uncoalesced (a) and coalesced (b) PE grown GaN layers for (0002)GaN reflection. PXM analysis of the coalesced PE grown sample 3 reveals two kinds of tilt boundaries in the layer. One is located in the region overgrown on top of the former column region with a tilt angle of 0.18° (Fig. 6 a). The second sharp tilt boundary with a tilt angle of 0.5° is formed at the wing/wing coalescence interface (Fig. 6 b).

Fig. 6 PXM Laue images of the (109) GaN and SiC Laue spots obtained from different locations of the coalesced PE grown GaN layer on SiC: column region (a) and wing/wing coalescence interface.

4 Summary PXM analysis of the GaN layers provided detailed information about local strain and strain gradient, misorientations and wing tilt in GaN layers. It was shown that the crystallographic tilt of the wings in GaN layers grown by CE on Si (111) or by PE on SiC (0001) substrates is upward for both

uncoalesced and coalesced GaN layers. In CE grown samples the (001) out-of-plane GaN surface normal is crystallographically tilted relative to the Si (111) substrate plane normal. Such tilt is not observed for PE samples. Two types of interfaces were observed in the coalesced PE grown GaN layers with different misorientation angle across the boundary.

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