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Reduction of dislocation density in GaN films on sapphire using AlN interlayers

J. CHAUDHURI*, J. T. GEORGE
Mechanical Engineering Department, Wichita State University, Wichita, KS 67260-0133, USA
E-mail: chaudhur@me.twsu.edu

D. D. KOLSKE, A. E. WICKENDEN, R. L. HENRY
Code 6800, Electronics Science and Technology Division, Naval Research Laboratory, Washington, D. C. 20375, USA

Z. REK
Stanford Synchrotron Radiation Laboratory, Stanford, CA 94305, USA

GaN (00.1) thin films, of thickness 1.25 to 2.25 μm grown on sapphire substrate (11.0) by metallo organic chemical vapor phase deposition (MOCVD) with different number of AlN interlayers, were characterized by triple crystal diffractometry and synchrotron white beam x-ray topography (SWBXT). The full width at half maximum (FWHM) of x-ray rocking curves from symmetric and asymmetric reflections was used to estimate the dislocation density in GaN films. It has been found that the edge dislocation density decreased from 1.63×10^{10} cm^{-2} to 1.23×10^{10} cm^{-2} and the screw dislocation density decreased from 2.0×10^{8} cm^{-2} to 1.1×10^{8} cm^{-2} when one AlN interlayer was inserted between the high temperature GaN layer. The dislocation density decreased further with the increase in number of interlayers. On the other hand the compressive stress in the GaN film increased from −0.29 GPa to −0.86 GPa. The compressive stress further increased as the number of interlayers increased but no cracking in the GaN film was observed. This could be due to better adhesion between the film and substrate due to interlayers. SWBXT in transmission from a GaN(00.1)/Al$_2$O$_3$(11.0) sample confirms the orientation of GaN and indicates that it is a single crystal with high dislocation density. SWBXT from the Al$_2$O$_3$ substrate shows cellular structure of dislocations. © *2002 Kluwer Academic Publishers*

1. Introduction

Significant potential of GaN as a short wavelength light emitter has been demonstrated by the pioneering work by Maruska and Tietjen [1] and Pankove *et al.* [2]. Bright blue light emitting diode (LEDs) and green LEDs have already been commercialized, and purple, deep purple and even ultraviolet (UV) laser diodes have been fabricated. Micro-wave field effect transistor (FEDs) and solar blind UV detectors are also available. These results are remarkable considering 16% lattice mismatch between GaN and sapphire substrate and are possible because of the low temperature buffer layer approach [3, 4]. Nevertheless, GaN grown on sapphire using low temperature buffer layer still contains high threading dislocations (TDs) of the order of 10^8–10^{10} cm^{-2} that originate at the interface between GaN and sapphire [5, 6].

Recently, Amano *et al.* found that insertion of low temperature interlayers of GaN and AlN between high-temperature-grown GaN layers reduces TDs [7]. Koleske *et al.* [8] have shown that the electron mobility in the top Si doped GaN increases from 440 to 725 cm^{2}V^{-1}s^{-1} as the number of interlayers increases. Photoluminesence spectra of undoped and Si doped GaN films with multiple interlayers have small yellow band intensity. Cross-sectional transmission electron microscopy (TEM) images revealed a significant reduction in the screw dislocation density in GaN films grown with interlayers. High resolution x-ray rocking curve method can be used to estimate dislocation density in epitaxial films [9]. In this paper, dislocation density and stress, in GaN thin films grown using different number of interlayers, are calculated using x-ray rocking curve method. Additionally, Synchrotron white beam x-ray topography (SWBXT) were taken to reveal defects present in the samples. For comparison, a GaN film grown with an AlN buffer layer is included.

2. Experimental

Details of the growth technique can be obtained elsewhere [8]. The GaN films were grown in a close-spaced

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*Author to whom all correspondence should be addressed.

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TABLE I Growth condition, strained lattice parameters, strain and stress in GaN thin films

		Thickness (μm)		Lattice parameters[a] (Å)		Strain (%)		
Sample no.	Growth conditions	[a]	[b]	a	c	$\epsilon_{\|\|}$	$\epsilon_{\perp}$	Stress (GPa)
1	With an AlN buffer layer	2.0	N/A	3.1839	5.1846	−0.06	0.13	−0.29
2	With 1 NL and 1 IL	1.0	2.0	3.1804	5.1963	−0.18	0.35	−0.86
3	With 1 NL, 1 IL and a thin AlGaN layer	1.25	2.25	3.1807	5.1954	−0.17	0.33	−0.81
4	With 1 NL and 6 IL	2.25	8.25	3.1792	5.2002	−0.21	0.43	−1.0

[a] Strained lattice parameters.
[a] thickness of top GaN layer.
[b] total thickness of top GaN layer and GaN interlayers.

showerhead MOVPE reactor on a-plane (11.0) sapphire [10]. Trimethylgallium (TMGa), Trimethylaluminum (TMAl) and ammonia (NH_3) were used as precursors, with hydrogen (H_2) used as the carrier gas. A thin (220 Å) low temperature (≈800°C) AlN layer (IL) and a 1.0 μm thick high temperature (1030°C) GaN layer were grown alternately up to a maximum of 6 times [10]. Following the growth of the AlN IL and GaN layers, the top GaN film of thickness varying between 1.25 μm to 2.25 μm were grown. A description of the samples are included in Table I.

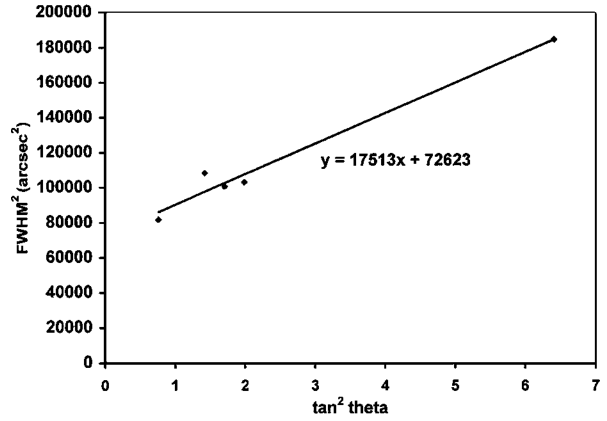

Figure 1 FWHM^2 versus $\tan^2\theta$ for the sample number three for asymmetric reflections only.

A Blake Industries high resolution triple crystal diffractometer with Cu Kα_1 was used for the x-ray analysis [9]. The second slit size was 1 mm by 2 mm and the detector was kept wide open. The structure, strain and dislocation density were obtained using a combination of symmetric and asymmetric reflections. The dislocation density in the GaN films was estimated from the FWHM of rocking curves and following a model by Hordon and Averbach [11]. Details of the dislocation density calculation are presented elsewhere. The corrected FWHM values were plotted against $\tan^2\theta$ where θ is the Bragg angle and a best fit straight line was obtained using least square method. Fig. 1 shows this plot for the sample number three. The intercept of the line is the rocking curve angular broadening parameter (caused by the angular rotation at dislocations), and the

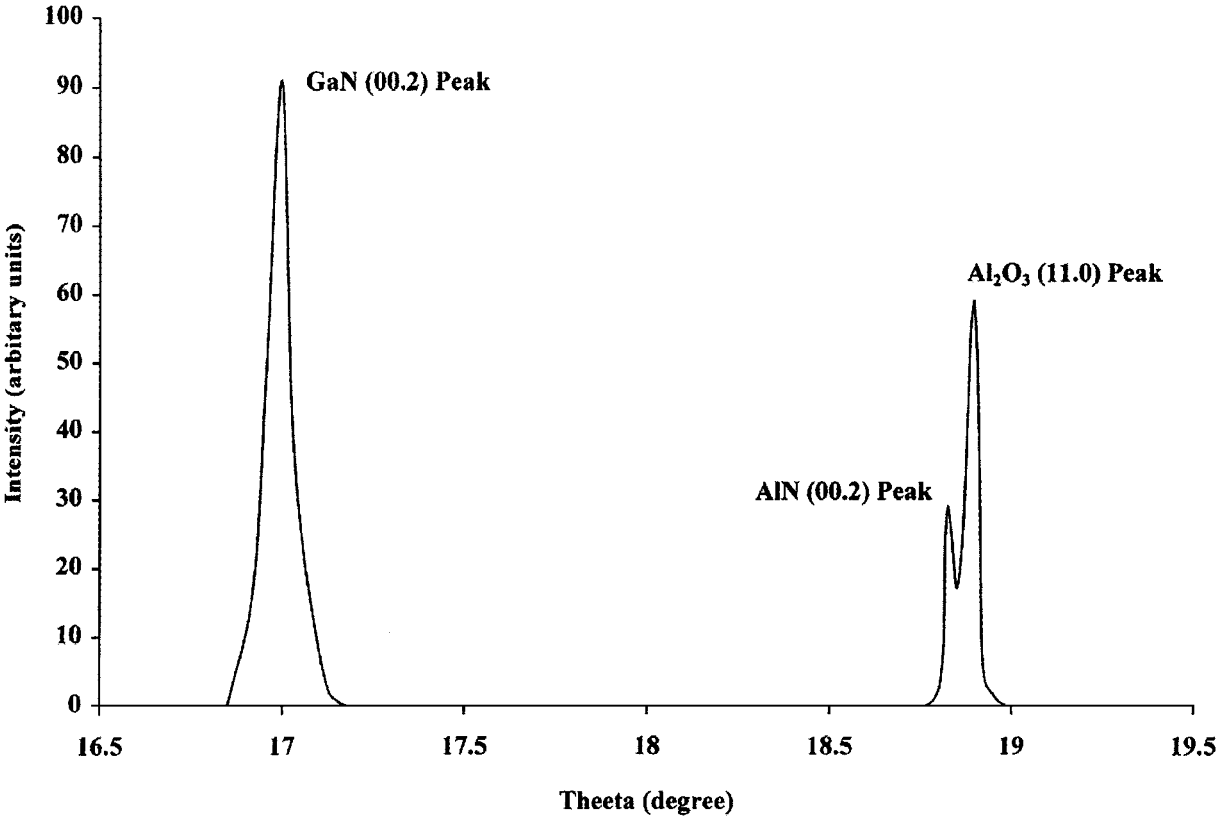

Figure 2 X-ray rocking curves from the sample number four showing GaN (00.2), AlN (00.2) and sapphire (11.0) peaks.

slope is the rocking curve strain broadening parameter (caused by the strain surrounding the dislocations).

The synchrotron radiation topography experiments [11] were carried out at the Synchrotron Radiation Topography experimental station 2.2 at the Stanford Synchrotron Radiation Laboratory, Stanford, CA. Transmission topographs were taken using Laue diffraction technique and white beam radiation. The incident beam was aligned parallel to the [11.0] direction of sapphire and the GaN film was on the exit side. Images were recorded in high resolution Kodak SR1 type films for all topographic work.

3. Results and discussions

Fig. 2 shows the symmetric x-ray rocking curves from the sample number four. The lattice parameters and strain in GaN films are calculated using symmetric and asymmetric rocking curve peak separations of the GaN film from that of the substrate and are listed in Table I, The stress in the GaN layers was calculated from the in-plane strain, $\epsilon_{||}$, and the following equation for the biaxial stress [13]:

$$\sigma_l = \frac{(c_{11} + c_{12})c_{33} - 2c_{13}^2}{c_{33}} \varepsilon_{\mathrm{II}} \qquad (2)$$

where c_{ij}s are elastic constants. The stress is compressive in nature and increases by approximately three times when one interlayer was used instead of a buffer layer (Table I). Also, the compressive stress increases with the number of interlayers. At the growth temperature GaN and sapphire are lattice matched and upon

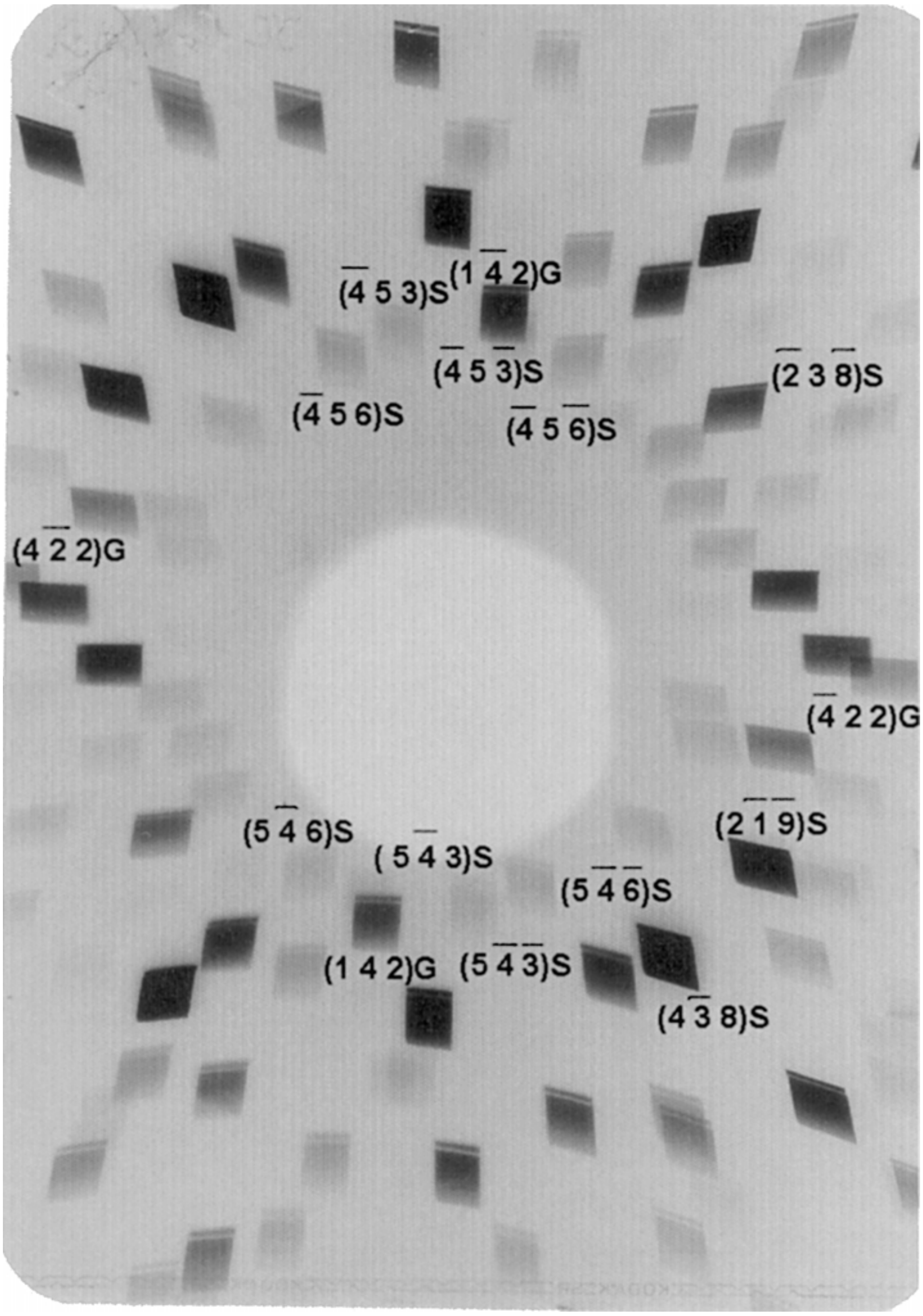

Figure 3 Synchrotron white beam x-ray transmission Laue topograph of the sample number three with the incident beam parallel to sapphire [11.0] direction, exposure time 10 sec and specimen to film distance 10 cm. The letters S and G indicates the spots from sapphire and GaN, respectively.

TABLE II Full width at half maximum (FWHM/$\sqrt{\pi}$) of rocking curves of different reflections and dislocation density in GaN thin films

Sample	AlN	Al_2O_3	FWHM/$\sqrt{\pi}$ GaN								Dislocation density ($10^9/cm^2$)	
no.	00.2	11.0	00.2	00.4	00.6	01.4	01.5	02.4	02.5	11.4	Screw	Edge
1		20	293	316	339	327	339	338	508	401	0.20	16.30
2		21	211	277	300	290	326	331	450	351	0.11	12.25
3		39	201	256	310	286	317	321	430	329	0.099	10.87
4	49	90	183	196	250	170	211	293	340	217	0.067	7.91

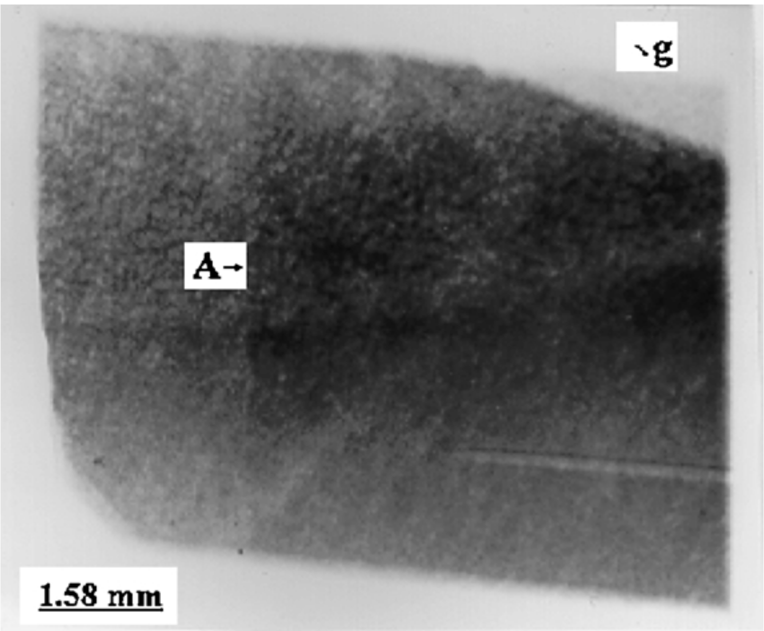

Figure 4 Synchrotron white beam x-ray transmission topograph from a GaN (00.1)/sapphire (11.0) sample, the image from sapphire only, diffraction vector $g = 2\bar{1}9$, $\lambda = 0.622$ Å, (A) cellular structure of dislocations.

cooling the difference in lattice expansion causes a stress which increases with the thickness of the GaN film. Usually a GaN film on sapphire thicker than 5.0 μm cracks but with the use of interlayers the films do not crack although it is highly stressed. The full width at half maximum (FWHM) of the [11.0] rocking curve from sapphire also increased with the number of interlayers (Table II) indicating that the substrate is also strained as the number of interlayers increased.

Table II shows the FWHM of rocking curves of different reflections and dislocation density in GaN films. Symmetric and asymmetric reflections were used to estimate screw and edge dislocation densities, respectively (Table II). The dislocation density, determined from the strain broadening parameter was larger than that obtained from the angular broadening parameter and thus the larger value was reported. It can be seen that the FWHM decreases when one interlayer was used instead of an AlN buffer layer. Both the screw and edge dislocation density decreased with the use of interlayers. The screw dislocation density decreased more than edge dislocation density with the increase of number of interlayers (sample number four). It could be that screw dislocations annihilate as they attempt to thread through the AlN interfaces, thereby removing the screw dislocations from the GaN film [8]. An additional AlGaN layer decreased the dislocation density slightly (sample number three). In comparison to earlier transmission electron microscopy (TEM) investigation [7] with the present study, the edge dislocation density in the GaN film with one interlayer is identical, where as much lower value was reported in reference 7 for six interlayers. This is because the x-ray diffraction technique measures average dislocation density over all the GaN layers instead of measuring it from the top layer.

SWBXT in Laue transmission mode from the sample number four using an area beam is shown in Fig. 3. Sapphire and GaN reflection spots are indexed by using a Laue computer simulation program [14]. It was confirmed that the GaN film is a single crystal with wurtzite structure and oriented along [00.1]. Each spot from GaN in figure three is quite smooth indicating presence of high dislocation density. Fig. 4 shows the SWBXT from a sapphire substrate. Cellular structure of dislocations and a scratch can be seen in this figure.

4. Conclusions

High resolution x-ray diffractometry and SWBXT were used to characterize defects in GaN thin films grown on sapphire substrate by MOCVD with different numbers of AlN interlayers. The dislocation density in GaN films was estimated from the FWHM of a number of symmetric and asymmetric reflections. Both the edge and screw dislocation density decreased with the use of interlayers as compared to a GaN film grown with a

AlN buffer layer. The compressive stress in GaN films increased when interlayers were used but no cracking was observed. SWBXT in transmission from a GaN (00.1)/sapphire (11.0) sample shows that GaN is a single crystal with high dislocation density and sapphire contains cellular structure of dislocations.

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