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Surface preparation of substrates from bulk GaN crystals

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Abstract

Large gallium nitride (GaN) crystals were grown using a hydride vapor phase epitaxy (HVPE) technique and were processed into substrates for device applications. Polishing procedures were developed for GaN substrates to produce surfaces prepared for epitaxial growth. Surface preparation of (0 0 0 1) and (0 0 0 $\bar{1}$) substrates was examined, along with preparation of (1 1 $\bar{2}$ 0) and (1 $\bar{1}$ 0 0) non-polar surfaces. For all surfaces, chemical mechanical polishing (CMP) resulted in an average root mean square (RMS) surface roughness on a $5\,\mu m \times 5\,\mu m$ scanning probe microscope (SPM) image of <0.2 nm. Characterization of the surfaces of polished substrates by cross-sectional transmission electron microscope (TEM) showed no sub-surface damage and no epitaxial defects generated at the substrate/epi interface during homoepitaxial growth. Cathodoluminescence (CL) imaging was used to verify the defect density and that no defects related to polishing were present. The average dislocation density of the substrates was $<5 \times 10^6\,cm^{-2}$.

Keywords: A1. Polishing; A1. Surfaces; A2. Growth from vapor; B1. GaN; B1. Nitrides

1. Introduction

Commercialization of gallium nitride (GaN)-based devices has steadily increased in the last several years. With the advent of blue and violet GaN-based laser diodes, devices using native, bulk GaN substrates have been reported [1–3]. These devices take advantage of the lattice matching and thermal expansion matching between the substrate and the device layers, along with the reduced defect density present in the bulk substrates. In order to achieve high-quality epitaxial device layers, fabrication of crystalline surfaces that are flat and free of damage or defects resulting from surface preparation is needed. Chemical mechanical polishing (CMP) has been used to produce smooth, scratch-free surfaces that are free of sub-surface polishing damage [4–6]. Recently, considerable attention has also been paid to the development of nitride epitaxial layers and heterostructures with non-polar surface orientations. GaN-based semiconductor materials and devices have historically been developed through crystal growth in the [0 0 0 1] direction (*c*-plane). The lack of mirror-like (or two-fold) symmetry of the hexagonal crystal structure gives rise to spontaneous polarization in this direction. Additionally, strain in lattice mismatched heteroepitaxially grown device layers for this orientation result in piezoelectric polarization. These polarization effects result in large built-in electric fields, hampering the performance of nitride-based devices. In optical devices, the built-in polarization field results in charge separation within quantum wells and decreases the recombination efficiency of electron–hole pairs and red shifts the emission wavelengths. For microelectronic devices, the spontaneous and piezoelectric polarization allows for the accumulation of a very high density of sheet charge in the conducting channel of GaN-based HEMTs; however, the surface of the device requires the appropriate passivation and is sensitive to the stress induced by passivation and thermal effects. As a result, researchers have investigated methods to eliminate the built-in fields of devices grown in the [0 0 0 1] direction.

GaN-based quantum structures grown along non-polar directions, such as the [1 $\bar{1}$ 0 0] (resulting in *m*-plane surfaces) and [1 1 $\bar{2}$ 0] (resulting in *a*-plane surfaces) have been shown to be free of the aforementioned polarization effects [7–9]. Using heteroepitaxially grown thin films and devices fabricated thereon, higher internal quantum

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efficiencies and lower EL peak sensitivity for LEDs have been demonstrated [10,11]. These and other initial results in this emerging technological area show great promise for non-polar substrates in impacting a number of commercial applications, such as laser diodes, visible and UV LEDs, and high-power electronic devices. For bulk non-polar materials, where material with low-defect densities have been reported [12], surface preparation of these substrate orientations is also important.

In this work we describe the surface preparation and characterization of polar (*c*-plane) and non-polar (*a*- and *m*-plane) surfaces grown by hydride vapor phase epitaxy (HVPE) and prepared using CMP processes.

2. Experimental procedure

The GaN bulk crystals were grown using a HVPE process described previously [13]. Aluminum nitride nucleation layers were deposited on sapphire substrates prior to HVPE growth of the GaN layers. Thick layers were grown and removed from the underlying sapphire to create free-standing, bulk material. For the *c*-plane substrates, the growth thickness was of the order of 1–2 mm; for the non-polar samples, the bulk GaN crystals were grown in the [0 0 0 1] direction up to 1 cm in thickness. The *a*- and *m*-plane substrates were prepared using a wire saw by making a vertical cut in the thick crystal along the [0 0 0 1] direction with the wire oriented along the [1 $\bar{1}$ 0 0] or [1 1 $\bar{2}$ 0] direction. Grinding and lapping using diamond media were employed to planarize the samples and to reduce the total thickness variation in the samples. A two-step mechanical polishing process was then used to prepare the surface for the final chemical mechanical polish. Ga-face *c*-plane (0 0 0 1), N-face *c*-plane (0 0 0 $\bar{1}$), *a*-plane (1 1 $\bar{2}$ 0), and *m*-plane (1 $\bar{1}$ 0 0) surface orientations were polished using the CMP process. A Pacific Nanotechnology Nano-R scanning probe microscope (SPM) in contact mode was used to characterize the roughness and surface features of the polished substrates. Non-destructive defect density measurements were made via cathodoluminescence (CL) imaging at room temperature on a JEOL JSM 6400 SEM with an Oxford MonoCL2 system. Cross-sectional transmission electron microscope (TEM) samples were prepared using a focused ion beam (FIB) process.

3. Results

CMP processes were developed to improve the surface roughness and polishing results over those achieved with mechanical polishing. While root mean square (RMS) surface roughness values <1 nm could be obtained using mechanical polishing with diamond media, scratches and sub-surface damage remain. Ga-face *c*-plane surfaces prepared with mechanical polishing yielded RMS surface roughness on the order of 0.2–0.6 nm on 5 μm × 5 μm scans; however, scratches were evident using a SPM, as shown in Fig. 1(a). Cross-sectional TEM images of mechanically polished samples revealed a sub-surface damage layer consisting of networks of dislocations near the surface of the sample. The thickness of this damage layer varied depending on the polishing conditions, and could be of the order of several microns thick. Epitaxial growth studies on similarly prepared mechanically polished samples resulted in defective epi with dislocations propagating in the epitaxial layers, increased surface roughness, and scratches highlighted in the epi morphology. These results combine to show that additional surface preparation is needed to obtain the desired epitaxial growth results.

Fig. 1(b) shows a 5μm × 5 μm SPM image of a Ga-face *c*-plane CMP surface. The RMS roughness is 0.18 nm. The figure shows that the CMP process was effective at removing the scratches that remained after the final mechanical polishing step. The chemistry of the polishing slurry in the CMP step, along with the polishing media material and size distribution, was optimized to provide a reactive environment that facilitated removal of material from the surface while eliminating sub-surface damage. A CMP process was developed for the Ga-face that had a 0.5 μm/h average removal rate. Due to the low removal rate, it was important to minimize the thickness of the

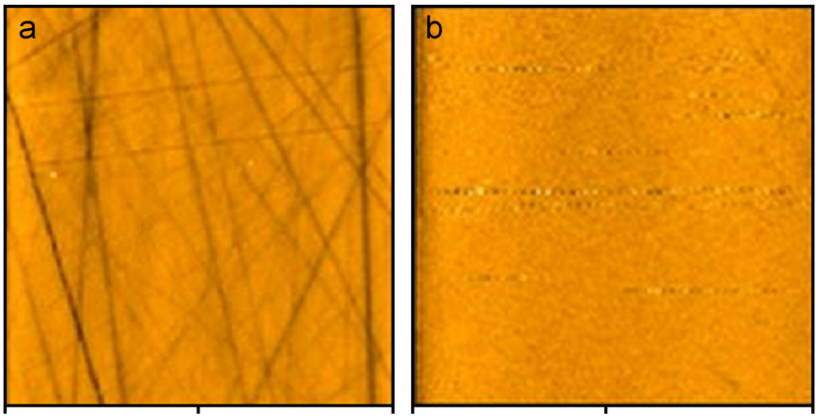

Fig. 1. 5 μm × 5 μm SPM images of polished Ga-face *c*-plane GaN surfaces: (a) mechanical polishing with diamond media only; (b) CMP prepared surface. The RMS roughness for the mechanically polished sample is 0.6 nm and the RMS roughness for the CMP prepared sample is 0.18 nm.