

InN layers grown by the HVPE

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We report on the properties of high quality HVPE InN and on successful subsequent MBE growth of InN layers with improved characteristics on HVPE InN template substrates. InN layers were grown by HVPE on GaN/sapphire HVPE templates. The (00.2) XRD rocking curve of the best InN layer (RC) had the FWHM of about 375 arc sec, being the narrowest XRD RCs ever reported for HVPE InN. Transmission Electron Microscopy (TEM) revealed that at the GaN/InN interface, the threading dislocations that come from GaN were transmitted into the InN layer. We estimated the dislocation density in HVPE grown InN to be in the low 10⁹ cm⁻² range.

Reflection high energy electron diffraction (RHEED) confirmed monocrystalline structure of the InN layers surface. Layers photoluminescence (PL) showed edge emission around 0.8 eV. Hall measured free electron concentration was in the range of $10^{19} - 10^{20}$ cm⁻³ and electron mobility was ~200 cm²/V s. MBE growth of InN was performed on the HVPE grown InN template substrate demonstrating the improvement of material quality in the case of homo-epitaxial growth of InN. Demonstration of the high quality HVPE InN materials opens a new way for InN substrate development.

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1 Introduction InN has attracted great interest as a promising material for THz emission, ultra high frequency electronics, IR light emitters and various sensors. Although high quality InN layers can be grown by molecular beam epitaxy, this material is not commercially available. Hydride Vapor Phase Epitaxy (HVPE) is known to produce high crystalline quality wide band gap nitride materials (GaN, AlN) with high growth rates. This method is attractive as a possible way of low cost production for InN template substrates for said applications. Results on HVPE of InN are limited and the quality of reported material is low. Recently we have reported on the HVPE growth of InN layers and nano-rods [1, 2]. In our current paper we focus on structural, optical, and electrical properties of HVPE grown InN layers and on InN layers grown by MBE on



HVPE InN material. Results of XRD including RSM, TEM, RHEED and PL characterization of InN layers are presented. Free electron concentration and mobility were estimated using Hall measurements.

2 Experimental InN layers were grown by HVPE in a temperature range from 500 to 600 °C on GaN/sapphire template substrates using NH₃ and HCl gas precursors and Ar as a carrier gas [1]. InN growth rate ranged from 0.3 to 0.5 μ m/hour. Layers with thickness from 0.02 to 2.5 microns were fabricated.

Grown layers were investigated by XRD measurements. A high-resolution triple-crystal configuration with the Ge (220) 2-crystal monochromator was used as the incidence optics. XRD spectra using $\omega/2\theta$ scan were meas-

ured between angles of 31° and 36° covering the range of InN and GaN (00.2) reflections. For XRD RSM measurement region was around the (10.2) reciprocal lattice point (RLP).

TEM was used to investigate the grown layers polarity and dislocation structure. Bright field and dark field imaging were employed. High resolution TEM images were also recorded for layers cross-sectional views.

RHEED patterns from the InN layer surface were registered at a survey angle of about 1° for incident electron beam along the (11.0) direction in the InN crystal.

Low temperature and room temperature PL spectra were registered for InN layers. Room temperature transmittance measurements were performed as well. The results of optical characterization were compared with few results of Hall measurements of free electron concentration and mobility. Homo-epitaxial growth of InN layers on HVPE grown template substrates was done by MBE.

3 Results and discussion Results of XRD measurements indicated high crystalline quality of the HVPE grown InN material. No diffraction peaks were observed around angle of $2\theta/\omega = 33^{\circ}$ that signifies no metallic In or polycrystalline inclusions in grown layer. For most HVPE grown InN on GaN on sapphire layers the FWHM of ωscan (00.2) XRD rocking curve (RC) does not exceed 500 Arc sec. in all range of growth temperatures (500-600 °C). For the 0.2 µm thick InN layer grown on GaN/sapphire at 550 °C, the ω-scan (00.2) XRD RC had the FWHM of about 375 arc sec, representing the narrowest XRD RC reported so far for HVPE InN. The asymmetric reflection (10.2) RC for this layer is about 1582 arc sec. The XRD reciprocal space map (RSM) was measured for this layer. For RSM (Fig. 1) upper and lower RLPs correspond to asymmetric (10.2) GaN and (10.2) InN reflexes, respectively. Transverse widths of the reciprocal lattice points (RLPs) are inversely proportional to the thicknesses of the layers while their longitudinal elongations are related to mosaic structure of the layers. The RLPs are almost exactly aligned on a vertical line that evidences equal inplane lattice parameters for InN and GaN. RSM confirms-



pseudomorphic growth of InN on GaN/sapphire template with fully strained (compressed) InN layer.

The monocrystalline surface of InN layers with a mosaic structure was confirmed by RHEED (Fig. 2). High crystalline quality of InN surface is an important factor for successful InN homoepitaxial overgrowth on the HVPE template substrates.



Figure 2 RHEED patterns of InN surface taken at survey angle 1° with incident beam along (11.0) direction in InN.

TEM measurements show that polarity of InN layers appears to be 'In', as there is no evidence of any inversion boundary at the InN/GaN interface and GaN layer measurements show Ga-polarity of the GaN template substrate. This result is consistent with results obtained by MBE [3] where only the In polarity InN layers could be grown at temperatures 550 °C and above. High resolution TEM reveals a high density of misfit dislocations at InN/GaN interface (Fig. 3). The threading dislocations from GaN are transmitted into the InN layer and others can be generated at the interface (Fig. 4).



Figure 3 High resolution, bright field TEM image of InN/GaN interface with misfit dislocations clearly seen.

In the GaN template layer a dislocation density in the low 10^8 cm⁻² range is measured. In InN layers this density is approximately one order of magnitude higher.

Figure 1 Reciprocal space map of the HVPE InN grown on GaN on sapphire template substrate around the (10.2) reciprocal lattice point.



The results of PL measurements of HVPE InN at room temperature using Argon laser excitation show the maximum of PL spectra at about 0.8 eV. This corresponds to the edge PL in InN with free electron concentration in the low 10^{19} cm⁻³ if consider the relationship of InN band gap on carrier concentration [4]. Hall measurements confirmed the free electron concentration in this HVPE InN layer to be about 5×10^{19} cm⁻³. The electron mobility of 212 cm² V⁻¹ s⁻¹ was measured for this layer.



Figure 4 Bright field TEM image of InN/GaN interface with threading dislocations penetrating from GaN and generated at the interface.

For the first time the homoepitaxial growth of InN was demonstrated by growing MBE InN on HVPE InN template substrates that result in improved structural quality and optical performance (PL intensity) of the MBE InN layers. As a comparison of the growth substrate, homoepitaxy versus heteroepitaxy, MBE grown InN layers, ~1 micron, were prepared on HVPE InN(0.1 µm)/GaN(2 μm)/sapphire and on GaN(2 μm)/sapphire templates. The homoepitaxial InN (MBE) layer results in a FWHM for the RC data of 460 arc sec, which is very close to that for initial thin HVPE InN template (440 arc sec). As a comparison, the MBE grown InN on the GaN/sapphire substrate, under the same growth conditions, yields a FWHM of 608 arc sec; whereby the FWHM of the GaN template substrate was ~380 arc sec. PL measurements of the InN layers grown on the two different substrates (InN/GaN/Sapphire versus GaN/sapphire) show a significant increase in intensity at 30 K and at room temperature for the homoepitaxial growth conditions (Fig. 5). This implies the improvement of material quality by using homoepitaxy InN/GaN/sapphire templates.



Figure 5 PL spectra of homoepitaxial MBE InN on HVPE InN template along with PL of heteroepitaxial InN on GaN layer grown under the same conditions.

4 Conclusion InN layers with record low for HVPE material XRD RC FWHM of 375 arc sec are demonstrated. Dislocation density in the low 10^9 cm^{-2} range is estimated for these layers. InN layers grown by the HVPE on Gapolar GaN are In-polar. Free electron concentration in these layers is about $5 \times 10^{19} \text{ cm}^{-3}$ and electron mobility is about 200 cm² V⁻¹ s⁻¹.

Successful homoepitaxial growth of InN by MBE on the HVPE grown InN template substrates is demonstrated, for the first time. Grown layers have improved structural and optical quality compared to the heteroepitaxial InN grown under the same conditions.

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