Buried *p*-type layers in Mg-doped InN

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Variable magnetic field Hall effect, photoluminescence, and capacitance-voltage (*CV*) analysis have been used to study InN layers grown by plasma assisted molecular beam epitaxy. All three techniques reveal evidence of a buried *p*-type layer beneath a surface electron accumulation layer in heavily Mg-doped samples. Early indications suggest the Mg acceptor level in InN may lie near 110 meV above the valence band maximum. The development of *p*-type doping techniques offers great promise for future InN based devices. © *2006 American Institute of Physics*.
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The controversy surrounding the band gap energy of InN has prompted a drive towards greater understanding of the material.[1,2] This encouraged more device-focused InN research with investigations into routes towards *p*-type InN. All as-grown unintentionally doped InN is *n* type. Donor-type native defects are thought to be prevalent as a result of the unusually low conduction band minimum.[3] The tendency of native defects in InN to form donors manifests itself particularly severely at surfaces where high levels of electron accumulation are observed. This accumulation layer is strongly localized within the first 5 nm of the film surface with a local carrier concentration as high as 10^{21} cm^{-3} in this region.[4,5] This highly conductive layer at the surface of InN films creates difficulties when assessing the impact of dopants, as electrical measurements are often dominated by the surface charge layer.

The first study of Mg-doped InN was by Blant *et al.* who used extended x-ray absorption fine structure analysis of Mg incorporation within InN during plasma-assisted molecular beam epitaxy (PAMBE) growth.[6] Mg on the In lattice site was identified as the most likely occupation, yet single field Hall effect measurements revealed *n*-type conduction. Although *p*-type material was not identified, Mamutin *et al.* also studied PAMBE InN:Mg, suggesting that Mg acts as a surfactant enhancing film growth.[7] At the time, little was known regarding intrinsic electron accumulation at InN surfaces. As a result, single field Hall effect measurements undertaken by the authors would have been heavily obscured by this accumulation charge.

More recently Jones *et al.* reported a combination of capacitance-voltage (*CV*) and photoluminescence (PL) data which accounted for the effect of the surface charge layer and provided the first indirect evidence for buried *p*-type layers in Mg-doped InN thin films.[8] *CV* analysis suggested possible depletion and hole layers in the near surface region, and PL quenching in Mg-doped layers were consistent with strong electric fields in the near surface region. Also, Cimalla *et al.* recently used sputter depth profiling and ultraviolet photoelectron spectroscopy to show a discontinuity in the conductivity of Mg-doped InN in the near surface region, consistent with a depletion region between *n*- and *p*-type layers.[9] In the present study we use a combination of variable magnetic field Hall effect, PL, and *CV* analysis to study the electrical and optical properties of a wide range of Mg concentrations in PAMBE grown InN, some of which exhibit distinct *p*-type conduction.

All films were grown at the University of Canterbury in a Perkin-Elmer 430 MBE system with a base pressure of 5×10^{-11} Torr. In and Mg fluxes were supplied from standard 60 and 2 cm^3 thermal effusion cells, respectively. Active nitrogen was provided from an Oxford Applied Research HD-25 rf plasma source operated at 150 W and 1.3 SCCM (SCCM denotes cubic centimeter per minute at STP) at a growth rate of about 150 nm/h. All films were grown on 1.6 μm thick metal-organic chemical vapor deposition GaN templates on sapphire substrates, after outgassing at 800 °C and two Ga flash-off cleaning steps. An additional 150 nm of MBE grown GaN was deposited before 30 nm of undoped InN followed by 500 nm of the Mg-doped InN layer. The growth temperature for all InN layers was 450 °C.

Six different Mg concentrations were studied with the In cell always operated at a flux of 1.5×10^{14} at. cm^{-2} s^{-1} as measured by a quartz crystal microbalance; the Mg cell temperatures were chosen for Mg concentrations of 6×10^{17}, 2×10^{18}, 6×10^{18}, 6×10^{19}, 3×10^{20}, and 1×10^{21} cm^{-3} assuming that all Mg is incorporated within the films. Mg content of the two most heavily doped films was determined using 2.5 MeV He+ ion particle-induced x-ray emission (PIXE), and found to be within a factor of 2 of the values predicted using theoretical flux curves. The reasonable correspondence between the two approaches gives confidence that most of the Mg flux is indeed incorporating within the film; all further references to Mg concentrations are therefore based directly on nonscaled theoretical flux calculations. The depth profile of the Mg cannot be determined from PIXE, but the recent report from Jones *et al.* described a similar approach resulting in homogenous Mg incorporation throughout the layers as determined by secondary ion mass spectroscopy.[8]

CV measurements were performed using an electrolyte to form a Schottky-like junction, as no metal deposition has

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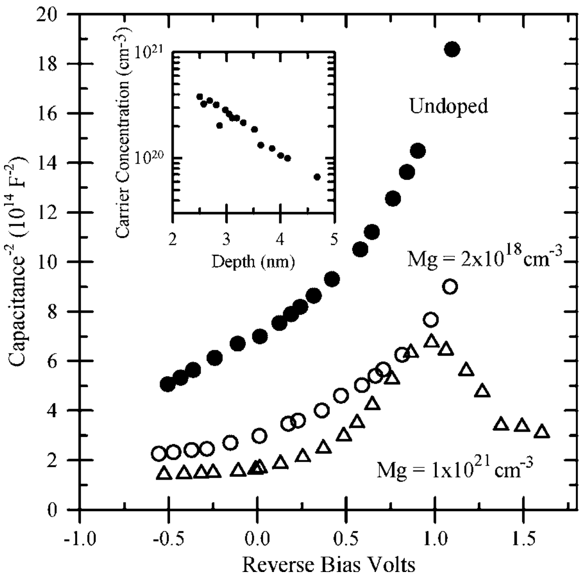

FIG. 1. Electrolytic capacitance-voltage measurements on unintentionally and Mg-doped InN films. Inset: concentration, due primarily to the near surface accumulation layer, is 1.8×10^{13} cm^{-2}, close to the 1.6×10^{13} cm^{-2} result obtained by others (Ref. 4).

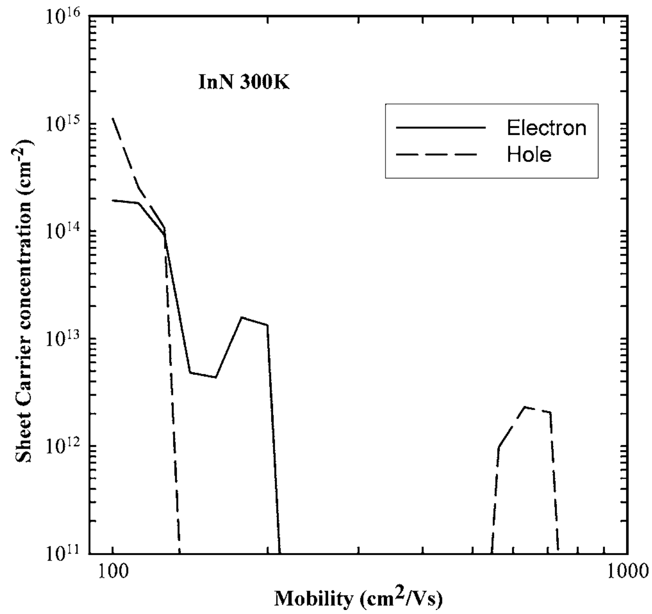

FIG. 2. QMSA spectrum of the 3×10^{20} cm^{-3} Mg-doped film at 300 K showing light hole, heavy hole, and electron conductions.

yet formed such a junction on InN without annealing at an excessively high temperature.[10] Etching in 32% HCl removed any In droplets on the surface and a solution of 0.2*M* KOH and 0.2*M* EDTA formed a rectifying contact with a diameter of 1 mm used to examine near surface charge density.[4,8] A 3 kHz, 50 mV signal was applied to a Pt electrode to drive the capacitance measurement.[11] When the reciprocal square of the capacitance (C^{-2}) increases with a greater reverse bias, then the mobile charge carriers are negative, while a decrease implies *p*-type carriers.[12]

As shown in the inset of Fig. 1, the surface sheet electron density and the spatial distribution of electrons of the unintentionally doped sample as obtained from *CV* measurements agree well with that observed previously.[4] The density and depth are calculated using the standard equations for depletion width,[12] and do not literally reflect the density or thickness of the accumulation layer, as a depletion layer does not begin to form until a reverse bias is sufficient to empty the accumulation layer. Nonetheless, the total amount of charge in the layer can be found from the integral of the capacitance over the applied reverse bias voltage. This is identical to the integral of concentration over depth, so the integrated concentration is indeed a measurement of the accumulation layer. Figure 1 compares the *CV* characteristic of the undoped film to two Mg-doped films. The reciprocal square capacitance has a slope which changes sign on the heavily doped sample, indicating that a *p*-type layer is buried beneath a *n*-type layer.

Variable magnetic field Hall measurements can be used to separate the influences of different, parallel conducting layers.[13] Resolution is limited by the squared product of mobility and maximum magnetic field, $(\mu B)^2$, which should be comparable to unity for unambiguous results.[14] The hole mobility in InN is expected to be an order of magnitude lower than that of the electron, corresponding to the difference in the calculated effective mass.[15] However, the light hole mass should be comparable to the electron mass, and the light hole band is predicted to be nearly degenerate with the heavy hole band. Thus, for sufficiently heavy acceptor doping, the light hole will be resolvable and can serve as an indication of *p*-type conduction. Quantitative mobility spectrum analysis, a fitting routine which allows for a layer's inhomogeneity,[16] as well as standard multiple carrier fitting (MCF) were used to analyze the field dependent data. Resistivity and Hall coefficient were measured, using the van der Pauw geometry, at logarithmically spaced fields up to a maximum 12 T.

The quantitative mobility spectrum analysis (QMSA) spectrum for the 3×10^{20} cm^{-3} Mg-doped sample is shown in Fig. 2. This sample, measured at 300 K, was clearly *p* type, exhibiting a spectrum indicative of both light and heavy holes along with an electron peak that may represent the surface conduction. This spectrum is reminiscent of that observed for *p* type HgCdTe with a *n*-type conducting layer also present.[17,18] MCF analysis indicated a heavy hole sheet concentration of 4×10^{15} cm^{-2} and mobility of about 50 cm^2/V s, and an electron with a sheet carrier concentration of 1×10^{13} cm^{-2} and mobility of 200 cm^2/V s. The close correspondence between the directly measured electron sheet concentration and that determined by *CV* strongly suggests that this is the surface electron. Similar measurements made on the highest Mg-doped sample also clearly showed a hole peak that could be associated with light hole conduction. Low mobility carriers are difficult to cleanly separate out. Yet, the presence of the light hole strongly suggests that there are *p*-type regions in this sample as well. The sample having Mg concentration of 6×10^{18} cm^{-3} was also measured; only *n*-type conductivity was observed in this case, similar to that observed in undoped samples.

Only films with Mg content less than 10^{19} cm^{-3} exhibited any detectable PL. Figure 3(a) shows the PL spectra of three such InN films. The spectra of layers with Mg contents of 6×10^{17} and 2×10^{18} cm^{-3} are typical of undoped InN, with peak intensities in the range of 0.65–0.7 eV and full width at half maximum (FWHM) of less than 50 meV. In contrast, the film with Mg content of 6×10^{18} cm^{-3} shows a peak PL signal at much lower energy, around 0.58 eV. The dip observed in the PL spectrum at 0.56 eV is an absorption artifact caused by the spectrometer; accounting for this, the

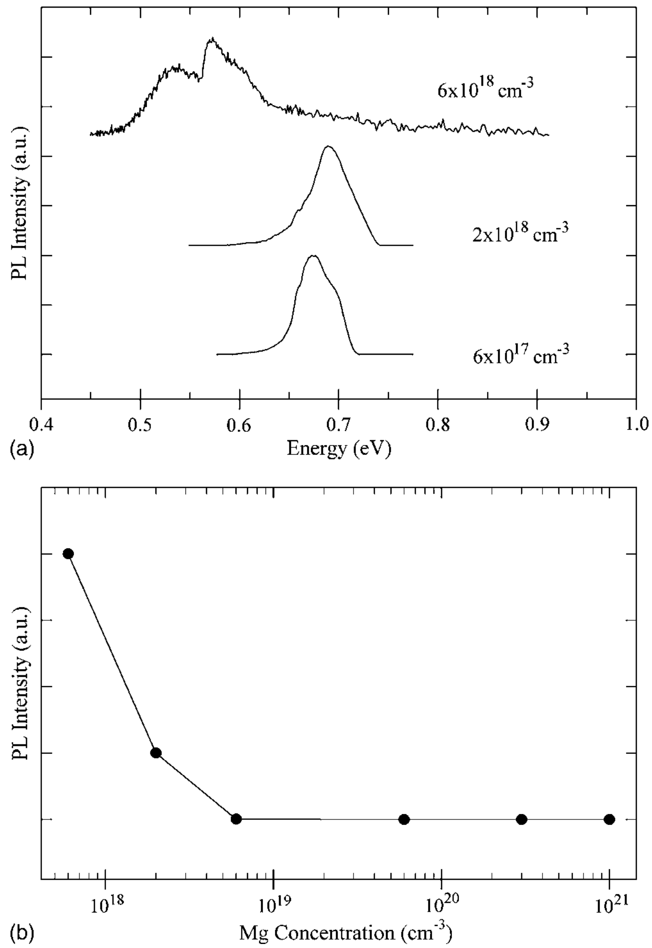

FIG. 3. (a) Photoluminescence signals (488 nm argon laser line, InSb detector) from three lightly Mg-doped InN films. (b) The 0.68 eV peak intensity measured from InN films as a function of Mg concentration.

PL signal actually peaks near 0.56 eV. The line shape of this signal is much broader than the lower Mg concentration samples, with a FWHM of 100 meV. If this feature is related to transitions between the conduction band edge and a Mg acceptor level, it would indicate a Mg acceptor level approximately 110 meV above the valence band edge. Figure 3(b) shows, as mentioned earlier, that quenching of PL occurs for Mg contents greater than 10^{19} cm^{-3}. For lower Mg content, the PL intensity is observed to increase with decreasing Mg content, until at 6×10^{17} cm^{-3} Mg, the PL intensity is comparable to that of undoped films. Jones *et al.* also observed quenching in heavily Mg-doped films,[8] although the 0.56 eV feature was not observed in their study.

While the Hall result suggests about 25% activation, higher than expected for a 110 meV activation energy, lower electrical activation energies are often observed in heavily doped samples due to screening, as with Mg in GaN. If the same relative activation holds for lower concentrations, then the sample with 6×10^{18} cm^{-3} Mg would have at most a *p*-type carrier concentration of 1×10^{18} cm^{-3}, which is comparable or lower than the background *n*-type doping, consistent with not obtaining a net *p*-type conductivity.

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