

Influence of growth conditions and polarity on interface-related electron density in InN

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Electron accumulation at the oxidized surface of In- and N-polarity indium nitride is shown to exhibit no dependence on the growth conditions (varied from In- to N-rich), revealing the surface Fermi level to be pinned 1.4 ± 0.1 eV above the valence band maximum for all cases. This is in contrast to the interpretation of recent multiple-field Hall effect measurements, which suggested almost an order of magnitude increase in the sheet density of the accumulation layer upon moving from In-rich to N-rich growth conditions, and sample thickness dependent single-field Hall effect measurements which suggested different surface sheet densities for In- and N-polarity samples. However, an increase in the electron density approaching the InN/GaN (buffer layer) interface was not considered in the analysis of these Hall effect measurements, and this is invoked here to reconcile the constant surface Fermi level with the variations in “excess” sheet density observed in the previous Hall effect studies. © 2008 American Institute of Physics. [DOI: [10.1063/1.3020528](https://doi.org/10.1063/1.3020528)]

I. INTRODUCTION

InN is known to exhibit a large downward bending of the conduction and valence bands at the surface, with respect to the Fermi level, accompanied by an extreme accumulation of electrons.^{1–3} This behavior, in contrast to the electron depletion observed at the surface of most other III-V semiconductors, results from the particularly low Γ -point conduction band minimum in InN, which lies well below the charge neutrality level.⁴ Although the majority of studies on the surface electronic properties of InN to date have focused on the wurtzite *c*-plane surface, there has been recent interest in other surface orientations. A pronounced universality of the electron accumulation has been observed at the clean surface of wurtzite In- and N-polar *c*-plane and nonpolar *a*-plane InN.⁵ Additionally, electron accumulation has been observed at the nonpolar *m*-plane surface of InN nanocolumns^{6,7} and at the surface of zinc-blende InN.⁵

Due to the significant variation in the carrier concentration with depth below the surface in InN, a single-field Hall effect (SFHE) measurement does not accurately characterize the bulk electrical properties of the material. Therefore, several investigations^{8–13} have employed multiple-field Hall effect (MFHE) measurements to separate the effects of carriers in different regions of the sample. In these studies, two electron species with differing mobilities and concentrations were identified, attributed to electrons in the surface electron accumulation layer (low mobility peak) and bulk electrons (higher mobility peak). The surface roughness is known to be highly dependent on the growth conditions for InN,^{14,15} with a reduction in surface roughness achieved for increasing In-flux during growth. Fehlberg *et al.*¹³ investigated the transport properties of In-polarity InN films grown under In-rich,

stoichiometric and N-rich conditions. They observed similar bulk electron concentrations in each film, although the concentration of the peak attributed to surface electrons was observed to increase with decreasing In-flux during growth, attributed to the increasing roughness of the surface.

Another method used to distinguish between surface and bulk conduction effects has been to investigate the sheet density determined from SFHE measurements as a function of sample thickness for samples grown under identical conditions. An extrapolation of the sheet density to zero thickness samples gives the “excess” sheet charge, that has previously been associated with the surface charge.¹ This procedure has recently been applied to In- and N-polarity samples grown under optimized In-rich conditions^{14,15} revealing a different excess sheet density in each case. This is seemingly in contrast to the universality of the surface electron accumulation observed at In- and N-polarity InN surfaces,⁵ and requires further investigation.

This article reports high resolution x-ray photoemission spectroscopy (XPS) measurements of In-polarity InN samples grown under In-rich, stoichiometric, and N-rich conditions and of N-polarity InN samples grown under In-rich and N-rich conditions. No change in the surface Fermi level position is observed for the different samples, indicating the same degree of surface electron accumulation independent of the growth conditions or polarity of the samples. This is reconciled with the recent Hall effect results^{13–15} by considering an increase in the electron density approaching the InN/GaN (buffer layer) interface.

II. EXPERIMENTAL DETAILS

In-polar (N-polar) InN samples were grown to a thickness of 1 μm by plasma-assisted molecular beam epitaxy on high-quality Ga-polar (N-polar) GaN templates provided by Lumilog. To produce a growth surface free from contamina-

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tion and achieve good interface quality, ~ 250 nm (~ 40 nm) thick GaN buffer layers were grown on the GaN templates under Ga-rich conditions at a temperature of 700 °C, prior to InN growth at the optimized temperature of 450 °C (540 °C). For In-polarity InN, samples grown under In-rich, stoichiometric, and N-rich conditions were studied, while for N-polarity InN, samples grown in the In-rich and N-rich regimes were investigated. Further details of the growth are reported elsewhere.^{14,15}

High resolution XPS measurements were performed at room temperature using a Scienta ESCA300 spectrometer at the National Centre for Electron Spectroscopy and Surface Analysis, Daresbury Laboratory, U.K. X-rays of energy $h\nu = 1486.6$ eV were produced using a monochromated rotating anode Al $K\alpha$ x-ray source. The ejected photoelectrons were analyzed by a 300 mm mean radius spherical-sector electron energy analyzer with 0.8 mm slits operated at a pass energy of 150 eV. The effective instrumental resolution is 0.45 eV. The binding energy scale is given with respect to the Fermi level calibrated from the Fermi edge of an ion-bombarded silver reference sample. The measurements were performed on oxidized InN samples to allow direct comparison with previous Hall effect studies.

The surface specificity of XPS results from inelastic scattering of photoelectrons in the sample, resulting in an exponential fall-off of intensity with depth below the surface from which the photoelectrons originate. The probing depth is therefore determined by the inelastic mean free path (IMFP) of the photoelectrons. For Al $K\alpha$ x-rays, photoelectrons excited from the valence band of InN have an IMFP of ~ 25 Å, calculated from the TPP-2M predictive formula of Tanuma *et al.*¹⁶ Consequently, in the normal emission geometry used here, approximately 65% of the photoemission signal comes from within ~ 25 Å of the surface and 95% from within ~ 75 Å of the surface, with the majority of the signal originating from photoelectrons generated closest to the surface.

III. RESULTS, ANALYSIS, AND DISCUSSIONS

Valence band photoemission spectra of the In-polarity samples are shown in Fig. 1. The leading edge of each valence band photoemission spectrum is coincident in energy for all of the In-polarity samples, indicating that the position of the surface Fermi level relative to the valence band maximum (VBM) is the same for the In-polarity samples grown under In-rich, stoichiometric and N-rich conditions.

The position of the surface Fermi level was determined by extrapolating the leading edge of the valence band photoemission to the background level in order to take account of broadening of the photoemission spectra.¹⁷ From this method, the VBM to surface Fermi level separation ξ is determined as 1.4 ± 0.1 eV. This is slightly lower than the recently reported⁵ universal value of 1.53 ± 0.1 eV observed at clean In- and N-polarity c -plane and at nonpolar a -plane samples. There are two possible reasons for this. First, the measurements reported here are from oxidized samples. Charge transfer from the accumulation layer to the more electronegative oxide layer may occur, slightly reducing the

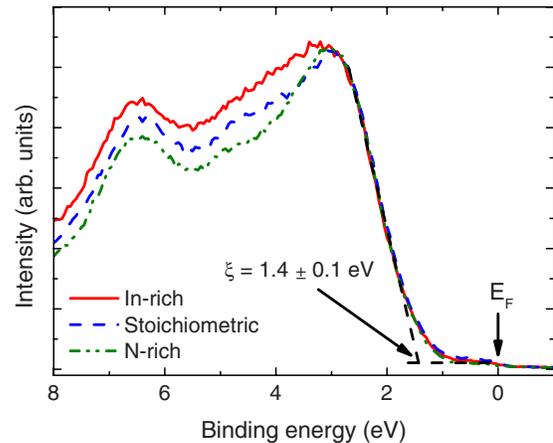


FIG. 1. (Color online) Valence band photoemission spectra of In-polarity InN samples grown under In-rich (solid line), stoichiometric (dashed line), and N-rich (dot-dashed line) conditions. The linear extrapolation used to determine the VBM to surface Fermi level separation ξ is also shown.

amount of electron accumulation. This is consistent with initial evidence that ozone-induced oxidation may reduce the degree of electron accumulation at InN surfaces.¹⁸ Second, the linear extrapolation method used here is known to slightly underestimate the value of the VBM to surface Fermi level separation when there is significant downward band bending.^{4,19} The measurements presented here are consistent with previous XPS results for oxidized InN samples determined from the linear extrapolation method.^{4,20}

The surface state density was determined by solving Poisson's equation within a modified Thomas-Fermi approximation (MTFA). Details of the calculations are reported elsewhere.²¹ A representative bulk carrier density of 4×10^{17} cm⁻³ was assumed, taken from the MFHE results of Fehlberg *et al.*¹³ for 1 μ m thick samples grown under similar conditions to those used here. The calculated band bending and carrier concentration profiles in the near surface region for this bulk carrier concentration and the surface Fermi level determined from the XPS measurements are shown in Fig. 2. The pinning of the Fermi level high above the VBM at the surface leads to a large downward bending of the bands relative to the Fermi level, accompanied by a distinct peak in the carrier density close to the surface as has been discussed in detail previously.^{2,4,5} It is the electrons in this accumulation layer that have previously been assigned as the low-mobility carrier species in the MFHE measurements.

The surface state density obtained from the Poisson-MTFA calculation presented here is $(9.2 \pm 2.1) \times 10^{12}$ cm⁻² for the In-polarity samples. This value is slightly lower than the universal value of 1.6×10^{13} cm⁻² observed at In- and N-polarity c -plane and at nonpolar a -plane InN surfaces,⁵ and this is again attributed to a possible small reduction in surface Fermi level position for oxidized compared to clean samples, and a slight underestimation of the surface Fermi level position using the linear extrapolation method of analysis as discussed above. The calculated surface state density was found to be relatively insensitive to the value of the bulk electron concentration used. Indeed, using bulk electron densities determined from SFHE measurements for these particular samples (an upper limit of the true bulk carrier con-

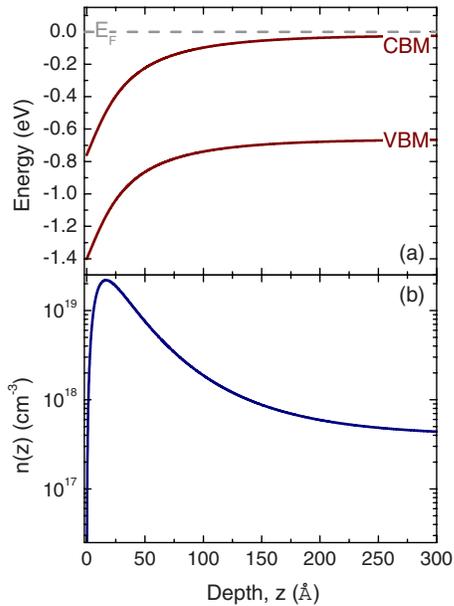


FIG. 2. (Color online) Poisson-MTFA calculations of (a) band bending relative to the Fermi level and (b) resulting carrier concentration variation as a function of depth z below the surface for the In-polarity InN samples grown under In-rich, stoichiometric, and N-rich conditions. A bulk carrier concentration of $4 \times 10^{17} \text{ cm}^{-3}$ from Ref. 13 has been assumed.

centration) yielded similar band bending and carrier concentration profiles to that shown in Fig. 2, with a surface state density within the stated errors of the surface state density calculated assuming the lower bulk carrier concentration.

The issue of band gap renormalization (BGR), whereby electron-electron and electron-impurity interactions cause a renormalization (shrinkage) of the band gap with increasing carrier density, has been extensively discussed for degenerate semiconductors, including InN.^{22–26} Due to the high carrier densities present in the accumulation layer, it has recently been suggested that this BGR may be particularly important at the surface of InN.²⁵ The most common approach to describe BGR within the bulk of a semiconductor,²⁴ recently extended to the surface under a number of simplifying assumptions,²⁵ is to use the analytical solutions of Inkson²² and Berggren and Sernelius²³ to estimate the amount of BGR. However, as Berggren and Sernelius themselves commented, these calculations significantly overestimate the amount of BGR. This is further supported by recent first-principles calculations of Walsh *et al.*²⁶ who showed that for the addition of electrons to the system, the BGR, defined as the difference between the Moss–Burstein (band-filling) shift and the total optical band gap shift, is actually very small (even for carrier concentrations up to $\sim 10^{21} \text{ cm}^{-3}$ in In_2O_3), provided a nonparabolic dispersion relation for the semiconductor conduction band is used. However, if a parabolic dispersion relation is used, the Moss–Burstein shift is drastically overestimated, causing a much larger BGR than really occurs to be invoked to reconcile theory with experimental results. In our case, however, we do use a nonparabolic ($\mathbf{k} \cdot \mathbf{p}$) conduction band dispersion relation, and so the effects of BGR should be small. Additionally, although the peak electron density in the accumulation layer is large, the carrier

wave functions must tend to zero amplitude at the surface, and so the carrier density and any BGR must also tend to zero here, limiting its influence on the electronic properties at the surface, in contrast to the approach of Yim *et al.*²⁵ Consequently, given the small contribution of BGR expected, and the lack of a consistent theoretical treatment with which to include it, BGR has been neglected in the calculations performed here. However, we do not expect this to lead to significant errors in the results.

Crucially, in any case, the surface state density, which is equal to the space charge of the accumulation layer in order to maintain charge neutrality, must be the same for the In-polarity samples grown under In-rich, stoichiometric, and N-rich conditions from the XPS data presented above. This is in contrast to the interpretation of the MFHE results of Fehlberg *et al.*¹³ where almost an order of magnitude increase in the sheet density of low mobility carriers between samples grown under In-rich (lowest determined surface electron concentration) and N-rich (highest determined surface electron concentration) conditions was attributed to an increase in the density of electrons in the surface electron accumulation region.

MFHE measurements have no inherent depth resolution. The low mobility peak is assigned as due to surface electrons from the prior knowledge of electron accumulation at InN surfaces. However, an increase in electron density approaching the interface is also thought to occur.^{27–32} Indeed, from detailed analysis of infrared reflectivity measurements, Ishitani *et al.*³² estimated the increase in carrier density at the InN/GaN (buffer layer) interface to correspond to a sheet density of the order 10^{13} cm^{-2} , and so this would be expected to have at least as significant an influence in Hall effect measurements as the surface electron accumulation. The carriers due to the interface would also likely be of lower mobility than those in the bulk, and it seems likely, therefore, that a combination of the surface and interface electron density gives rise to the low mobility peak in MFHE spectra. Indeed, it has previously been pointed out by Fehlberg *et al.*¹¹ that it is unlikely that the effects of an increase in electron density at the surface and interface could be resolved in MFHE measurements. However, they do not consider this further in their analysis of the MFHE results.

A change in the surface sheet density with growth conditions has been ruled out by the XPS measurements presented here. Additionally, no change in the high mobility peak of the MFHE measurements was observed with changing growth conditions, indicating that the concentration of “bulk” electrons does not change with growth conditions. As the samples investigated here were grown and measured under the same conditions as those of the MFHE study, we expect the surface electronic properties of these samples to be very similar, especially given the strong pinning of the Fermi level at the surface of InN. Consequently, we attribute the increase in the sheet density of the low mobility MFHE peak on moving from In-rich to N-rich growth conditions¹³ to the only remaining plausible candidate, namely, an increase in the sheet density of electrons due to the interface, as represented schematically in Fig. 3. Assuming the low mobility peak in these MFHE measurements therefore con-

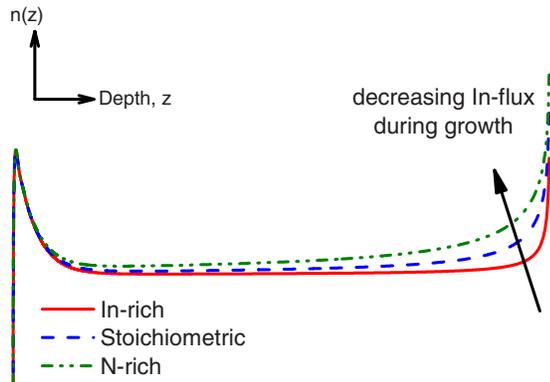


FIG. 3. (Color online) Schematic representation of the increase in sheet density of electrons due to the interface with decreasing In-flux during growth.

tains both the surface and interface contributions to the total carrier density, and taking the surface sheet density as $\sim 1 \times 10^{13} \text{ cm}^{-2}$ from above, the sheet density due to the interface can be estimated as increasing from $\sim 2 \times 10^{13}$ to $\sim 9 \times 10^{13} \text{ cm}^{-2}$ upon moving from In-rich to N-rich growth conditions. Additionally, a variation in mobility of the surface and/or interface carriers within a single sample and between samples grown under different conditions may cause inaccuracies in assessing the true sheet density variation; indeed, Fehlberg *et al.*¹³ pointed out that a very broad mobility spread in the MFHE results for the most N-rich sample makes accurate determination of the sheet density associated with the low mobility electrons difficult.

N-polarity InN, grown under In- and N-rich conditions, was also investigated here. The valence band photoemission from these samples is shown in Fig. 4. A linear extrapolation of the leading edge of this valence band photoemission to the baseline again reveals a surface Fermi level position $1.4 \pm 0.1 \text{ eV}$ above the VBM for both samples. Thus, the surface Fermi level is pinned in the same place at In- and N-polarity InN, in agreement with other recent observations.^{5,33} Additionally, this reveals that the growth

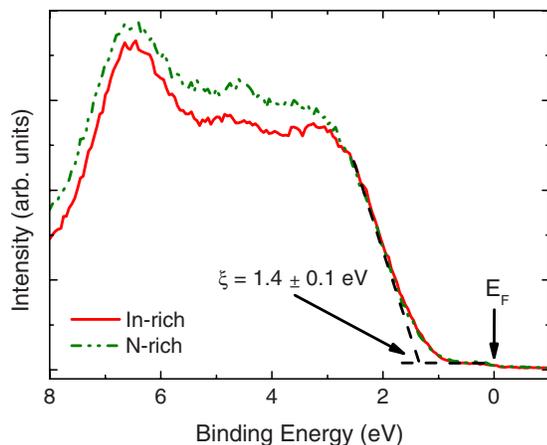


FIG. 4. (Color online) Valence band photoemission spectra of N-polarity InN samples grown under In-rich (solid line) and N-rich (dot-dashed line) conditions. The linear extrapolation used to determine the VBM to surface Fermi level separation ξ is also shown.

conditions (In- or N-rich) do not affect the degree of electron accumulation at the surface of N-polarity InN.

Sample thickness dependent SFHE measurements of samples grown under the same conditions as the In-rich samples investigated here have previously revealed an excess sheet density extrapolated for zero thickness samples of $\sim 5 \times 10^{13}$ and $\sim 3 \times 10^{13} \text{ cm}^{-2}$ for In- and N-polarity InN, respectively. From the surface sheet density of $\sim 1 \times 10^{13} \text{ cm}^{-2}$ determined here, this suggests an interface-related electron sheet density of $\sim 4 \times 10^{13}$ and $\sim 2 \times 10^{13} \text{ cm}^{-2}$ for In- and N-polarity samples, respectively, indicating a slightly higher interface-related electron density for In- rather than N-polarity samples. These results are consistent with infrared reflectivity measurements³² and the In-polarity value is within experimental error of that estimated using the MFHE results discussed above.

As the III-N materials are pyroelectric,³⁴ the influence of spontaneous polarization (SP) charges at the interface must also be considered. However, the SP coefficients of InN and GaN are rather similar (-0.042 and -0.034 cm^{-2} , respectively³⁵) and so this effect would not be large. Indeed, assuming opposite polarity of adjacent faces at the interface, an electron (hole) interface sheet density of $\sim 0.5 \times 10^{13} \text{ cm}^{-2}$ would be expected for In (N) polarity samples due to SP. Additionally, the invariance of the surface Fermi level position between the In- and N-polarity samples suggests that the SP charge may itself be screened at the surface by a high density of surface charges³³ and possibly also at the interface by defects and interface states. Thus, SP could only be partly responsible for the lower interface-related sheet density for N-polarity as compared to In-polarity samples. The variations in interface-related electron density with growth conditions is therefore likely dominated by differences in defect or impurity concentrations at the interface.

IV. CONCLUSIONS

Electron accumulation has been investigated at the surface of In- and N-polarity InN samples as a function of growth conditions, varied from In-rich to N-rich. No change in the surface Fermi level position, and hence in the degree of surface electron accumulation, was observed as a function of the In-flux during growth. This is in contrast to the recently reported interpretation of MFHE measurements, which suggested almost an order of magnitude increase in the surface sheet density. This discrepancy is explained by considering the sheet density associated with an increase in electron concentration approaching the InN/GaN (buffer layer) interface which increases upon moving from In-rich to N-rich growth conditions.

Additionally, the surface Fermi level was shown to be located in the same position for both the In- and N-polarity samples, supporting the assignment of the electron accumulation as a universal feature of as-grown or *ex situ* prepared InN surfaces. A comparison of the surface sheet density with the excess sheet density for zero thickness samples determined from SFHE measurements again revealed an interface-related electron density, with a slightly higher concentration for In-polarity than N-polarity samples attributed

to a lower density of defects or impurities at the interface for N-polarity samples.

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