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Correlation between switching to *n*-type conductivity and structural defects in highly Mg-doped InN

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The effect of Mg doping on the microstructure of InN epitaxial films in relation to their free-charge carrier properties has been investigated by transmission electron microscopy (TEM) and aberration corrected scanning TEM. We observe a direct correlation between Mg concentration and the formation of stacking faults. The threading dislocation density is found to be independent of Mg concentration. The critical Mg concentration for the on-set of stacking faults formation is determined and found to correlate with the switch from *p*- to *n*-type conductivity in InN. Potential mechanisms involving stacking faults and point defect complexes are invoked in order to explain the observed conductivity reversal. Finally, the stacking faults are structurally determined and their role in the reduction of the free electron mobility in highly doped InN:Mg is discussed. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4922301>]

Accurate control of *p*-type doping in InN is a critical issue on the way to material implementation in advanced electronic devices such as next generation solar cells, light emitting diodes,^{1,2} and terahertz emitters.³ So far, only Mg has been shown as a working acceptor for InN.⁴ Due to peculiarities of energy band structure, intrinsic InN is extremely prone to *n*-type behavior. Therefore, obtaining *p*-type InN proves a challenge and large concentrations (10^{18} – 10^{20} cm^{−3}) of Mg need to be incorporated to overcome the inherent *n*-type conductivity. Furthermore, reliable measurements of free hole properties with standard contact-based electrical methods are difficult due to the inversion electron layer formed at the InN surface, as a result of Fermi energy pinning.⁵ To detect *p*-type conductivity and assess the free-charge carrier (FCC) properties, non-trivial methods are used such as electrolyte capacitance-voltage (ECV) measurements,⁴ thermopower measurements,⁶ or, as it was shown recently, infrared spectroscopic ellipsometry (IRSE).^{7,8}

It has lately been demonstrated that doping of InN with Mg results in a *p*-type conductivity window in the range of 1.0×10^{18} cm^{−3} ≤ [Mg] ≤ 2.9×10^{19} cm^{−3}.^{7–9} Holes from ionized Mg acceptors in concentrations less than 1.0×10^{18} cm^{−3} are not sufficient to compensate for the electrons from intrinsic donor defects and such InN material is *n*-type. Once Mg concentration reaches 1.0×10^{18} cm^{−3}, InN becomes *p*-type. Switching of conductivity again from *p*- to *n*-type at [Mg] ≥ 1.8×10^{20} cm^{−3} was tentatively ascribed to Mg induced donor defects. Indications of zinc-blende formation^{7,10} and polarity inversion^{11,12} accompanied the switch to *n*-type conductivity at high Mg doping. Despite existing reports,^{10,12–14} the relationship between Mg doping, defect formation, and FCC properties in wurtzite InN is not fully understood and requires further investigation.

In this paper, we present a comprehensive (scanning) transmission electron microscopy [(S)TEM] investigation of InN layers doped with Mg through a wide concentration range in relation to their FCC properties. A total of six samples were investigated, one of which was undoped, intrinsically *n*-type, while the remaining had the following Mg concentrations: 1.0×10^{18} cm^{−3}, 5.6×10^{18} cm^{−3}, 2.9×10^{19} cm^{−3}, 1.8×10^{20} cm^{−3}, and 8.0×10^{20} cm^{−3} (Table I). The three lower doped samples were *p*-type, whereas the two higher doped ones—*n*-type, as was previously confirmed by IRSE,⁷ ECV, and FTIR.¹⁵ The InN samples were wurtzite, *c*-plane oriented and grown with intended *In*-polarity on 4.1-μm-thick GaN/*c*-Al₂O₃ templates by plasma-assisted molecular beam epitaxy at 480 °C. The growth of 400-nm-thick Mg-doped InN was always preceded by 50-nm-thick undoped InN layer. Detailed information about the growth procedure has been reported elsewhere.¹⁵

Cross-sectional samples for the (S)TEM investigations were prepared by conventional methods, starting with mechanical cutting, gluing, and polishing, followed by low energy Ar-ion milling to electron transparency. TEM studies were performed using FEI Tecnai G2 TF20 UT 200 keV FEG microscope. The TEM images were taken in conventional bright-field TEM mode with electron beam parallel to $\langle 11\bar{2}0 \rangle$, which allows the visualization of *a*, *c*, and *a* + *c* dislocations with a predominant *c*-type nature.¹⁶

High-angle annular dark field (HAADF) STEM imaging was performed using the Linköping double corrected FEI Titan³ 60–300 at 300 kV. STEM probe had a 21 mrad semi-angle convergence at a camera length of 145 cm. Typical image size was 2048 × 2048 pixel with dwell time of 20 μs (total frame time of 100 s). The typical beam current was about 50–100 pA.

Figures 1(a)–1(f) show overview TEM images of the ~400-nm-thick InN epitaxial layers with increasing Mg doping from (a) to (f), where (a) represents the undoped

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TABLE I. Properties of the studied set of InN samples: Mg concentration, [Mg], bulk free-charge carrier concentration, N , mobility, μ , threading dislocation (TD) density, and stacking fault (SF) density.

Sample	[Mg] (cm^{-3})	N^7 ($\times 10^{17} \text{cm}^{-3}$)	μ^7 ($\text{cm}^2/\text{V}\cdot\text{s}$)	TD density (cm^{-2})	SF density (cm^{-1})
E925	Undoped	11.6 ± 0.5 (n)	1601 ± 87	7×10^9	0
E942	1.0×10^{18}	3.3 ± 0.1 (p)	21 ± 1	6×10^9	0
E941	5.6×10^{18}	1.6 ± 0.1 (p)	24 ± 1	7×10^9	0
E940	2.9×10^{19}	3.3 ± 0.1 (p)	30 ± 2	5×10^9	0
E924	1.8×10^{20}	9.20 ± 0.04 (n)	1079 ± 40	6×10^9	4×10^{10}
E930	8.0×10^{20}	77 ± 0.3 (n)	203 ± 4	4×10^9	6×10^{10}

intrinsically n-type sample. Threading dislocation (TD) densities were assessed from these images and are summarized with other sample properties in Table I. As can be seen from the figures, the apparent TDs nucleate at the InN/GaN interface, supposedly due to lattice mismatch and propagate all the way through the Mg doped InN layer. The estimated TD densities are in the range of $4\text{--}8 \times 10^9 \text{cm}^{-2}$ and vary negligibly through the doping range. The density of TDs in the same set of samples was previously measured by XRD.⁷ The numbers of edge type dislocations, which are the predominant type of dislocations in InN, were slightly higher, in the $1\text{--}2 \times 10^{10} \text{cm}^{-2}$ range, but also proved to be independent with Mg doping.⁷ In the sample with the highest Mg content (Fig. 1(f)), TDs may be partly obstructed by the high density of stacking faults (SFs) present in the layer. This can explain a small decrease in the number of TDs estimated from this specimen.

Apart from TDs that are present in all studied samples, SFs were found in the two samples with the highest Mg content; no SFs were observed in the *p*-type or intrinsic *n*-type InN samples. To estimate the SF density and determine their structure on the atomic level, aberration corrected STEM was employed. In Fig. 2(a), an overview STEM image of the sample with $[\text{Mg}] = 1.8 \times 10^{20} \text{cm}^{-3}$ is shown. The visualization of specimen microstructure is promoted by enhanced diffraction contrast conditions in STEM mode. A clearly visible V-shaped contrast appears at the interface between the 50-nm-thick undoped InN buffer layer and the $\sim 400\text{-nm}$ -thick

Mg doped InN. In the top part of the Mg doped layer, SFs are identified as white horizontal lines. Further magnifications are shown in Fig. 2(b) and atomic resolution is shown in Fig. 2(c). In this figure, primarily, the heavier In atoms are resolved due to the $\sim Z^2$ promoted mass-contrast. A single column is highlighted for reference and present SFs are identified by yellow horizontal lines. As can be seen, the stacking sequence changes several times from the wurtzite AaBb sequence (capital letters denote In atoms, while small letters—N atoms) to AaBbCc— I_1 type SF, which can be viewed as a local sheet of zinc-blende InN. This stacking fault requires the least amount of energy compared to other SF types and is consequently the most commonly observed. Additional SF geometries include a type 2- I_2 fault; however, only a single I_2 type fault is observed in this image. An I_2 type fault constitutes a change of stacking from AaBb to AaBbCcAa and requires about twice the formation energy as for I_1 SF energy and is therefore less likely to form.¹⁷ The slight misalignment of the atom sequence of I_2 in Fig. 2(c) may be related to image drift due to charging during the acquisition. However, additional factors play a role too. For instance, using a high camera length to increase the signal-to-noise ratio leads to additional diffraction contrast resulting in slightly different appearances of different symmetry positions.

Fig. 3(a) is an overview high resolution STEM (HRSTEM) image of the highest doped sample ($[\text{Mg}] = 8 \times 10^{20} \text{cm}^{-3}$). The highlighted rectangle in Fig. 3(a) is magnified to atomic resolution in Fig. 3(b). This micrograph

FIG. 1. Cross-sectional TEM images showing TDs in the undoped InN—(a), and the InN films doped by Mg with concentrations: $1.0 \times 10^{18} \text{cm}^{-3}$ (b), $5.6 \times 10^{18} \text{cm}^{-3}$ (c), $2.9 \times 10^{19} \text{cm}^{-3}$ (d), $1.8 \times 10^{20} \text{cm}^{-3}$ (e), and $8.0 \times 10^{20} \text{cm}^{-3}$ (f). The dark spot in Fig. 1(c) appears due to the redeposition of the material during ion-milling.

FIG. 2. HRSTEM images showing SFs in InN film with Mg concentration of $1.8 \times 10^{20} \text{ cm}^{-3}$. The bright lines in Fig. 2(b) are SFs.

reveals a higher density of SFs of both types compared to the previous sample. Locally, I_1 -type SFs also form bundles. From Fig. 4, acquired from the highest doped sample, it is apparent that the formation of SFs is initiated immediately after the onset of Mg doping. The observed numerous SFs, constituting local zinc-blende sheets, can explain peaks which were previously observed by reciprocal space mapping and attributed to zinc-blende InN inclusions in these samples.⁷

From the present observations, it is apparent that Mg doping of InN at concentrations above $1.8 \times 10^{20} \text{ cm}^{-3}$ leads to material quality deterioration and the nucleation of SFs. However, there can be several mechanisms responsible for SF formation—directly or indirectly linked to Mg atom incorporation in the InN lattice. As one can see from the TEM images in Figs. 1(e) and 1(f) and, at larger magnification, in Figs. 5(a) and 5(b), the Mg doped InN layer is delineated from the undoped InN buffer with V-shaped contrast. Such V-shaped defects were previously confirmed to be a sign of polarity inversion in InN.^{11,12} InN surface with In- and N-oriented polarities has different bonding configuration and adsorption/desorption properties.¹⁴ Consequently, different III/V ratios and growth temperatures are used to grow In- and N-polar InN. Change of polarity induced by Mg doping means that N-polar material starts to grow in

conditions unoptimized for N-polar InN. Therefore, it is reasonable to assume that two major sources of nucleation for SFs can be point defects formed due to non-optimal growth conditions as well as an increasing number of Mg atoms in the InN lattice.

The present results show that the on-set of SF generation coincides with the switching from *p*- to *n*-type conductivity in InN. The SF can be regarded as a very thin zinc-blende layer embedded in the wurtzite matrix, i.e., as a polytypic quantum well. The band offsets at the interface between the wurtzite and zinc-blende InN have been calculated to be type I with 104–130 meV and 57–93 meV for the conduction and the valence bands, respectively.¹⁸ The large valence band offset implies that holes can be confined in the zinc-blende quantum well-like region, which can lead to reduction of free hole concentration. Furthermore, the discontinuity of the spontaneous polarization across the interface shows that the band bending will produce a triangular potential in the barrier which may also confine holes. Similar localization should also occur for electrons. Rigorous first principle calculations have shown that in contrast to electrons, holes are localized not only in the zinc-blende but also in the wurtzite InN.¹⁸ This suggests that the localization of holes due to SFs may play a role for the conductivity reversal. However, other factors, such as point defects and impurity complexes, are

FIG. 3. HRSTEM images showing SFs in the InN film with Mg concentration of $8.0 \times 10^{20} \text{ cm}^{-3}$.

FIG. 4. HRSTEM image showing the interface structure of the InN film with Mg concentration of $8.0 \times 10^{20} \text{ cm}^{-3}$.

FIG. 5. TEM images showing V-shaped contrast which indicates switching from In- to N-polarity.

likely to have impact on the conductivity type in InN doped with high concentrations of Mg.

According to theoretical calculations, substitutional Mg_{In} acceptor atom tends to form complexes with an O_{N} donor atom.¹⁹ Typically, InN films contain oxygen in the 10^{17} – 10^{19} cm^{-3} concentration range.²⁰ Oxygen incorporation can be further enhanced by switching to N-polarity during growth. In general, the crystallographic orientation strongly affects the impurity and dopant incorporation in III-nitrides.²¹ For instance, it is well known that N-polar GaN and InN tend to incorporate higher impurity concentrations compared to Ga- and In-polar material, respectively.^{21,22} MgO , Mg_2O_2 , and Mg_2O_3 are the complexes with the lowest formation energies in InN grown under In-rich conditions (our case).¹⁹ MgO and Mg_2O_2 are neutral complexes, while Mg_2O_3 is a donor with -1 charge state. All these three defects can contribute to the acceptor compensation and the observed switching to n -type conductivity at $[\text{Mg}] \geq 1.8 \times 10^{20} \text{ cm}^{-3}$. Other point defects that can account for n -type conductivity at high Mg concentrations are single nitrogen vacancies or complexes of such. They were theoretically predicted to have the lowest formation energies among all point defects in n -type material grown under In-rich conditions.²³

Experimentally, vacancy-type defects in Mg doped InN were studied by Uedono *et al.*²⁴ by positron annihilation spectroscopy. In their case, the switch to n -type conductivity occurred at $[\text{Mg}] \geq 3 \times 10^{19} \text{ cm}^{-3}$, and it was attributed to In-vacancy and N-vacancy clusters— $\text{V}_{\text{In}}(\text{V}_{\text{N}})_3$.²⁰ Such complex, however, is expected to exhibit a very high formation energy as predicted by first-principles calculations²³ and therefore more difficult to form.

Charge carrier mobility is known to be affected by the concentration of point defects in semiconductors. As can be seen from Table I, electron mobility in the n -type samples with the two highest Mg concentrations experience a substantial decrease compared to the undoped n -type InN—from $1601 \pm 87 \text{ cm}^2/\text{V}\cdot\text{s}$ in the undoped InN to $1079 \pm 40 \text{ cm}^2/\text{V}\cdot\text{s}$ for the sample with $[\text{Mg}] = 1.8 \times 10^{20} \text{ cm}^{-3}$ and $203 \pm 4 \text{ cm}^2/\text{V}\cdot\text{s}$ for the sample with $[\text{Mg}] = 8 \times 10^{20} \text{ cm}^{-3}$. The TD densities in these two highest doped samples are very similar (Table I). SF density, on the other hand, increases with Mg content. As the number of SFs correlates with the

deterioration of the InN FCC properties, we propose that SFs may affect the electron mobility themselves or serve as a signature for the increased concentration of point defects that affect charge carrier properties of the material.

In conclusion, we have presented a (S)TEM investigation of the structural properties in a set of Mg doped InN films in relation to their FCC properties. TD densities are found to be independent of Mg doping. On the other hand, SFs have been identified only in the samples with $[\text{Mg}] \geq 1.8 \times 10^{20} \text{ cm}^{-3}$ and the p -type InN films have been found to be free of SFs. Additionally, the onset of SF formation coincides with the switch in conductivity type from p - to n -type. The SF density also correlates with the drop in FCC mobility in comparison with the undoped n -type InN (see Table I). We have discussed possible mechanisms involving SFs or point defects that may be responsible for the switching to n -type conductivity in InN at high Mg doping. SFs have also been suggested to play a role in the deterioration of electron mobility parameters or serve as a signature for increased concentration of point defects.

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