Direct Imaging of p–n Junction in Core–Shell GaN Wires

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Supporting Information

ABSTRACT: While core–shell wire-based devices offer a promising path toward improved optoelectronic applications, their development is hampered by the present uncertainty about essential semiconductor properties along the three-dimensional (3D) buried p–n junction. Thanks to a cross-sectional approach, scanning electron beam probing techniques were employed here to obtain a nanoscale spatially resolved analysis of GaN core–shell wire p–n junctions grown by catalyst-free metal–organic vapor phase epitaxy on GaN and Si substrates. Both electron beam induced current (EBIC) and secondary electron voltage contrast (VC) were demonstrated to delineate the radial and axial junction existing in the 3D structure. The Mg dopant activation process in p-GaN shell was dynamically controlled by the ebeam exposure conditions and visualized thanks to EBIC and VC imaging. The Mg doping levels were locally determined to be $N_p = 3 \times 10^{18} \text{ cm}^{-3}$ and $N_d = 3.5 \times 10^{18} \text{ cm}^{-3}$ in both the axial and the radial junction. Results from EBIC and VC are in good agreement. This nanoscale approach provides essential guidance to the further development of core–shell wire devices.

KEYWORDS: GaN wire, core–shell p–n junction, doping level, diffusion length, electron beam induced current, voltage contrast

Wire-based devices have been considered as a promising new route toward improved electronic and optoelectronic applications thanks to their desirable intrinsic properties such as small footprints and improved strain relaxation. In addition, wire synthesis provides exciting new degrees of freedom for the fabrication of heterostructures with the advent of facet-selective shell growth. Along with a careful axial and/or radial design of doping levels and (alloy) compositions at the single wire level, an increased versatility is offered to wire devices as compared to planar layer devices. The p–n junctions in wires with either axial or core–shell geometry have been reported to provide the building block of optoelectronic applications. The latter geometry leads to a three-dimensional (3D) core–shell p–n junction thanks to a shell generally deposited on both the sidewalls and the top of the core. Taking advantage of the wire high aspect ratio, this increases the active area, an improvement that is expected to alleviate the efficiency droop issue in light-emitting devices (LEDs) and to increase light absorption in solar cells. Because GaN and its alloys are the materials of choice for optoelectronic applications, III-Nitrides nano- and microwire core–shell devices have been focusing much attention. In the context of light emission, this interest is particularly driven by the use of the nonpolar facets (m-plane) as templates for the growth of multiple-quantum wells yielding sidewall light emission free of polarization effects.

Similar to the planar epitaxial case, measurements of material properties such as doping levels and minority carrier (or exciton) diffusion lengths are crucial to fabricate efficient optoelectronic devices. Minority carrier diffusion lengths in wire semiconductors have been probed using different methods such as cathodoluminescence (CL), time-resolved scanning photocurrent microscopy (SPCM), SPCM combined with a near-field scanning optical microscopy (NSOM), a combined atomic force microscope (AFM)/NSOM system, or ultrafast optical microscopy. However, no diffusion length study has been reported on core–shell GaN wire p–n junctions. Electrical properties of a p-doped shell have been recently measured in a core–shell InP nanowire using Hall-effect experiments performed on a single nanowire. Although the electrical properties of n-GaN wires have been studied, measurement of doping levels and mobility of the p-GaN shell in radial p–n junction has not been yet reported. This is not surprising, as obtaining efficient Mg doping is a known problem in GaN. Surprisingly, as obtaining efficient Mg doping is a known problem in GaN, Mg activation process in GaN core–shell has not been yet reported. For instance, Mg dopant activation process in p-GaN shell was dynamically controlled by the ebeam exposure conditions and visualized through electron microscopy (EBIC) imaging. The Mg doping levels were locally determined to be $N_p = 3 \times 10^{18} \text{ cm}^{-3}$ and $N_d = 3.5 \times 10^{18} \text{ cm}^{-3}$ in both the axial and the radial junction. Results from EBIC and VC are in good agreement. This nanoscale approach provides essential guidance to the further development of core–shell wire devices.
challenge in GaN, with the additional activation annealing step in the case of metal–organic vapor phase epitaxy (MOVPE) growth. Because of their 3D geometry, the full potential of core–shell wire devices relies on a nanoscale spatially resolved understanding of their properties.5,31,32

In this Letter, we report on spatially resolved scanning electron beam probing techniques to measure minority carrier/exciton diffusion lengths and doping levels in both n- and p-regions of a cleaved GaN core–shell microwire grown by MOVPE. First, the cross-sectional approach is presented, and results from low-temperature CL are described. In a second section, EBIC measurements are shown to dynamically monitor Mg dopants activation in p-GaN as well as to spatially delineate the p–n junction in this core–shell device. The ability to measure both the minority carrier/exciton diffusion lengths and the total depletion width is also illustrated. Finally, secondary electron voltage contrast technique is shown to map the electrostatic potential in the vicinity of the junction. By using classical p–n junction theory, both donor and acceptor doping levels close to the p–n junction were evaluated and compared to doping levels deduced from EBIC results.

Core–shell GaN wire p–n junctions were grown using catalyst-free MOVPE35,34 on a N-polar GaN freestanding substrate (sample #1) or a n-type (100) silicon substrate (sample #2). As shown in Figure 1a, a selective area growth through a mask was employed to grow wurtzite n-doped GaN wires with a radius chosen in the range of 500–1200 nm (defined as hexagon side length) and a length chosen in the range of 5–10 μm. As shown schematically in Figure 1a, this n-GaN core was covered by the growth of a p-GaN shell on both sidewalls (c-plane radial junction) and top of the core (c-plane axial junction). Wires were grown along [0001] that is, GaN wires have N polarity. Trimethylgallium (TMG) and NH3 were used as III- and V-precursors, respectively. SiH4 and Cp2Mg gas flows were used as precursors to achieve n-type and p-type doping, respectively. Ex situ annealing to activate Mg dopants was then performed on sample #2 while no specific annealing was performed on sample #1. GaN wires were processed in the same way as to fabricate a regular device. Metal contacts were deposited on both the wires p-shell and the conductive substrate to allow for appropriate current injection through the substrate. Devices include numerous wires in parallel and no effort to contact a single wire is required in this study. Thanks to the improved mechanical strength from the contact process steps, it was found that a straightforward manual cleaving of a sample leads statistically to numerous wires cleaved in half as depicted in Figure 1a, which were appropriate for characterization. This simple cross-sectional approach provided access to the 3D buried junction and was found to be successful for GaN substrates as well as for Si substrates.

The sample could then be studied thanks to a FEI Inspect F50 Schottky Field emission scanning electron microscope (FESEM) equipped with a secondary electron (SE) Everhart-Thornley detector and a homemade CL system fitted with a GATAN cryogenic stage. The wire ensemble device was biased using a Keythley 2611 sourcemeter. A typical I–V curve for sample #2 is reported in Figure 1e. Clear rectification properties were observed and the value of the turn-on voltage ~3 V was typical of a standard GaN diode behavior with appropriate ohmic contacts. Figure 1f shows the energy band diagram of a typical p–n junction with acceptor (Nd) and donor (Nd) doping levels9,18 such that Nd = Nd = 1018 cm−3. From the associated electric field, depletion width on both n-side and p-side can be directly inferred. For EBIC measurement of a device under bias, a lock-in technique was employed to get rid of dark current. A SR-DG535 pulse generator with typical frequency of 2–5 kHz allowed to synchronize a homemade electron beam blander and a SR830 lock-in current amplifier in order to extract the EBIC signal (typically 1 nA) from the
background dark current (6 nA at −1 V, 30 nA at −2 V, and 200 nA at −3 V).

Figure 1b depicts schematically the SE and EBIC contrasts. Secondary electron voltage contrast between the two sides of the junction dramatically increases and becomes the dominant contrast source as the junction is reverse biased. This was used to delineate the p−n junction and map the electrostatic potential in its vicinity. By mapping the electric field, EBIC signal also spatially delineates the p−n junction revealing the core−shell structure.

Results from electron beam probing techniques such as EBIC, VC, and CL not only depend on the energy band structure of the device under study but also on the distribution of the electron beam in the GaN material. It is therefore essential to infer both the energy and spatial distribution of the electron beam in order to simulate the generation of excess carriers and draw reliable conclusions. Shown in Figure 1c is the spatial distribution of excess carriers generated in GaN material with a 4 keV ebeam as obtained by Monte Carlo simulations of electron trajectories using CASINO software v2.48. This distribution was used to simulate the measured EBIC signal (discussed later). We also used this simulation to infer if the device was investigated under weak-injection conditions (WIC), which means that the amount of excess carriers generated by the electron beam in the sample was negligible with respect to the majority carrier concentration. Under high-injection conditions, the diffusion length will result from both minority and majority carriers and the energy band structure could be modified, preventing any meaningful analysis. The procedure to calculate the excess carrier distribution Δn, p has been previously reported in detail. From these calculations (detailed in Supporting Information), it was not obvious that WIC are fulfilled in p-GaN. Therefore, an experimental approach was employed by varying the ebeam current and measuring the EBIC values in p-GaN. EBIC values were found to scale linearly with ebeam current (see Supporting Information). We concluded that WIC were satisfied and that the ebeam measurements under these conditions were suitable to characterize the sample.

Low-temperature CL was used to first structurally characterize the GaN material from sample #2. The CL signal was produced by the radiative recombination of the excess carriers generated during ebeam exposure. CL is a suitable technique to probe nano- and microstructures, thanks to its nanometer resolution, and has been applied recently on a GaN axial p−n junction nanowire. A typical low-temperature (10 K) CL spectrum acquired on an entire cleaved wire with a 5 keV electron beam and current ~20 pA is reported in Figure 1d. Donor-bound exciton at 3.47 eV originates primarily from the n-region while the dominant transition at 3.27 eV is attributed to donor−acceptor pair (DAP) recombination in the p-region. The two satellites peaks are red shifted from each other by 91 meV. This behavior is ascribed to phonon replicas with LO phonon energy in GaN equal to 91 meV.

CL signal at 5 K has been reported to be sensitive to the electric field existing close to a Schottky contact in ZnO nanowires and was used to measure the exciton diffusion length. It is however difficult to readily apply this method on GaN p−n junction. Indeed, at 5 K the p-GaN is insulating because of depth of the Mg acceptor while at room-temperature DAP luminescence weakens dramatically as acceptors become thermally ionized. Rather than CL, cross-sectional EBIC measurements were thus employed on core−shell GaN wire p−n junction devices in order to infer minority carrier/exciton diffusion lengths.

While originally applied in microelectronics, the measurement of EBIC has been very popular for the assessment of semiconductor junction devices as it maps the existence of an electric field in the material, giving information on the junction activity and locally active defects. This field separates electron−hole pairs (or possibly free excitons in GaN) generated by the ebeam leading to a current measured by a current amplifier. Using a cross-sectional approach, EBIC measurement is therefore effective to localize the electric field region and the active defects with a nanometer resolution limited by the ebeam interaction volume.

Figure 2a represents a FESEM picture taken at 10 keV of a connected wire-based device from sample #1 (no ex situ annealing) whose first-rank wires were cleaved to access their buried junctions. The cleaving step essentially led to wires cleaved in half as schematized in Figure 1a.

Figure 2b shows the corresponding EBIC map simultaneously acquired where the yellow color maps the current. EBIC measurement demonstrates the presence of a radial junction even on uncleaved wires while inspection of cleaved wires also reveals the existence of an axial junction at the top of the wire. The knowledge of excess carrier distribution is thus essential in order to understand the charge collection. At this angle of incidence, excess carriers generated in the top metal contact were not reaching the p−n junction. More importantly, it demonstrates the interest of the cross-sectional approach to readily image the p-type shell presence all around the n-type core, as well as the p−n junction activity in core−shell wire-based devices. Such information is not accessible without cleaving.
Figure 3. (a) EBIC profile in log scale of the radial junction in a wire from sample #2 under ebeam energy of 4 keV and current 20 pA. Best fit is achieved using a depletion width $W = 45$ nm, diffusion length on p-side $L_p = 55$ nm and n-side $L_n = 15$ nm. (b) EBIC profile of the same radial junction as a function of applied voltage. Best fitting parameters ($W, L_p, L_n$) for each curve are reported in (c) where it is shown that both diffusion lengths do not depend on applied voltage whereas the depletion width increases with $(V_{bi} - U)^{1/2}$ in agreement with abrupt junction theory. The best fit is obtained for $W(0 \text{ V}) = 45$ nm corresponding to $N_{diff} \approx 1.6 \times 10^{18} \text{ cm}^{-3}$.

![Image](image.png)

Figure 2b shows that the width of EBIC signal equals the p-GaN thickness ($\sim 500$ nm) in sample #1. In the case of an abrupt p–n junction, the expression of depletion width writes

$$W = \left[\frac{2\varepsilon_{\text{GaN}}(V_{bi} - U)}{q} \times \left(\frac{1}{N_{diff}}\right)\right]^{(1/2)}$$

(1)

where the applied voltage is $U$ (V), the effective doping level $N_{diff} = \frac{N_p N_n}{N_p + N_n}$ where $N_p, N_n$ are the acceptor and donor doping levels, $\varepsilon_{\text{GaN}} \approx 9 e_0$ is the GaN permittivity, and $V_{bi} = \frac{(k_b T)/q}{\ln[(N_p \times N_n)/n_i^2]}$ is the built-in potential where $k_b$ is the Boltzmann constant, $T$ is the absolute temperature, and $n_i = 1.9 \times 10^{-10} \text{ cm}^{-3}$ is the intrinsic carrier density in GaN. Using eq 1, p-GaN doping level in the non ex situ annealed Sample #1 was estimated to be lower than $N_p = 2 \times 10^{16} \text{ cm}^{-3}$, taking $N_n = 10^{18} \text{ cm}^{-3}$.

Figure 2c depicts a schematic where the top region defined by the black rectangle was exposed to a larger ebeam dose as compared to the rest of the wire. Keeping the same imaging condition ($E_0 = 5 \text{ keV}, L_0 = 50 \text{ pA}$), increasing the dose to typical value of $10^5 \text{ e}^{-}/\text{cm}^2$ (image area $\sim 4 \text{ mm}^2$) allowed to dynamically image the activation of Mg dopants over a time scale of a few seconds. In Figure 2d, EBIC mapping following the complete exposure of the region defined by the black rectangle reveals that the exposed region exhibited a much more localized EBIC signal.

The EBIC signal maps the electric field existing in the p–n junction depletion region of width $W$. A larger acceptor doping level results in an increase of $N_{diff}$ for a constant donor doping level. From eq 1, it is clear that $W$ decreases as $N_{diff}$ increases. The narrower EBIC signal in the exposed region therefore evidences the ebeam induced Mg activation resulting in a larger acceptor doping level on the p-side.

During MOVPE growth of Mg-doped layer, it has been reported that interstitial hydrogen is incorporated, creating a H–Mg acceptor complex forms which passivate the acceptor.41,42 The ebeam exposure (or so-called low-electron energy beam irradiation) led to the first demonstration of p-type conductivity in MOVPE grown GaN material,43 a key milestone for further GaN bipolar device development.

Figure 2e is a simulation using NextNano++ software44 of the electric field in such a structure where the core doping $N_p = 10^{18} \text{ cm}^{-3}$ is constant while shell doping is either $N_p = 10^{16} \text{ cm}^{-3}$ (not ebeam activated) or $N_p = 10^{18} \text{ cm}^{-3}$ (ebeam activated region). The simulation does not take into account the diffusion of minority carriers/excitons at the proximity of the depletion region, which contributes to the apparent EBIC signal width. The difference between the simulation and the experiment at the border of the exposed region could therefore be attributed to minority carrier diffusion.

In this cross-sectional approach, EBIC measurement was therefore effective to spatially delineate the p–n junction in the three-dimensional structure. It could be used as a way to check for Mg dopants activation in wires grown by MOVPE and to study dynamically the activation process in core–shell wire devices.

Under WIC, one can infer quantitative information from EBIC signal if a sufficient spatial resolution and signal-to-noise ratio are obtained. An EBIC profile from the radial junction in a wire from sample #2 is reported in Figure 1a. The semiconductor properties that control the measured EBIC signal are the depletion width $W$ (related to the doping levels by eq 1), the minority carrier/free exciton diffusion lengths on n-side $L_n$ and p-side $L_p$ as well as the associated surface recombination velocities $v_n$ and $v_p$.

In order to determine these properties, a modified version of Bonard and Ganière model45 was employed where the particular geometry in the radial junction was taken into account. The degree of discrepancy between this geometry and the usual planar-collector configuration is found to be small (discussed in Supporting Information). This model is reliable as it takes into account the spatial distribution of the generated excess carriers and therefore the contributions to the EBIC signal of excess carriers generated in the depletion region as well as in the neutral n- and p-regions. This is of particular importance in devices with depletion width in the range of the ebeam interaction volume (typically for depletion width below 100 nm at 4 keV).

In planar p–n junctions, doping levels from both sides are known quantities and EBIC measurements generally focus on evaluating $L_n, L_p$ and sometimes $v_n$ and $v_p$.46 However, in the case of wire devices the evaluation of depletion width is relevant to estimate the unknown doping levels. Ebeam energy has therefore to be chosen low-enough to allow the accurate determination of $W$. For this reason, 4 keV ebeam was found to provide a fair spatial resolution while providing sufficient signal-to-noise level. Thanks to a previously reported technique47 based on the Monte Carlo simulation shown in Figure 1c, a realistic spatial distribution of excess carrier distribution was implemented in the model by using broadening parameters $\sigma_x = 38 \text{ nm}$ and $\sigma_z = 12 \text{ nm}$ in the model. Literature values for GaN surface recombination velocities are in the range $v = 10^4$
cm$^{-1}$. Being 2 orders of magnitude lower than in GaAs surface recombination was not found to play a role in the fit of EBIC profile at 4 keV and one can therefore neglect this effect in the simulation. A previous study\(^5\) in planar devices also reported another EBIC modeling with negligible surface recombination and ascribed it to the passivation of the surface by the polymerization of the hydrocarbon layer produced during the scanning of the electron beam over the surface of the sample.

Fitting of the profile in Figure 1a gives \( W = 45 \text{ nm} \), \( L_p = 55 \text{ nm} \), and \( L_n = 15 \text{ nm} \). Measured diffusion lengths values are in line with reported values \((L_p = 23 \text{ nm}, L_n \approx 30 \text{ nm})\) in literature for cross-sectional EBIC in planar GaN p–n junctions at low ebeam energy (1 keV)\(^{46}\) but larger values \((L_p = 80–950 \text{ nm}, L_n = 70–250 \text{ nm})\) were reported in studies with larger ebeam energies \((5, 10 \text{ keV})\).\(^{19,50}\)

EBIC signal was also recorded as a function of applied voltage \( U \). During a single image acquisition \((-500 \text{ nm along the junction})\), reverse bias was varied by step of 1 V from 0 to \(-3 \text{ V}\). The four curves are reported in Figure 1b. Subsequent fitting gives \( W, L_p \), and \( L_n \) for each curve. These parameters were plotted in Figure 3c. It is seen that the diffusion lengths do not depend on the applied voltage \( U \) showing that in the neutral region, diffusion dominates over drift in this voltage range. Interestingly, \( W \) as a function of \( U \) exhibits a \((V_{th} - U)^{1/2}\) variation as expected from abrupt p–n junction theory. This is illustrated by the plotted fit obtained for depletion width \( W(0 \text{ V}) = 45 \text{ nm} \) (corresponding to \( N_{eff} \approx 1.6 \times 10^{18} \text{ cm}^{-3} \)). In abrupt junction theory, eq 1 shows that depletion width \( W \equiv \sqrt{[(N_n + N_d)/(N_n N_d)]^{1/2}} \). If \( N_n \gg N_d \) (respectively \( N_d \gg N_n \)), \( (N_n + N_d)/(N_n N_d) \approx 1/N_n (1/N_d)\). Taking \( W = 45 \text{ nm} \), lower value for doping levels on each side considering the contribution of the other side negligible are therefore \( N_{nd} = 1.6 \times 10^{18} \text{ cm}^{-3} \). Although values of depletion width \( W \) were measured from EBIC, it is not possible to assess the exact values for n- and p-side doping levels without assumption. In order to thoroughly evaluate the doping levels, another characterization technique needed to be employed. For this reason, secondary electron voltage contrast measurements were performed.

The SEM is the most widely used tool for visualization at nanoscale. Imaging with secondary electrons highlights primarily topographic contrast. Over several decades, it has been however consistently reported that secondary electron voltage contrast (VC) could arise,\(^{5,51-55}\) particularly in p–n junction devices where the p-side appears systematically brighter than the n-side.\(^{4,54}\) The main mechanism for this effect is believed to be due to a difference in the escape barrier height for secondary electrons.\(^{55}\) In addition, the measured contrast value is influenced by several experimental parameters such as electron dose, ebeam energy, working distance, extractor voltage, and surface treatment as previously reported by several groups.\(^{56}\) Recently, VC profiles along a p–n junction have been demonstrated not only to map the electrostatic potentials across a junction with identified doping levels,\(^{54}\) but even to yield the doping levels in axial GaN nanowire p–n junction where these properties were unknown.\(^4\)

In this study, the configuration is the following: an Everhart–Thornley detector with 258 V grid bias, 5 mm typical working distance, \(\sim 1 \mu \text{m}^2\) image dimension, and 4–5 keV and 20 pA ebeam energy and current, respectively. The resolution is ultimately limited either by the SE imaging or by the Debye length. The Debye length was estimated to be 2 nm for doping level in the \(3 \times 10^{19} \text{ cm}^{-3}\) range,\(^{57}\) so that the resolution should be limited by the SE imaging (and therefore below 10 nm).

First, we were interested to infer whether the SE image was really mapping the electrostatics in the vicinity of the junction. This was verified by applying a bias to the device in order to change the band structure at the p–n junction location. We noticed that multiple high-magnification imaging at the same location resulted in contamination-induced changes in the contrast value between n- and p-region. This prevented the comparison with subsequent images. In order to get rid of this issue and keep the contamination level constant, a single image.

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**Figure 4.** (a) SE image and (b) SE contrast profile of GaN wire top junction as a function of applied voltage. The contrast between the n- and p-region increases with the reverse bias. (c) Linear variation of p–n junction voltage contrast as a function of applied voltage. (d) SE contrast profile and its derivative for an applied voltage \( U = -2 \text{ V} \). Good agreement with abrupt p–n junction theory is obtained for \( N_n = 3.0 \times 10^{18} \text{ cm}^{-3} \) and \( N_d = 3.5 \times 10^{19} \text{ cm}^{-3} \). Total \( W_p \), n-side \( (W_{np}) \), and p-side \( (W_{p}) \) depletion widths measured in the case of (e) an axial junction and (f) a radial junction.
acquisition procedure was used where the applied voltage was changed during the imaging along the junction. It means that the effect of the applied voltage is inferred in a region of length a few hundred nanometers along the studied junction. The SE signal $S$ for an axial junction in a cleaved wire from sample #2 is reported in Figure 4a where the bias has been varied from $-3$ to $2$ V. The contrast $C$ is generally defined as:

$$
C = \frac{S - S_{\text{ref}}}{S_{\text{ref}}} = \frac{S - S_p}{S_p}
$$

(2)

where the reference signal $S_{\text{ref}}$ is chosen in this study to be the signal in the neutral p-side region $S_p$. Figure 4b then shows the calculated contrast $C$ as a function of the distance along the junction for different applied voltages $U$ (shifted for clarity to the same contrast level on the n-side). It can be inferred that the SE yield is always larger (brighter SE signal) on the p-side as reported in the literature. A junction contrast $C_{pn}(V) = (S_{pn}(V) - S_{fi}(V))/S_{fi}(V)$ is defined to quantify this effect. The dependence of $C_{pn}$ on applied voltage $U$ is reported in Figure 4c where it is shown that $C_{pn}$ strongly increases with the reverse bias. $C_{pn}$ tending toward 0 at $U = U_{\text{bi}}$. This supports the observed SE contrast as being due to the applied voltage directly dropping at the p–n junction depletion region (with negligible voltage drop at contacts). Surface states have been reported to play a role in the observed VC for silicon p–n junction devices, in particular by reducing the apparent built-in voltage. In our experiment, the contrast observed for $U = 1$ V and $U = 2$ V are rather similar. It might reflect that contrast from surface effects dominates from $U \approx 2$ V. However, the large increase in contrast by reverse biasing the junction and the agreement between the theoretical and apparent built-in potential evidence that the p–n junction electrostatics dominates the SE VC in the reverse bias regime. Consequently, the SE signal profile is directly related to the spatial distribution of electrostatic potential across the p–n junction.

Similarly to Heath et al. approach, the SE profile was mapped to energy level by linearly fitting $C_{pn}(V) = a \times V + b$ and then defining the calibrated energy as $S_{\text{cal}} = C/a$ where $S_{\text{cal}}$ was in electronvolts. The calibrated SE signal and its spatial derivative are reported in Figure 4d for an applied bias $U = -2$ V. Because the electric field is defined as $E = 1/q \times \frac{dE}{dx}$ where $E$ is the energy and $x$ is the position, the spatial derivative of the calibrated SE signal represents a measurement of the electric field. As illustrated in Figure 1f, the depletion width on p- and n-side could then be directly extracted from the electric field profile. Figure 4e shows the total depletion width $W$, the depletion widths on p-side $W_p$ and n-side $W_n$ as a function of the applied voltage in the same axial junction. $W_p$, $W_p'$ and $W_n$ exhibit a $(N_n - U)^{1/2}$ variation in agreement with abrupt junction theory. Moreover, the ratio $r = W_p'/W_n = N_d/N_n = 1.2$ is constant as expected from the charge neutrality condition. Knowing $r$, eq 1 is finally used to fit $W$ and yields $N_d = 3.0 \times 10^{18}$ cm$^{-3}$ and $N_n = 3.5 \times 10^{18}$ cm$^{-3}$.

The simulated curves using the derived doping levels are compared to the calibrated SE signal $S_{\text{cal}}$ and its derivative in Figure 4d. A good agreement is obtained except at the borders of the depletion region. Effect of patch fields could be responsible for the observed difference.

Figure 4f describes the apparent $W$, $W_p'$ and $W_n$ measured for a radial junction in sample #2. A reverse bias $U < -4$ V was necessary for the SE voltage contrast to fully dominate over other contrast sources. Similar to EBIC, the particular geometry should be taken into account for the case of a radial junction. In Supporting Information, it is shown that the geometry-induced change in the electric field profile in the first 10 nm below the surface was not negligible. We accounted for this effect by using NextNano++ simulations. In this radial junction, the estimated doping levels are $N_n = 2.8 \times 10^{18}$ cm$^{-3}$ and $N_d = 3.4 \times 10^{18}$ cm$^{-3}$. These values are similar to the doping levels determined in the axial junction.

A comparison between the different depletion widths ($W$, $W_p$, $W_n$) in the radial and axial junction obtained from VC is plotted as a function of applied voltage in Figure 5. In this figure, the y-axis is linear with $(W)^2$, so that the obtained linear characteristics demonstrate that the abrupt junction theory is appropriate. Similar donor and acceptor doping levels are inferred in the axial and radial junctions. The depletion width values for the radial (m-plane) junction estimated from VC (blue full circle symbols) are found in good agreement with the values derived from EBIC experiments (gray star symbols) on a similar wire, which demonstrates the consistency of both approaches.

In both the axial and radial junctions, we demonstrated that the p–n junction electrostatics could be satisfactorily described by a simple abrupt p–n junction model with constant acceptor and donor doping levels. For each junction, doping levels were constant over the several hundred nanometers used for this imaging procedure. A spatially resolved evaluation of doping levels is possible from a single SEM picture with the device under a known reverse bias chosen to increase the contrast. The ability to measure locally doping levels is an essential indication in order to improve core–shell devices. Here, no difference in n- and p-doping levels was found, for example, for material deposited on m-plane and c-plane.

A recent report on secondary electron voltage contrast in axial GaN p–n junction nanowire used the same approach except that the authors assumed to be evaluating directly electron and hole concentration instead of donor and acceptor doping levels. These two quantities differ because of the dopant ionization energy. While the donor doping level $N_d$
corresponds essentially to the electron concentration $n$ (negligible ionization energy), there exists a dramatic difference in the case of p-doping due to large acceptor ionization energy of Mg in GaN (100–200 meV). A study on MBE c-axis p-GaN samples illustrates that in our samples, $N_a \approx 3 \times 10^{18}$ cm$^{-3}$ probably corresponds to hole concentration $p \approx 7 \times 10^{16}$ cm$^{-3}$.

Besides, SE VC imaging provides no information on the Mg atom concentration [Mg]. Comparison of the derived acceptor doping levels with reported literature in p-GaN planar layers grown on c-plane by MOVPE indicates that either [Mg] $\approx N_a \approx 3 \times 10^{18}$ cm$^{-3}$ or [Mg] $\approx 8 \times 10^{17}$ cm$^{-3}$. For large [Mg], only a fraction of the dopant atoms are actually incorporated as Mg–H complexes while the remaining part likely incorporates under the form of pure Mg or Mg clusters due to an increased compensation at this dopant atom concentration. As shown in Figure 1d, DAP emission in the p-GaN is centered at 3.27 eV in Figure 1d, DAP emission in the p-GaN is centered at 3.27 eV and depletion width ($W$) were spatially estimated at $N_a \approx 3 \times 10^{18}$ cm$^{-3}$ and $N_a \approx 3.5 \times 10^{18}$ cm$^{-3}$ in both the axial and the radial junctions. Their values corroborate the lower-limit doping level inferred from EBIC measurements, which highlights the consistency of these ebeam probing techniques. The ability to image the 3D p–n junction properties with a nanoscale spatial resolution paves the way to the development of more efficient core–shell wire p–n junction devices.

**ASSOCIATED CONTENT**

**Supporting Information**

Additional information, figures of EBIC values as a function of ebeam current, simulation of EBIC profiles, and electric field mapping and profiles. This material is available free of charge via the Internet at http://pubs.acs.org.

**AUTHOR INFORMATION**

Notes

The authors declare no competing financial interest.

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