Structural Changes in a Single GaN Nanowire under Applied Voltage Bias

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Supporting Information

ABSTRACT: GaN nanowires (NWs) are promising building blocks for future optoelectronic devices and nanoelectronics. They exhibit stronger piezoelectric properties than a bulk GaN. This phenomena may be crucial for applications of NWs and makes their study highly important. We report on an investigation of the structure evolution of a single GaN NW under an applied voltage bias along polar [0001] crystallographic direction until its mechanical break. The structural changes were investigated using coherent X-ray Bragg diffraction. The three-dimensional (3D) intensity distributions of the NWs without metal contacts, with contacts, and under applied voltage bias in opposite polar directions were analyzed. Coherent X-ray Bragg diffraction revealed the presence of significant bending of the NWs already after metal contacts deposition, which was increased at applied voltage bias. Employing analytical simulations based on elasticity theory and a finite element method (FEM) approach, we developed a 3D model of the NW bending under applied voltage. From this model and our experimental data, we determined the piezoelectric constant of the GaN NW to be about 7.7 pm/V in [0001] crystallographic direction. The ultimate tensile strength of the GaN NW was obtained to be about 1.22 GPa. Our work demonstrates the power of in operando X-ray structural studies of single NWs for their effective design and implementation with desired functional properties.

KEYWORDS: GaN nanowires, coherent X-ray Bragg diffraction, piezoelectric effect, finite element method

Semiconductor nanowires (NWs) based on gallium nitride (GaN), indium nitride (InN), and indium gallium nitride (InGaN) have promising applications for light-emitting diodes, low-cost solar cells, transistors, single photon sources, and other devices.^{1–6} The wurtzite (WZ) (hexagonal) crystal structure of GaN NWs is noncentrosymmetric and has an internal electric field along the [0001] crystallographic direction.^{7,8} Local deformation of the GaN unit cell leads to a formation of an internal piezoelectric field and vice versa.⁹ Nitride-based light-emitting diodes (LEDs) grown on *c*-plane GaN substrate usually show the blue shifts with increased injected current. This effect is usually attributed to the screening of the piezoelectric field when the injected electron density is high, which balances the band bending. It was also demonstrated that a single GaN NW exhibits stronger piezoelectricity¹⁰ than a bulk GaN.¹¹ Integration of the NWs into an electric circuit by metallic contacts may induce additional strain and, therefore, may lead to additional piezoelectric effects in the structure. This may dramatically influence electron–hole pair recombination and alter the efficiency of optoelectronic devices based on GaN NWs.¹²

Moreover, better knowledge of the relation between piezoelectric effect and strain field in single GaN NWs with the sizes of hundreds of nanometers could potentially contribute to understanding of inefficient recombination of electron–hole pairs in quantum wells.^{13–15} Therefore, investigation of the influence of applied voltages on the structure of a single GaN NW is of significant importance.

Different methods may be employed to reveal structural changes in NWs under applied voltage such as scanning electron microscopy (SEM) or transmission electron microscopy (TEM).¹⁶ X-ray nanodiffraction, developed recently at synchrotron sources, is an alternative approach that allows one to determine structural properties of single NWs in a nondestructive way.^{17–20} Unfortunately, resolution of this method is limited by the X-ray beam size. Newly developed X-ray coherent scattering methods such as Bragg coherent X-ray diffractive imaging (CXDI) and ptychography^{21–24} allow to

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Figure 1. Scheme of the Bragg coherent scattering measurement geometry. The incoming X-ray beam K_0 is scattered by a GaN NW at the Bragg angle θ_B and resulting diffraction pattern is recorded by the 2D detector in the far-field. In the inset (a) the diffraction map of 111 Au Bragg reflection used for location of the NW is shown. Region A corresponds to the large Au bond pad; region B corresponds to the contacts deposited by lithography and thermal evaporation of Ti and Au. Region C has a gap, where the GaN NW is located. The inset (b) schematically demonstrates two configurations of the NW connection to the power supply.

determine strain and deformation of single nanostructures with the spatial resolution reaching 10 nm.^{25–30} Moreover, these techniques allow nondestructive investigation of nanostructures in situ and in operando.³¹ In this work, we applied coherent X-ray Bragg diffraction to investigate the influence of an applied voltage bias on the structure of a single GaN NW. The samples were provided by the NanoLund Laboratory at Lund University, Sweden. The GaN NWs with a diameter of 350–400 nm and a length 3–4 μm were grown on a 1 μm thick GaN template on top of a Si(111) substrate using metal organic vapor phase epitaxy (MOVPE).³² The structure of NWs was expected to be pure WZ without dislocations and stacking faults. After the growth, GaN NWs were removed from the original GaN/Si substrate and deposited on a Si(111) chip with a 100 nm thermally grown SiO₂ layer on top, as an insulating layer. Further, 220 nm thick metallic contacts were deposited to connect the ends of single NWs to the pads using electron beam lithography and thermal evaporation of Ti and Au. First a 20 nm thick layer of Ti was deposited on SiO₂ and GaN NW to provide good adhesion between Au and GaN, and then 200 nm thick layer of Au on top of Ti layer. The position and orientation of the NWs with respect to the main contacts on the substrate could not be controlled during their deposition. We implemented a sample holder, which provided the electrical connections to the selected single NWs (see Supporting Information). Electric measurements prior to the X-ray experiment revealed the resistance of the NWs with contacts to be about 10¹² Ohm. Because of high resistivity, the current through the NWs was on the order of a picoampere, and additional cooling of the sample was not necessary. The experiment was performed at the coherence beamline P10 at the PETRA III synchrotron facility (DESY, Hamburg, Germany). The geometry of the experiment is presented in Figure 1. The X-ray beam with photon energy of 9.6 keV and flux of about 10¹¹ ph/s was focused at the sample down to one micrometer in size at full width at half-maximum (fwhm) using compound refractive lenses. Characterization of the focus was performed by knife edge scan at the sample position. The measurements were performed at the six circle diffractometer.

It was equipped with a two-dimensional (2D) X-ray pixel detector Lambda (pixel size of 55 × 55 μm²) positioned at a distance of 1.96 m from the sample in Bragg geometry. An evacuated flight tube was mounted between the sample and detector to reduce air scattering. The sample with GaN NWs was mounted on the diffractometer using an adapter with wires connected to a power supply providing bias voltage (see Supporting Information). The experimental setup allowed measurements of 10 $\bar{1}0$ Bragg reflection of GaN with the reciprocal lattice vector $H_{10\bar{1}0}$ being normal to the substrate (see Figure 1).

First, a free lying NW without Au electrodes was investigated using 3D Bragg CXDI as a reference measurement for further comparison with the NW under applied voltage. After the X-ray beam was positioned at the center of the NW, the rocking curve scan was performed in the angular range of ±0.4° in the vicinity of the Bragg angle with 160 angular steps. At each angular position diffraction patterns were recorded with an exposure time of 20 s.

The 3D intensity distribution around 10 $\bar{1}0$ GaN Bragg peak for the free lying nanowire is shown in Figure 2a (see SEM images of this NW in the inset in Figure 2b). The isosurface of intensity distribution around the Bragg reflection demonstrates almost perfect hexagonal symmetry with the fringes originating from the opposite facets of the free lying NW. The maximum resolution achieved in the measurement was estimated to be about 12 nm from the maximum Q-value reached in 3D reciprocal space. The CXDI reconstruction of the reference NW is presented in Figure 2b (see Supporting Information). The reconstructed object has a hexagonal shape with the distance between opposite side facets about 350 nm which is in a good agreement with the SEM data. Additionally, after the X-ray experiment, the same GaN NW was studied by the selected area electron beam diffraction (see Figure 2c). The dark-field transmission electron microscopy (DF-TEM) of 4131 Bragg diffraction of the GaN NW, which is originating only from WZ GaN crystal structure (see Figure 2d), confirmed absence of defects and presence of only wurtzite phase in the GaN NW (see Supporting Information).

Figure 2. Study of the GaN NW without contacts. (a) Isosurface of the 3D intensity distributions around $10\bar{1}0$ Bragg reflection of GaN. The isosurface is plotted at 20% of the maximum value of the normalized logarithmic scale. The $[0001]$ crystallographic direction is shown by an arrow. (b) Isosurface of the amplitude of the reconstructed NW. The corresponding SEM image of the studied NW is shown in the inset. (c) Electron beam diffraction on the same GaN NW performed after the X-ray experiment. Bragg reflections are indexed and direction $[0000]$ corresponds to the direct electron beam. (d) Dark-field transmission electron microscopy (DF-TEM) image of the GaN NW for the $\bar{4}131$ Bragg diffraction. The DF-TEM image demonstrates the absence of defects and confirms the presence of only wurtzite phase in the GaN NW.

Next, GaN NWs with deposited metal contacts were investigated (see SEM image in the inset in Figure 3a). In order to find such NWs, diffraction mapping of gold with the step size of $1\ \mu\text{m}$ was performed in Bragg conditions of $111\ \text{Au}$ reflection ($\theta_B = 15.92^\circ$). Position of the selected NW was identified as a gap in a gold signal (see inset a in Figure 1). After the NW was located, the geometry of the setup was changed to the Bragg conditions of a $10\bar{1}0$ GaN reflection ($\theta_B = 13.54^\circ$). This procedure was repeated for each NW with metal contacts.

The 3D reciprocal space maps in the vicinity of the Bragg angle were recorded by rocking the sample around y -axis and recording far-field diffraction patterns on the 2D detector. The angular range of the rocking scan for the NW with deposited metal contacts was extended to $\pm 0.5^\circ$ around Bragg peak position and 200 equally separated angular steps were measured. An isosurface of the corresponding 3D intensity distribution is presented in Figure 3a and reveals similar hexagonal symmetry as in the case of the free lying NW. However, the intensity of fringes was lower, decaying faster, and slightly disturbed in comparison to the intensity distribution of the reference free-lying NW (compare Figure

3a and Figure 2a). At the same time, we observed new features of the scattered intensity. The intensity distribution from the NW with the deposited contacts consisted of two, not equally intense, 6-fold “stars” with the separation between them along the $[0001]$ direction of the NW. We will call further the more intense “star” of the Bragg peak as peak I and the second one with lower intensity as peak II (see Figure 3). Similar features were recently reported in refs 33 and 34 where nanodiffraction studies of the single NWs were performed and were associated with the bending of NWs. We also assign the peaks separation in reciprocal space to scattering from different parts of the GaN NW, caused by the NW bending (Figure 4a). Because no chemical interaction or diffusion between Au and GaN is expected,³⁵ the observed deformation of the NW may be predominantly attributed to the metal contacts depositing process.

Further, the coherent scattering intensity distribution from several GaN NWs with deposited metal contacts were investigated as a function of the applied voltage bias. First, we applied the positive polarity to the tip of the GaN NW in $[0001]$ direction (see configuration I in the inset b of Figure 1). In this case, the GaN lattice shrinks due to the piezoelectric

Figure 3. Isosurface of the 3D intensity distribution around $10\bar{1}0$ Bragg reflection of GaN NW under applied voltage bias of 0 V (a), 5 V (b), 10 V (c), and 15 V (d). The intensity is presented in logarithmic scale and the isosurface values are taken at 20% of the maximum intensity value. The $[0001]$ crystallographic direction is indicated by an arrow and two separated Bragg peaks, peak I and peak II, are depicted in the plots.

Figure 4. Schematics of X-ray scattering on a bent NW and bending angle θ dependence on the applied voltage bias. (a) Schematics of X-ray scattering on a bent NW. The reciprocal lattice vector $H_{10\bar{1}0}$ is defined by the incident K_0 and diffracted K_H scattering vectors. The scattering vectors Q_I and Q_{II} of the Bragg peaks I and II correspond to the X-ray diffraction from different parts of the bent NW. Vector q defines separation between the scattering vectors Q_I and Q_{II} in reciprocal space. (b) Bending angle θ between the scattering vectors Q_I and Q_{II} as a function of the applied voltage bias.

Figure 5. (a) Equilibrium shape of the NW with the displacement $u_z(x)$ determined from the analytical solution of the elasticity theory. Displacement in this figure is scaled 100 times for visibility. (b) Comparison of the experimental 10 $\bar{1}0$ GaN reciprocal space map corresponding to the voltage bias of 1 V (top) with the simulated one from the FEM model (bottom). The 3D scattered intensity of the GaN Bragg reflection was projected on Q_x - Q_y plane. The scale bars on the reciprocal space maps correspond to 0.2 nm^{-1} . (c) Displacement profile $u_z(x)$ of the NW along its length determined by analytical solution and FEM model for different voltage values. (d) Dependence of the maximum value of the tensile stress in the slice of the NW at $x_{\text{max}} = l/3$ as a function of the applied voltage bias. The ultimate tensile strength of the GaN NW of 1.22 GPa was reached at the maximum voltage of 15 V after which the NW was broken. Stress values in a slice of the NW at the same position simulated by the FEM model for the voltage bias of 1 V is shown in inset. The positive values (red color) of stress are denoted to tension and negative (blue color) to compression of the GaN NW.

effect. Already at bias value of few volts the NW and gold were disconnected due to the NWs contraction (see Supporting Information). Next, we applied a negative polarity to the tip of the GaN NW (see configuration II in the inset b in Figure 1). As a result, the GaN lattice expanded in the [0001] crystallographic direction. In this case, it was possible to study evolution of the 3D Bragg peak intensity of the GaN NW under applied voltage until its breakdown at 15 V. Evolution of the intensity distribution of the 10 $\bar{1}0$ GaN Bragg peak for the applied voltage bias values of 5, 10, and 15 V is shown in Figure 3b–d (see Supporting Information). The damage of the NW was confirmed by the SEM studies at DESY NanoLab³⁶ after the X-ray scattering experiment (see Supporting Information).

Direct reconstruction of the coherent scattering data shown in Figure 3 by applying conventional reconstruction routines turned out to be not feasible due to a large amount of strain accumulated in the NWs with contacts and under applied voltage bias (see Supporting Information).

In order to follow evolution of the intensity distribution of the 10 $\bar{1}0$ GaN Bragg peak, we focus our attention on the scattering vectors $\mathbf{Q}_I$ and $\mathbf{Q}_{II}$ of the corresponding peaks and the relative distance between them defined by the vector $\mathbf{q}$ (see Figure 4a). The modulus of the scattering vectors of the peaks $|\mathbf{Q}_I|$ is related to the GaN unit cell lattice parameter a as $|\mathbf{Q}_I| = 2\pi/d_{10\bar{1}0} = 4\pi/a\sqrt{3}$, where $d_{10\bar{1}0}$ is an interplanar distance between scattering planes corresponding to 10 $\bar{1}0$ GaN Bragg peak. The calculated unit cell lattice parameters a_I and a_{II} of the peaks were found to be almost the same, independent from the voltage bias, and equal to $a_I = a_{II} = 3.1886 \pm 0.0003 \text{ \AA}$ (see Supporting Information). This value was by 1.33% larger than the lattice parameter of the free lying GaN NW $a = 3.1462 \pm 0.0003 \text{ \AA}$ that means that the lattice of the NW with contacts is expanding. The bending angle, defined as $\theta \simeq |\mathbf{q}|/|\mathbf{H}|$, where $\mathbf{H}$ is the reciprocal space vector of undistorted NW (see Figure 4a), was estimated to be about 0.4° without applied voltage for the GaN NW with metal contacts and increased until 1.7° when the voltage was 225

changed to 15 V. Its variation as a function of the applied voltage is presented in Figure 4b.

Bending of the GaN NW under the applied voltage is the major effect observed in our experiment. The NW was modeled with its dimensions determined from the SEM studies, such as, its size between opposite facets equal to $d = 350$ nm and length $l = 3.5$ μm . Because of its diameter-to-length ratio (about 0.1) the NW can be considered as a thin rod. We introduce the displacement field $u_z(x)$ in such a rod which in our coordinated system is perpendicular to the surface of the substrate and is along z -axis. According to the elasticity theory,³⁷ the displacement field in the absence of any shear forces is defined by the differential equation $u_z''''(x) = 0$, where the prime sign denotes derivative over the coordinate. Solution of this equation with corresponding boundary conditions is given by (see Supporting Information for details) $u_z(x) = u_0[(x/l)^3 - 2(x/l)^2 + (x/l)]$, where $u_0 = 27u_{\text{max}}/4$ and u_{max} is the maximum value of the function $u_z(x)$. From this equation, we can directly determine that the maximum value u_{max} is located at the position $x_{\text{max}} = l/3$. Interestingly, this position is in a good agreement with the length of the remaining part of the broken NW determined by the SEM measurements (see Supporting Information).

To relate now analytical solution of the bend NW with the split of the Bragg peak observed in the experiment and caused by this bending, we performed finite element method (FEM) simulations.³⁸ Material elastic properties and lattice constants of the NW were obtained from corresponding GaN bulk material values⁹ (see Supporting Information). The equilibrium shape of the NW used in FEM simulations and given by the analytical solution discussed above is shown in Figure 5a. The incident Gaussian beam profile with one micrometer fwhm and 3D displacement field from the FEM model based on analytical solution were used to simulate the intensity distribution in reciprocal space, which was compared with the experimental one (see Figure 5b). Varying parameter u_{max} in the model, the best correspondence between the angular split of the Bragg peaks in the model simulations and experimental data were determined as it is shown in Figure 5b for the 1 V bias (see Supporting Information for other values of voltage). Finally, the displacement field $u_z(x)$ of the NW was determined for each applied voltage value and is shown in Figure 5c. As we see from this figure, after applying contacts to the nanowire without any voltage already introduced a bend of the NW with the maximum displacement value $u_{\text{max}} = 3.3$ nm. By applying voltage, this maximum displacement was substantially increased up to $u_{\text{max}} = 12.9$ nm for the voltage value 15 V.

We used our analytical solution to the elasticity equation for a thin rod to determine elongation Δl of the NW as a function of the applied voltage bias (see Supporting Information). By that we found that the average value of the piezoelectric constant d_{33} of the GaN NW in [0001] crystallographic direction defined by its elongation Δl was about $d_{33} = 7.7 \pm 2.2$ pm/V (see Supporting Information). The determined value of the piezoelectric constant of the GaN NW is lower than the one measured previously for GaN NWs with sizes 64–191 nm by piezoresponse force microscopy,¹⁰ which was about 12.8 pm/V, but is significantly higher than the bulk GaN value of 2 pm/V¹¹ (see for review ref 9).

Bending of the NW induces tensile and compressive strain fields in the NW.^{37,39} As it was discussed above, we expect that the failure of the GaN NW is more likely to start at the top

facet of the NW at the position $x_{\text{max}} = l/3$ of the maximum deformation value u_{max} . Using the FEM model, stress values σ in the slice of the NW at x_{max} were determined for all voltages. Such slice for the voltage bias of 1 V is shown in the inset in Figure 5d. The maximum tensile stress σ_{max} values on the top facet of the NW were determined for different applied voltage values and vary from 0.31 GPa at 0 V to 1.22 GPa at 15 V (see Figure 5d). Because the mechanical fracture of the GaN NW in our experiment occurred at 15 V, the ultimate tensile strength of the GaN NW was estimated to be about 1.22 GPa.

In summary, our measurements of the single GaN NWs with and without deposited metal contacts demonstrated a visible distortion of the 10 $\bar{1}0$ GaN Bragg peak intensity due to the bend of the NW caused by the contacts. The bending angle increased as a function of the applied voltage leading to evolution of the diffraction pattern. Comparison of the experimentally measured intensity distributions and the FEM simulations allowed us to determine piezoelectric constant in [0001] crystallographic direction with the value of 7.7 pm/V and ultimate tensile stress σ_{max} of about 1.22 GPa that was the origin of a mechanical break of the NW.

Our work demonstrates the power of in operando X-ray structural studies of single NWs for their effective design and implementation with desired properties. For our future experiments, we plan to study the influence of the surface to volume ratio of the NWs on their electro-mechanical properties.

■ ASSOCIATED CONTENT

● Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.nanolett.8b01802.

Description of the sample holder, reconstruction of the free-lying GaN NW, transmission electron microscopy of the free-lying GaN NW, SEM study of the GaN NWs after the applied voltage bias, diffraction pattern of the GaN NW evolution under applied voltage, analytical calculation of a thin rod bending, FEM simulation (PDF)

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Notes

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