

## Nanoscale Imaging of Structural and Optical Properties Using Helium Temperature Scanning Transmission Electron Microscopy Cathodoluminescence of Nitride Based Nanostructures

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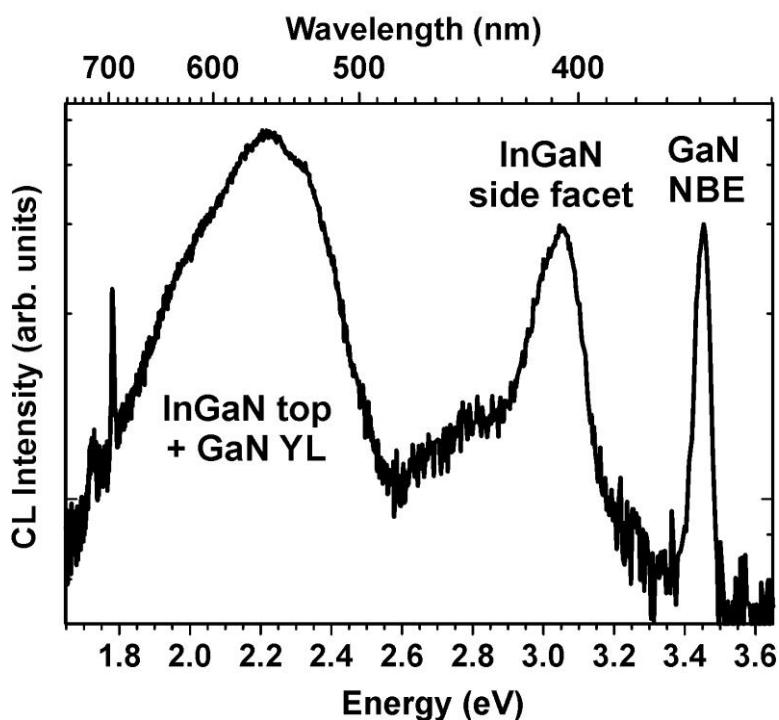
We present the direct nanoscale correlation of structural and optical properties of InGaN/GaN core-shell microrods using highly-spatially resolved cathodoluminescence spectroscopy (CL) in a scanning transmission electron microscope.

For a comprehensive understanding of complex semiconductor heterostructures and the physics of devices based on them, a systematic determination and correlation of the structural, chemical, electronic, and optical properties on a nanometer scale is essential. Luminescence techniques belong to the most sensitive, non-destructive methods of semiconductor research. The combination of luminescence spectroscopy – in particular at liquid He temperatures - with the high spatial resolution of a scanning transmission electron microscopy (STEM) as realized by the technique of low temperature cathodoluminescence microscopy in a STEM (STEM-CL), provides a unique, extremely powerful tool for the optical nano-characterization of quantum structures. Our CL-detection unit is integrated in a FEI STEM Tecnai F20 equipped with a liquid helium stage ( $T = 10\text{ K} / 300\text{ K}$ ) and a light collecting parabolic mirror. Panchromatic as well as spectrally resolved (grating monochromator) CL imaging is used. In CL-imaging mode the CL-signal is collected simultaneously to the STEM signal at each pixel. The TEM acceleration voltage is optimized to minimize sample damage and to prevent luminescence degeneration under electron beam excitation.

The core-shell microrod sample under investigation has been grown by a combination of top-down and bottom-up processes. In a first step, regularly ordered GaN micropillars were obtained by inductively coupled plasma etching of a GaN/sapphire template. Subsequently, the GaN micropillars were overgrown with GaN using plasma assisted molecular beam epitaxy. Finally, a thick InGaN layer was deposited on the whole structure. Scanning electron microscope measurements of the sample reveal a homogeneous array of hexagonally shaped microrods with a density of  $1.2 \times 10^7\text{ cm}^{-2}$ . The mean diameter of the microrods could be estimated to  $1.3\text{ }\mu\text{m}$ .

The cross-sectional STEM image in annular dark field contrast (ADF) of the microrods clearly shows an InGaN capped region of about 750 nm thickness with a low crystal quality directly on top of the GaN microrod. In addition, we observe a thin InGaN shell layer on the side facets of the microrod with a thickness gradient from 70 nm to 140 nm towards the microrod topside. Structural investigations reveal the presence of threading dislocations propagating from the GaN template through the microrods towards the apex.

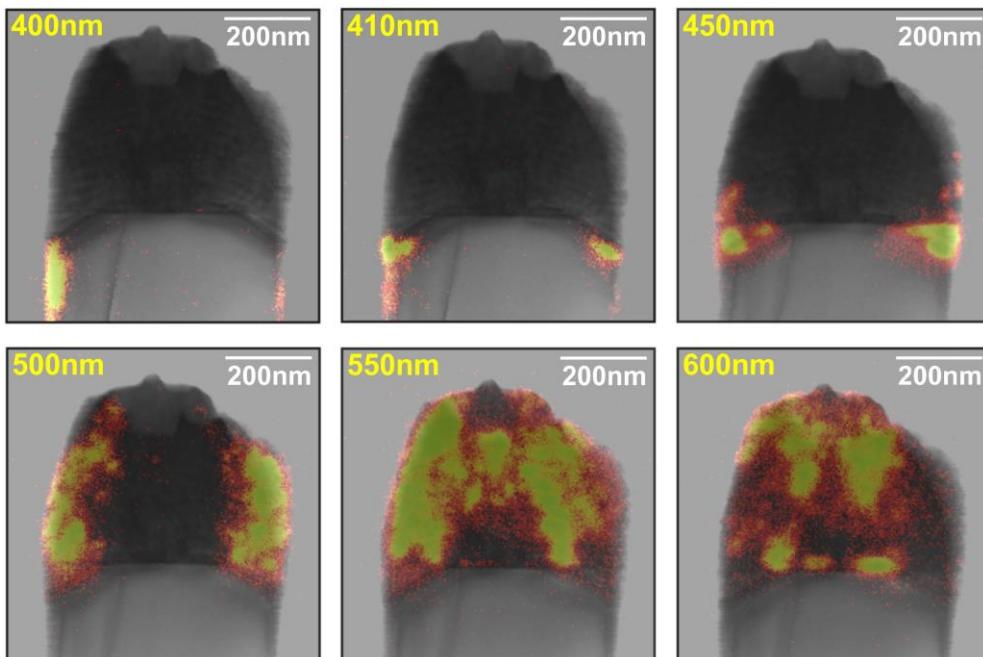
Direct comparison of the ADF image of a single microrod with the panchromatic CL intensity image at 16 K reveals the highest CL intensity coming from the InGaN on top as well as on the side walls of the



**Figure 1.** Spatially integrated CL spectrum at  $T = 16$  K exhibits a near band edge emission of GaN, as well as two broad InGaN luminescence contributions. The side walls emit at 3.05 eV whereas the InGaN top shows emission at 2.21 eV.

microrod. The integral CL spectrum from a single InGaN/GaN microrod exhibits three distinct luminescence contributions: the near band edge emission (NBE) of GaN and two InGaN peaks. The distribution of the peak wavelength exhibits a dominating NBE emission at 359 nm (3.45 eV) as well as the yellow defect band at 560 nm (2.21 eV) out of the center of the microrod. On the side facets one can observe the high energetic InGaN CL at 407 nm (3.05 eV).

In contrast, the cap layer emits a broad luminescence band between 510 – 640 nm (2.43 – 1.94 eV). This difference of the InGaN emission on the side facet compared to the cap can be attributed to different InGaN compositions and/or strain conditions. Furthermore, a red shift of the InGaN luminescence in both InGaN regions is observed indicating a gradual increase of the In concentration during the growth due to the lattice pulling effect.



**Figure 2.** Greyscale high-angle annular dark field images superimposed by monochromatic CL Intensity ( $T = 16$  K) maps in orange contrast: different Indium compositions are directly seen with changing wavelength.