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Quantitative measurement of Indium atom distribution inside InGaN QWs with atomic sensitivity

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In light emitting diodes (LED) consiting of indium gallium nitride (InGaN) quantum wells (QWs) in a GaN matrix, the atomic In distribution inside the wells defines the performance. The analyzed samples (LED structures) were nominally grown with 17% In composition. A number of reports [1,2] have shown that in InGaN wells with a nominal 10-20% In composition a phase separation takes place up to forming small clusters (70-80% InN) on nm scale [1,2].
One issue affecting the success of our quantitative measurements is the sample preparation. We establish the Ga^{2+} Focused Ion beam (FIB) method with a subsequently wet etching procedure [3]. To prepare the $[11\bar{2}0]$ InGaN/GaN cross-section samples we cut a trench with a 2-step FIB process: at 30keV and a 'cleaning' procedure at 10keV. The wet chemical etching with 25% KOH solution leads to a fast removing of the by FIB damaged surface layer. The results are atomically flat surfaces with almost no amorphous layer (see FIG.1), which enables the separation of the Ga and the N atomic columns (see line scan FIG. 1b) .
Along with strain analysis on single high-resolution (HR) TEM lattice images [1,2] (see FIG. 2), we determine the In distribution on exit-plane wave (EW) images (amplitude and phase) by measuring the number of In atoms in each atomic column inside the quantum well. To reconstruct the EW function we took a focal-series of HRTEM lattice images on our CM300 FEG/UT (OAM) using its unique signal-to-noise ratio for recovered phase magnitudes [4] and implement the focal-series reconstruction with the FEI program package "TrueImage" based on the PAM/MAL algorithm [5]. We benefit from a resolution extension to the information limit into the sub-Ångstrøm region and a high EW phase sensitivity tested on a gold sample [4].
The EW phase oscillations depending on the atomic number (extinction distance, see FIG. 3) make it possible to extract the chemical information between Ga and In in an atomic column at a well defined thickness (about 50Å, see FIG. 3). We use multi-slice calculations on model structures plus simulated EW images to test and discuss the sensitivity, limits and error bars.
Using the comparison with simulations we determine in the EW images the amount of In segregation (deviation from Poisson distribution) and the In atom content in an atomic column with about 1 In atom sensitivity.

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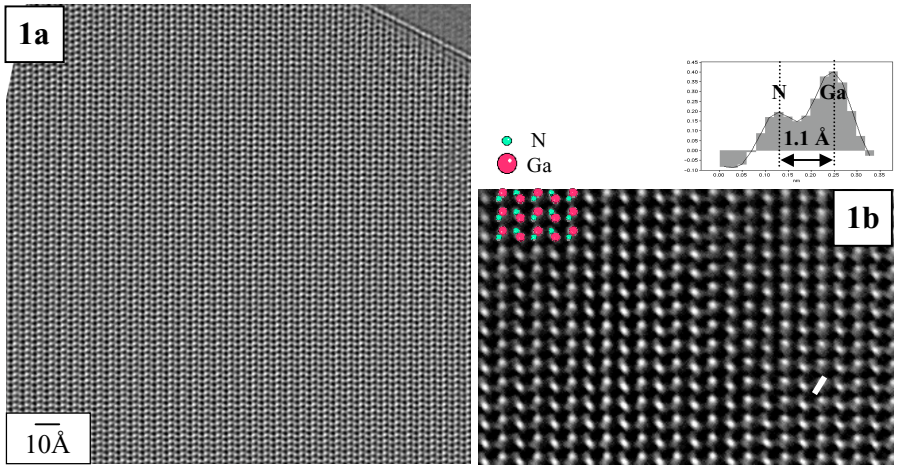

FIG.1 Reconstructed EW phase images in [11$\underline{2}$0] zone axis orientation ((a) with lower magnification than (b)) of the GaN matrix after FIB specimen preparation with subsequential KOH etching procedure. The in (b) inserted structure model and the line scan show the separation of the Ga and the N atomic column.

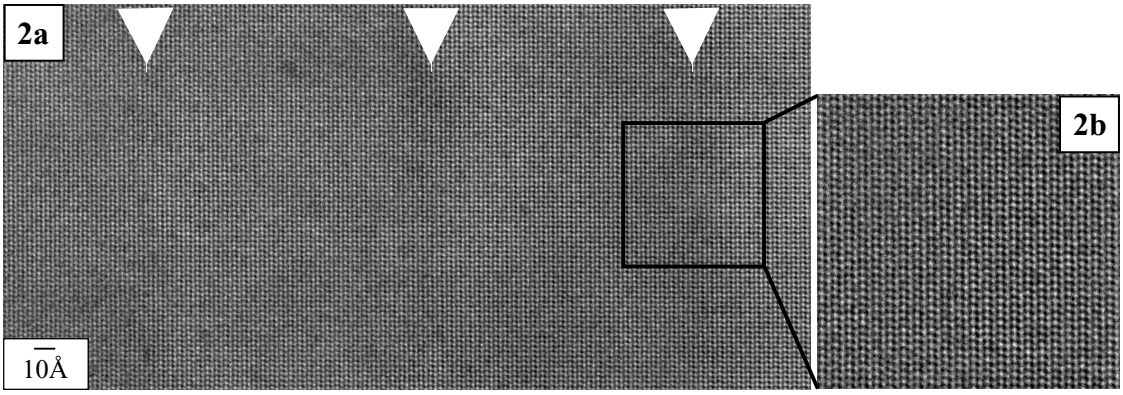

FIG.2 HRTEM lattice images in [11$\underline{2}$0] zone axis orientation of the active LED region consisting of multi InGaN QWs (marked by arrows) inside the GaN matrix ((b) one QW with higher magnification) showing the perfect pseudomorphical structure (no dislocations, no defects). The stress due to the larger size of the In atoms is compensated by strain in c-axis direction of the hexagonal 2H structure.

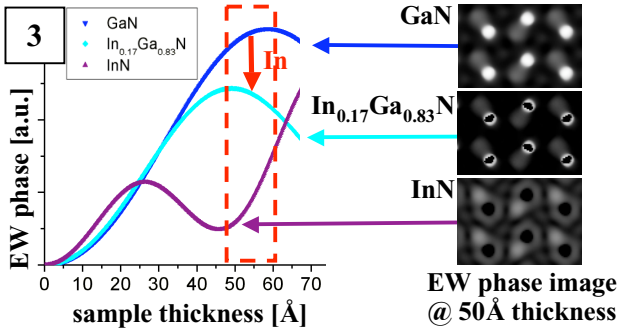

FIG.3 EW phase oscillations vs. thickness of GaN, $In_{0.17}Ga_{0.83}N$, and InN. In the (red) marked thickness region the phase value allows to determine the In content.

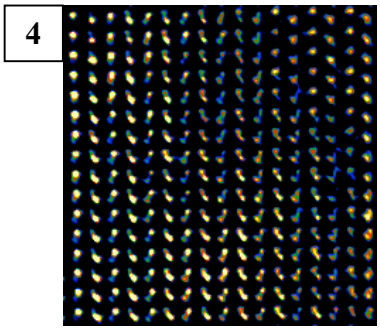

FIG.4 EW phase image in color code of an InGaN QW. Higher phase values (yellow) show the GaN region. More In content leads to lower phase values (darker).