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High Spatial Resolution Energy Dispersive X-ray Spectroscopy and Atom Probe Tomography study of Indium segregation in N-polar InGaN Quantum Wells

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N-polar grown III-nitrides are very interesting materials for the fabrication of heterostructures devices such as transistors, photodetectors, solar cells or optoelectronic devices. In GaN/(In,Ga)N/GaN heterostructures with thin (In,Ga)N layers, polarization engineering allows to achieve interband tunneling. Thereby the tunneling probability is proportional to the indium concentration in the InGaN layers. Indium incorporation is higher for N-polar InGaN films than for the typically grown Ga-polar ones. N-polar III nitride films are often grown on misoriented substrates enabling the growth of smooth, high quality layers [1]. The crystal misorientation leads to the formation of surface steps, and misorientation angles of 4°-5° can result in up to 3-4 unit cell high steps. The growth of N-polar InGaN films is further complicated by the necessary reduced growth temperatures and the required absence of hydrogen in the growth ambient. In quantum well structures, however, hydrogen, which acts as surfactant and promotes the growth of smooth layers, can be introduced during GaN barrier growth, allowing the deposition of thick multiple quantum well (MQW) stacks. Ga-polar InGaN films have been extensively studied and are known for their local fluctuations in the indium composition [2]. Not much is known about the uniformity of N-polar InGaN layers.

This study deals with a structural and compositional characterization of 3 period InGaN/GaN MQWs. The sample in this study was grown in an atmospheric pressure two-flow metal organic chemical vapor deposition (MOCVD) reactor. In order to achieve smooth surface morphologies in the N-polar orientation, the sapphire substrates used were 4 degrees miscut in the *a*-direction, resulting in a (In,Ga)N layer of 4 degrees miscut in the *m*-direction. Following the growth of a 1-2 µm thick N-polar GaN base layer, a triple quantum well stack was grown at a well growth temperature of 725 °C using triethyl gallium (TEGa), trimethyl indium (TMIn), and NH_3 as precursors. The first 4 nm of the GaN barrier layer were grown in N_2 under the same conditions as the InGaN QW, while the following 6 nm of the barrier were deposited in H_2 at 755 °C.

TEM cross-sectional samples and APT tips were prepared by dual-beam FIB/SEM by the standard lift-out method. The lamella was prepared in order to visualize the InGaN steps along the <11-20> direction. Atomic STEM-HAADF images were obtained in a JEOL ARM200F microscope operated at 200 kV. Energy dispersive x-ray spectroscopy was performed with an Aztec Energy Advanced Microanalysis System and by using an analytical silicon drift detector to collect the X-rays. APT analysis was performed with a Cameca 3000X HR Local Electrode Atom Probe (LEAP) operated in laser-pulse mode [3]. Both spot and line scan EDX experiments were performed, in order to evaluate the composition gradient across the vertical structure (barriers/wells) and to study local composition variation along the well. An acquisition time below 1.5 secs/pixel was used in the acquisition of line scans, in order to minimize sample damage and contamination.

HAADF and BF STEM images allowed to visualize the structure of the MQW at the atomic scale (Figure 1(a)). The steps in the wells are evident. The contrast of HAADF images depends on the atomic species, a heavier element producing a lighter contrast. A contrast modulation is evident in the wells, with lighter areas located at the step edges, compatible with In segregation in these areas. Vertical line scans allowed to detect a low concentration of In also in the first part of the barrier layer on top of each well as evidenced in figure 1(b).

The reconstruction of the APT volume after evaporation of the three InGaN QWs is optimized thanks to the TEM images as shown in figure 2(a). The average In composition in the wells directly obtained after the indexation of the APT mass spectrum is 29.6 at.% ± 1.0%. As already evidenced by EDX, the presence of In in the bottom part of the GaN barrier layers is observed in the reconstruction. The distribution of indium within the QWs is obtained by 2D concentration maps as shown in figure 2(b) These maps highlight the segregation of indium in rich regions situated along the growth steps. The lateral spacing between steps is about 8.5 nm closely corresponding to the prediction for a sample grown with a 4° miscut.

EDX and APT were successfully utilized to evaluate indium composition variations in N-polar InGaN quantum wells. Both techniques were able to evidence the presence of In in the bottom section of the GaN barrier layers, but also the lateral segregation of In at surface step edges of the growing crystal [4].

References:

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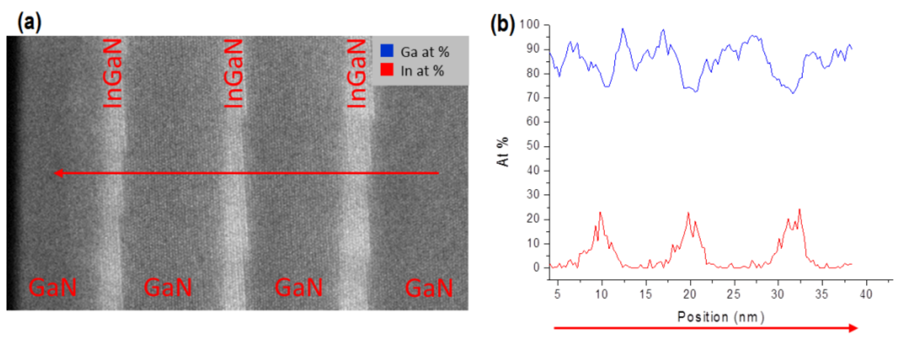
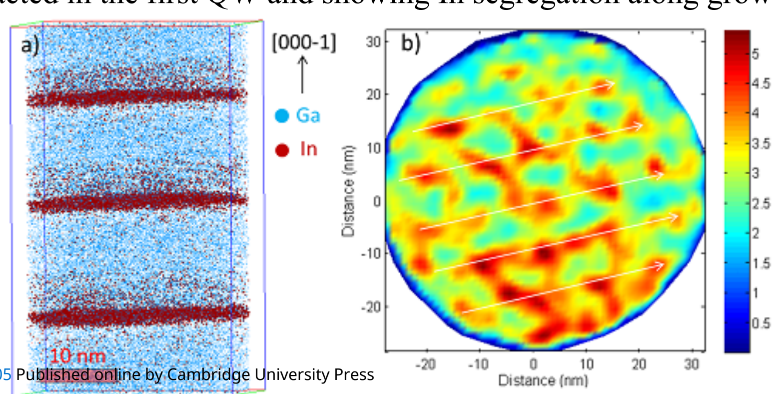


Figure 1. (a) HAADF image showing the vertical structure of the sample. (b) In (red) and Ga (blue) concentration profiles going from the bottom well to the top one.

Figure 2. (a) Atomic 3D reconstruction of the InGaN QWs obtained from APT. (b) 2D projection of the In fraction extracted in the first QW and showing In segregation along growth steps.



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