doi:10.1017/S143192761700808X
Microsc. Microanal. 23 (Suppl 1), 2017

Phase Identification of III-N Thin Films Grown by Molecular Beam Epitaxy and Migration Enhanced Epitaxy using Precession Electron Diffraction

Eduardo Ortega[1], Y. L. Casallas-Moreno[2], M. López-López[3] and Arturo Ponce[1]

[1.] Department of Physics and Astronomy, University of Texas at San Antonio, San Antonio, Texas 78249, United States
[2.] Escuela Superior de Física y Matemáticas, Instituto Politécnico Nacional (IPN), Ciudad de Mexico 07738, Mexico
[3.] Physics Department, Centro de Investigación y de Estudios Avanzados del IPN, Ciudad de Mexico 07360, Mexico.

Group III nitrides are semiconductor materials with a wide application in electronic and optoelectronic devices. The study of these materials has been carried out using mainly the stable phase, which has a hexagonal (wurtzite) structure. However, this crystal phase presents large spontaneous and piezoelectric polarization fields that limit the free-carrier recombination efficiency. Recently, in order to avoid the intrinsic polarization fields in electronic devices, intensive efforts have been made to grow the group III nitrides in the metastable phase, which has a cubic (zinc blende) structure. One of the most important compound for III'N thin films is Gallium Nitride (GaN), this material together with its alloys are suitable for developing photovoltaic and optolectronic applicatins with a high efficiency. This is mainly due their direct energy gap, that covers a large part of the solar spectrum, from infrared with InN to the ultraviolet with GaN and AlN. Another important compound for III-N thin films is Indium Nitride (InN) which has the highest electronic mobility, the best doping properties, the lowest phonon scattering, and smallest direct band gap among the III-N family [1,2]. However, nowadays a higher crystalline quality and smaller hexagonal inclusion in c-InN samples are still challenging due to a low dissociation temperature of the compound and the metastability of the cubic phase due lattice mismatch.

Some growth techniques have been used to improve the crystalline quality and phase purity of c-InN, such as Molecular Beam Epitaxy (MBE). Under this technique, different methods have been implemented to overcome lattice mismatch problems with different substrates. In the case of GaN, a low temperature c-GaN nucleating layer is deposited prior to the high temperature epitaxial one. In the case of the InN, the Migration Enhanced Epitaxy method (MEE) applied to MBE is used to achieve a high crystalline quality of c-InN. The MEE method consists of an alternating sequence of group III atoms (In) followed by group V atoms (N). The alternating periods allow In atoms to increase their surface migration length at low growth temperatures.

The crystalline structure of the thin films was studied using a Scanning/Transmission Electron Microscopy (HR-S/TEM) JEOL ARM 200F microscope operating at 200 kV equipped with an ASTAR system capable of perform Precession Electron Diffraction (PED), for phase mapping of the thin film. Using PED increases greatly the quality of the orientation/phase maps as their patterns contain more information in the diffraction spots than in normal SAED/NBD patterns. With precession the intensities of the diffraction spots are proportional to the square modulus of the structure factor of the material. And when is compared to the theoretical simulations this will result in high index correlation values. The precession angle can be increased to obtain a more kinematical diffraction pattern, but the beam will suffer from beam broadening and the resolution of the map will degrade, for that reason a compliance between the two parameters is required [3]. On this work, the diffraction patterns were recorded every 1 nm in an automated way by scanning the precessed beam 0.4° at 100 Hz along the sample over an area of 200 nm x 200nm with nanobeam diffraction (NBD) conditions and a spot size of 1.1 nm. Then, the set of diffraction patterns was indexed with the ASTAR software considering the possible structures present, until obtaining the phase map. Sample preparation for TEM analysis was realized using a Focus Ion Beam, Zeiss Crossbeam 340, working with 30, 5 and 2kV gallium ions.

In Figure 1, the phase map is combined with the index map to show the degree of confidence. The map illustrates with colors the phases present in the GaN film. The dark regions correspond to lower reliability values, which are due to overlapping of both phases (cubic and hexagonal) because of high density of stacking faults or because of overlapped orientations contributing to the diffraction pattern making it hard to index. The c-GaN samples shows, overall, a cubic phase structure (blue color) with some inclusions of the hexagonal phase (green color). At the first stage of growth, the film presents only the cubic phase, this means that the hexagonal inclusions are not formed in the nucleating layer. Therefore, the nucleating process of cubic GaN on GaAs(001) substrates is optimal. For the InN sample, the hexagonal inclusions (yellow color) can be seen in Figure 2. As in the previous map, the red color illustrates the GaAs substrate. We observe the high purity of cubic phase represented with the purple color in the map. The nucleation of the hexagonal phase inclusions in the cubic InN samples are due to misorientations or stacking faults created during growth and are found in a columnar shape. [4].

References:
[1] T. Nakamura, et al, Phys. Stat. Sol. (b) 243 (7) (2006) 1451–1455.
[2] Y. L. Casallas-Moreno, et al, J. Appl. Phys. 2013, 113, 1.
[3] F. Ruiz-Zepeda, et al, Microsc. Res. Tech. 2014, 77, 980.
[4] The work was supported by SENER-CONACYT Project number 151076 and by the National Institute on Minority Health and Health Disparities of the NIH grant G12MD007591.

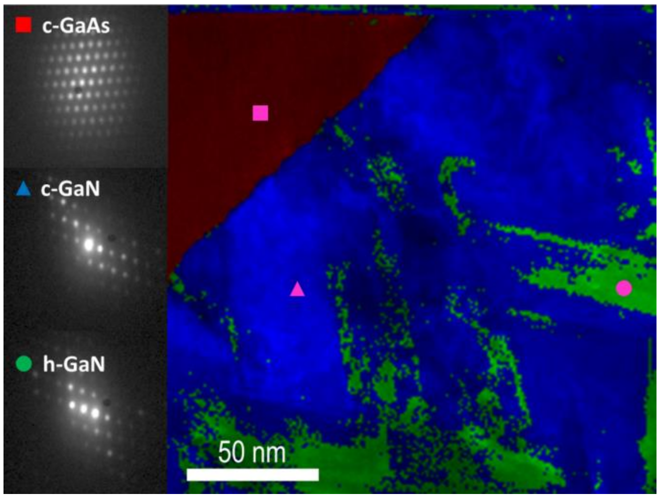

Figure 1. Phase map (combined with the index map) obtained with PED. The colors correspond to the different phases of GaN in the film and the GaAs substrate. As recorded PEDs from the corresponding points marked in the phase map are displayed to the left.

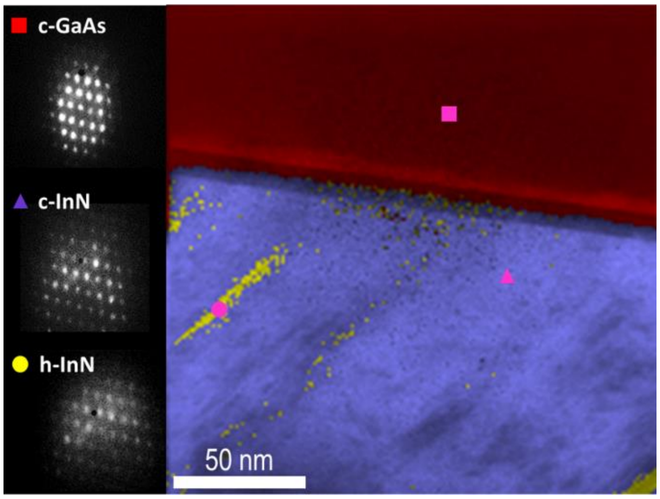

Figure 2. InN/GaAs Phase map (combined with the index map) obtained with PED. Left DPs' correspond to the selected points on the map.

https://doi.org/10.1017/S143192761700808X