4.1.3 X-Ray Diffraction of GaN Layers and Nanostructures

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Introduction

The band gap of GaN (WG = 3.4 eV) can be widely modified using alloy nitride semiconductors such as AlxGa1-xN and InxGa1-xN. Therefore, the GaN material system is a very important material for light emitting devices and photo detectors in the whole visible wavelength range and beyond [1]. Unfortunately, AlxGa1-xN and especially InxGa1-xN exhibit a strong dependence of the lattice constant on the composition and it is very difficult to epitaxially grow nitride based heterostructures offering the full theoretically band gap range of 0.7 eV < WG < 6.1 eV.

GaN based nanowires are widely accepted as a promising candidate to relax the lattice mismatch constraints of nitride based alloy semiconductors. Up to now nitride based nanowires are mainly grown by molecular beam epitaxy. In May 2010 a new Metal Organic Vapor Phase Epitaxy apparatus (MOVPE) for nitride nanowire growth was installed in our facility in order to study MOVPE for nanowire based light emitting devices. The crystal lattices structure was examined by high resolution X-ray diffraction (HRXRD). The aim of this work is to evaluate first grown GaN samples and to study the applicability of X-ray diffraction for GaN nanowires.

Experimental Setup

All samples were grown by MOVPE. Most layers and structures were produced in Duisburg, while the nanowires were provided by Axitron (Aachen). The nanowires were grown on silicon and sapphire (Al₂O₃), while the layers had sapphire as substrate. X-ray diffractometry was performed on a computer-controlled Stoe STADI P double-crystal diffractometer [2]. Two GaAs wafers in a parallel (400) setting act as monochromator and as a collimator to extract the CuKα₁ radiation (λ = 0.1540598 nm) and a scintillation counter was used as detector. The ω-scan changes the position of the sample and gives accurate information on the crystal quality while the coupled 2θ-ω scan is changing the angles of both detector and sample providing information about multiple layers, different materials and tensions [3].

Results

Various GaN, AlₓGa₁₋ₓN-, and InₓGa₁₋ₓN-layers and heterostructures were grown during system start-up. The tension within a single GaN-layer can be identified by examining the Bragg peak position of GaN in the rocking curve. This way also the lattice parameter c can be determined. The GaN Bragg peak was found at the theoretical position of about 34.57 ° proving that all samples are nearly relaxed beyond a thickness of 1 µm. The full width half maximum (FWHM) of the peak is around 270 ° (in a ω-scan). A FWHM between 250 ° and 350 ° indicates a good GaN-crystal with little defects.
The samples with an Al$_x$Ga$_{1-x}$N-layer were grown on top of a GaN-buffer layer. Figure 1a) shows three different rocking curves. The Bragg peak gives direct information on the incorporated Al-concentration. With increasing Al-concentration the AlGaN-peak appears at higher angels. It changes position between the two extremes GaN (34.57 °) and AlN (36.04 °). The samples show an almost linear relationship between offered TMAI and embedded Al in the layer.

Fig. 1. Rocking curve of GaN heterostructures

a) GaN/AlGaN samples with different Al-concentration
b) InGaN-MQW rocking curve and simulation: In-content 27 %

InGaN thickness 2.3 nm; GaN thickness 16.6 nm

Samples with In$_x$Ga$_{1-x}$N/GaN multiple quantum wells (MQW) for LED application were grown. The growth starts with a GaN-buffer layer followed by five periods of In$_x$Ga$_{1-x}$N/GaN-layers to form a MQW. These five periods create a superlattice. Figure 1b) shows a corresponding rocking curve. The highest peak corresponds to the GaN buffer layer. The smaller peaks are satellite peaks and belong to the superlattice. The distances between the peaks depend on the period length. If the distance between all satellite peaks is equal, the superlattice is equally spaced. All samples have an equally spaced superlattice. The simulation-software is supposed to determine In-concentration and the thickness of each layer. While it is easy to determine the period length, ascertaining the thickness of each individual layer is difficult. The position of each satellite peak depends on In-concentration and the thickness of each layer. The envelope provides information of the concentration and thickness, but it is difficult for the simulation software to take the envelope in account. The same challenge appears with LEDs, since they also use a InGaN-MQWs.

Second task of this thesis was to examine GaN nanowires. This was done in two different steps. First different AuGa alloys were analyzed, which support the growth of GaN nanowires via VLS-growth. The Au nanoparticles were placed on the substrate. Afterwards TMGa was exposed to the gold. Depending on the exposure time, Gallium supply and temperature different alloys are formed, which influence the growth of the nanowires. With HRXRD it is possible to detect those alloys and determine the lattice parameters. Temperatures higher 900 °C produced atom-atom distances from
0.222-0.223 nm and could form the alloys Au$_7$Ga$_2$ or Au$_{0.79}$Ga$_{0.21}$. At 744 °C distances of 4.31 and 3.51 nm were found and could be Au$_7$Ga$_2$, AuGa$_2$ or Au$_2$Ga.

In the second step fully grown nanowires were studied. Several challenges exist in analyzing GaN nanowires via X-ray diffraction. First the density of nanowires on the provided samples is often low. Secondly between the wires grew crystals (visible in Figure 2a) with the same lattice parameter like the wires and thirdly the rocking curves look almost equal. Figure 2b) shows two rocking curves, one of a sample with nanowires and one without. There are only two differences between both curves. One shows an additional gold-peak. This gold is used to catalyze the growth of nanowires. The second difference is that the GaN-peak of the Nano-wires has a higher FWHM-value. The ω-scan had for different nanowires samples FWHM-values of 900-950 ° and 216-350 ° for the 2θ-ω scan. It was only possible to examine GaN nanowires grown on sapphire, because those grown on Si did not show any GaN in their rocking curves. While other measurement methods prove that the wires are made from GaN. The crystal structure is not equally enough to be studied with HRXRD. Also GaN nanowires with an additional InGaN coat showed no difference in their rocking curve. So at the moment it is impossible to state any parameters based on the rocking curves.

**Fig. 2.**  

a) Microcraft of GaN nanowires on sapphire side view;  
b) rocking curves of GaN nanowires (top) and a GaN-crystal (bottom)

**Conclusion**

It is possible to examine the tension and crystal quality in a GaN-layer. The Al-concentration in Al$_x$Ga$_{1-x}$N-layer can be determined. It is difficult to give information about thickness and In-concentration in In$_x$Ga$_{1-x}$N-layers. GaN nanowires have at the moment too many unwanted crystals between the wires and too little density of nanowires to give useful information. GaN nanowires still need more research.
Acknowledgement

This work is part of the NaSoL Project. Special thanks to AIXTRON for providing the GaN nanowire samples.

References

