In-situ Control of MOCVD Nitrides Deposition via Spectroscopic Ellipsometry

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Within the frame of an EU project we had the possibility to install a MOCVD system for the growth of GaN and its ternary alloys containing In and Al. As dopand sources we installed Si for n-type and Mg for p-type. The main purpose of the project was the installation of an *in situ* monitoring of the layer thickness and composition during growth. This could be managed by spectroscopic ellipsometry.

1. Introduction

In February and March of this year we had a very busy time with the transport, setup and installation of the MOCVD machine. The machine was shipped to our lab, where we had to overcome the limits of our building, which allowed only a transport through the windows, due to the size of the components.

At the end of April of this year we had the start-up procedure of the MOCVD equipment, where we could grow the first layers of GaN and GaInN together with p- and n-doping. Although we had a delay of 4 months at the time of setup of the MOCVD equipment in comparison to the work plan of the project, we managed to catch up the time schedule within one month.

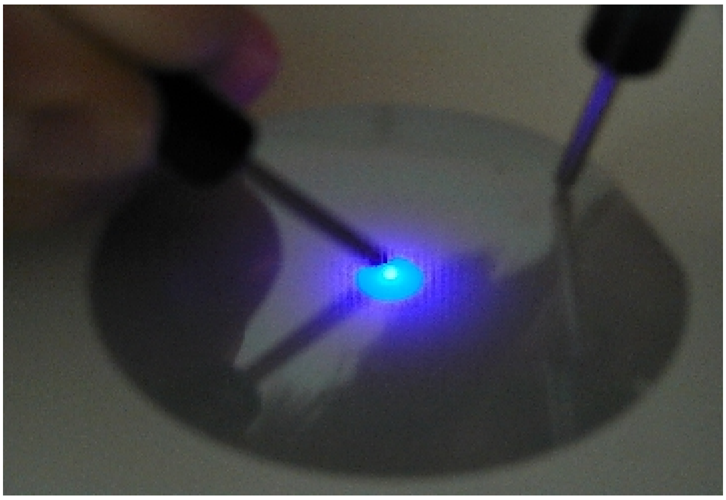

Fig. 1: Blue light emission from the first LED structure grown in our MOCVD reactor after thirteen growth runs.

2. Startup of the MOCVD System

To get some experience with ternary compounds the startup engineer from Aixtron introduced us to the growth of GaInN, since the Al source was not available at this time. So all components for the growth of an LED with a small GaInN quantum well were tested. Finally in the 13th growth run a simple LED structure was grown. The obtained result can be seen in Fig. 1.

The mounting of the optical components for ellipsometry measurements needed the construction of a special holder structure. The fixing turned out to be quite tricky because of the limited space in the reactor cabinet. Even more sophisticated was the exact alignment of the ellipsometer, which needed the help of a specialist from the ellipsometer supplier to optimize the output of the optical signal.

In all our experiments, whole 2 inch (0001)-oriented Al_2O_3 substrates are employed, and the growth stages are constantly monitored with the final goal of developing a closed loop control of the nucleation process. Special effort has been devoted to the monitoring of the initial stages of GaN growth (e.g. a low temperature GaN nucleation layer deposited onto the substrate in order to promote the transition between substrate and GaN device layers) and to the interface formation (nucleation layer/GaN, GaN/ternaries). The growth parameters have been optimised and at present we can fabricate state-of-the-art pure hexagonal GaN (FWHM of XRD rocking curve of 250”), p- and n-type doped GaN and ternary compounds (AlGaN, InGaN). The ‘yellow luminescence’, characteristic of nitride materials and detrimental for device performance, has been dramatically reduced compared with the first deposition runs and we are able to routinely produce blue LEDs (one example is shown in Fig. 1).

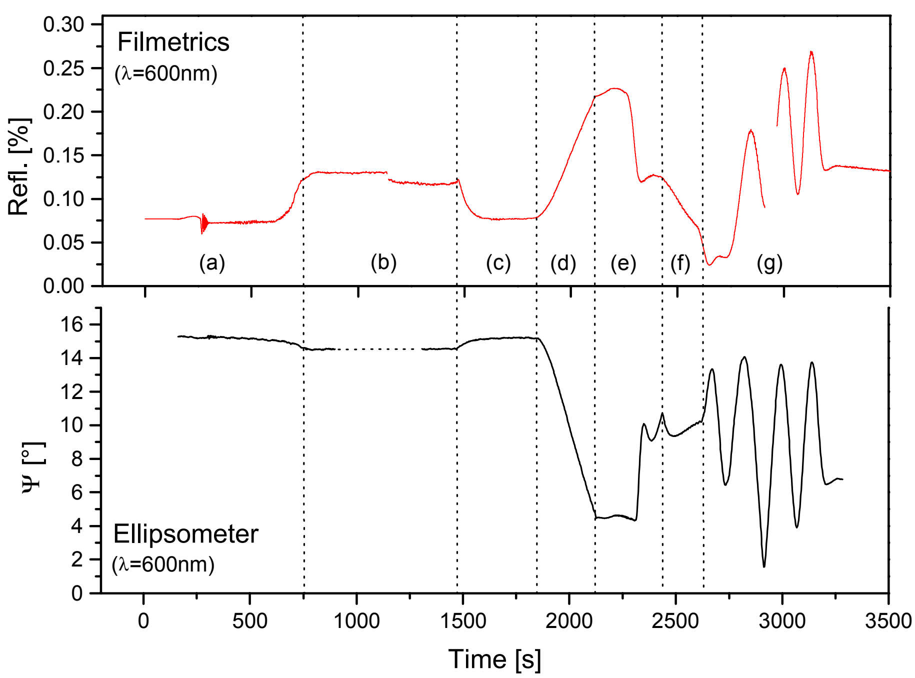

Fig. 2: Comparison between reflectivity (up) and kinetic SE acquired during the fabrication of GaN on sapphire.

3. In Situ Monitoring of the Growth

Concerning the in-situ monitoring of the growth process, special attention has been devoted to the comparison between SE and reflectometry measurements performed in parallel during deposition. Figure 2 gives an example of the two sets of data acquired during the fabrication of a standard GaN layer on Al_2O_3. All the information obtainable from reflectivity is also contained in the SE optical response. In addition, since reflectivity is intensity dependent, the signal is crucially influenced by wobbling of the sample (unavoidable in the reactor) and by light emission during the heating stages. On the contrary, SE is polarisation dependent and therefore much less sensitive to light interference and wobbling, so a far better signal-to-noise ration can be obtained (at the same wavelength). By *in situ* monitoring of GaN and ternaries deposition, we can obtain reproducible data on device quality material.

One of the goals of the in-situ monitoring is to have the possibility to determine in real time during growth the actual composition of the depositing layers.

In this perspective, series of AlGaN (and InGaN) layers with different concentrations have been grown. Kinetic SE measurements (ellipsometric angles/dielectric function vs. time) have been taken above the energy gap during the whole growth procedure, and an example of the results is reported in Fig. 3, where the imaginary part (ε_i) of the dielectric function is given as a function of the real part (ε_r). The obtained exponential spirals are typical of an interface which is continuously overgrown, and their radius is determined by the amplitude of the growth oscillations. The spirals convergence points give, in the complex plane, the values of the dielectric function of the deposited material and, therefore, the actual alloy composition.

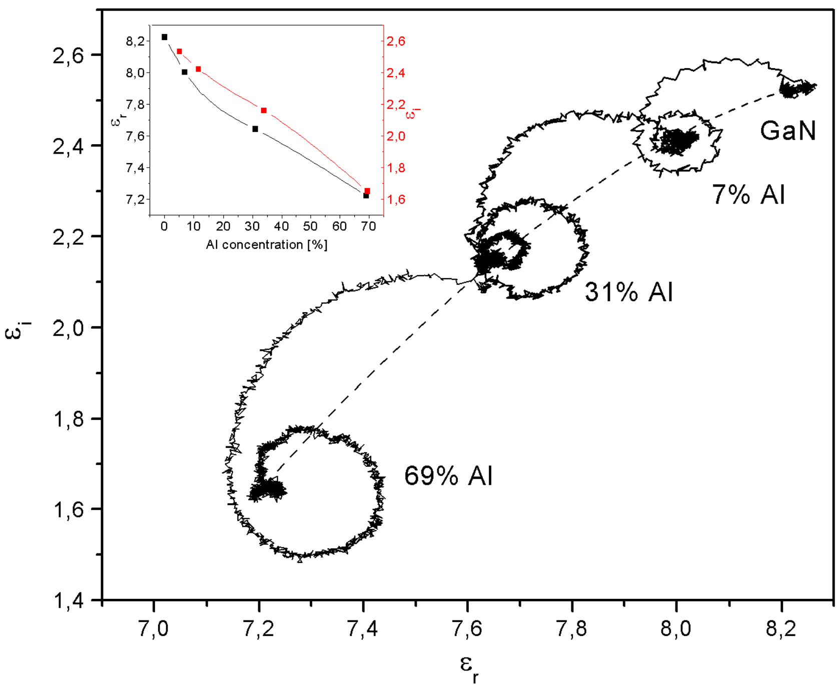

Fig. 3: Imaginary (ε_i) vs. real (ε_r) part of the dielectric function for layers with different Al content. The inset shows ε_i and ε_r respectively, as a function of the Al concentration.

Furthermore, we recently developed an algorithm for the real time determination of the compound concentration of ternary GaN (AlGaN, InGaN) during growth using the virtual interface (VI) model proposed by D. Aspnes for optical absorbing semiconductor crystal layers.

The last development regards the growth of pure cubic GaN we carried out on MBE-grown templates on GaAs (001). To our knowledge the first cubic MOCVD GaN in Europe.