Fourier Transformation Applied on *in-situ* Laser Reflectometry during MOCVD Growth

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We present an additional aspect of laser reflectometry by introducing Fourier transformation in order to analyze the kinetic reflectometry spectra taken during metal organic chemical vapor deposition. We can show that offset errors due to background radiation can be completely removed by the method itself without using filters or lock-in amplifiers. Additionally calibration of the reflected intensity is needed as long as the response of the detector is linear to the reflected intensity of the sample. By analyzing the time dependent part of the signal growth rate, layer thickness and the refractive index of the growing layer can be deduced.

Introduction

In-situ monitoring with high accuracy and real time feedback are important requirements for the reproducibility of the deposition steps. The possible spectrum of *in-situ* diagnostic tools is quite narrow, since the metalorganic chemical vapor deposition (MOCVD) procedure, being carried out at almost ambient pressure, excludes all techniques requiring ultra high vacuum, like reflection high electron diffraction. Only optical methods like spectroscopic ellipsometry (SE) [1], laser reflectometry [2] – [4] and x-ray diffraction [5] are successfully applied to gain information on layer thickness, layer composition and crystalline quality. Laser reflectometry represents nowadays one of the standard methods for optical *in-situ* investigations and is delivered with most of the MOCVD reactors. In this article we report on the Fourier analysis of *in-situ* reflectometry data leading to dramatic simplifications of the hardware and of the calibration processes.

Theory

Fresnel's equations and the applied transfer matrix formalism [2] are the roots of the reflectometry analysis of multi layer structures containing n different layers, assuming each of them to be homogeneous and isotropic. By mathematical considerations it can be shown that the time dependent reflectivity $R_{p,n}(t)$ of a growing layer can always be described as an Airy function if we assume a static history of layers 1..(n-1) and real refractive indices for the growing layer n which can be expanded in a Fourier series as shown in [6]:

$$\omega_{g,n} = \frac{4\pi v_{g,n}}{\lambda}\sqrt{\varepsilon_n - \varepsilon_0 \sin^2 \varphi}$$

$$R_{p,n}(t) = R_c + (2R_c - 1)\sum_{k=1}^{\infty}(-\rho_n)^k \cos(k\omega_{g,n}t - k\Phi_{n,0})$$

(1)

where ρ_n, $\Phi_{n,0}$ and R_c represent time invariant material constants recursively defined by the dielectric function of the growing layer ε_n, the ambient ε_{amb} and the material constant of the previous grown layer structure ρ_{n-1} [6]. The time invariant parameter $\omega_{g,n}$ represents the angular growth speed which is connected to the optical properties of the growing layer ε_n, the wavelength λ and angle of incidence φ of the reflectometer as defined in equ. (1).

Thus, the experimental signal can be described as a constant offset R_c and a sum of cosine functions where the argument is linearly dependent on the growth time t. Therefore, fast Fourier transformation (FFT) of the kinetic signal gives direct access to the angular growth velocity including high frequency noise reduction by the method itself. Higher harmonics of $\omega_{g,n}$ are damped by a factor of $(-\rho_n)^k$ as $\rho_n \leq 1$ [6] and allow to conclude that for the analysis the absolute scale of the detector is not important, as long as the response is linear with the intensity of the signal, since the relation between the intensity of two harmonics can be defined as:

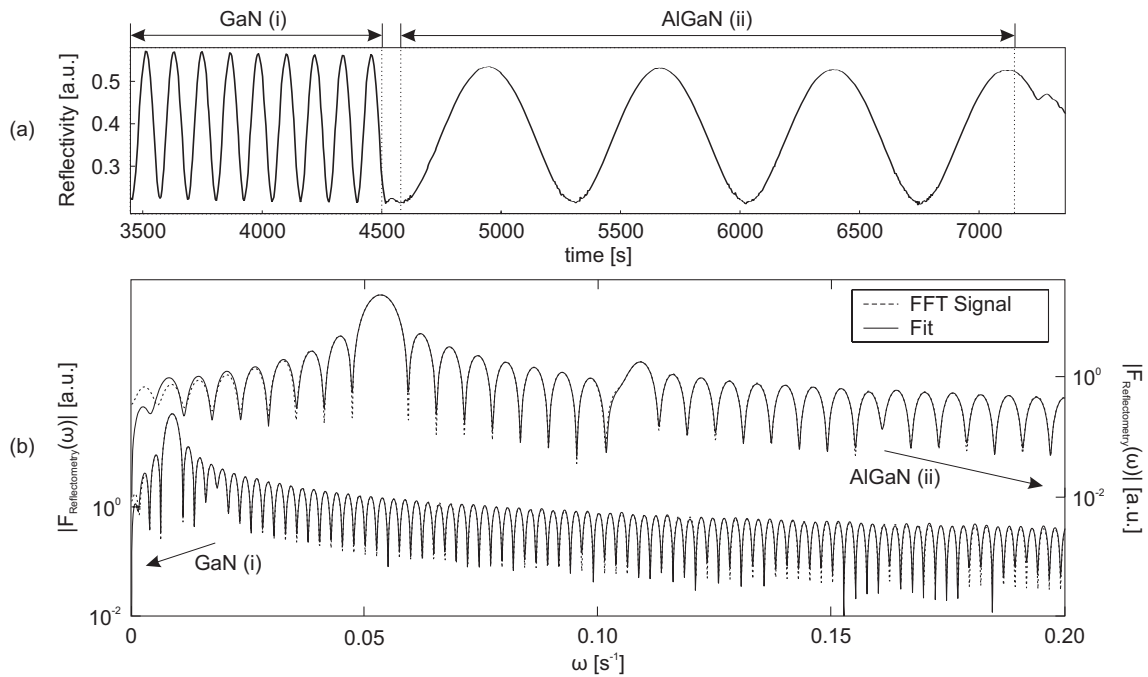$$I_{k+1} = -\rho_n I_k \tag{2}$$

Experimental results

Fig. 1: (a) reflectometry spectrum as a function of time acquired during GaN and Al-GaN growth. The time scale is taken relative to the start of the growth procedure including the thermal cleaning process of the substrate. (b) The FFT signal (dashed line) and the fit to the data (solid line)

Figure 1(a) shows a typical reflectometry spectrum taken during the deposition of a GaN layer followed by AlGaN. A GaN buffer is grown on sapphire substrate (i) with a growth rate of 3.6 µm/h. On the GaN buffer layer AlGaN is deposited using a lower growth rate, as evidenced by the onset of a longer period of the thickness oscillations (ii). After the removal of the offset part of the signal, the data are transformed into frequency domain by using FFT plotted in Fig. 1(b) as a power spectrum. The time range of the transformation is indicated by vertical dotted lines in the reflectometry spectrum.

For fitting the data, a sum of four harmonics has been employed and is in agreement with the measured data, due to a strong damping of the higher harmonics as expected from equ. (2). The fit is indicated by solid lines in the Fourier spectra.

In Table I we summarize the results for the fitted angular growth velocity ω_k and the damping factor $|\rho_k|$ together with the calculated values for the refractive indices n_k and the growth rates v_k. The refractive index for sapphire has been taken from literature [7] and the nitrogen and hydrogen atmosphere in the growth chamber has been assumed to be comparable to ambient. A comparison with previous works [8], [9] leads to a very good agreement of the measured refractive indices for GaN.

	k	ω_k [s^{-1}]	$|\rho_k|$	V_k [nm s^{-1}]	n_k
Al_2O_3	0	-	0	-	1.78
GaN	1	0.0533	0.0614	1.094	2.403
AlGaN	2	8.68×10^{-3}	0.0557	0.182	2.351
Ambient	3	-	-	-	1

Tab. I: Experimental results of $|\rho_k|$, the angular growth velocity, the growth rate and the resulting refractive indices for GaN and AlGaN layer at 1050°C.

The refractive index of $Al_xGa_{1-x}N$ shows a strong dependence on the achieved Al concentration x which was determined to be x=20.7% in the considered sample as proven by x-ray diffraction. The relative change at the GaN/AlGaN interface of $\Delta n = -0.052$ is comparable with literature data [10] for similar Al concentrations at room temperature.

Conclusion and Outlook

We have demonstrated that Fourier analysis of kinetic reflectometry measurements acquired during a MOCVD process leads to fast and accurate information about the deposited layers, including optical properties, growth rate and layer thickness. The method eliminates offset errors resulting from thermal radiation from the reactor chamber and does not need careful calibration. Furthermore the measured signal is independent on an absolute scale. Further potential of applied Fourier transformation on kinetic reflectometry data is expected for periodic grown structures as shown in ref. [6].

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