SVT ASSOCIATES

Application Note

In-Situ 4000 Process Monitor Measurement of GaN Growth Rate as a Function of Substrate Temperature

Abstract

The growth of GaN on a (0001) sapphire substrate has successfully been monitored for temperature and growth rate using the In-Situ 4000 Process Monitor. The growth rate has been measured in real time by fitting the optical reflectance oscillations to an analytical model. The growth rate is found to drop substantially as the substrate temperature is raised even while gallium rich growth conditions are preserved.

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Introduction

One unique feature of the In-Situ 4000 is its ability to simultaneously measure both the substrate temperature and the film growth rate. The In-Situ 4000 optical system contains both a near-infrared pyrometer and a specular reflectometer and combines the information from both systems to produce an accurate temperature measurement which is independent of thin film optical interference effects. The specular reflectance information is also analyzed by a set of sophisticated algorithms to compute the film growth rate in real-time.

Traditional pyrometers simply measure the intensity of radiated energy in a selected wavelength range. If the emissivity of the surface under measurement is known, then the temperature may be calculated from Planck's Law. Semiconductor substrates are not opaque at all wavelengths so it is critical to choose a measurement wavelength in which the substrate is opaque. Traditional semiconductor substrates are transparent in the near infrared and longer wavelengths, so the In-Situ 4000 pyrometer uses a 950 nm wavelength which is optimized for GaAs, InP, and silicon substrates. In this experiment however, the substrate used is sapphire which is transparent at 950 nm, so the back surface of the sapphire substrate was coated with an opaque film of a refractory metal which provides a good surface for pyrometry.

A major complication of accurate pyrometry during epitaxy occurs in the case of heteroepitaxy where the deposited film has a different optical index of refraction from the substrate. During heteroepitaxial film growth, optical interference will occur between the film interface and the surface and the pyrometric radiation from the substrate will alternately be enhanced and suppressed as the film becomes thicker. This effect appears to a traditional pyrometer as a change in temperature when in fact it may not be. The In-Situ 4000 performs 950 nm reflectance measurements simultaneous with 950 nm pyrometry and uses the known reflectance to compensate for the "varying emissivity" of the thin film stack and produce a more accurate temperature reading.

Measurement Setup

The In-Situ 4000 was used to measure the substrate temperature and growth rate of a GaN film deposited onto a 50 mm diameter sapphire (0001) substrate. The In-Situ 4000 optical sensor was mounted at the central pyrometry viewport of a molecular beam epitaxy system. The Ga flux was provided by an SVT Associates hot lip effusion cell and the atomic nitrogen flux was provided by an SVT Associates RF plasma source fed by N_2 gas. The unpolished back surface of the substrate was coated with a Ti film to create an opaque, emitting surface for the pyrometry measurement. The substrate was radiatively heated from the back side to temperatures up to 850°C and substrate rotation was used to ensure good film uniformity. The atomic fluxes were adjusted to ensure that the film growth was nitrogen flux limited, that is there was excess Ga flux.

Measurement Results

Gallium nitride films were grown under seven different substrate temperatures ranging from 700°C to 850°C. During the film growth, the optical reflectance was monitored at 470 nm and 952 nm wavelengths. Since the index of refraction of GaN is approximately 2.5 and the index of sapphire is about 1.8, there are strong oscillations in reflectance as the film becomes thicker. The period of oscillation, P, is proportional to the film index of refraction, n, and the growth rate, G, by the expression:

$$P = \frac{2nG}{\lambda}$$

Since the measurement wavelength, λ, is known, the product of n and G is found from fitting the oscillations to a theoretical model[1]. This model may be used such that the index is specified (if it is known) and the growth rate is calculated from the oscillation period. This method is useful even if the absolute reflectance measurement is not well calibrated since it is the oscillation period which contains the growth rate. If the film index is not well known then it may be included in the fitting algorithm to yield both the index and the growth rate. In this experiment, the index was assumed to be known and the growth rate calculated from the oscillation period.

Six films were grown each at different substrate temperatures. Figure 1 shows the substrate temperature as a function of time during the deposition process.

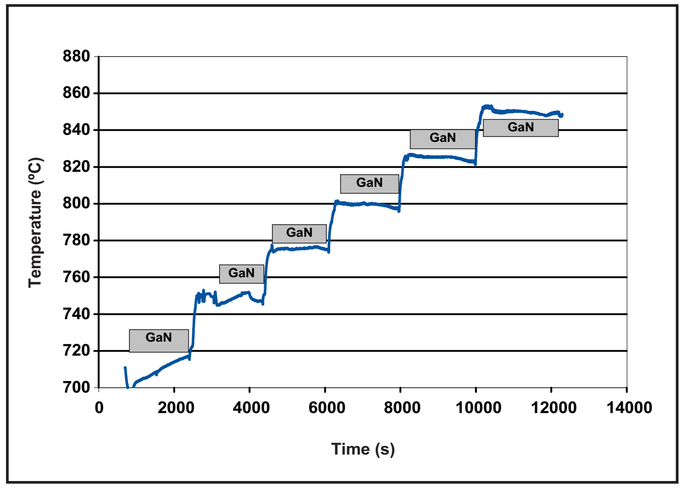

Figure 1 - Measured substrate temperature during the growth of six GaN films from 700°C to 850°C. The boxes indicate the portions of time when GaN growth occurred.

The measured temperature using a traditional pyrometer would have shown substantial upward and downward swings as the films were deposited. The In-Situ 4000 temperature shows no interference induced temperature errors as is seen more clearly in Figure 2.

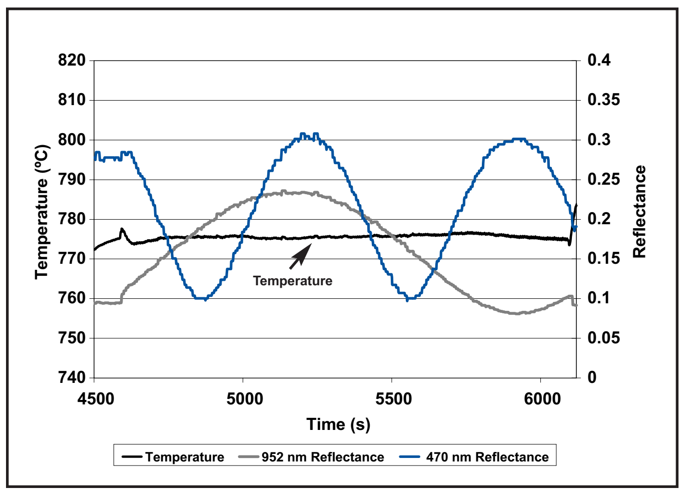

Figure 2 - Substrate temperature, 952 nm reflectance, and 470 nm reflectance curves measured by the In-Situ 4000 during one of the GaN film growths. The temperature reading is stable in spite of the presence of large oscillations in the reflectance due to thin film interference.

The results of the reflectance oscillation fitting for both the 470 nm and 952 nm measurements are shown in Figure 3. The product of G·n is clearly seen to drop as substrate temperature rises.

[1] W.G. Breiland and K.P. Killeen, J. Appl. Phys. 78(11), p. 6726 (1995).

Since the two curves of Figure 3 are not coincident with each other and the film growth rate must be the same, the ratio of refractive index between the two wavelengths may be calculated. With the exception of the film grown at 800°C, the ratio is consistently 1.084 which is effectively an estimate of the dispersion of GaN at growth temperature. Ex-situ measurement of the physical film thickness results in a GaN index of refraction of 2.264 at 952 nm, and 2.465 at 470 nm which are very close to published room temperature values.

The drop in G·n can be shown to be due to a drop in G alone due to the fact that the absolute reflectance intensity measured for all the films did not change as is seen in Figure 4. If the drop in G·n was due to a change in index, then the average reflectance would have changed which was not observed. The damping of the 470 nm reflectance oscillations at the higher temperatures is presumed to be due to an increasingly rough surface morphology.

The 470 nm reflectance growth rate measurement at 800°C deviates from the trend due to an insufficient Ga flux rate to maintain Ga rich growth conditions. Upon closer inspection of the 470 nm reflectance data, the oscillation period is seen to vary during the 800°C growth and the analytical model to which the data is fit assumes a constant growth rate. The 470 nm reflectance is twice as sensitive to growth rate as compared to the 952 nm measurement and the fit algorithm for the 470 nm data did not converge onto a stable value for growth rate.

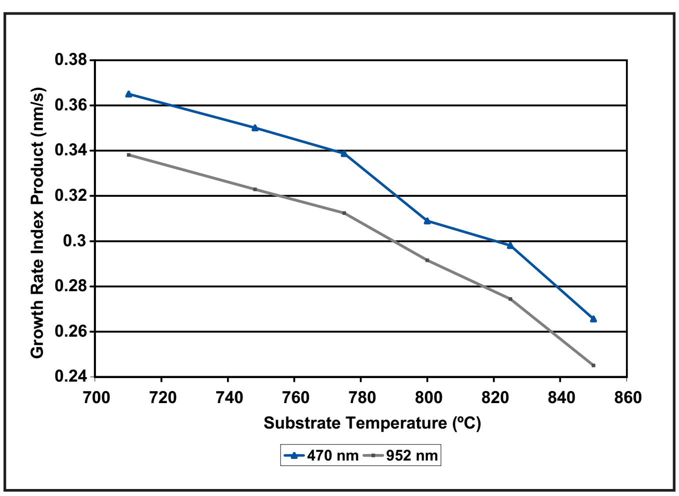

Figure 3 - Plot of growth rate - index product (G·n) vs. substrate temperature for the two reflectance wavelengths. The values for G·n result from fitting the reflectance oscillations to the analytical model.

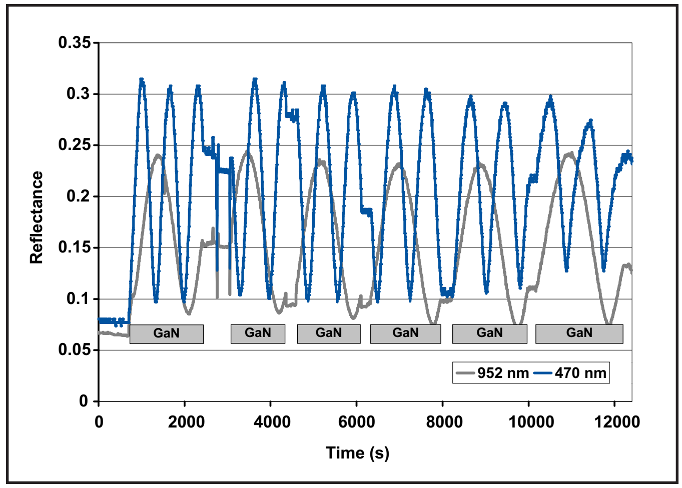

Figure 4 - Plot of reflectance at 470 and 952 nm during the growth of GaN films at varying substrate temperatures.

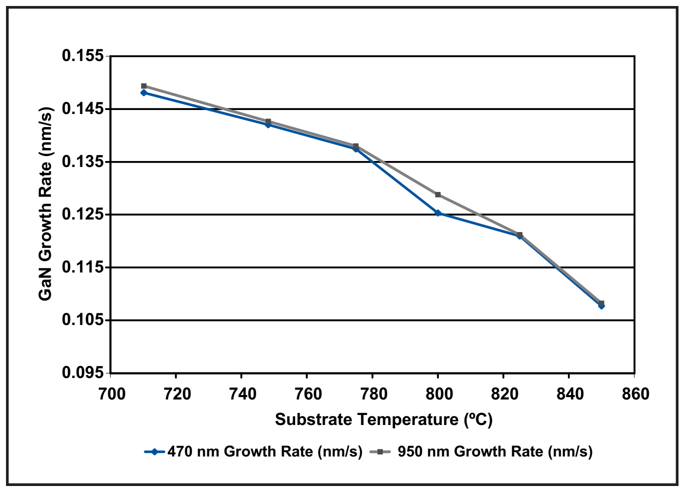

Figure 5 - GaN growth rate calculated from fits to the reflectance data of Figure 4 and an index of refraction calculated from an ex-situ thickness measurement.

Summary

The growth rate of GaN films has been successfully measured as a function of substrate temperature using the In-Situ 4000 Process Monitor. The temperature is accurately measured even in the presence of strong thin film interference effects which normally cause false readings with traditional pyrometers. The growth rate is measured in real time by fitting the oscillating reflectance information to an analytical model. The growth rate is found to strongly depend on substrate temperature, even under gallium rich growth conditions.

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