SVT ASSOCIATES

Application Note

In-Situ 4000
Monitoring the Growth of Nitride Films on Transparent Sapphire Substrates

Abstract

The In-Situ 4000 Process Monitor has been used to accurately measure substrate temperature during the growth of GaN on sapphire. The emissivity compensation feature provides an improved temperature measurement over traditional pyrometry. The specular reflectance information provided in real-time allows film thickness to be determined *in-situ*.

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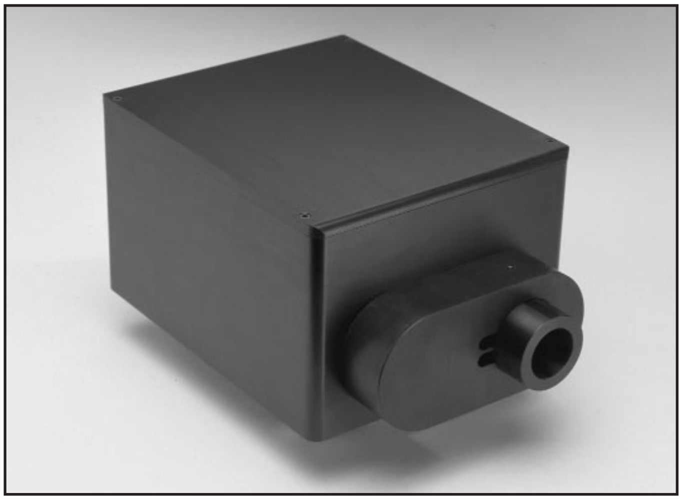
Measurement Challenge - Transparent Substrates

The In-Situ 4000 Process Monitor is well suited to provide both temperature and film thickness information in real-time during growth of III-V Nitride semiconductor thin films on transparent sapphire substrates. Pyrometry normally cannot be used for temperature measurement on transparent substrates for two reasons. The first problem occurs if the transparent substrate is radiatively heated. Here the optical radiation from the substrate heater passes through the substrate and reaches the pyrometer. The pyrometer cannot then distinguish between substrate radiation and heater radiation and a higher than actual temperature reading results. The second problem is that the emissivity of the transparent substrate is very small, yielding a very low “black body” radiance, even at very high temperatures.

Our approach to solving the problem of transparent substrates is to coat the back side of the substrate with an opaque, high temperature compatible metal film, in this case titanium. The film must be sufficiently opaque to prevent any radiation from the substrate heater from passing through to the pyrometer. This backside metal film then becomes the absorber of the heater energy and then thermally conducts the heat to the sapphire substrate. The emissivity of the film will typically be much larger than the transparent substrate so that the back side film becomes the black body radiator which the pyrometer measures. We then assume that the thermal conductivity of the substrate is high enough to prevent any significant temperature difference between the measured back side and the front side where film growth occurs.



In-Situ 4000 Process Monitor

Measurement Challenge - Pyrometry of Thin Films

Pyrometry during the deposition of semiconductor thin films can suffer from a measurement error when the index of refraction of the thin film differs from the index of refraction of the substrate. This optical interference effect, which varies with time as deposition occurs, changes the optical transmission of the film and thus alters the quantity of black-body radiation reaching the pyrometer. This "changing emissivity" effect will produce an error when using a pyrometer to measure the substrate temperature and will, in many cases, actually change the substrate temperature since it alters the rate at which radiative cooling takes place. Since knowledge and control of the substrate temperature is key to high quality film growth, improved temperature measurement is often required. In fact, time varying emissivity can be especially severe when high reflectance mirrors are grown which consist of multiple periods of alternating materials of low and high index of refraction as the emissivity varies strongly as the layers are deposited.

The In-Situ 4000 process monitor corrects for the emissivity errors in temperature measurement by actively measuring the emissivity both before and during growth. The measured emissivity can then be used to correct the pyrometry signal to achieve a correct temperature reading. Figure 1 below illustrates the effect. In this experiment, a film of GaN grown on sapphire was grown while the temperature was measured with the In-Situ 4000 Process Monitor. The substrate temperature reading of both traditional uncompensated pyrometry and emissivity compensated pyrometry were measured at 950 nm wavelength. For the uncompensated measurement, the apparent temperature of the substrate oscillates as the layer is grown due to the strongly changing emissivity of the growing thin film. The In-Situ 4000 compensated measurement shows the actual substrate temperature.

The substantial difference between the curves represents the measurement error present in the conventional pyrometer.

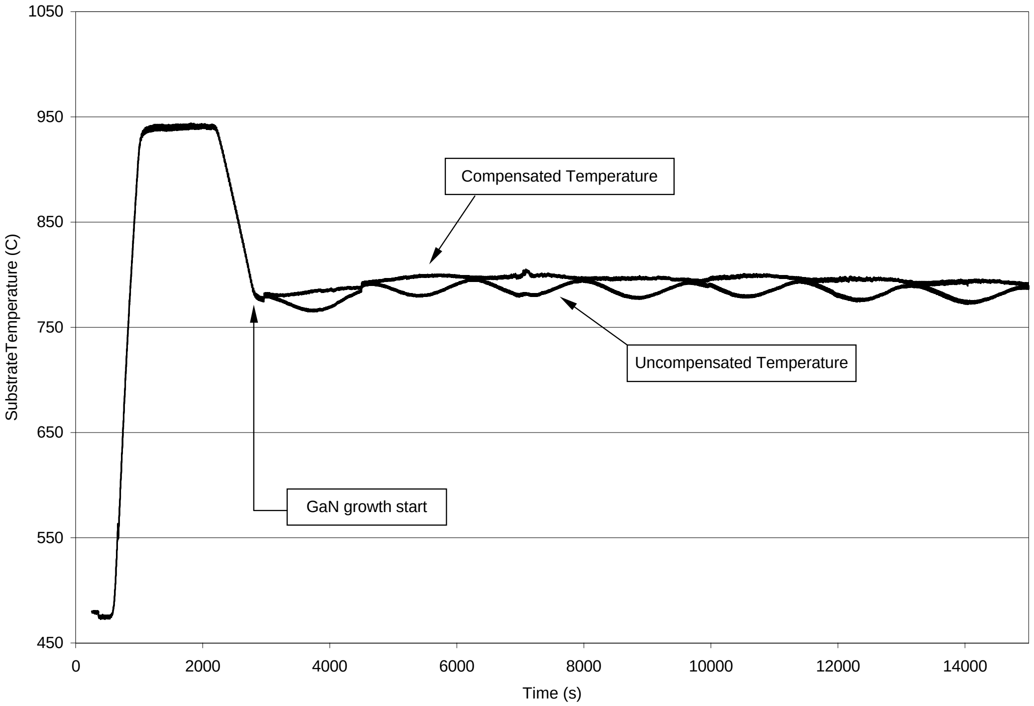


Figure 1 - Measurement of substrate temperature during the growth of GaN on sapphire. The back surface of the sapphire substrate was coated with titanium. The deposition of GaN on the front surface causes optical interference to alter the emissivity of the substrate producing temperature errors. The curve labeled "Uncompensated Temperature" is the reading a traditional pyrometer produces and the curve labeled "Compensated Temperature" is the corrected temperature reading of the In-Situ 4000 Process Monitor.

Measurement Challenge - In-Situ Film Thickness

There is a clear need for real-time film thickness information during MBE growth processes. Specular reflectometry is an accurate and convenient method to compute film thickness during growth. One major advantage of the In-Situ 4000 Process Monitor is the ability to perform specular reflectometry at two wavelengths simultaneously with pyrometry so that neither the film thickness nor the substrate temperature measurement is compromised. Both long (950 nm) and short (470 nm) wavelengths are used in the reflectometry so that sensitivity is maximized for the thinnest of films.

For this example of the growth of GaN on sapphire, the reflectometer is calibrated to the known reflectance of the sapphire substrate which is 7.7%. Once nitride films are deposited, optical interference will cause the reflectance to change and this evolution of reflectance can be used to compute film thickness and even index of refraction at growth temperature. Figure 2 below shows reflectance during the growth of a thin AlN buffer layer, and the subsequent growth of a GaN film and an AlGaN film. The first 1000 seconds of GaN growth were performed at a lower growth rate at which point the growth rate was increased. At the 5000 second point, the film composition was changed to AlGaN with the same growth rate as GaN and the 470 nm reflectance shows lower oscillation amplitude due to the lower index of refraction.

Summary

The In-Situ 4000 Process Monitor has successfully measured the substrate temperature during the growth of GaN films on metal-backed sapphire substrates. The built-in reflectometer corrected the emissivity fluctuation which would have otherwise produced a temperature error of approximately 20°C. The two wavelength reflectometer followed the growth of both GaN and AlGaN films allowing calculation of the growth rate in-situ.

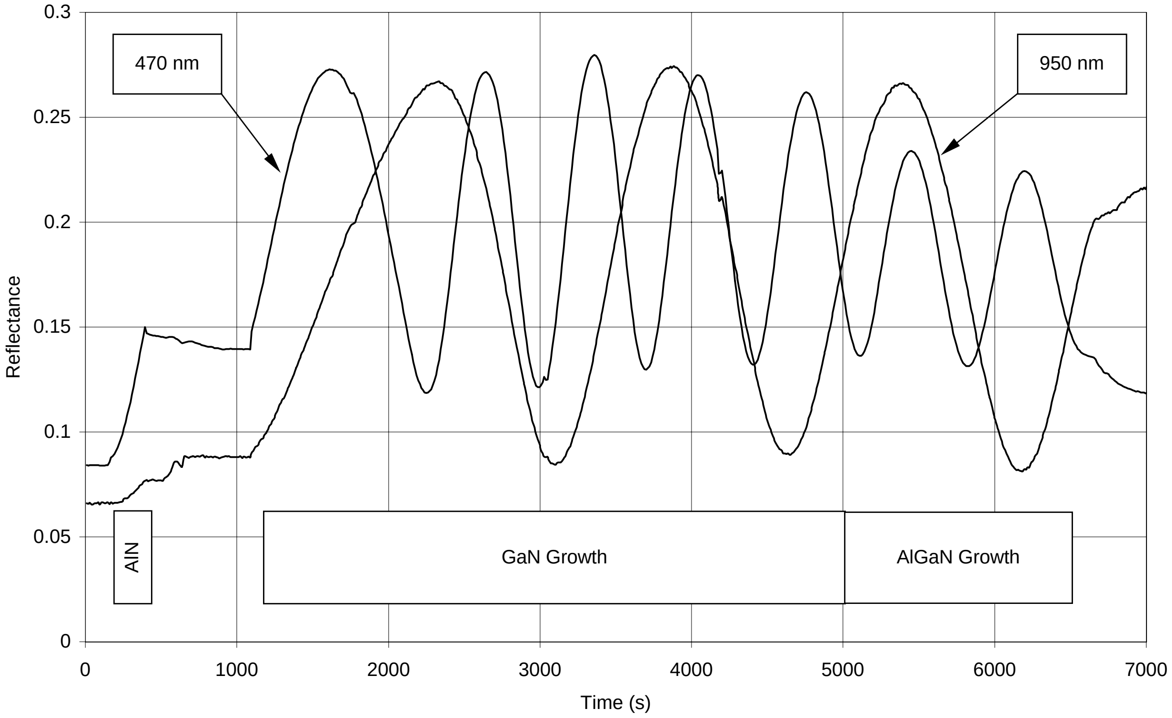


Figure 2 - Specular reflectometry at both 470 nm and 950 nm measured during the growth of GaN and AlGaN films on a sapphire substrate. The GaN growth begins at 1100 seconds at a relatively slow rate. The GaN growth rate was increased to a higher rate at 2000 seconds. At 5000 seconds, the aluminum source shutter was opened yielding an AlGaN film with the same growth rate as the GaN, but with smaller reflectance modulation due to the lower index of refraction of the AlGaN film as compared to the GaN film.

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