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IN SITU REFLECTANCE AND VIRTUAL INTERFACE ANALYSIS FOR COMPOUND SEMICONDUCTOR PROCESS CONTROL

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We review the use of in-situ normal incidence reflectance, combined with a virtual interface model, to monitor and control the growth of complex compound semiconductor devices. The technique is being used routinely on both commercial and research metal-organic chemical vapor deposition (MOCVD) reactors and in molecular beam epitaxy (MBE) to measure growth rates and high temperature optical constants of compound semiconductor alloys. The virtual interface approach allows one to extract the calibration information in an automated way without having to estimate the thickness or optical constants of the alloy, and without having to model underlying thin film layers. The method has been used in a variety of data analysis applications collectively referred to as ADVISOR (Analysis of Deposition using Virtual Interfaces and Spectroscopic Optical Reflectance). This very simple and robust monitor and ADVISOR method provides one with the equivalent of a real-time reflection high energy electron reflectance (RHEED) tool for both MBE and MOCVD applications

INTRODUCTION

State-of-the-art device structures, particularly structures used in optoelectronic applications, require extraordinary control over thin film thickness, chemical composition, and doping level. Such control is more easily achieved with real-time, in-situ process sensors. In this paper, we review the use of reflectance interferometry as an in situ monitor of thin film deposition and its use in compound semiconductor process control.

The methods described in this paper represent an evolution from traditional methods used in thin film fabrication toward a future goal of fully-implemented, real-time process control. The fabrication of semiconductor thin films using CVD or MBE has traditionally been accomplished by metering source materials into the reaction chamber using a series of timed sequences for a fixed set of reactor conditions. Calibration runs are used in an iterative "dead reckoning" control strategy to make adjustments in the device structure recipe. Most calibrations are performed first with test structures and finally with repeated trials on the actual device structure using post-process ex situ analysis tools such as microscopy, photoluminescence, and X-ray diffraction methods. It is often very difficult to determine the origin of a failure or process deviation using post-

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growth analysis during the final fine tuning of the device structure recipe. The grower is often forced to guess what portion of the recipe needs alteration and then test this guess with another growth run. A large portion of the time and expense of thin film CVD is thus spent in calibration and post-process failure analysis.

The above deficiencies may be avoided with a pre-growth calibration method that uses normal incidence reflectance. In many respects the method assumes the same role as reflection high-energy electron diffraction (RHEED) does in molecular beam epitaxy. The instrumentation is simple, inexpensive, and robust. Its use greatly simplifies the pre-growth calibration process by allowing one to measure the growth rate from each source in a single, relatively short deposition run. The reflectance probe also serves as a valuable real-time diagnostic of the state of the wafer throughout the device structure growth.

The paper is divided into two sections. The first section describes the theory behind the calibration method and details of the data analysis. The second section illustrates the practical use of the method with brief examples from CVD and MBE applications. Most of the concepts presented in this review have been described in more detail in references [1-5].

VIRTUAL INTERFACE ANALYSIS OF REFLECTANCE

The Virtual Interface Concept

It is straightforward to model the reflectance from a smooth semiconductor substrate with an arbitrary number of smooth, homogeneous films deposited on it [6,7]. The only parameters required are the complex refractive index for the substrate and the thickness and refractive index of each layer. If a growing film is monitored, reflectance interference oscillations are observed as the topmost film thickness changes with time. This is also straightforward to model by expressing the topmost film thickness as the product of a growth rate and the time.

There are, however, severe practical limitations that arise when such a straightforward approach is used to model the in situ reflectance waveform of a growing multiple-layer film. First, the optical constants of most semiconductor materials are not well known at the temperatures typical for semiconductor growth. This is particularly true for compound semiconductor alloys. Second, the thickness of a layer is not necessarily known precisely, particularly during a calibration run. In a multiple-layer reflectance model, errors in the refractive index and thickness of each layer contribute additively to uncertainties in the description of the growth of the topmost layer. This leads to a growth rate determination that is progressively less accurate with each additional calibration layer.

The solution to the multiple-layer reflectance modeling problem is to use a virtual interface concept, which is illustrated in Fig. 1. One chooses a virtual interface position (dashed line) that lies anywhere within the topmost film. It is then possible to rigorously describe the effects of all underlying layers as a single effective complex refractive index,

N_{vi}, of a virtual interface effective substrate. The precise value of N_{vi} can, in principle, only be calculated from a complete knowledge of all the refractive indexes and thicknesses of the underlying layers. However, if N_{vi} is taken to be an unknown, it is always true that any multiple-layer structure requires only two parameters, the real and imaginary parts of N_{vi}, to describe the effects of all underlying layers below the virtual interface boundary. Analysis of the topmost layer is thus made completely independent of the optical constants and interface positions of underlying layers. By choosing a new virtual interface position with each new layer, cumulative effects are eliminated in the analysis of a growing multiple-layer film structure.

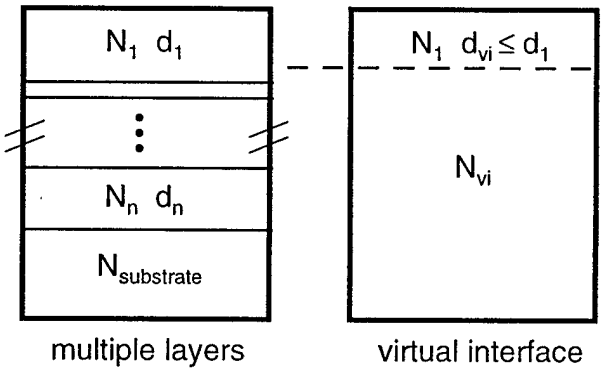

Fig. 1. Any multiple-layer thin film structure having refractive indexes, N_i, and thicknesses, d_i, may always be modeled as a single-layer film on a virtual substrate with an effective refractive index, N_{vi}. All the optical effects of the various N_i and d_i are lumped into the value for N_{vi}.

Aspnes [8] first suggested the use of a virtual substrate in the analysis of in situ ellipsometry of growing thin films. A slightly modified version of this concept was applied to normal incidence reflectance by Breiland and Killeen [1].

Growth Rate Extraction

The mathematical details of the virtual interface as it applies to normal incidence reflectance have been described in detail in refs [1,4]. It is straightforward to extend this approach to include all incidence angles and both "s" and "p" polarization.

The time-dependent complex reflectance, $r(t)$, for a growing film has the same functional form for either polarization:

$$r^{s,p}(t) = \frac{r_{\infty}^{s,p} + r_{vi}^{s,p} Z}{1 + r_{\infty}^{s,p} r_{vi}^{s,p} Z}, \quad Z = \exp\left(-i4\pi N_{eff} Gt / \lambda\right),$$

where θ is the angle of incidence, $N_{eff} = \sqrt{N^2 - \sin^2\theta}$ is the effective refractive index, $N = n - ik$ is the actual complex refractive index of the film, G is the growth rate, and λ

is the vacuum wavelength of light. The above expression contains two complex reflectance values: a materials-dependent reflectance for an infinitely thick film:

$$r_{\infty}^{s} = \frac{\cos\theta - N_{eff}}{\cos\theta + N_{eff}}, \quad r_{\infty}^{p} = \frac{N^2\cos\theta - N_{eff}}{N^2\cos\theta + N_{eff}},$$

and a virtual interface reflectance, r_{vi}^{s}, or r_{vi}^{p}, that contains the cumulative effects of the substrate and all underlying layers below the topmost film. The virtual interface reflectance may be calculated if all details of the structure are known, but it may also be treated simply as a two-parameter fitting coefficient that requires no knowledge of underlying film structure or materials properties. Note that the virtual interface reflectance takes on different values for each polarization. An experiment performed with mixed polarization would need to extract both of these values to enable the accurate determination of the topmost film optical constants and growth rate.

The topmost film growth rate and optical constants may be determined with the following scheme: The observed reflectance, $R^{s,p}(t) = \left|r^{s,p}(t)\right|^2$, is first cast into a waveform that closely resembles a damped cosine by performing a bilinear transform [9]:

$$B^{s,p} = \left(1 + R^{s,p}\right)/\left(1 - R^{s,p}\right) \cong a - be^{-\gamma t}\cos\left(\omega t - \sigma\right).$$

values for $a, b, \gamma, \omega, \sigma$ are obtained from the positions of extrema and inflections in the experimental waveform using digital filter methods. Estimates of the virtual interface reflectance, optical constants, and growth rate are obtained from the following expressions:

$$r_{vi}^{s,p} = r_i e^{i\sigma}, \quad r_i = b\left(1 - R_{\infty}\right)/4\sqrt{R_{\infty}}, \quad R_{\infty} = (a-1)/(a+1)$$

$$n_{eff}^{s} = \rho\cos\theta, \quad n_{eff}^{p} = \rho/2\cos\theta + \sqrt{\left(\rho/2\cos\theta\right)^2 - \sin^2\theta}, \quad \rho = \left(1 + \sqrt{R_{\infty}}\right)/\left(1 - \sqrt{R_{\infty}}\right)$$

$$G = \omega\lambda/4\pi n_{eff}, \quad k_{eff} = \gamma\lambda/4\pi G$$

$$n^2 = \left(\frac{n_{eff}^2 - k_{eff}^2 + \sin^2\theta}{2}\right) + \sqrt{\left(\frac{n_{eff}^2 - k_{eff}^2 + \sin^2\theta}{2}\right)^2 + n_{eff}^2 k_{eff}^2}, \quad k = n_{eff}k_{eff}/n.$$

The estimates, $r_{vi}^{s,p}, n, k, G$ are finally used as starting values in a non-linear least-squares fit to the exact expression for $R^{s,p}(t) = \left|r^{s,p}(t)\right|^2$.

The above extraction scheme becomes extremely useful for routine pre-growth calibrations. The calibration procedure is as follows: A multiple-layer film is grown and a single-wavelength in situ reflectance interferogram is recorded. Each layer is grown thick enough to provide several extrema of interference oscillations. The order of the layers and timing is chosen to yield good contrast between the interferograms in each

layer. For example, a GaAs growth calibration layer is done after an AlAs calibration layer is placed on the GaAs substrate. Segments of data from each layer are chosen for analysis. The starting time for each segment is arbitrary, provided that it does not include the transition from one layer to the next. The choice of starting time changes only the value of r_{vi}, which is of no physical interest. Typically, the starting segment is chosen to be several seconds beyond the time at which the analyzed layer is known to have started. The stopping time is chosen to be a few seconds before the next layer is known to have started. The procedure outlined above is used to provide starting estimates for the five-parameter fit, and a least-squares analysis is then done to fit the data segment to $R(t) = |r(t)|^2$. This can all be accomplished without prior knowledge about the growth rates or optical constants of previously deposited materials. The process is then repeated for each layer in the calibration run. Fitting takes less than a second for each layer. Fig. 2 displays the screen output from a program designed to analyze reflectance spectra using the virtual interface method. With this program, analysis consists of selecting the layer and pressing the "Do Fit" button. This method of analysis has been dubbed ADVISOR for "Analysis of Deposition using Virtual Interfaces and Spectroscopic Optical Reflectance".

The accuracy of G is related directly to the accuracy of the absolute reflectance. A one percent error in reflectance results in approximately a one percent error in growth rate. It is thus very important to self-calibrate the reflectance at the beginning of each run and to maintain instrument stability throughout the run.

With ideal signal-to-noise (10^4), it is possible to obtain one percent accuracy in the growth rate with a film thickness on the order of $\lambda / 4n$. Accuracy improves exponentially with film thickness up to $\lambda / 2n$, at which point it becomes noise limited. These thicknesses may be excessive if one is considering real-time control of thin structures, but there is no such limitation on the thickness of a pre-growth calibration film.

Reactor Models

An important aspect of the ADVISOR scheme is to use the growth rate measurements to determine the empirical behavior of the reactor. This can range from a simple confirmation that growth rate is proportional to source flux to more complicated relationships such as the etching effect that a dopant gas such as CCl4 has on GaAs growth. The reflectance monitor may be used to quickly perform these studies because many growth rate measurements for a variety of deposition conditions may be performed within a single run. Fig 3 illustrates such a procedure for verifying the linear dependence of growth rate on trimethylgallium (TMG) and trimethylaluminum (TMA) flow rates in a CVD reactor. The slopes extracted from the fits on the left graph may be used to construct a deposition recipe to grow an $Al_xGa_{1-x}As$ alloy of any composition, x.

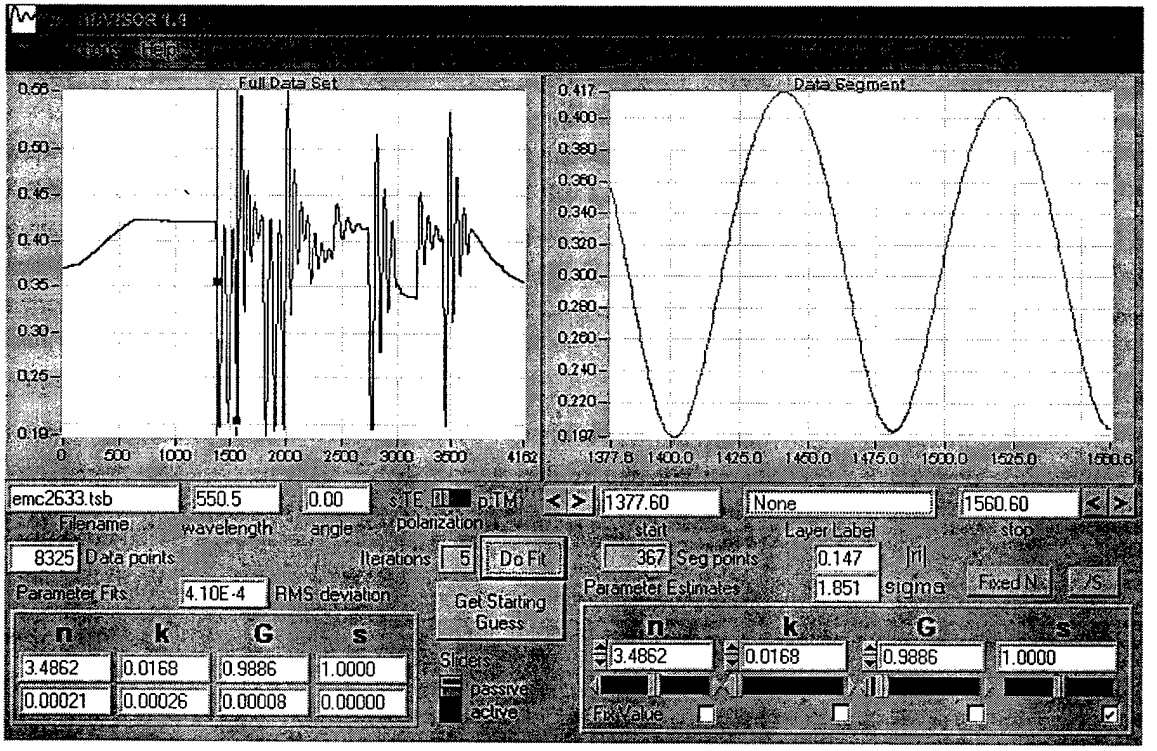

Fig 2. Screen output from a program designed to automatically extract the growth rate and optical constants from a reflectance waveform. The left plot displays the entire calibration run. The right plot displays a segment of reflectance from one layer. The fit overlays the data. The screen also displays parameter values extracted from the fit.

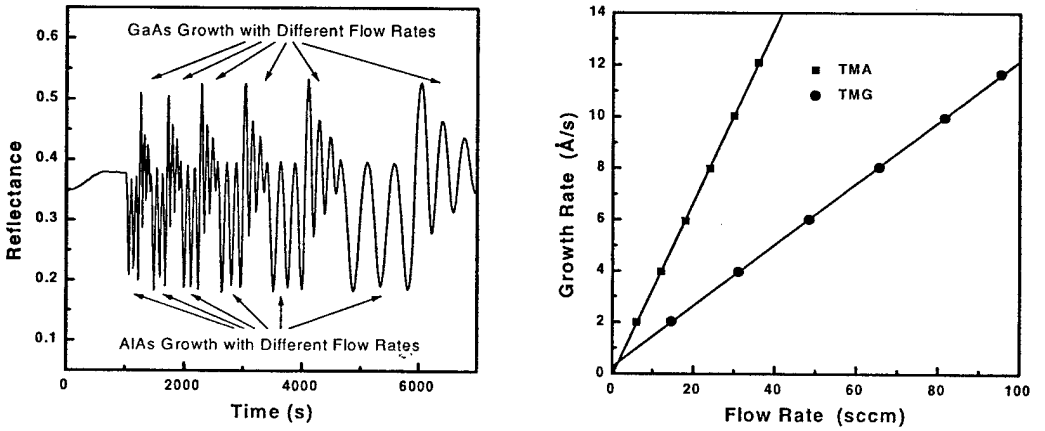

Fig. 3. Left: Reflectance from twelve layers of alternating AlAs(2500 Å)/GaAs(2500 Å) thin films. Different alkyl flow rates were used for each layer. Right: Growth rates as a function of alkyl flow rate extracted from the data using ADVISOR.

APPLICATIONS

A schematic of the reflectance apparatus is shown in Fig 4. Normal incidence reflectance requires only one window, is insensitive to incidence angle, (allowing one to monitor "wobbling" substrates) and is also insensitive to light polarization. The light source is a 5 Watt tungsten halogen lamp coupled to the probe with multimode optical fibers. DC detection of the light is made through an interference filter and silicon photodiode. It is therefore simple, robust, and well suited to a production environment. Absolute reflectance is obtained by referencing the starting signal to the known reflectance of the substrate before deposition begins. This provides a self-calibration at the beginning of each growth run.

Also shown in Fig. 4 is a reflectance waveform from an InGaAs / InAlAs distributed Bragg reflector (DBR) structure grown by MBE The signals are comparable to those obtained in CVD systems illustrated in Refs [3,4].

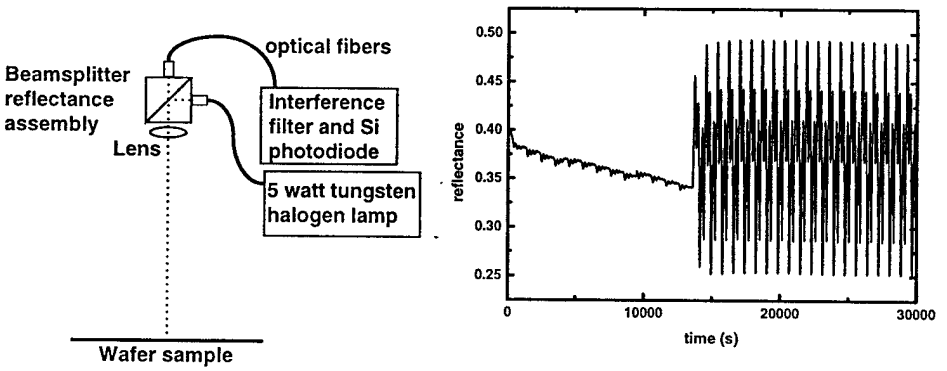

Fig 4. Left: schematic of apparatus used to record normal incidence reflectance during deposition. Right: 550 nm reflectance waveform from the step-graded buffer (first half of waveform) and the distributed Bragg reflector (second half of waveform) of an InGaAs / InAlAs structure grown by MBE

References [3,4] have described how the ADVISOR technique has been used to develop growth recipes for vertical cavity surface emitting lasers (VCSEL's). Very high yields and run-to-run reproducibility of ±0.3% was demonstrated for these very complex structures. Similar results, ±0.5%, have been achieved with the growth of infrared DBR structures using MBE [5]. It is easiest to demonstrate the value of the ADVISOR technique by looking at the corrections that are made after ADVISOR calibration from the "dead reckoning" that would have been used without corrections. Because the device yield is very high with the reflectance adjustments, the corrections are essentially a measure of how far off the device structure would have been had no corrections been made. Fig. 5 shows such plots for MOCVD and MBE applications of the ADVSIOR method.

The plot on the right of Fig 5 shows ADVISOR corrections for bubbler sources in an MOCVD machine over the course of approximately 600 growth runs. The corrections

needed to achieve a desired growth rate reflect the qualitative behavior of MOCVD systems familiar to most growers: Bubbler sources slowly drift as they are depleted, and large jumps in source behavior occur after new sources are added or major reactor maintenance is performed. Note that variations up to ten percent are exhibited by these sources, demonstrating the need for a simple calibration tool if 1% control is required.

The plot on the left of Fig. 5 illustrates how the ADVISOR method has been used in the MBE growth of a reflectance modulator structure [5]. In this instance, the optical growth rate, nG, was measured for several of the lower layers in the DBR structure during the growth run. These values were used along with an empirical model to make corrections in the deposition recipe for subsequent layers. The growth process was slow enough to allow these modifications to be made without interruption. Also shown are the very limited tolerances placed on this particular device. This short series of runs, which included fine-tuning the empirical model, increased the yield of useful structures by a factor of 2.6. The success of this "recipe update" approach illustrates that real-time control of deposition may not require sophisticated rapid feedback or PID methods to be useful, even for state-of-the art compound semiconductor device structures.

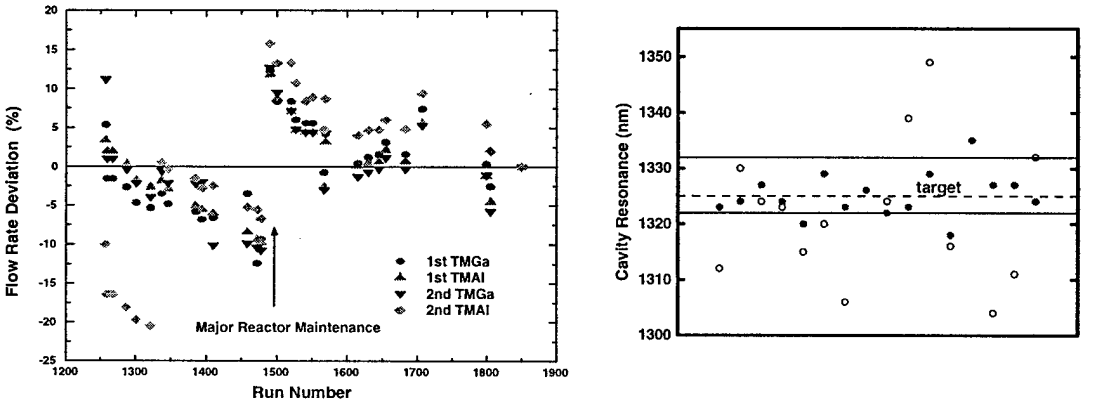

Fig. 5. Left: Corrections made to flow rates for four MOCVD sources based on ADVISOR calibrations. Right: Measured cavity resonance position (solid circles) in MBE-grown device obtained with ADVISOR corrections compared to the estimated cavity position (hollow circles) that would occur if corrections were not made. Dashed line is target value, solid lines bound region for which this device will operate.

SUMMARY

In situ reflectance has become an indispensable routine tool for pre-growth calibration and growth monitoring in our laboratory. It has proved to be an efficient and very precise method for calibrating growth rates for the reactor. Analysis of the complex waveforms produced by reflectance interferometry of multiple-layer films is simplified substantially with the application of the "virtual interface" concept. This concept allows one to extract growth rate and optical constant data from the topmost layer of a growing thin film without having to know any details of the materials or interface positions of underlying layers. As a real time monitor, reflectance has saved a great deal of time and

expense in detecting and identifying problems during growth. Our experience with VCSEL and reflectance modulators demonstrates that there are opportunities to achieve excellent process control without having to resort to sophisticated real time strategies

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