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**"EPITAXIAL LAYER THICKNESS
MEASUREMENTS USING REFLECTION
SPECTROSCOPY"**

by

Lawrence E. Tarof, B Sc.

**A thesis submitted to the Faculty of
Graduate Studies and Research in partial
fulfillment of the requirements for the
degree of**

Master of Engineering.

**Ottawa-Carleton Institute for Electrical
Engineering
Department of Electronics
Faculty of Engineering
Carleton University**

April, 1989

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ISBN 0-315-51170-2

The undersigned hereby recommend to the Faculty of Graduate Studies
acceptance of the thesis

"Epitaxial Layer Thickness Measurements Using Reflection Spectroscopy"

in partial fulfillment of the requirements for the degree of Master of
Engineering.

Prof. N. G. Tarr
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ABSTRACT

The trend toward full wafer processing of III-V semiconductor compound device structures has created a need to obtain pertinent information about epitaxial layers in a non-destructive manner. Since semiconductors are transparent at wavelengths above their bandgap wavelengths the well-known principles of thin film interference can be applied to analyze these layers. Using reflectance spectroscopy, epitaxial layer thickness information has been obtained on a range of III-V compounds incorporated into a variety of heterostructures, including single epitaxial layers, multilayers and layers delineated by very thin markers. Also, it has been shown that the modulation amplitude of the reflectance spectrum is related to the refractive indices of both the epitaxial layer and the substrate, allowing in principle for refractive index determination from reflectance spectroscopy alone.

ACKNOWLEDGEMENTS

In addition to my thesis supervisor, Dr. N.G. Tarr, the assistance of the Advanced Technology Laboratory at Bell-Northern Research is greatly acknowledged. In particular, Dr. A.J. SpringThorpe and P. Mandeville provided the MBE growth, Dr. C. Blaauw and Dr. N. Puetz the MOCVD growth, Dr. I. Bassignana and A. Margittai provided the etched AlGaAs samples and C.C. Tan performed the SEM measurements. Dr. C.J. Miner, to whom my greatest debt is owed, is acknowledged for the modifying the photoluminescence apparatus and performing the measurements on that system, as well as providing constructive comments during the preparation of the manuscript. Dr. C.J. Miner and Dr. A.J. SpringThorpe are further acknowledged for collaboration and useful discussion. Dr. F.R. Shepherd and Dr. H.W. Willemsen are acknowledged for making available the physical resources necessary to do this work, and for useful discussion. Many of the figures and much of the textual material in this thesis is either taken directly from or paraphrased from two publications (Refs. [1,2]).

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LIST OF SYMBOLS AND ACRONYMS

β	quantity denoting optical path at a given wavelength (radians)
Δ	before a variable, denotes a difference quantity
κ	imaginary part of complex index of refraction
λ	wavelength, may be subscripted
$\varnothing$	angle of incidence
θ	angle of incidence
$f/\#$	f-number
i	imaginary number
IR	infrared
m	order of interference, may be subscripted
MBE	molecular beam epitaxy
MOCVD	metal-organic chemical vapor deposition
n	refractive index, may be subscripted
N	complex refractive index, may be subscripted
NA	numerical aperture
r	reflectivity, may be subscripted
r_{ij}	reflectivity at interface ij (Fresnel reflection coefficient)
R	reflectance, may be subscripted
R_0	reflectance of bare substrate
R_1/R_0	modulation amplitude (usually normalized to the reflectance of the substrate)

$R_{\lambda/4}$	reflectance of a " $\lambda/4$ layer" -- i.e. a layer whose thickness at the given wavelength corresponds to a half-integral order of interference
$R_{\lambda/2}$	reflectance of a " $\lambda/2$ layer" -- i.e. a layer whose thickness at the given wavelength corresponds to an integral order of interference
s	as a subscript, refers to the substrate
SEM	scanning electron microscopy
TEM	transmission electron microscopy
$\text{Al}_x\text{Ga}_{1-x}\text{As}$	In this usage, x refers to the mole fraction of the group III (as in this case) or the group V element in a ternary or quaternary alloy

1. INTRODUCTION

1

The trend in optoelectronic device development toward full wafer processing of III-V compounds dictates a need to obtain information about the epitaxial layers over the entire wafer [1,2]. Currently used layer thickness measurement methods such as scanning electron microscopy (SEM), transmission electron microscopy (TEM) and stylus profilometry are destructive in nature, and allow for measurement only at the cost of sacrificing wafer area.

In the case of SEM, a bar is cleaved from the wafer, stained and mounted on a stub prior to actual SEM observation. Only a one-dimensional analysis is possible from a given cleaved bar, and the wafer is no longer whole. Two-dimensional analysis is possible only from cleaving many bars, at the expense of further reducing the size of the wafer remaining. There is also significant sample preparation time required. In the case of TEM the wafer must be cleaved, chemically thinned and ion milled prior to the actual analysis. This preparation is a difficult and time consuming process. Only a few data points per sample are possible, and as in the case of SEM the wafer is no longer whole. In the case of stylus profilometry, the wafer is patterned (usually by standard photolithography) to allow for the selective removal of the epitaxial layers. This allows more easily for two-dimensional analysis wherever the measurement is performed, and the wafer remains whole. However the area of wafer sacrificed increases with the density of the measurement points, there is also actual processing required to selectively remove the epitaxial layers, and the actual act of profiling can damage the surface. Thus with currently available thickness measurement techniques, it is necessary to damage the wafer in order to perform the measurement.

In many applications a critical process step, such as the placement of a Zn diffused junction depth, requires accurate epitaxial thickness information which must be obtained prior to processing the wafer. Obtaining this information in advance is incompatible with retaining the entire wafer intact, and necessitates the development of a non-destructive method for measuring epitaxial layer thickness.

For the wavelengths greater than the bandgap wavelength of a given semiconductor, the semiconductor is transparent and may be regarded as an optical thin film which obeys the well-known laws of thin film interference. Hence a measurement technique based on this principle has the potential for fast, non-destructive thickness evaluation.

Such measurements have been routinely carried out for many years to determine the properties of dielectric layers deposited on a substrate, and to determine the thickness of Si [3] and GaAs [4] epitaxial layers upon a substrate of the same material with a substantially different doping concentration. Dielectric properties have been readily obtained from thin film interference methods typically using visible and near infrared ($<1\ \mu\text{m}$) wavelengths. Two of the most common methods used are spectrophotometry and ellipsometry [5,6]. Usually the thickness alone is determined by spectrophotometry, and both the thickness and refractive index are obtained by ellipsometry. The resolution of the optical techniques is limited by the wavelength used for the analysis; in principle more precision can be obtained by using shorter wavelengths. For Si and GaAs, however, measurements are usually made in the intermediate infrared ($>5\ \mu\text{m}$) because the free carrier effects which give rise to the refractive index differences between media of different doping only become significant at longer wavelengths. For Si and GaAs epitaxial layer thickness measurement a Fourier Transform Infrared (FTIR) apparatus is

typically used for thickness determination only. The precision of the measurement is limited by the long wavelengths used for the analysis.

Similar techniques can in principle be applied to compound heterojunction structures, but in their case, unlike homo-epitaxial Si or GaAs, refractive index discontinuities at the epitaxial layer interfaces are sufficiently large that sharp interference spectra are discernable at much shorter wavelengths. For GaAs- and InP-based alloys these effects become apparent at wavelengths as short as 0.6-2 μm , dependent on the specific alloy. The shorter wavelengths allow for precise non-destructive measurement of epitaxial layers of any composition in these materials systems. Moreover, it is not generally realized that the modulation amplitude of the interference spectra may be interpreted in order to obtain refractive index information from spectrophotometry data alone.

In this work the theory is developed along the lines indicated in the previous paragraph, in order to use reflectance spectroscopy to determine non-destructively both the thickness and refractive index of epitaxial layers in principle. While the thicknesses of dielectric layers and homoepitaxial layers are routinely measured by reflection spectroscopy, in this work the technique is applied to single and multiple epitaxial layers of arbitrary composition in the GaAs- and InP-based materials systems. Although ellipsometry could be used to determine the refractive index of these materials, in this work the theory is developed which allows reflectance data alone to determine refractive index. For the GaAs- and InP-based materials systems, a very simple relationship between the modulation amplitude of the reflectance spectrum and refractive index is developed for the first time, which allows for refractive index determination in the GaAs- and InP-based alloys at any wavelength in the transparent region without the necessity of deconvoluting

ellipsometric measurements. Three different spectrometer systems are used in this work to verify the theory -- a commercial spectrophotometer (Nanometrics AFT-20), a modified photoluminescence (PL) apparatus and a commercial FTIR system. -- to perform Measurements are performed on a variety of III-V heterostructures of GaAs- and InP-based alloys. Fig. 1.1 shows the interrelationship of the spectrometer systems and the particular alloys. Experimental results are given which corroborate and further develop the theory, showing how the technique can be applied to single epitaxial layers, polycrystalline material, multilayers and layers delineated by very thin marker layers.

Fig. 1.1: Relationship among wavelength, appropriate semiconductors and spectroscopic systems used in this work. At the top of the figure the wavelength range covered by the three spectroscopic systems is illustrated. The bandgap wavelength range of the alloys in the AlGaAs and InGaAsP materials systems is illustrated by the diagonal lines in the bottom portion of the figure. For any given bandgap wavelength along the diagonal lines, the material is transparent for wavelengths to the right and opaque for wavelengths to the left. The technique of reflectance spectroscopy is most easily applied at wavelengths for which the epitaxial layers which are transparent.

2. BACKGROUND AND THEORY

2.1 Thin film interference

Thin film interference arises from the refractive index discontinuity between any two media [7,8]. Typically this phenomenon is used to advantage in the measurement of the thickness and refractive index of dielectric films. Since at wavelengths above their bandgaps semiconductors may be regarded as optical thin films, the same principles may be extended to the refractive index discontinuity between an epitaxial layer and the substrate.

2.2 Applications

There are many common applications of thin film interference in the semiconductor field. As noted above, the phenomenon is used in the measurement of the thickness and refractive index of dielectric films. Among the most common are SiO_2 and Si_3N_4 . Also the thickness of polysilicon films may be measured. Commercial units are readily available for these measurements using both spectroreflectometry and ellipsometry. These techniques are discussed in the next section.

Additionally it has long been recognized that epitaxial layer thicknesses of Si on a Si substrate and GaAs on a GaAs substrate (of different doping levels) can be measured in the intermediate or far infrared wavelength range [3,4]. Typically an FTIR system is used at these wavelengths. This is an

advantage over classical reflection spectroscopy in the infrared because all the light is collected at once, allowing for a greater signal-to-noise (S/N) ratio. This is an advantage only in the case where the measurement is detector noise limited. In the visible wavelength region, where bright sources and Si photomultiplier tubes are available, the system is shot noise, not detection noise limited, and the Fourier transform does not provide an advantage [5].

Epitaxial layers in heterostructures are characterized by refractive index discontinuity at shorter wavelengths than for the corresponding homostructures, and so a reflectance spectrum should undergo oscillations. This prediction is illustrated in Fig. 2.1 for the case of a higher index epitaxial material on a lower index substrate. Analysis of the reflectance spectrum should be possible by using the well-known Fresnel coefficients, or equivalently by using transfer matrix analysis. This will be discussed below.

2.3 Spectrophotometry and ellipsometry

Two of the most common methods used to determine thin film properties are spectrophotometry (typically thickness only) and ellipsometry (thickness and refractive index). With spectrophotometry the reflection or transmission of the sample is measured as a function of wavelength. With ellipsometry the polarization of the light incident upon the sample is varied, typically the reflected light is analyzed.

The most commonly seen ellipsometric apparatus uses a single wavelength (often the 632.8 nm output of a low power He-Ne laser) and measures the reflectance as a function of polarization. Recently the same principle has also been used by Sagax [6] with a broadband rather than monochromatic light source. Another recent development in ellipsometry is (variable angle) scanning ellipsometry [6,9], which allows reflectance to be measured not only as a function of polarization, but of angle of incidence and wavelength as well. Aspnes [10] is currently using spectroscopic ellipsometry as an in-situ growth analytical tool. The Sagax method involves no computation but requires a variable thickness calibration standard as an integral part of the measurement. All the other methods involve the measurement of the so-called delta and psi values, from which both the refractive index and thickness of the layer may be calculated.

Ellipsometry measures both amplitude and phase information using so-called delta and psi values [5,9]. Ellipsometry is potentially a very powerful tool for obtaining information about thin layers but has its own set of drawbacks. For layers of optical path $2m\pi$ (m an integer) no information can be obtained [5], and near these values the measurements contain considerable error as all the possible solutions converge. The same delta and psi are

measured for films which differ in optical thickness by any multiple of 2π , and it is not possible to determine which is the correct multiple of 2π without using multiple angles or wavelengths. Also, it is not easy to develop an intuitive feel for what delta and psi values mean.

Spectrophotometry measures amplitude information alone, and is not usually used to measure the refractive index of the dielectric films. Rather, usually the thickness alone is determined. Throughout the spectral range the reflectance (most commonly used) exhibits oscillations periodic with respect to the wavenumber (reciprocal wavelength) as illustrated in Fig. 2.1. This allows for easy and intuitive interpretation of the spectra.

In this work spectrophotometry alone is used to determine properties of epitaxial thin films. This is both for practical reasons and by design. Although spectroscopic ellipsometry is currently available, the technique is new and most ellipsometers commercially available today function at a single wavelength only. The epitaxial thin films investigated in this work have a wide range of bandgaps, and therefore the optimal wavelength to use is different depending on the material(s) used. While the appropriate wavelength range is nearly covered by semiconductor diode lasers, the best information is obtained for collimated light, introducing an added complexity to using ellipsometry above and beyond even that of interchanging several lasers as sources. The shortest commercially available wavelength transparent to all the semiconductors investigated here is a HeNe laser which operates at a wavelength of $2.4\text{ }\mu\text{m}$, for which a quarter wavelength is approximately $0.2\text{ }\mu\text{m}$ in the semiconductor. This would pose an unnecessary restriction of both how thin an epitaxial film and how precisely this film can be measured. On the other hand, spectrophotometry offers the capability of a continuous

range of wavelengths, so the particular wavelengths desired are readily available.

The main reason why ellipsometry is often used rather than spectrophotometry is because the refractive index n can be extracted [5,6]. It is not generally realized that n may also be extracted from the spectrophotometry data as will be discussed in Section 2.5. Furthermore, both the thickness and refractive index information are intuitive and easy to interpret from spectral data, whereas δ and ψ values are not intuitive and therefore easy to misinterpret. Both thickness and refractive index measurements by spectrophotometry are discussed below.

Fig. 2.1: Simulated reflectance spectrum for 2.5 μm thick InGaAs on InP normalized to the reflectance of the InP substrate. Minima occur where m , the order of interference, is an integer (where the normalized reflectance is 1). Maxima occur where m is a half-integer. The normalized reflectance at the maxima depends on n and n_s , both of which vary slightly as a function of wavelength. For this reason the modulation amplitude varies slightly throughout the spectral range, which is given in μm . [2]

2.4 Thickness measurement

The transfer matrix method [7,8] is used as follows. For a single film layer the matrix elements are given by

$$M = \begin{bmatrix} A & -iB \\ -iC & D \end{bmatrix} = \begin{bmatrix} \cos \beta & -in \sin \beta \\ -(i/n) \sin \beta & \cos \beta \end{bmatrix} \quad (2.1)$$

where

$$\beta = 2\pi n t \cos \theta / \lambda \quad (2.2)$$

where n is the refractive index, t is the film thickness, θ is the angle of incidence and λ is the wavelength of light. For multilayer stacks the transfer matrix is given as the product of the individual transfer matrices:

$$M = M_1 M_2 M_3 \dots M_N \quad (2.3).$$

The reflectivity r of an arbitrary stack of dielectrics is given from the transfer matrix method as

$$r = \frac{An_0 + iBn_0n_s - iC - Dn_s}{An_0 + iBn_0n_s + iC + Dn_s} \quad (2.4).$$

The reflectance R is the the modulus squared of r . In general r is complex. For epitaxial III-V layers on GaAs or InP, absorption may be neglected in the near IR (see section 2.6), greatly simplifying the measurement of film thickness. In this case R is given by

$$R = \frac{(An_0 - Dn_s)^2 + (Bn_0n_s - C)^2}{(An_0 + Dn_s)^2 + (Bn_0n_s + C)^2} \quad (2.5).$$

For the case of a single film layer equation (2.5) may be rewritten as

$$R = \frac{\cos^2\beta(n_0 - n_s)^2 + \sin^2\beta((n_0n_s/n) - n)^2}{\cos^2\beta(n_0 + n_s)^2 + \sin^2\beta((n_0n_s/n) + n)^2} \quad (2.6).$$

From equation (2.6) it can be seen R is periodic with respect to β , or $1/\lambda$. This periodicity is illustrated in Fig. 2.1. Extrema of reflectance occur for those wavelengths at which

$$2nt \cos \theta = m \lambda \quad (2.7),$$

where m is the order of interference of the film. At normal incidence the $\cos \theta$ term may be neglected, and equation (2.7) becomes simply

$$2nt = m \lambda \quad (2.8).$$

The case where m is an integer corresponds to films of thickness $\lambda/2$, λ , $3\lambda/2$,.... The reflectance $R_{\lambda/2}$ is the same as for the reflectance R_0 of the bare substrate, given in this case by

$$R_{\lambda/2} = R_0 = \frac{(n_0 - n_s)^2}{(n_0 + n_s)^2} \quad (2.9).$$

The case where m is a half-integer corresponds to films of thickness $\lambda/4$, $3\lambda/4$,

$5\lambda/4, \dots$. The reflectance $R_{\lambda/4}$ is given in this case by

$$R_{\lambda/4} = \frac{(n_0 n_s - n^2)^2}{(n_0 n_s + n^2)^2} \quad (2.10).$$

It is apparent from examination of equations (2.9) and (2.10) that $R_{\lambda/4} > R_{\lambda/2}$ if $n > n_s$, and vice versa. The thickness of the film may be determined by using equation (2.7) or (2.8) for two wavelengths λ_1 and λ_2 . If the indices of the film at these two wavelengths are given by n_1 and n_2 and the orders of interference are m_1 and m_2 respectively then analysis of equation (2.8) yields

$$t = \frac{\Delta m}{2} \frac{\lambda_1 \lambda_2}{n_1 \lambda_2 - n_2 \lambda_1} \quad (2.11),$$

where

$$\Delta m = m_1 - m_2 \quad (2.12).$$

Suppose the refractive index dispersion is at most linear with respect to wavelength, i.e.

$$n(\lambda) = n(0) + \frac{dn}{d\lambda} \lambda \quad (2.13),$$

where $n(0)$ is the refractive index extrapolated back to zero wavelength [11]. In this case equation (2.11) may be written in the more simple form

$$t_{\text{optical}} = n(0)t = \frac{\Delta m}{2} \frac{\lambda_1 \lambda_2}{\lambda_2 - \lambda_1} \quad (2.14),$$

where in this equation the optical thickness t_{optical} [1] is defined for n at zero wavelength (t_{optical} varies as n with wavelength). An algorithm based on equation (2.14) allows for a simpler epitaxial layer thickness calculation. If

equation (2.13) is not true then $n(0)$ changes for different sets of (λ_1, λ_2) . Therefore any algorithm for thickness measurement based on equation (2.14) must use a fixed (λ_1, λ_2) if the refractive index dispersion is non-linear.

2.5 Refractive index measurement

The epitaxial layer thickness of a film is commonly measured in many applications as outlined above. It is less well known that not only the thickness, but also the refractive index of a film may be measured with this technique [5,6].

As pointed out in the last section the reflectance R oscillates between two extremes given by $R_{\lambda/4}$ and $R_{\lambda/2}$ from equations (2.9) and (2.10). In other words the reflectance modulates from R_0 , the reflectance of the bare substrate, with a modulation amplitude R_1 (not yet normalized to R_0), which may be defined as

$$R_1 = |R_{\lambda/2} - R_{\lambda/4}| \quad (2.15).$$

The ratio of the unnormalized modulation amplitude to the base amplitude (R_1/R_0), henceforth referred to as the modulation amplitude, is thus given by

$$\frac{R_1}{R_0} = \frac{R_{\lambda/2} - R_{\lambda/4}}{R_{\lambda/2}} = 1 - \left(\frac{(n_0 n_s - n^2)(n_0 + n_s)}{(n_0 n_s + n^2)(n_0 - n_s)} \right)^2 \quad (2.16).$$

This equation may be solved for general n , but a more simplified expression can be derived for the case of an epitaxial layer upon a substrate, for which n is close to n_s and $n_0=1$. If the refractive index difference Δn between the substrate and epitaxial layer is defined as

$$\Delta n = n - n_s \quad (2.17),$$

then for $\Delta n \ll n, n_s$, analysis of equation (2.16) yields

$$\Delta n = \frac{R_1}{R_0} \frac{n_s^2 - 1}{8} \quad (2.18).$$

Equation (2.18) and the noise in the optical measurement place a constraint on the smallest refractive index difference that can be resolved. This in turn determines the smallest compositional difference between substrate and epitaxial layer which will give rise to discernable oscillations. Another application of Eq. (2.18) is to measure in principle Δn solely from the modulation amplitude of a reflectance spectrum, and hence n as well if n_s is known. Usually this sort of measurement is restricted to ellipsometric methods.

2.6 Errors in measurement

It is important to realize that there are experimental errors involved in the reflection spectroscopy measurement technique.

The first consideration with this technique is knowing under what conditions the absorption of the substrate may be safely neglected. Consider a substrate of complex index of refraction $N_s = n_s + i\kappa_s$. In the absence of a film upon the substrate, for normal incidence in air, the Fresnel reflection coefficient r_{0s} is given by

$$r_{0s} = |r_{0s}|e^{i\phi_{0s}} = \rho_{0s}e^{i\phi_{0s}} = \frac{1-N_s}{1+N_s} \quad (2.19),$$

where ϕ_{0s} is the phase change upon reflection and ρ_{0s} is the amplitude of the reflectivity. Since $e^{i\phi_{0s}} = \cos \phi_{0s} + i \sin \phi_{0s}$, this phase change may be calculated from

$$\tan \phi_{0s} = \frac{\sin \phi_{0s}}{\cos \phi_{0s}} = \frac{\text{Im}(r)}{\text{Re}(r)} \quad (2.20).$$

Substituting equation (2.19) into (2.20) gives the phase change ϕ_{0s} :

$$\phi_{0s} = \tan^{-1} \left(\frac{2\kappa_s}{1 - n_s^2 - \kappa_s^2} \right) \quad (2.21).$$

where $\tan^{-1}(0)$ is π as κ_s approaches zero since $n_s > n_0$. The reflectance is given as $R = |r|^2$ from equation (2.19):

$$R = \frac{(1-n_s)^2 + \kappa_s^2}{(1+n_s)^2 + \kappa_s^2} \quad (2.22).$$

Note that for a transparent substrate Eqs. (2.21) and (2.22) simplify to the familiar Eq. (2.9) and

$$\phi_{0s} = \tan^{-1}(0) \quad (2.23).$$

The error in R , ΔR , in assuming $\kappa_s=0$ is given by

$$(\Delta R)^2 = \left(\frac{\kappa_s^2}{n_s - 1} \right)^2 + \left(\frac{\kappa_s^2}{(n_s + 1)^2} \right)^2 \quad (2.24).$$

Rearranging the above equation for κ_s ,

$$\kappa_s = \left(\frac{(n_s + 1)^4 (n_s - 1)^4}{(n_s + 1)^4 - (n_s - 1)^4} \right)^{0.25} (\Delta R)^{0.5} = 2.24 (\Delta R)^{0.5} \quad (2.25),$$

assuming $n_s=3.2$. For a reflectance accuracy of $\Delta R < 10^{-4}$, the absorption must satisfy $\kappa_s < 2.24 \times 10^{-2}$. Note that κ , the imaginary part of the complex refractive index, not α , the absorptance, is specified in Eq. (2.25), although α and κ are related by $\alpha = 4\pi\kappa/\lambda$. This requirement (Eq. (2.25)) is met for many of the materials used in this study, especially in the transparent regime. Thus the index of the substrate is accurately given by inverting equation (2.19) with $\kappa_s=0$:

$$n_s = \frac{1 + R^{0.5}}{1 - R^{0.5}} \quad (2.26).$$

In an experimental situation the accuracy with which n_s is determined depends on the signal-to-noise (S/N) ratio -- i.e the accuracy to which R may be measured.

For the case of a transparent film on an absorbing substrate the reflectance R is given by ([7], p.632, Eq. (6))

$$R = \frac{r_{01}^2 + \rho_{1s}^2 + 2r_{01}\rho_{1s}\cos(\phi_{1s} + 2\beta)}{1 + r_{01}^2\rho_{1s}^2 + 2r_{01}\rho_{1s}\cos(\phi_{1s} + 2\beta)} \quad (2.27)$$

where ϕ_{1s} is the phase change upon reflection and ρ_{1s} is the amplitude of the reflectivity at the film-substrate interface. Note that the two interfaces indicated by the subscripts are the air-film interface (01) and the film-substrate interface (1s). Since both of the below are true

$$-1 \leq \cos(\phi_{1s} + 2\beta) \leq +1 \quad (2.28)$$

$$0 < |r_{01}|, |\rho_{1s}| < 1 \quad (2.29)$$

it may be shown that equation (2.20) has extrema where the cos term is ± 1 . If the two extrema are termed R_{-1} and R_{+1} it evident that

$$R_{+1} = \frac{(r_{01} + \rho_{1s})^2}{(1 + r_{01}\rho_{1s})^2} \quad (2.30).$$

$$R_{-1} = \frac{(r_{01} - \rho_{1s})^2}{(1 - r_{01}\rho_{1s})^2} \quad (2.31)$$

Eqs. (2.30, 2.31) simplify to Eqs. (2.9, 2.10) respectively for the case where $\kappa_s = 0$. Simulations of R vs. λ using Eq. (2.27) have been done for various κ_s in the range 0 to 0.3. The results are shown in Fig. 2.2. Unlike the case of the substrate alone, for which κ_s has a small effect on the reflectivity, the modulation amplitude of the spectra depends more sensitively on the value of κ_s . Note also that the actual reflectance at the $\lambda/2$ extremum overshoots R_0 .

Thus for the case of an absorbing substrate Eq. (2.18) could not be used to predict the refractive index of the film.

Usually it is not the refractive index of the substrate but rather the thickness or refractive index of the epitaxial film which is of experimental interest. Since $R_{\lambda/4}/R_{\lambda/2}$ in equation (2.16) is of order 1, the primary error in experimentally measuring Δn is the accuracy of $R_{\lambda/4}$ relative to $R_{\lambda/2}$, and not the absolute value of $R_{\lambda/2}$ itself, which from equation (2.19) has a relatively weak effect on an experimentally measured n_s . Thus systematic errors in calibrating absolute values of R will tend to nearly offset each other for purposes of determining Δn . Note that it is Δn which is of importance for both alloy composition determination and certain waveguiding applications. Independent errors in $R_{\lambda/4}$ and $R_{\lambda/2}$, however, will tend to have a more significant effect on the accuracy of Δn . To illustrate, for $\Delta n = -0.1$, $n_s = 3.6$, the measured R_1/R_0 value should be 7%. If both $R_{\lambda/4}$ and $R_{\lambda/2}$ are off by 1% in the same direction, the change in Δn is dominated by the n_s^2 term in equation (2.18), and the error in Δn has a value of 1%, or 0.001. If, however the experimentally measured R_1/R_0 value is 6% or 8%, the calculated Δn would be in error by 14%, or 0.014.

Another source of error involves the tradeoff in choice of bandpass. Using a small bandpass prevents truncation of the peaks of the reflectance spectrum which would reduce the measured value of R_1 , but at the expense of less signal, which reduces the accuracy with which R_1 may be determined. The optimum value of bandpass to use depends on the actual system configuration. To illustrate the extent to which R_1 may be truncated in a typical experimental situation, consider the example of an epitaxial film for which a peak occurs at a certain wavelength (λ_{peak}). From equation (2.11) or (2.14) it is possible to determine the wavelength spacing, defined as $\Delta\lambda_{\text{peak}}$,

between adjacent peaks. Define the bandpass as $\Delta\lambda$. Assuming a sinusoidal reflectance function near the extrema, then the truncation at the peak would be given by

$$\frac{\text{measured } R_1}{\text{theoretical } R_1} = \frac{\int \cos x' dx'}{\int dx'} \quad (2.34)$$

where the limits of integration of $x' = (\lambda - \lambda_{\text{peak}}) / \Delta\lambda_{\text{peak}}$ are $\lambda_{\text{peak}} - \Delta\lambda/2$ and $\lambda_{\text{peak}} + \Delta\lambda/2$. The right hand side of Eq. (2.34) is given by $\frac{\sin x}{x}$, which tends to 1 in the limit of small x . The Taylor series expansion is easily seen to be

$$\frac{\text{measured } R_1}{\text{theoretical } R_1} = 1 - \frac{x^2}{3!} + \dots \quad (2.35)$$

For the example of $\Delta\lambda, \Delta\lambda_{\text{peak}} = 5 \text{ nm}, 100 \text{ nm}$ respectively, the ratio expressed in Eq. 2.35 is 0.9998, or a truncation of 2×10^{-4} . A similar truncation would occur at the opposite extreme as well, so the total truncation would be 4×10^{-4} for this particular example. If the peaks were much closer together than $0.1 \mu\text{m}$ (i.e. a thicker epitaxial layer) then this effect would be significant.

A similar argument applies to the non-normal incidence due to the finite focal length of a given lens. A range of incident angles is used, weighted more toward the larger angles (further from normal) because each successive angular increment fills a larger solid angle Ω . In this case the error in assuming normal incidence is given by the average of the cos function in standard spherical coordinates:

$$\text{Mean } (\cos \Omega) = \frac{\int \cos \Omega' d\Omega'}{\int d\Omega'} \quad (2.36)$$

κ in substrate:

—————	0
-----	.1
- . - . - .	.2
-----	.3

Fig. 2.2: Simulated reflectance spectra for various κ_s . The index of the substrate and film are taken as 3.55 and 3.2 respectively and is meant to simulate InP on InGaAs. At a wavelength of 1 μm , κ of 0.1 corresponds to α of 0.13 μm^{-1} .

$$= \frac{\int_0^{2\pi} \int_0^\theta \cos \theta' \sin \theta' d\theta' d\phi'}{\int_0^{2\pi} \int_0^\theta \sin \theta' d\theta' d\phi'} \quad (2.37) \quad 23$$

$$= \frac{1 + \cos \theta}{2} \quad (2.38),$$

where θ is the maximum half-angle of the cone of solid angle Ω . For small θ the quantity in Eq. (2.38) is easily seen to be $1 - \theta^2/4$. Some examples of Eq. (2.38) are as follows. For an NA of 0.33, 0.25, or 0.1, the error is 3.2%, 1.6%, or 0.3% respectively. Thus it is seen that for a sufficiently low NA lens or not filling a somewhat higher NA lens, the correction is negligible.

2.7 Film thickness, wavelength and bandpass requirements

There are two constraints on film thickness requirements. Films as thin as a quarter wavelength of the bandgap ($\lambda_g/4n$) may be measured without resorting to the use of Fresnel coefficients. In the III-V materials systems most commonly used this translates to 50 nm of AlGaAs or 70-120 nm of InGaAsP lattice matched to InP, depending on the specific composition. At the opposite extreme Nyquist's theorem, which states that a given frequency of modulation (in this case spatial) may be measured only for a sufficiently small sampling bandpass, places an upper limit on the film thickness, which in practice is hundreds of μm . These limits may be summarized as follows:

$$\lambda_g/4n < t < \lambda^2/4n\Delta\lambda \quad (2.39)$$

where $\Delta\lambda$ is the bandpass of the monochromator. The range of wavelengths which may be used is in between the bandgap wavelength λ_g and the point where free carrier absorption of the film becomes significant. Free carrier absorption [12] increases with both wavelength (proportional to $\lambda^{1.5}-\lambda^2$) and doping level, so the upper wavelength which may be used is dependent on the doping level of the film.

2.8 Limits to light throughput

The limits to light throughput are defined by the source, the monochromator system (including lensing) and the sample. As a reference point for how much light is needed, the noise equivalent power (NEP) of a typical PbS detector capable of operating from 1-4 μm is roughly 1 pW, if used with a lock-in amplifier at an operating frequency of 750 Hz. The sample limits the light available for detecting simply because of its reflectivity. For the samples studied in this work, this is approximately 30% of the light received through the monochromator. It is worth discussing the remaining limits in more detail.

The source output is limited by the Planck blackbody radiation limit. For a tungsten filament operating at 3000°C, the total output density per unit filament area over the entire spectrum is limited to approximately 10 W/mm². According to Wien's law, the maximum output of the spectrum is at the wavelength given by (2898 $\mu\text{m}\cdot^\circ\text{C}/\text{temperature}$), which is approximately 1 μm , declining by a factor of 10 at 3 μm , in this case. A common misconception is that the maximum output at any given wavelength is at the temperature for which that wavelength is a spectral

maximum from Wien's law. In fact, elementary examination of the Planck blackbody equation shows that for every wavelength, the higher the temperature of the source, the greater the spectral output, assuming constant emissivity. Therefore the intensity achievable in a broadband source in any wavelength regime is ultimately limited by the temperature of the source. Another common misconception is that a higher power source operating at a given temperature will necessarily deliver more usable power to the monochromator. Actually, a higher power source merely means a larger filament, but the usable power to the monochromator is limited by the size of filament which can be imaged onto the monochromator entrance (larger for a higher NA lensing system) and the f number ($1/(2 \text{ NA})$) of the monochromator. The lower NA of these two system components imposes the actual system limit, and it is only worth increasing the output power of the source until the point where the monochromator entrance aperture is filled.

For a given source of finite output, the f number of the monochromator imposes the theoretical system limit on how much light the sample receives. Consider first the light output from the monochromator. For a given monochromator slit width there is a tradeoff between using a high f number (low NA) to achieve better monochromaticity due to lower dispersion of the grating or using a low f number (high NA) to achieve more throughput due to higher grating dispersion. For sufficiently thin slit widths ($< 1 \text{ mm}$) where the dispersion is linear, the difference in grating dispersion may be offset by changing the slit width to achieve the same output, with somewhat less stray light from the higher f number monochromator. Next consider the light input to the monochromator. The use of a lensing system in directing the light from the source to the monochromator can only

approach the theoretical limit of having the source adjacent to the monochromator entrance, and not improve upon it. Note, though, that the use of back-reflection from the source can in principle double the available light within the specified NA. The solid angle of the light output from the source which may be usefully received is matched to the NA of the monochromator.

As an example of how the monochromator choice affects a practical system designed for high throughput, consider a tungsten filament source. For a slit size of 1mm width and 5 mm height, the maximum power at a wavelength of 1 μm for monochromators of f numbers $f/4$ and $f/7$ is 78 and 25 μW , respectively. The bandpass is approximately 12 and 7 nm respectively, assuming 600 line/mm gratings. These light levels would be reduced by the emissivity of the source, grating efficiency, reflections within the monochromator and off all the optics, as well as any beamsplitters for viewing optics or simultaneously acquiring a reference (substrate) reflectance spectrum. Allowing a factor of between 10 and 100 for all of this, it should still be theoretically possible to obtain a S/N ratio of more than 10^5 at 1 μm wavelength, 10^4 at 3 μm wavelength.

3. EXPERIMENTAL

3.1 Growth of material

Epitaxial layers of InGaAsP were grown on 51 mm InP substrates by metal-organic chemical vapor deposition (MOCVD) in a modified CVT horizontal reactor. The lattice matching was verified from X-ray rocking curve data. Epitaxial layers of AlGaAs and GaAs were grown on 76 mm GaAs substrates by molecular beam epitaxy (MBE) using a VG-80 reactor. As well, GaAs layers were grown on Si substrates by MBE.

3.2 Spectral measurement systems

Reflectance data were recorded in the spectral region 0.5-10 μm by use of three distinct spectral systems: a commercial spectrophotometer (Nanospec AFT-20), a modified photoluminescence (PL) setup and a conventional Fourier Transform Infrared (FTIR) spectrometer (Perkin-Elmer 1800). Each system will be discussed below.

The commercial spectrophotometer covers the wavelength range 0.5-0.8 μm and is routinely used for the measurement of dielectric film thicknesses. Since the normal output to the user is a film thickness rather than a digitized reflectance spectra, the unit was somewhat less versatile than a genuine research tool might have been. However the reflectance spectra is available to the user in the form of an analog chart recorder output. The S/N obtained was better than 2000:1 (measurement limited) although there was an

apparent shift in overall output of 1-2 percent from scan to scan. The spot size diameter was 35 μm . The spot size is determined by the numerical aperture (NA) of the lens; there is a decrease in spot size as the NA (and magnification) increases. For this spot size the NA of the lens is 0.33, but the lens is not nearly filled, resulting in an actual NA of less than 0.1. From the results of Sec. 2.6, corrections for non-normal incidence may to a good approximation be neglected. This spot size is small enough, however, to allow for microscopic information about the wafer to be obtained.

To test the sensitivity of a measured thickness to both microscope objective magnification (NA) and focussing error (which fills more of the lens, resulting in a higher NA), an 1200 $\text{\AA}$ SiO_2 film was measured using the standard Nanospec routine at 5, 10 and 20x magnification (NA of 0.1, 0.33 and 0.67 for the lens, respectively) in focus, and at 10x magnification up to 10 μm out of focus. The measured thickness varied by a few $\text{\AA}$ with respect to magnification and 10 $\text{\AA}$ with respect to focal distance. This is experimental evidence that non-normal incidence may be neglected for wafers in focus.

The modified PL setup, as illustrated in Fig. 3.1 and described in reference [2], can be used in the wavelength range 0.5-2 μm . The spot size for the 0.25 NA lens selected was 35 μm . These spectral limits are determined by the grating selected for dispersion of the light, the spectral output obtainable from a standard tungsten filament source (which may be regarded as a blackbody source whose spectral output peaks at a wavelength of 1 μm), and the usable spectral range of the cooled InAs detector. The monochromator used had an f number of f/6.4, so the error in assuming normal incidence is 0.2% from Eq. (2.34). The S/N ratio using a lock-in detection system was 1000:1 in the range 1-1.5 μm . This could be improved considerably by use of a lensing system (Sec. 2.8). One key feature of the PL system is that conventional PL

Fig. 3.1: Schematic for modified photoluminescence (PL) system. The chopped light is detected and amplified using a lock-in amplifier. Two illumination sources are used: a tungsten lamp for the reflectance spectroscopy and a HeNe laser for standard PL. The spot size is 35 μm . The PL and reflectance spectra may be recorded in exactly the same place on the wafer. [2]

may be done in addition to reflectance spectroscopy at the same point on the wafer.

The FTIR system operates in the wavenumber (wavelength) range 5000-450 cm^{-1} (2-21 μm). For doped substrates the plasma frequency [12] was observable for wavelengths greater than 10 μm , so the scans were generally terminated at a shorter wavelength to avoid confusion in interpreting the results. This particular system is a double beam spectrometer which in principle allows for the instantaneous ratio of the spectra with and without the epitaxial film. This is strictly true for transmission, but as only one reflectance attachment was available the spectra were acquired in single beam mode. Even so, the S/N ratio of the ratioed scans was greater than 1000:1 over the spectral range 4700-1000 cm^{-1} . Note that the use of a lower temperature source (glowbar of approximately 1600° C) and higher NEP detector (HgCdTe) than in the other systems means that the maximum theoretical S/N ratio is also lower. The spot size diameter was approximately 6mm, much larger than in the other systems. This could be reduced to tens of μm by the use of an infrared microscope attachment, but such an attachment was not available for these experiments. The large spot size, while not convenient for studying epitaxial layer uniformity, guaranteed a low system NA. Therefore the refractive index could be studied without worrying about corrections for non-normal incidence.

The wavelength range for the Nanospec unit can be used for the most precise possible measurements of $\text{Al}_x\text{Ga}_{1-x}\text{As}$ thicknesses ($x < 0.2$), because this range covers the region just above the bandgap wavelength of the $\text{Al}_x\text{Ga}_{1-x}\text{As}$ layers. The wavelength range for the modified PL system can be used for analyzing most $\text{Al}_x\text{Ga}_{1-x}\text{As}$ and $\text{In}_x\text{Ga}_{1-x}\text{As}_y\text{P}_{1-y}$ compositions, but for the greatest S/N ratio this unit works best for InP or any $\text{Al}_x\text{Ga}_{1-x}\text{As}$ composition.

The wavelength range for the FTIR unit is appropriate for either the $\text{Al}_x\text{Ga}_{1-x}\text{As}$ or $\text{In}_x\text{Ga}_{1-x}\text{As}_y\text{P}_{1-y}$ system. The refractive index dispersion is nearly linear in this wavelength range, so $n(0)$ is nearly constant. However, thinner layers cannot be measured as precisely in this system as in the other systems, as the wavelengths are larger.

4. RESULTS AND DISCUSSION

To illustrate the use of reflection spectroscopy in the practical evaluation of epitaxial layer thicknesses, measurements made on several distinct heteroepitaxial systems are described. In these examples confirmation of the technique through corroborating evidence is presented, and different aspects of the technique are highlighted, as applied to single epitaxial layers, non-stoichiometric materials, multilayers and layers delineated by very thin markers.

4.1 GaAs on Si

The deposition of GaAs on Si has recently attracted much attention due to the potential for optical and electronic integration of these two materials [13]. These heteroepitaxial structures are also simple systems for the application of reflection spectroscopy. Furthermore, it is possible to carry out accurate mechanical thickness measurements on the same structure by stylus profilometry to confirm the optical measurements. In this way comparative measurements of GaAs on Si have been made both to validate the reflection spectroscopy technique and to establish the uniformity of flux distribution in the MBE system.

Following the deposition of approximately 1 μm of polycrystalline GaAs onto 76 mm diameter Si substrates sharp interference spectra were recorded in the wavelength range 1-1.4 μm using the modified PL system in

order to measure the variation in thickness across the wafer, and hence the flux variation within the reactor. Spectral scans were carried out at 2 mm intervals across the wafer diameter. Despite the polycrystalline nature of the GaAs, the epitaxial surfaces were optically smooth. Some of these measurements are illustrated in Figure 4.1 where, for clarity, only every fourth scan is shown. The change in wavelength ($\Delta\lambda$) of the spectral peak of any given order, from scan to scan, is directly related to the change in both thickness and refractive index of the layer. From equation (2.8) this is seen to be

$$\Delta(nt) = (m/2)\Delta\lambda \quad (4.1)$$

Thus it is only necessary to follow the variation of the peak position of one particular order across the wafer to map the thickness variation. This is illustrated in Figure 4.1 by the dashed lines, which join peaks of a particular order of interference on adjacent scans. Since for the case of polycrystalline GaAs on Si refractive indices are not accurately known, only optical thickness (nt) variations are determined from the above equation. If the variation in n is small across a wafer then an evaluation of relative thickness uniformity is obtained. Experimentally the lack of significant variation in R_1 (see Section 2.5) is evidence that such is indeed the case. For this particular wafer the variations are so large that the thickness variation is obviously being measured. For a wafer with less variation the relative contributions of thickness and composition uniformity should be examined more carefully.

A mechanical cross-check of the optical measurement was carried out by photolithographically defining 100 μm wide channels, at 2 mm intervals, in the deposited layer. The GaAs in the channels was selectively removed by etching with an ammonia/hydrogen peroxide based etch, which does not

attack Si. Dektak (stylus profilometry) measurements were then made of the channel depths. A comparison of the relative thickness variation obtained by the mechanical and optical techniques is shown in Figure 4.2. As can be seen, the results of the optical and (destructive) mechanical techniques are in excellent agreement. They also clearly demonstrate the poor flux uniformity of the MBE deposition system used to prepare the samples.

This algorithm (based on equation (4.1)) is similarly valid even for very complex heterostructures provided only a relative measurement of thickness uniformity across the wafer is required. In this case (nt) in equation (4.1) is replaced by the sum of (nt) over all the layers. Note that the contribution to (nt) can arise both from thickness and compositional variation. For the cases where the compositional variation is small, thickness uniformity information alone is obtained.

Fig. 4.1: Spectra at successive points on a wafer are recorded for epitaxial GaAs on a Si substrate using the modified PL system. Adjacent scans corresponds to points spaced 8 mm apart across a whole wafer. Each dashed line joins peaks of a particular order of interference. By following just one of these peaks across a wafer the relative thickness uniformity across the wafer may be determined, as shown in the next figure. The top scan corresponds to a measured (stylus profilometry) thickness of 3.907 μm , the middle to 4.083 μm and the bottom to 3.937 μm . [2]

Fig. 4.2: Wafer map: comparison of relative thickness uniformity of an MBE wafer using reflectance spectroscopy and stylus profilometry [2].

4.2 AlGaAs on GaAs

The deposition of $\text{Al}_x\text{Ga}_{1-x}\text{As}$ on GaAs is of interest for a variety of specific optoelectronic applications, and such structures are convenient to demonstrate the reflection spectroscopy technique. Unlike Si, GaAs is easily cleaved into bars with cleavage surfaces smooth enough for microscopy without extensive additional wafer preparation such as thinning. This easily permits a physical cross-check of the optical measurements with methods other than stylus profilometry. To demonstrate the use of reflectance spectroscopy over a range of epitaxial thicknesses, samples of differing layer thicknesses of specific Al compositions have been prepared. In this way comparative measurements of $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$, $\text{Al}_{0.5}\text{Ga}_{0.5}\text{As}$ and $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ have been made.

Approximately $2.5\text{ }\mu\text{m}$ of $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ and $3.4\text{ }\mu\text{m}$ of $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ were deposited onto 76 mm GaAs substrates. The compositions were nominal, but the error in x is expected to be no more than 0.03. To generate samples of differing layer thicknesses in the range 0.1 to 4 mm, the wafers were cleaved into several pieces which were wet chemically etched for different lengths of time. Bars of these samples were oxide coated, cleaved and stained, and then measured both by scanning electron microscopy (SEM) and reflectance spectroscopy. The JEOL-35C unit, which was calibrated, is accurate to $0.05\text{ }\mu\text{m}$ at a magnification of 10000x. Reflectance spectra were recorded in the wavelength range 480-800 nm using the Nanospec.

Figure 4.3 shows typical spectra. Note that the spectral range in which sharp interference can be recorded is only a subset of the scanned range. The useful spectral range for $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ is greater than for $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ because λ_g is lower for greater Al content. The interference fringes allow the layer

Fig. 4.3: Measured reflectance spectra for AlGaAs on GaAs recorded using the Nanospec. (a): 2.23 μm thick $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$; (b): 0.80 μm thick $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$. The usable spectral range of the $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ is greater than for the $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ owing to the different bandgaps. [1]

thickness to be calculated from equations (2.11) or (2.14). Using Eq. (2.14), which relates the optical thickness and the refractive index extrapolated to zero wavelength, it is important to keep in mind that $dn/d\lambda$ is negative in this wavelength region and therefore $n(0)$ should be greater than n_1 or n_2 . To standardize the measurement and therefore reduce the error in $n(0)$ resulting from non-linear dispersion (see section 2.4), λ_1 was chosen near λ_g and λ_2 was chosen near 800 nm. Three measurements of t_{optical} were made per sample. The results of t_{optical} (from Eq. (2.14)) vs. t (from SEM measurements) are shown in Fig. 4.4. The linearity of the data is excellent, as the correlation coefficient is greater than 0.99 for both compositions. The empirical $n(0)$ values determined from the slope of the lines in Fig. 4.4 (see Eq. (2.14)) are 5.08 for $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ and 3.97 for $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$. For the former case this agrees well with the value of 5.03 (using $\lambda_1, \lambda_2 = 700, 790$ nm) which can be extrapolated from reference [14]. Thus the thickness of the epitaxial layer may be determined by the ratio of t_{optical} to $n(0)$.

As a further demonstration of the technique the wafer uniformity of an $\text{Al}_{0.5}\text{Ga}_{0.5}\text{As}$ epitaxial layer grown by MBE as measured by the former algorithm is plotted in Fig. 4.5. The dashed line is similar to data from similar MBE growths, for example that shown in Fig. 4.2. While the main trend of the epitaxial layer thickness variation is demonstrated in Fig. 4.5, it is evident that the data in Fig. 4.2 is better. This will be discussed presently.

Although the above algorithm demonstrates the reflection spectroscopy technique, λ_1 and λ_2 must be within a certain range for the above algorithm to have maximum accuracy because of non-linear dispersion of the $\text{Al}_x\text{Ga}_{1-x}\text{As}$ films (Sec. 2.4). However for films of varying thickness λ_1 and λ_2 must necessarily be different for different thicknesses. Thus $n(0)$ itself is a function of the thickness of the epitaxial film. A more accurate approach

Fig. 4.4: Optical thickness vs. measured SEM thickness for $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ and $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$. [1]

Fig. 4.5: Wafer map of optical thickness of a $\text{Al}_{0.5}\text{Ga}_{0.5}\text{As}$ wafer grown by MBE.

[1]

would be to use equation (2.11), which requires the wavelength dependent refractive index to be calculated for each extreme wavelength. This has been done for the $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ composition using the refractive index equations from reference [15], and the results presented in Fig. 4.6. Once again the linearity is excellent, and in this case it is seen that the optically measured epitaxial layer thickness agrees well with the measured SEM thickness without normalizing an optical thickness to an empirically determined $n(0)$. It is worthwhile to quantify the extent to which the optical and measured thicknesses agree in Fig. 4.6 in order to gauge how much improvement in the technique is necessary to apply it toward actual samples for processing. A histogram of the ratio of (t_{optical}/t) is presented in Fig. 4.7. The increments in the histogram are 4%, centered at 1.00. One data point was out of the range of the histogram. The agreement was within $\pm 2\%$ in 40% of the trials, within $\pm 5\%$ in 75% of the trials and within $\pm 10\%$ in 95% of the trials. If 0.99 rather than 1.00 is taken as the midpoint, the agreement is within $\pm 3\%$ in 65% of the trials. This agreement is quite good since the original extrema from the Nanospec chart recorder output are subject to a 1 nm digitizing error each, resulting in a $\pm 2\%$ experimental error from extrema location alone. Also the composition of the AlGaAs is nominal and subject to a $\pm 1\%$ Al deviation from the nominal value. This would affect the optically measured thickness by a further $\pm 1\%$. Additionally the measurement of the SEM photographs are subject to a digitizing error which on average is $\pm 2\%$. Although the reflectance spectroscopy technique can provide refractive index information, it was not convenient to extract the necessary data from the Nanospec machine. A demonstration of this portion of the technique is deferred to the next section. Using digital output (e.g. from either the

Fig. 4.6: Comparison of absolute thicknesses for $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ using reflectance spectroscopy and SEM

Fig. 4.7: Histogram of the ratio of thicknesses measured by reflectance spectroscopy and SEM

modified PL system, the FTIR, or for that matter more recent versions of the Nanospec) should significantly reduce the error in locating the extrema. In that case the thickness accuracy is expected to be better than in Figs. 4.6 and 4.7.

In summary, then, it is evident that both the optical and (destructive) cleaving and staining technique are in excellent agreement, similar to the GaAs on Si case. However in this section it has been clearly demonstrated that an accurate absolute thickness can be obtained if either the refractive index at the extrema is known or a calibration curve for the particular composition, to determine $n(0)$, is available.

4.3 InGaAsP and InGaAs on InP

As with the previous materials systems described, the deposition of InP-based materials is of considerable importance for optoelectronic applications. The wavelength range is such that either the modified PL system or the FTIR system may be used to acquire the spectral data. For thicker films the FTIR system may be appropriate, where the refractive index dispersion is minimized. Alternately, the use of the modified PL system in the study of these epitaxial structures allows for both conventional emission wavelength measurement and reflectance spectroscopy at the same points on the wafer. If the sample is a ternary, knowledge of the PL wavelength is sufficient to uniquely specify the composition of the layer [16]. Similarly if lattice matched conditions can be assumed, then the PL wavelength also can be used to determine the composition of the quaternary. This in turn specifies the refractive index as a function of wavelength [16]. Note that this is also true for AlGaAs. Absolute thicknesses may then be obtained by using equation (2.11), as in the previous section. Two examples of InP-based materials are discussed in this section.

Samples of approximately 1-3 μm of lattice matched $\text{In}_{.28}\text{Ga}_{.72}\text{As}_{.62}\text{P}_{.38}$ (emission wavelength 1.3 μm) and $\text{In}_{.53}\text{Ga}_{.47}\text{As}$ (InGaAs) on InP were prepared using low pressure MOCVD. Reflectance spectra, recorded in the wavelength range 5000-2000 cm^{-1} , are shown in Fig. 4.8. Note the similarity between these spectra and that expected from Fig. 2.1.

In the manner described above, absolute thicknesses were calculated by using refractive index values from Ref. [16] and Eq. (2.11), which agreed well with what was expected from previously calibrated MOCVD growth rates. The calculated thicknesses were independent (t within less than 1%) of the

Fig. 4.8: Reflectance spectra for two compositions of InGaAsP recorded by FTIR. The modulation amplitudes of the two spectra are different because the the different compositions have different refractive indices.

Fig. 4.8: Reflectance spectra for two compositions of InGaAsP recorded by FTIR. The modulation amplitudes of the two spectra are different because the different compositions are of different refractive indices. See Eq. (2.18).

extrema chosen to calculate the thickness. As another cross-check of the self-consistency of the optical thicknesses, the refractive index values calculated from reference [16] and the calculated thicknesses from equation (2.11) were used in equation (2.8), arranged to solve for m , for each extremum. These calculated m values were whole or half-integer values to within 0.02, i.e. within experimental error. Furthermore the peaks corresponded to half-integer values and the valleys to whole-integer values, as expected from Sec. 2.4.

To further validate the reflection spectroscopy technique, the modulation amplitude R_1/R_0 (measured at the extremum nearest 4000 cm^{-1}) was compared to the theoretical value as developed in Section 2.5. The line drawn in Fig. 4.9 shows the theoretical R_1/R_0 predicted from equation (2.18), using refractive index values for the InP substrate calculated from reference [16]. The squares in the same inset are the measured R_1/R_0 values. It is necessary to compare Eq. (2.18) directly with the more exact Eq. (2.16) as well as with the experimental data. The actual values of $R_{\max}/R_{\min}$ from Eqs. (2.16), (2.18) and the experimental spectra in Fig. 4.9 are given in Table 4.1. It is evident that the theoretical modulation amplitude expected from Eqs. (2.16) and (2.18) are in reasonable agreement. The absolute error introduced in the case of the $\text{In}_{.53}\text{Ga}_{.47}\text{As}$ is approximately double the error for the case of the $\text{In}_{.28}\text{Ga}_{.72}\text{As}_{.62}\text{P}_{.38}$. This is consistent with a larger Δn for the former, not satisfying the condition in Eq. (2.17) as well. This translates to a relative difference in R_1/R_0 ($(R_{\max}/R_{\min})-1$) values of 3% for both the InGaAsP and the InGaAs, which could in principle be corrected for. The relative difference between the theoretical (Eq. (2.16)) and experimental (FTIR) R_1/R_0 values is 10% for both the InGaAsP and the InGaAs, significantly larger than the error introduced by the approximation of Eq. (2.18). If the experimental R_1/R_0

Fig. 4.9: Theoretical vs. measured modulation amplitude for material in the $\text{In}_x\text{Ga}_{1-x}\text{As}_y\text{P}_{1-y}$ system. The As content as given corresponds to y , where $y=2.2x$.

Table 4.1. Theoretical and measured values of maxima/minima ratio near 2.5 μm

Epitaxial layer	Theoretical (Eq. 2.16))	Approximation (Eq. (2.18))	Experimental (FTIR data)
InGaAsP (1.3 μm emission)	1.158	1.154	1.174
InGaAs	1.266	1.258	1.297

Table 4.2. Theoretical and measured values of Δn near 2.5 μm

Epitaxial layer	Theoretical (Ref. [16])	Experimental (Eq. (2.18))	Theor./Expt. ratio
InGaAsP (1.3 μm emission)	0.1714	0.1894	1.105
InGaAs	0.2891	0.3230	1.095

values were less than the theoretical values, this might be due to truncation errors from both the finite (not zero) bandpass and the non-uniformity of the epitaxial layers. However the trend is in the opposite direction. This point will be returned to.

In a material quality control situation, the parameter of interest is not $R_{\max}/R_{\min}$ or R_1/R_0 , but rather n or Δn . Table 4.2 presents the theoretical (from Ref. [16]) and experimental (from Eq. (2.18)) values of Δn , as well as the ratio of these two values.

Even though Tables 4.1 and 4.2 contains only two data points, it is worth noting that the difference between theoretical and experimental R_1/R_0 and Δn values is in the same direction for both and in the same ratio. Note that the actual discrepancy is closer to 7% than the 10% in the last column of Table 4.2, owing to the 3% theoretical error shown in Table 4.1.

The constant ratio in the last column of Table 4.2 may indicate that the theoretical value somewhat underestimates these values. This is a real possibility in light of the observation that the overall quality of epitaxial layers has improved over the years, and it may be that material from some time ago contains defects or voids which would reduce the apparent refractive index. It is important to note that the effective refractive index in the case of voids is not simply the volume average of the material. Rather, effective medium theory [5] must be used, which weights the refractive index more in the direction of the lower index material.

Another possibility is that the substrate absorption, while small, is enough to influence the modulation error by enough to affect the apparent Δn value. This might be expected considering Fig. 2.2 and the analysis of Sec.

2.6. However the growths were on semi-insulating substrates, so it is anticipated that the substrate absorption should be very small.

Even assuming the theoretical values from Eq. (2.18), corrected by the known difference (from Table 4.1) between Eqs. (2.18) and (2.16), to be absolutely correct, the present results show that if lattice matched conditions can be assumed, the P content of an InGaAsP layer may be determined to within roughly 7% from modulation amplitude alone. Further investigation could be done to hopefully improve this tolerance and identify the reason(s) for this discrepancy. If a 1% tolerance in composition accuracy could be achieved, this technique could be usefully applied to the InP-based material system in combination with either photoluminescence data or X-ray rocking curve data [17] in a production environment to determine the composition of an epitaxial layer. Also, in a waveguiding structure, the refractive index is a critical parameter. Therefore it is evident that the results in Tables 4.1 and 4.2 represent a significant trend.

In summary, in this section the reflectance spectroscopy technique was applied to single epitaxial layers of InGaAsP on InP. It was conclusively shown that the modulation amplitude of the spectrum depends on the refractive index of the film, although there is an unresolved quantitative discrepancy between the predicted and measured modulation amplitudes. The high degree of self-consistency of these measurements and calculations is further evidence of the validity of reflection spectroscopy as a technique for epitaxial layer thickness measurement and indicates the potential for refractive index measurement.

4.4 GaAs/InP on AlAs quantum barriers/InGaAsP quantum wells on GaAs/InP

Reflection spectroscopy for determining homojunction layer thicknesses is typically done at intermediate infrared wavelengths. However, by inserting a very thin marker layer of epitaxial material other than GaAs in between the GaAs epitaxial layer and the substrate, interference effects are apparent at much shorter wavelengths. The largest modulation amplitude occurs for the case where the refractive index discontinuity is maximized; hence AlAs rather than any other AlGaAs composition is the most suitable for the marker layer if the GaAs thickness is to be determined.

As an independent prediction of the spectral data, computer simulations of the reflectance spectra may be obtained using the transfer matrix method [7,8]. In this way a comparison of the theoretical and experimentally measured reflectance spectra is possible.

The theoretical reflectance spectrum bears some similarities and differences to the simple spectrum of one epitaxial layer upon a substrate. As for the single epitaxial layer case, the interference oscillations in the marker layer spectrum are periodic and the extrema are an integral or half-integral number of orders of interference apart except at very long wavelength. Therefore the epitaxial layer thickness may be calculated using equation (2.11). In the single epitaxial layer case for which $n < n_s$, minima correspond to $R_{\lambda/4} = 1 - (R_1/R_0)$ and the maxima correspond to $R_{\lambda/2} = R_0$. The converse is true if $n > n_s$. For the marker layer case both the thick epitaxial layer and the substrate are of the same material, and so $n = n_s$. The simulations show that in the limit

Table 4.3. Theoretical values of m and R_{extreme}/R_0 ratio between 2 and 3 μm

λ	R/R_0	----- m values -----		
		total thickness	GaAs	average
2.01	0.962	10.265	10.237	10.250
2.059	1.000	10.016	9.989	10.000
2.112	1.038	9.764	9.738	9.751
2.168	1.000	9.510	9.484	9.486
2.225	0.964	9.264	9.239	9.250
2.286	1.000	9.015	8.991	9.011
2.351	1.034	8.764	8.740	8.752
2.421	1.000	8.501	8.486	8.488
2.492	0.968	8.263	8.241	8.250
2.570	1.000	8.012	7.990	8.011
2.652	1.030	7.763	7.742	7.752
2.741	1.000	7.508	7.488	7.489
2.834	0.971	7.262	7.242	7.252
2.935	1.000	7.011	6.992	7.009

of small AlAs thickness both $R_{\lambda/4}$ and $R_{\lambda/2}$ are equal to R_1/R_0 , and the oscillations reach extrema at one-quarter- and three-quarter-integer values of m . The amplitude R_1 in this case depends on the ratio of the AlAs thickness to the wavelength of the extremum. As this ratio decreases, so does R_1 . This is illustrated in Fig. 4.10 for the example of 3.1 μm of GaAs on a 10 nm AlAs marker layer. It is evident that R_1 tends asymptotically to zero at long wavelength, as the ratio of the AlAs thickness to the wavelength approaches zero. In Table 4.3 the actual simulated values for m and R_1/R_0 in the wavelength range 2-3 μm are shown. It is seen in this and similar simulations that the m values for the average of the GaAs thickness and the sum of the GaAs and AlAs thicknesses (or the GaAs thickness plus one-half the AlAs thickness) are nearly quarter-integers, whereas the m values for either the GaAs thickness alone or the sum of the GaAs and AlAs thicknesses are not exactly quarter-integers.

It is interesting to simulate what happens if the AlAs thickness is varied, keeping the total GaAs and AlAs thickness constant, so a given m occurs at nearly the same wavelength in each case. Figs. 4.11 and 4.12 show some results for AlAs thickness ranging from 1-500 nm, in the wavelength range 2-3 μm . In Fig. 4.11, the maximum AlAs thickness is 0.1 μm . It is observed that the R_1/R_0 values for each of the quarter- or three-quarter-integers are similar. Spectral maxima occur at three-quarter-integers and spectral minima occur at quarter-integers. Curvefitting has been done with the result that for any m in this wavelength range, R_1/R_0 is a quadratic function of AlAs thickness with a correlation coefficient of greater than 0.99 in this thickness range. Thus in addition to measuring the GaAs thickness from the method demonstrated in Sec. 4.2, it should also be possible to

Fig. 4.10: Theoretical and experimental normalized reflectance spectra for 3.09 μm of GaAs on 10 nm of AlAs on a GaAs substrate. The modulation amplitude decreases to zero as wavelength increases. The extrema correspond to quarter-integer orders of interference. The points where the normalized reflectance is 1 ("zero crossings") correspond to half-integer orders of interference. This is different from the case of a single epitaxial layer. [2]

Fig. 4.11: Simulated modulation amplitude of the GaAs on thin AlAs on GaAs system for AlAs thickness ranging from 1-100 nm

Fig. 4.12: Simulated modulation amplitude of the GaAs on AlAs on GaAs system for AlAs thickness ranging from 1-500 nm

simultaneously measure the AlAs thickness uniquely from the modulation amplitude alone in this wavelength and thickness range.

However as the AlAs thickness increases beyond $0.2\ \mu\text{m}$, there is more than $\lambda/2$ thickness of AlAs for some of the wavelengths in this spectral region. In this case, as illustrated in Fig. 4.12, the maxima shift from three-quarter-integers for less than $\lambda/2$ of AlAs to quarter-integers for more than $\lambda/2$ of AlAs. Not shown in Fig. 4.12 is the interesting phenomenon that a maxima occurs at an R/R_0 value of 1 in the transition, where the AlAs thickness is exactly $\lambda/2$.

For any given GaAs thickness, the simulation shows that increasing the AlAs thickness causes an increase in R_1 throughout the spectrum, but reduces the exactness of m to quarter-integers. In practical situations it appears that an AlAs thickness of 10 nm is a good compromise between observable interference oscillations and closeness of m to quarter-integers.

Also shown in Fig. 4.10 are the optical spectra experimentally recorded on the FTIR system. It is evident that the agreement between theory and experiment is quite good. In this case no attempt was made to quantitatively determine the goodness of fit. However it was observed that the agreement between the experimental data and the simulated spectra for a 3.08 or $3.11\ \mu\text{m}$ thick layer was not as good as for the spectra simulated for 3.09 or $3.10\ \mu\text{m}$. The $3.09\ \mu\text{m}$ simulated spectrum appeared to fit the experimental data slightly better than the $3.10\ \mu\text{m}$ simulated spectrum. Thus the technique can determine the thickness of the GaAs layer to within $0.01\ \mu\text{m}$. It should also be pointed out that the AlAs marker layer was grown to a nominal thickness of 10 nm but this thickness was not independently verified.

Although the AlAs is a quantum barrier and not a well, the band edge discontinuity, if sufficiently abrupt, allows for 2-D electron gases at the

interfaces which could conceivably have an impact on the refractive index discontinuity. Similarly, sufficiently non-abrupt interfaces might reduce the index step. These effects would be apparent in the measured modulation amplitude. Since R_1 for both the theoretical and experimental spectra agree, either neither of these effects are present, they offset or the AlAs thickness is significantly different from its intended nominal thickness.

If neither effect is present then as mentioned above it should in principle be possible to determine the thickness of the AlAs from the modulation amplitude alone. This same principle could be applied to the InP materials system as well. Recently an absorption technique has been reported [18] that determines the thickness of an InGaAsP active layer of an InP-InGaAsP-InP sandwich structure (similar to the experimental structure in this section), which is intended for use in a laser. The reflectance spectroscopy technique is another alternative, in which the active layer thickness may be determined directly from the modulation amplitude of the spectrum. In this case the InGaAsP thickness would be of nominal thickness 100-200 nm.

To determine the suitability of this approach for the analysis of thin layers, simulations of epitaxial stacks of InP on thin InGaAsP on InP have been carried out using the transfer matrix technique [7,8], with similar results to the GaAs on thin AlAs on GaAs case. Interestingly, in this case the maxima occur at quarter-integers and the minima at three-quarter integers for less than $\lambda/2$ of InGaAsP. This is consistent with the observation that the refractive index for InGaAsP is greater than for InP, whereas the refractive index for AlGaAs is less than for GaAs. Again, the total epitaxial layer thickness was kept constant so the extrema would occur at similar wavelengths for differing thicknesses of the thin layers. It was also observed that for two different total epitaxial layer thicknesses (1.15 and 1.65 μm), the

simulated modulation amplitude depends on wavelength only, and is independent of the InP thickness. For this technique to be usefully applied, the composition of the InGaAsP would have to be accurately known. However, by using reflectance spectroscopy rather than the absorption technique reported in Ref. [18], it should be possible to simultaneously measure the thickness of both the active layer and the InP layer. Since in any manufacturing process there is a certain range which is acceptable for both the active layer and InP thicknesses, such a measurement can in principle help determine the suitability of wafers for processing prior to actual processing, thus reducing waste of valuable resources. Furthermore this technique may be combined with a PL intensity measurement to determine non-destructively the PL intensity per unit volume, a measure of material quality. Also, knowledge of the InP thickness is a valuable aid to determining Zn diffusion conditions or how deep to etch the ridge in a ridge waveguide laser structure. Other examples of optoelectronic device structures where this technique could be applied include quantum well lasers and heterojunction bipolar transistors.

Even more accurate than using modulation amplitudes to determine the thickness of a thin AlGaAs or InGaAsP layer sandwiched in between GaAs or InP, if a spectral range can be used in which at some point the thin layer is of thickness $\lambda/2$, then the actual wavelength for which the normalized reflectance has an extremum at 1 may be used to accurately determine the thickness of the AlGaAs or InGaAsP layer directly from Eq. (2.8). For typical InGaAsP lasers the appropriate thickness of 100-200 nm is $\lambda/2$ at 0.65-1.3 μm ; thus absorption would be significant and the application of this $\lambda/2$ algorithm difficult.

In summary, reflectance spectroscopy has been successfully applied to the study of the heteroepitaxial system consisting of a GaAs layer on a very thin AlGaAs layer on a GaAs substrate and InP on a very thin InGaAsP layer on an InP substrate, with interesting results which suggest that both layer thicknesses may be individually determined if the composition of the thin layer is known. There appears to be good agreement between theory and experiment for the case of a $3.09\text{ }\mu\text{m}$ GaAs layer on a 10 nm AlAs layer on a GaAs substrate. There exists the potential to determine laser active layer thicknesses as well as how deep to place a Zn diffusion in laser structures, and how deep to etch a ridge in a ridge waveguide laser structure.

4.5 Multilayers

As one example of the application of reflection spectroscopy to a specific multilayer device structure, the system of InP on InGaAs on InP can be used. This system is of particular interest because it can be used in the fabrication of optoelectronic photodetector devices [19]. Accurate knowledge of both the InP and InGaAs epitaxial layer thicknesses is critical in determining the placement of the Zn diffused junction, predicting the transit time in the InGaAs and predicting the quantum efficiency of the device. As for the GaAs on marker AlAs layer on GaAs case, computer simulations may be done as an independent cross-check of the spectral data.

The theoretical reflectance spectra, as illustrated in Fig. 4.13, contain more information than in any of the previous cases, because oscillations arise with frequencies related to the total layer thickness as well as the layer thicknesses of the individual layers. The highest spatial frequency component (e.g. the frequency between two adjacent extrema) is due to the combined layer thicknesses.

If integral and half-integral orders of interference for the extrema could be assumed, then the individual layer thicknesses could be assigned uniquely in the following way. A combined optical layer thickness (nt) could be obtained from two neighboring peaks. The requirement that m be exactly an integer or half-integer could be used to assign n at the wavelengths of both extrema. This n would be the thickness-weighted average of the InP and InGaAs refractive indices, and thus would uniquely specify both the InP and the InGaAs thickness. This method could be applied to an arbitrary number of epitaxial layers providing at least as many extrema are present within the spectral range as there are epitaxial layers. However, the computer

Fig. 4.13: Theoretical and measured normalized reflectance spectra for 2.65 μm of InP on 3.8 μm of InGaAs on an InP substrate. Below 1.65 μm the InGaAs layer is opaque and reflectance spectra may be interpreted in terms of a single epitaxial layer of InP. [2]

simulations show that the extrema do not correspond exactly to integral and half-integral orders of interference, similar to the GaAs on thin AlAs on GaAs case studied in Sec. 4.4; they can differ by as much as 0.02 for the structures investigated. Nonetheless the combined layer thickness can be determined. By taking two extrema sufficiently far apart the relative error in using equation (2.11) to determine the combined optical layer thickness can in principle be reduced to less than 1%, although the individual layer thicknesses cannot be assigned in this simple way. The actual combined and individual layer thicknesses may be estimated to an accuracy which depends on how well the ratio of layer thicknesses is known. For thick enough layers or for a large enough spectral range the above algorithm which depends on exact knowledge of m might still be applicable through an averaging process.

There is another simple way to determine the individual thickness of both epitaxial layers of this particular structure. Two wavelength regions are scanned: one where the InP is transparent and the InGaAs is not and the other where both materials are transparent. The former scan determines the thickness of the InP. The extrema of the latter scan yield a combined optical thickness of InP and InGaAs. Knowing the thickness of the InP, the InGaAs thickness is uniquely determined. Fig. 4.13 shows both a computer simulation and experimentally measured spectra recorded on both the modified PL and FTIR systems for this structure with InP and InGaAs epitaxial layer thicknesses of 2.65 and 3.8 μm respectively. As for the GaAs on thin AlAs on GaAs case no attempt was made to measure the goodness of fit, but it is apparent from inspection of the two curves that once again the agreement between theory and experiment is excellent. To further illustrate the application of this technique a wafer map of the InP layer alone on the uncleved, unprocessed wafer is presented in Fig. 4.14. These data could be

Fig. 4.14: Wafer map of InP thickness of InP on InGaAs on InP system

used, for example, to determine what Zn diffusion conditions to use to place the p-n junction in the proper place.

This simple algorithm could be easily applied to an arbitrary number of epitaxial layers which become successively transparent as the wavelength increases: for example an InP layer on a quaternary layer on an InGaAs layer. Other multilayers of arbitrary complexity could be measured by using curvefitting techniques, thereby extending the use of reflection spectroscopy to a wide variety of device structures.

These techniques are not restricted to the InP materials system. For example, the experimentally measured optical spectrum of 0.9 μm of GaAs on 3 μm of AlGaAs on a GaAs substrate, grown by MBE, has been obtained. Such a structure can be used in the fabrication of an optical rib waveguide, for which the critical parameters are the channel width and residual GaAs thickness following an etch step. The channel width is accurately controlled by standard photolithographic techniques, but controlling the residual GaAs thickness has traditionally been a problem because the GaAs layer thickness must be accurately known before the etch step. A non-destructive solution is to obtain the initial GaAs thickness by reflection spectroscopy prior to the etch step, so the depth to be etched may be accurately determined. Initially, though, it is important to know the thickness uniformity of the epitaxial layers over the entire wafer. This information, derived from the optically measured data by using equation (2.11), is shown in Fig. 4.15 using a variety of algorithms. Note the excellent agreement among the different algorithms, demonstrating that the layer thickness uniformity obtained is independent of the choice of algorithm used for the computation.

Reflection spectra give spatial frequency information about the epitaxial layers. Therefore the Fourier transform of the spectra, or in the case

4.15 Wafer map of total epilayer thickness of GaAs on $\text{Al}_{18}\text{Ga}_{82}\text{As}$ on GaAs system along two perpendicular directions of the wafer. Three different algorithms are used: using the 29th and 30th order of interference as in Sec. 4.1 and the optical thickness as in Eq. (2.14). The discrepancy in one point might be compositional variation in that portion of the wafer.

of the FTIR system the original interferogram, should give direct spatial information about the epitaxial layer thicknesses. For maximum effectiveness as large a spectral region as possible should be used, as ideally the Fourier transform should be integrated from zero to infinite wavelength. There is, however, a tradeoff between a large spectral range and using a spectral region where the non-linear refractive index dispersion is minimized, because the Fourier transform should actually give optical thickness (nt) information, and variations in n should blur the peaks. An effective use of the Fourier transform might be to supply initial guesses of individual layer thicknesses to a curvefitting routine for a very complex multilayer system.

5. CONCLUSIONS

Reflectance spectroscopy has been demonstrated as a reliable, self-consistent and useful technique for determining epitaxial layer thicknesses. To illustrate the use of the technique measurements have been made on several distinct heterostructures composed of III-V semiconductor compounds deposited onto Si, GaAs and InP substrates, and in a range of wavelength regimes. A variety of experimental apparatus including a commercial spectrophotometer, a modified photoluminescence system and an FTIR system, covering a variety of wavelength regimes in the visible, near- and mid-infrared, have been used.

Measurements on single epitaxial layers have demonstrated the validity of reflection spectroscopy in several controlled cases in which the actual epitaxial layer thickness could be independently verified by two distinct and standard destructive physical techniques: stylus profilometry and cleave and stain (scanning electron) microscopy. The high degree of self-consistency of the optical measurements and the excellent agreement with the physical measurements reinforce the confidence level of the technique. Furthermore it was shown for the first time that the modulation amplitude of the spectrum is related directly and nearly linearly to the refractive index of the film, and therefore there exists the potential to measure the composition of deposited material by reflection spectroscopy, although at present there is a 7% discrepancy between the predicted and measured modulation amplitudes. Further work would resolve the reasons for this discrepancy. The refractive

index dispersion of III-V materials could be measured and compared with presently accepted theoretical calculations.

To illustrate the use of the technique measurements have been made on some multilayer systems. Several interesting properties have been found. For the case of a thin marker layer sandwiched in between a GaAs or InP layer and substrate, it appears that for the first time it is possible to simultaneously measure the GaAs or InP thickness from the wavelength or wavenumber separation of the peaks and the marker layer thickness of known composition from the modulation amplitude, thus avoiding the need to resort to curvefitting the spectrum or ellipsometry. Thus the potential exists to measure non-destructively certain parameters necessary for the control of semiconductor laser, heterojunction bipolar transistor, photodetector and waveguide manufacturing. Excellent agreement between computer simulations and experimental data has been demonstrated for two multilayer systems, including a structure suitable for the fabrication of photodetectors. Also a structure suitable for fabricating waveguides has been measured. In both the photodetector structure and the waveguide structure a wafer map of epitaxial layer thickness uniformity has been obtained.

As reflection spectroscopy is non-destructive and lends itself to automation, it is an attractive alternative to currently used techniques such as SEM, TEM and stylus profilometry. Using this technique whole wafers may be screened prior to processing. Thickness uniformity of the epitaxial layers may easily be determined in the form of a two dimensional wafer map. Additionally critical processing parameters -- e.g., a Zn diffused junction depth for a photodetector or a GaAs etch depth for a waveguide -- may be targetted prior to the process step with confidence on a whole wafer. Further

studies on other multilayer structures may broaden the application of this technique.

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