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Optical approaches for controlling epitaxial growth

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Abstract

Optical spectroscopy of surface and near-surface regions has advanced to the stage where detailed measurements can be made and analyzed in real time. Here, we discuss reported and potential applications of optical probes for sample-driven closed-loop feedback control of semiconductor epitaxy. Parameters that have been controlled include temperature, thickness in both deposition and etching, and composition, including continuously graded compositions. Although considerable progress has been made, much remains to be done before these techniques become viable production tools. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Epitaxial growth; Optical spectroscopy; OMCVD

1. Introduction

The drive toward higher performance and increasing complexity in semiconductor devices, together with the trend toward chemical-beam deposition methods such as organometallic chemical vapor deposition (OMCVD) and chemical beam epitaxy (CBE), are providing strong incentives to develop a better understanding of growth processes and better methods of monitoring growth [1]. Real-time information about a growing crystal is useful for various reasons, including a posteriori diagnosis of failed devices. Although much effort is presently being directed toward developing predictive approaches where sample parameters such as layer thicknesses and compositions are calculated from accurately measured process parameters through the use of accurate growth models, this strategy is clearly limited in its capacity to deal with complex nonlinear processes such as OMCVD, where the surface plays an integral role in decomposing precursor species and small changes in the composition of the ambient can affect growth substantially [2,3]. A more definitive approach is to base process decisions on information about the growing sample itself, leading ultimately to sample-driven closed-loop feedback control of epitaxy.

Optical probes are of interest for sample-driven applications because they are nondestructive, noninvasive, and can be used in any transparent ambient. However, optical probes have several well-known limitations. Most reflectance-based techniques are relatively insensitive to surface processes, and the

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spectral range accessible with common quartz-optics technology is relatively limited. Sensitivity is determined approximately by the ratio of the penetration depth of the light, rarely less than 100 Å, to the thickness of the surface layer, about 1 Å. Thus the information contained in a reflected beam will be divided roughly as 99% bulk and 1% surface. To improve our capabilities of isolating the surface contribution and determining it more accurately, much recent effort has gone into inventing new and refining established optical techniques. Those that are relatively new yet now relatively well developed include reflectance-difference (-anisotropy) spectroscopy (RDS/RAS), surface photoabsorption (SPA), p-polarized reflectance spectroscopy (PRS), and laser light scattering (LLS). Second-harmonic generation (SHG) is currently undergoing rapid refinement. These rely either on symmetry (RDS/RAS, LLS, SHG) or enhancement of surface sensitivity by operation at the Brewster (PRS) or pseudo-Brewster (SPA) angle to suppress the bulk response in favor of the surface component. These techniques are described in more detail in recent reviews [4–9].

Although growth is a surface process, the emphasis on the use of surface-analytic approaches for *control* of epitaxial growth does not result from practical considerations but is historical, stemming from the use of reflection high energy electron diffraction (RHEED) in molecular beam epitaxy (MBE), where 'RHEED oscillations' generally have a 1-to-1 relationship with monolayer (ML) growth. Although the analogous monolayer-sensitive experiment has been done with RDS [10], the main practical considerations in growth and deposition are layer thicknesses and compositions. These are *bulk* properties, which are more efficiently and accurately determined by bulk probes such as reflectometry and ellipsometry, especially when used in the spectral mode. Reflectometry and single-wavelength ellipsometry have long been used to determine average thicknesses and compositions of deposited films, and reflectometry is currently used routinely for monitoring and controlling thin-film deposition in optical thin-films technology, where materials are mainly transparent and layers are thick enough to generate distinctive interference patterns. Recently, reflectometry has also been adapted and extended to the analogous configuration in semiconductor epitaxy, the Bragg reflectors of vertical cavity surface emitting lasers (VCSELs) [11,12].

However, for control purposes one needs not average compositions but *fluctuations* of these compositions from target values, ideally of just-deposited material in a region where the thickness is vanishingly small. These *near-surface* data have recently become available through the development of virtual-interface (V-I) theory [13]. Here, kinetic ellipsometric (KE) data and their derivative with respect to layer thickness are used to determine the dielectric response of near-surface material down to the ML level, even if nothing is known about the underlying material. By circumventing the instability inherent in the direct inversion of the Fresnel equations, this approach also enabled the first (and so far only) demonstration of sample-driven closed-loop feedback control of the epitaxy of films of continuously graded compositions [14]. Applications are now being made of more extended data-analysis schemes [15–17], which require thicker layers but which return growth rate as well not only for semiconductor epitaxy [1] but also for active feedback control of the growth of optical thin films [16,17].

Since monitoring is often confused with control, we define terms as follows. By monitoring we refer to the real-time determination of information about the process (ambient), sample surface, and/or the sample itself. By control we refer to the use of this information to make real-time decisions about and modifications of growth or deposition parameters. Control can occur at several levels depending on the type of real-time information available. In principle, such information can be obtained about any of the regions relevant for semiconductor epitaxy: the ambient, the unreacted and reacted parts of the surface reaction layer (SRL), and the near-surface and bulk regions of the sample, as shown in Fig. 1. The unreacted part of the SRL consists of (mainly) physisorbed species that are weakly bound to the substrate and not in registry with it, and the reacted part of (mainly) chemisorbed species that are strongly bound to the substrate and acquire the symmetry of its outermost atomic layer. In particular, process-state

[1] [18]; G.N. Maracas, C.H. Kuo, in Ref. [1], p. 476.

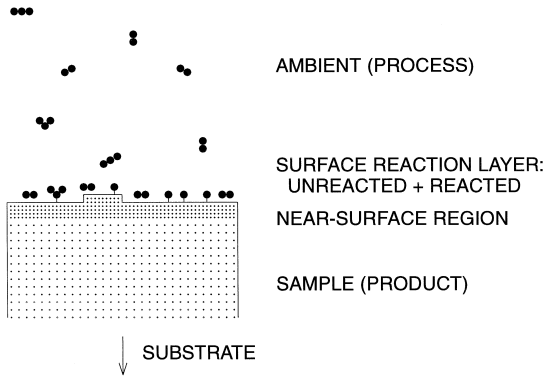

Fig. 1. Schematic diagram of the various regions involved in epitaxial growth.

and wafer-state control refer to the use of information about the ambient and sample, respectively.

The potential effectiveness of various control strategies can be assessed by considering the various regions and their interrelationships. The ambient consists of the carrier gas (if any) and the nutrient species providing the constituent elements to the growth surface. Some growth chemistry, such as thermal cracking of precursor species, may also occur here. The nature and composition of the weakly and strongly bound parts of the SRL depend on the competition between the two adjacent regions and on the reactions taking place within the regions themselves. The reaction between the strongly bound part of the SRL and the crystal determine the properties of the near-surface region and the rate of growth of the crystal. Although information about all of the regions can be used to advantage, it is clear that the weakest and strongest links in the control process are those involving the ambient and near-surface regions, respectively. Control approaches based on wafer-state measurements also have the advantages of bypassing the need to accurately measure process parameters and to model the intervening processes leading to species incorporation. In addition, they can automatically correct for small changes as they occur, and are portable, not being affected by peculiarities associated with a particular reactor. However, the disadvantages (usually fatal) of wafer-state measurements, particularly ellipsometric measurements, are the need to modify growth chambers to allow optical access to the sample and the relatively complicated nature of ellipsometry itself.

In this work we assess recent progress and outline current technological problems that must be solved to make sample-driven closed-loop feedback control viable. Much still remains to be done, but progress continues. Spectroscopic ellipsometric (SE) data have been used to control sample temperature in an MBE chamber through the temperature dependences of optical spectra [18]. [1] The first experiments involving sample-driven closed-loop feedback control, those done at Bellcore to hold compositions x of Al_xGa_{1-x} As layers constant over extended periods of time [19], have now been duplicated under more challenging conditions, specifically for the growth of In_xGa_{1-x} As layers on InP in a multiwafer OMCVD reactor under production conditions [20]. Ellipsometric control of layer thickness has been demonstrated in both etching [2] and epitaxy [18,22,23], [1] and growth-rate control has been demonstrated as well [24]. Multiwavelength reflectance monitoring of III–V Bragg reflectors, although not directly used for control, provides an excellent example of the advantages of multiwavelength analysis [12,25]. Some of these approaches are beginning to make inroads into production. [3]

2. Growth processes

One possible list of parameters needed to characterize growth is given in Table 1. The primary category contains only bulk parameters: layer thicknesses, compositions, and uniformities, on the grounds that if specifications cannot be met at this level the remaining parameters do not matter. The secondary category includes surface-determined parameters: efficiency of dopant incorporation, possibility of atomic ordering of nominally random alloys, interface widths, etc. The tertiary parameters are those related to the process itself, for example type, pressure, and flow rate of the carrier gas, the partial pressures of active species, temperature of the sample, etc. Tertiary parameters have received the most attention simply because they are easiest to measure

[2] [21]; W.M. Duncan, M.J. Bevan, S.A. Henck, in Ref. [1], p. 467.

[3] J.C. Bean, in Ref. [1], p. 459.

Table 1
Parameters involved in crystal growth

Parameter	Category	Technique
Primary		
Thickness	Bulk	SR, SE
Composition	Near-surface	KE
Uniformity	Proximal	Imaging, PR
Secondary		
Dopant incorp.	Surface-determined	RHEED,
Atomic ordering	Surface-determined	LS/LLS, RDS,
Interface widths	Surface-determined	SPA, PRS, SHG
Tertiary		
Sample temp.	Process-related	TC, pyrometry,
Ambient pressure, temperature, species, fluence...	Process-related	ultrasonics, LIF, IRAS, CARS, UVAS, MS, etc.

and can be measured without significant, or even any, modification to growth reactors.

Our ability to use any of these parameters for control is predicated on our capability of obtaining information about them. Process-directed measurements include temperature determinations by thermocouples (TCs) and ambient-composition determinations by laser induced fluorescence (LIF), infrared absorption spectroscopy (IRAS), coherent anti-Stokes Raman scattering (CARS), ultraviolet absorption spectroscopy (UVAS), and mass spectrometry (MS). Surface information can be obtained by RDS/RAS, SPA, PRS, LLS, SHG, and in uhv environments, RHEED. Bulk probes include spectroreflectometry (SR), SE, photoreflectance (PR), and various imaging techniques. Near-surface analysis can be performed by KE in connection with V-I theory. The unreacted part of the SRL generally can be accessed by *surface-oriented* probes such as SPA and PRS, where surface information is obtained by changing the state of the surface. Surface effects are also detectable by SR and SE, but mainly at nuisance levels. Information about the reacted part of the SRL can be obtained from *surface-specific* probes such as RDS, LLS, and SHG, which can return information about the surface under steady-state conditions. In principle IRAS can provide information about both parts of the SRL, but IR absorption is so weak that multiple internal reflectance techniques must be used, thereby limiting its applicability to energies and temperatures where the sample is transparent. Grazing-incidence X-ray spectroscopy (GIXS) [26] returns information about long-range order but at present can only be performed at synchrotron sources.

3. Examples

3.1. Thickness control based on the SRL

In Fig. 2 we show RD data obtained during the OMCVD growth of a 30-period InGaAs/GaAs superlattice, where each period consists of 5 ML of $In_{0.11}Ga_{0.89}$ As and 10 ML of GaAs [10]. The top part of the figure shows the RD data for the entire 30 periods, and the lower part the data for the 26th layer on an expanded scale. The lower data clearly show the growth of the individual MLs, along with the growth interruptions between $In_{0.11}Ga_{0.89}$ As and GaAs. The flows of the In- and Ga-containing precursors were terminated at 5 and 10 ML completions, respectively. Subsequent X-ray diffraction data on the finished sample indicated that the thicknesses were accurate to better than 5%, and that the interfaces were sharp. This showed that the transients that occurred at the beginning and ending of the metal-precursor flows were not due to roughness but to a change of surface reconstruction driven by an exchange of chemisorbed species. The overall thickness of the layers can also be determined from the Fabry–Perot oscillations in the envelope of the data at the top.

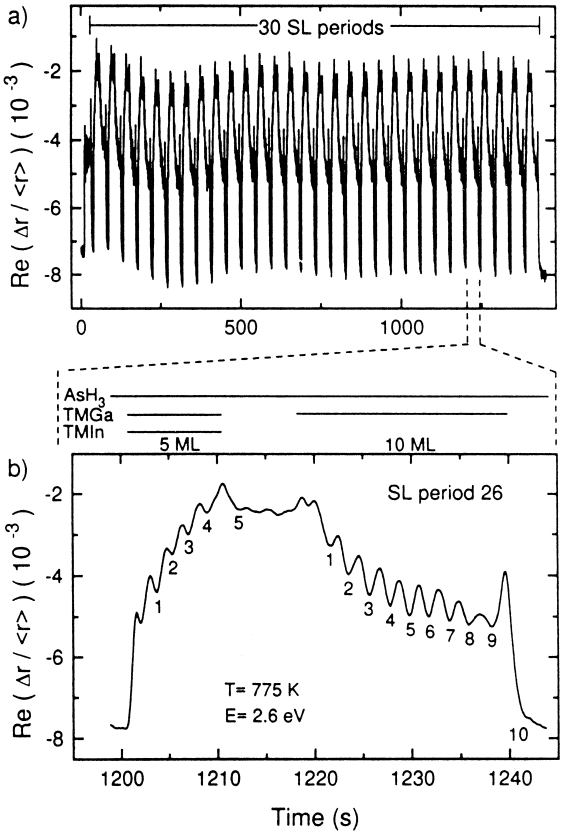

Fig. 2. Top: RD response measured during the growth of a 30-period InGaAs/GaAs superlattice consisting of 5 and 10 ML of InGaAs and GaAs, respectively. Bottom: expanded scale showing the RD oscillations allowing growth to be followed on a ML scale (after Ref. [10]).

3.2. Compositional control based on the near-surface region

The near-surface region is effectively accessed only through ellipsometric measurements, which return both amplitude and phase of the complex reflectance ratio $\rho = r_p/r_s$, where r_p and r_s are the complex reflectances for s- and p-polarized light, respectively. When plotted in the complex plane with time (thickness) as the running variable, complex optical data such as r_s, r_p, and the ellipsometrically determined pseudodielectric function $\langle \varepsilon \rangle$, appear as sequential exponential spirals, as shown in Fig. 3 [27]. Fig. 3 shows KE data obtained at 2.6 eV during the growth of Al_xGa_{1-x} As layers of increasing Al composition grown sequentially on a GaAs substrate. Each spiral converges to a focus where $\langle \varepsilon \rangle$ equals the value of the bulk dielectric function ε of the layer when the layer becomes optically thick. It is seen from the foci that compositions can easily be determined from ε to within 1% with the given signal-to-noise ratio of these data.

The possibility of using KE data for compositional control was recognized in 1979 by Theeten et al. [28] and Hottier and Laurence [29], who studied Al_xGa_{1-x} As layers deposited by OMCVD. Here, the near-surface value of ε, and thus the near-surface composition, was obtained by taking advantage of the fact that an exponential spiral has no memory, i.e., that at any point on the spiral, regardless of sample history, the angle between any point on the spiral and its focus is fixed and, for constant deposition and measurement rates, the distance between the point and the focus is proportional to the spacing between data points. Theeten et al. and later, Hartley

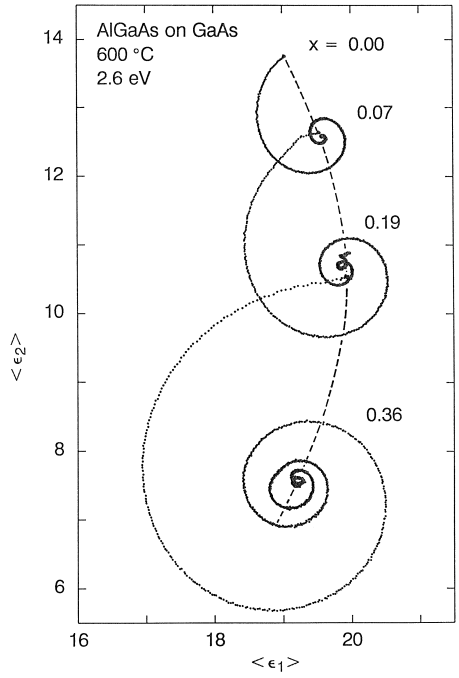

Fig. 3. Evolution of the complex dielectric function ε as a function of time (layer thickness) for a series of Al_xGa_{1-x} As layers grown sequentially on a GaAs substrate (after Ref. [27]).

[30], obtained a condition on ε independent of deposition rate by using the phase of the ratio of the derivative to the value. However, neither Theeten et al. or Hartley controlled growth: their algorithm fails completely in the limit of optically thick films [31].

The first demonstration of compositional control was done by Aspnes et al. [19], who used a more accurate exponential-spiral algorithm that used the deposition rate explicitly. In investigating other possible ways of analyzing $\langle\varepsilon\rangle$ data they also developed V-I theory, where for s- and p-polarized light the underlying sample structure is summarized by virtual reflectances r_{vs} and r_{vp}, respectively, at a virtual interface within the layer, where r_{vs} and r_{vp} were treated as parameters to be determined from the data along with ε [13]. This can be done exactly for r_s and r_p from their values and thickness derivatives at any point on a complex-plane trajectory, but not $\langle\varepsilon\rangle$ since both r_{vs} and r_{vp} are involved. However, for applications to semiconductor epitaxy ε could be determined from $\langle\varepsilon\rangle$ to better than 0.1% in the virtual substrate approximation (VSA), where r_{vs} and r_{vp} were assumed to result from a single virtual substrate of dielectric function ε_v. The determination of additional parameters such as the deposition rate required second or higher derivatives, adequate for controlling thick films but not ML films. However, these more general approaches have been useful for analyzing KE data of relatively thick films [15] and for controlling the thicknesses of optical films [16,17] and epitaxial layers [18,22,23]. [1] A detailed discussion of various approaches, including hybrid V-I and Fresnel approaches, is given in [31].

By eliminating the feed-forward instability of conventional Fresnel analysis, the V-I approach made sample-driven closed-loop feedback control possible. The Bellcore group used the VSA version to obtain the results shown in Fig. 4. These data were obtained during such growth of a 200 Å wide Al_xGa_{1-x} As parabolic quantum well by organometallic molecular beam epitaxy (OMMBE), where the flow of the Al-containing species, triisobutyl (TIBAl), was controlled by real-time analysis of the composition of the near-surface region [14]. The solid line in the top part of the figure shows the target composition as a function of position in the structure. The points represent the composition determined from the ellipsometrically measured pseudodielectric function $\langle\varepsilon\rangle$.

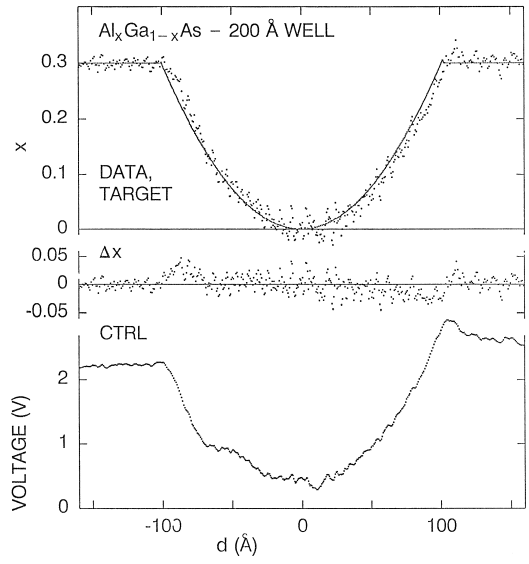

Fig. 4. Compositional data for a 200 Å wide parabolic quantum well grown by sample-driven closed-loop feedback control of epitaxy. Top: data and target values; middle: difference; bottom: control voltage. Growth and measurement parameters are given in the text (after Ref. [14]).

The middle is the difference between target and experimental values, and shows that composition was held to target values to within about 3%. The bottom shows the control voltage fed to the TIBAl mass flow controller while the experiment was in progress, the flow rate of which was used to establish the composition. The control voltage exhibits a noticeable asymmetry, probably due to the gettering of TIBAl as the flow increased. These results were obtained with a deposition rate of 0.95 Å/s, a sampling plus analysis interval of 650 ms, and 5-point averaging, thus x was obtained with the analysis of about 3.1 Å (approximately 1 ML) of material. Although this project was terminated in 1991, these data are still state-of-the-art.

Recent compositional-control work has concentrated on developing KE and specifically spectroscopic kinetic ellipsometry (SKE) for use in more realistic production conditions, which generally involves sample rotation for improved uniformity. Fig. 5 shows the In composition of three AlInAs alloys grown on InP in a commercial rotating-platen OMCVD reactor as determined by SKE [20]. Although these data pertain to monitoring, the system is now

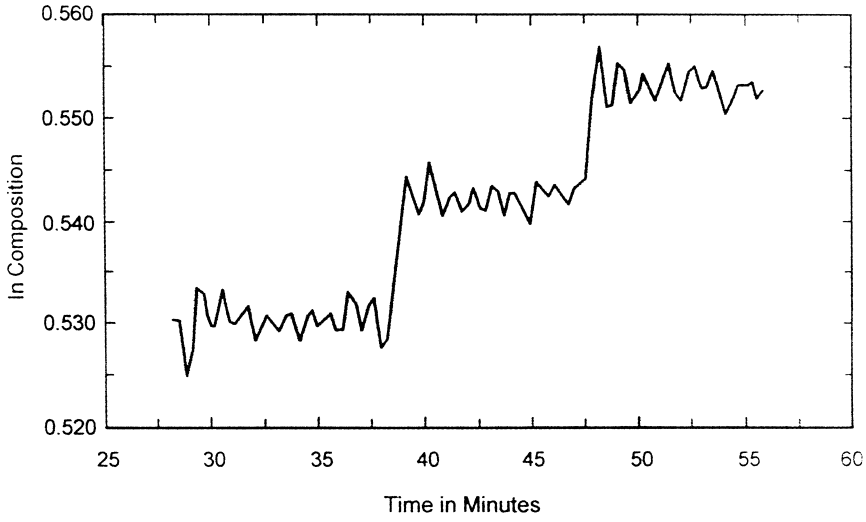

Fig. 5. In composition of InAlAs alloys grown on an InP substrate as a function of time, as determined under production conditions in a commercial OMCVD reactor. The step changes in composition were due to changes in the flow rate of the In precursor (after Ref. [20]).

being used to actively control In composition during the growth of relatively thick InGaAs buffer layers on InP. These data are significant because (1) InGaAs is lattice-matched to InP at only a single composition, and therefore potentially involve strain; (2) they were obtained on three three-inch wafers, with the wafers and the blank spaces between being swept under the optical beam once per revolution; and (3) the data obtained at different wavelengths were acquired and analyzed simultaneously. The latter capability will be necessary for controlling deposition of quaternary materials such as $In_xGa_{1-x}As_{1-x}P_y$. For the $Hg_{1-x}Cd_x$ Te system, a combination of data analysis and simulation calculations indicate an ultimate multiwavelength sensitivity of $\pm$ 0.0008 [32].

3.3. Other controlled parameters

As mentioned above, KE has been applied in various ways for controlling the thicknesses of optical films [16,17], epitaxial layers [18,22,23], [1] and layers removed by etching [21]; [2] deposition rates [24]; and sample temperatures [18]. [1] All but the latter involve the extraction of parameters from a sliding least-squares fit of generalized V-I expressions, and the use of this information to adjust appropriate system parameters.

3.4. Monitoring by reflectance

Lum et al. [25] recently described an application of the SR configuration of Killeen and Breiland [12]

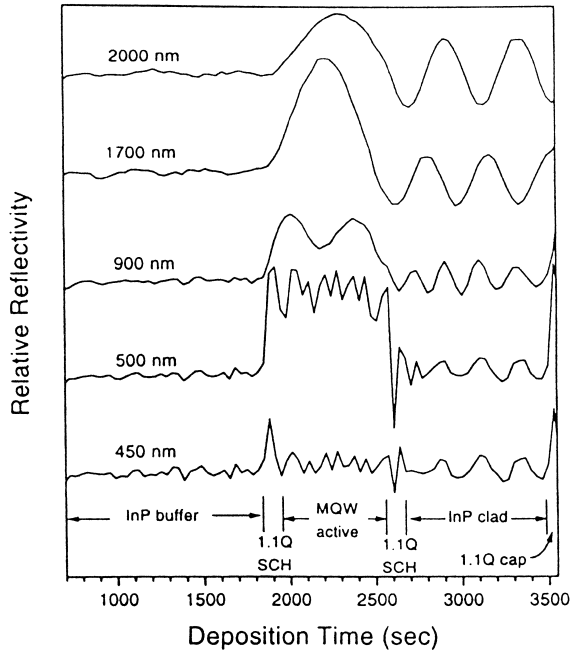

Fig. 6. Multiwavelength reflectance spectra obtained during the OMCVD growth of an InP-based multiquantum well laser device structure (after Ref. [25]).

to monitor the growth of a 1.3 μm InGaAsP laser device structure. Here, optical fibers were used to collect the reflected light and transmit it to a Si/PbS dual detector with a combined wavelength range of 400 to 2500 nm. Data obtained at several wavelengths are given in Fig. 6. This device structure consisted of an InP substrate, a InGaAsP confinement layer with composition giving a 1.1 μm bandgap, an InGaAsP/InP multiquantum well stack with a repeat period of 17 nm, a second 1.1 μm InGaAsP confinement layer, an InP cladding layer, and a final 1.1 μm InGaAsP confinement layer. The data obtained at 450 and 500 nm resolve the individual MQW structures, while those at longer wavelengths allow overall thicknesses to be determined. Interference oscillations in the envelope also provide information about layer thicknesses.

3.5. Monitoring by p-polarized reflectance spectroscopy

PRS is one of the ways that the unreacted part of the SRL can be accessed directly. The second, third, fourth, and lowest levels of Fig. 7 show the time dependence of the molar concentrations $n_i(t)$, thickness d_{SRL}, the real ε_1^r and imaginary ε_1^i parts of ε, and the p-polarized reflectance R_p for the growth of GaP on Si under pulsed chemical beam epitaxy (PCBE) conditions [33]. The sequence of exposure of the sample to tertiarybutylphosphine (TBP) and triethylgallium (TEG) is shown at the top. The n_i are the solution of the coupled differential system describing the decomposition process of TBP and TEG using a reduced order kinetic model, and R is calculated in a four-layer model at a wavelength of 632.8 nm, an angle of incidence of 74.1°, and a growth rate of 0.81 Å/s. A complete analysis of these data would require a spectral capability and either an atlas of spectra of the different species or an accurate theoretical calculation of such spectra. As mentioned below, accurate theoretical calculations are a difficult challenge, but one that needs to be met if the full diagnostic power of surface-optical spectroscopy is to be realized.

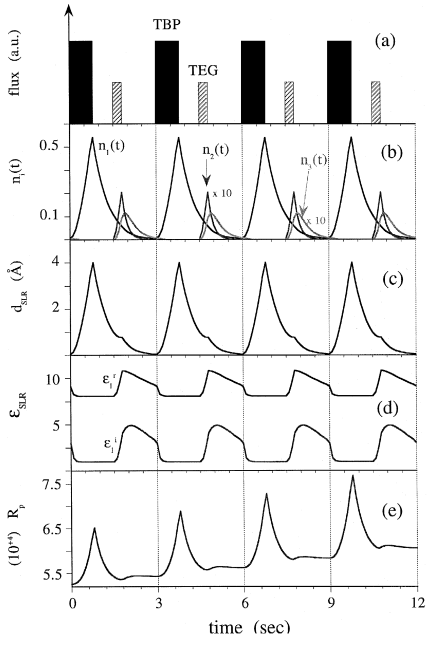

Fig. 7. Modeling of PR response during GaP growth on Si under PCBE conditions at 350°C: (a) precursor cycle; (b) molar concentrations of constituents in the SRL; (c) thickness dependence; (d) dielectric function dependence; (e) four-layer reflectance calculated as described in the text (after Ref. [33]).

4. A general control approach

Given the complexity involved, it is clear that control of semiconductor epitaxy must evolve into a multifaceted process where information from each region is obtained by specific probes and reduced by model calculations to required parameters. A general schematic is given in Fig. 8, with time increasing along the *region* axis from left to right and the *information* axis from top to bottom. We assume that information about the ambient is obtained by MS, the unreacted part of the SRL by PRS, the reacted part of the SRL by a combination of RDS, PRS, and LLS, and near-surface ε from KE with V-I theory. The analytic modules, scattering theory and surface chemistry, deal with the interpretation of the optical data and the modeling of the growth process, respectively. The scattering-theory model makes use of a database of the wavelength-dependent polarizabilities of the different species that in principle make up the unreacted and reacted parts of the SRL, allowing these concentrations to be determined. In principle these calculations are done at the level of individual polarizable species, and can therefore accommodate local-field effects, chemical complexes, and non-

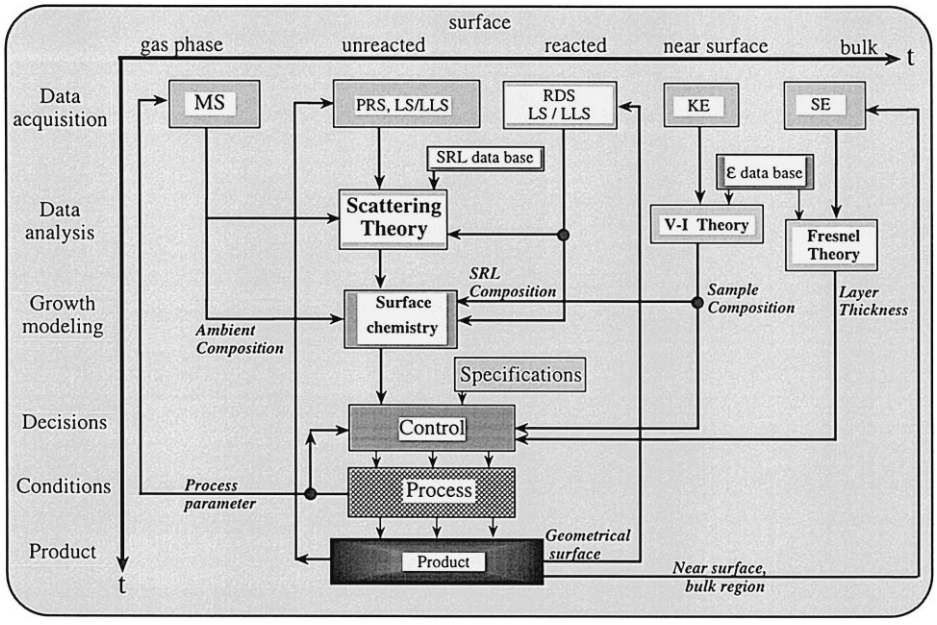

Fig. 8.

specular scattering by rough surfaces. The surface-chemistry module includes and extends the conventional approach where growth is modeled on the basis of process information alone. The control module compares the analyzed information to target values and adjusts process parameters accordingly.

Although this system remains to be implemented, the component capabilities have already been demonstrated to sufficient accuracy with the possible exception of the scattering theory and surface chemistry modules. The main challenge now is to develop these modules to the point where their capabilities match those of the probes.

5. Challenges

As can be seen from the above examples, considerable progress has been made in the control problem, yet a number of challenges remain. A major challenge, particularly with respect to quaternary materials such as $In_xGa_{1-x}As_{1-y}P_y$, is to enhance signal-to-noise ratios to where anion as well as cation compositions can be determined, especially in actual reactors where samples are rotated for uniformity and mechanical vibration may occur as well. Recent developments allow these to be addressed [18], [1] as well as the problem of window strain [34,35].

Surface-optical spectra must also be interpreted to achieve a quantitative understanding of growth chemistry. The quartz-optics spectral range, about 1.5 to 6.0 eV, is severely limited. Without a suitable atlas of visible-near ultraviolet spectra of surface species it has been necessary to rely on theoretical calculations. The task of calculating surface-optical spectra is extremely difficult even in the linear approximation, and recent experiments suggest that nonlocal and many-body effects must be considered as well [36]. However, new theoretical approaches, where the system is treated as a coherent superposition of scattering centers, offer some prospect that this problem will be solved, or at least addressed in more appropriate terms [37,38]. IR spectroscopic data would be welcome, but IR technology is not sufficiently sensitive to allow MLs to be measured routinely. Finally, techniques for probing the SRL, the closest region to the product, remain to be fully developed and tested. Accessing and understanding the properties of this region will be one of the interesting surface-physics challenges of the next several years.

However, the biggest challenge will be to win acceptance of these approaches in production. The difficulties faced in adapting control technology to production here are well summarized by Bean. [3] The major objective of growth is the production of devices, and success is measured in new capabilities and improved yields. It is unlikely that the advances discussed above, particularly any but the simplest, will be accepted until improved yields are demonstrated or devices that cannot currently be fabricated are produced.

Acknowledgements

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