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Real-Time Optical Diagnostics of Epitaxially Grown Semiconductors

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ABSTRACT

Various optical techniques are being developed to meet new challenges in epitaxial growth. I discuss representative examples involving the determination of bulk properties (layer composition and thickness), near-surface properties (compositional fluctuations), and surface properties (surface chemistry).

Epitaxial growth technology is facing increasingly difficult challenges. New materials such as wide bandgap semiconductors and epitaxial metals, more complex device structures such as vertical cavity surface-emitting lasers, increasingly stringent tolerances on layer compositions and thicknesses, emphasis on selective-area epitaxies for growing quantum structures, a general trend away from simple physical to complex chemical deposition methods, and the need to maintain satisfactory yields despite increasing economic pressures are moving the field toward real-time monitoring and control, i.e., toward obtaining accurate information about the process and/or sample itself during epitaxial growth. While present efforts tend to be concentrated primarily on accurately determining process parameters and modeling growth processes, the ultimate objective will be the accurate, real-time determination of the sample parameters themselves. Sample-oriented measurements are essential for closed-loop feedback control. They would also greatly relax demands on the determination of process parameters and on process modeling, because long-term drifts could be detected and corrected as they occur and complex details of the highly nonlinear growth process would not need to be considered. Even before the ultimate goal of closed-loop sample-driven feedback control is realized, the more modest and currently achievable target of real-time monitoring would allow conditions to be modified on-line to meet composition and thickness specifications, and would provide records by which causes of downstream failures of complex devices could be identified and eliminated.

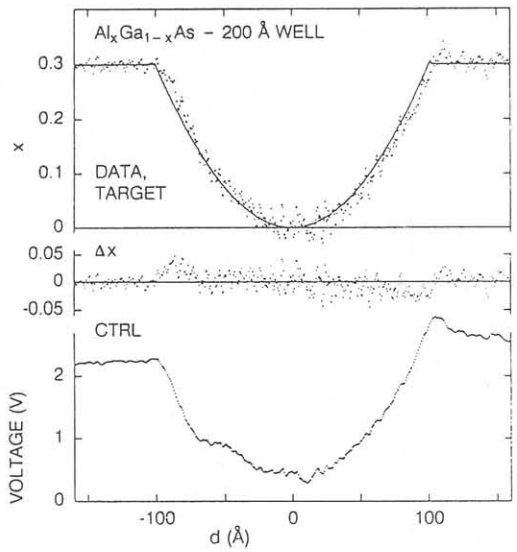
To meet these challenges attention is being directed toward optical techniques, which are nondestructive, noninvasive, and can be used in any transparent ambient. Of the various optical probes that could be used to monitor epitaxial growth, those that are receiving the most current attention are the bulk probes spectroreflectometry (SR)[1] and spectroellipsometry (SE),[2-6] the near-surface probe kinetic ellipsometry (KE),[7,8] and the surface probes laser light scattering (LLS),[9-11] surface photoabsorption (SPA),[10,12,13] and reflectance-difference (-anisotropy) spectroscopy (RDS/RAS).[14,15]

The bulk probes SR and SE return information integrated over the entire penetration depth of light. In particular, SR determines the scalar reflectances $R_s = |r_s|^2$, $R_p = |r_p|^2$, or $R = |r_n|^2$, where r_s, r_p, and r_n are the complex reflectances for s-polarized, p-polarized, and normally incident light, respectively. SE determines the complex reflectance ratio $\rho = r_p/r_s$. Reflectometry has long been used by the optical-coatings industry for real-time characterization of thicknesses and average compositions of depositing films[16] and has recently been adapted to the analogous problem in semiconductor technology, which is the fabrication of distributed Bragg reflectors.[17] However, much of semiconductor technology is based on samples consisting of layers that may be only a few Å thick or may have graded compositions. For these applications an optical technique that can measure phase as well as amplitude, i.e., ellipsometry, must be used.

For control purposes the primary quantities of interest are film thicknesses and compositions, which are bulk properties. The film-thickness measurement problem is that of endpoint detection. To perform this function quickly and accurately rapid-scan SE systems have recently been developed that can determine process switching points for either deposition or etching.[4] For thickness measurements a wavelength must be chosen where the material is transparent, because thickness information is encoded as the interference pattern between the waves reflected from the front and back surfaces of the film. For complex data such as ρ measured by KE, these interferences appear as exponential spirals when the data are plotted in the complex plane. For real data such as R obtained by kinetic reflectometry (KR), they appear as interference oscillations when the data are plotted as a function of time. Although either KE or KR data could be used in principle to detect thickness changes to within fractions of an Å, the advantage lies with KE because sufficient KR data must be taken to determine the maxima and minima of the interference oscillation so as to be able to locate the present datum on the pattern.

Compositional determination, and specifically the determination of compositional fluctuations, is a more difficult challenge. For control applications the composition of interest is not the average of a layer, as determined by conventional Fresnel analysis, but rather fluctuations of the composition of the most recently deposited material. These fluctuations should be measured over a thickness that is ideally vanishingly small, and certainly much less than the thicknesses of most films. In conventional Fresnel analysis this problem is resolved by dividing the layers into sections that are much thinner than the layers themselves, then determining the dielectric functions of the sections sequentially by solving the Fresnel equations in terms of the new data and the previously determined properties of the sample. Unfortunately, this feed-forward approach is unstable if the sections are chosen to be too thin, for example less than 25 Å.

The stability problem was recently solved with the development of virtual-interface (V-I) theory, which has significantly advanced analytic capabilities by providing a method of determining near-surface properties of depositing materials from optical data without any knowledge whatever of the underlying sample structure.[18] In the V-I approach the contribution of the underlying structure to the overall reflectance is summarized in one or two complex parameters, called virtual reflectances, which are determined along with the dielectric response of the section. This can be done no matter how complicated the underlying structure. Because the V-I approach does not use previously determined sample parameters, the layers can be made arbitrarily thin without introducing instabilities. This is precisely what is needed for sample-driven closed-loop feedback control. As an example of the power of this approach, the Bellcore group used V-I analysis of KE data to grow $Al_xGa_{1-x}As$ quantum structures with continuously graded compositions under sample-driven closed-loop feedback control, where the composition x was regulated to within several percent by analysis of the running outermost 3 Å of depositing material.[8] Data for a 200-Å-wide structure are shown in Fig. 1.



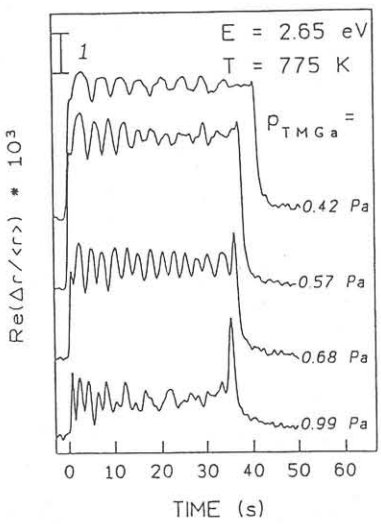


Fig. 1. Compositional data for a 200-Å-wide parabolic quantum well grown by sample-driven closed-loop feedback control. Top: data and target values; middle: difference; bottom: control voltage (after ref. 8).

Fig. 2. RD oscillations observed during OMCVD growth on (001) GaAs at H_2 and AsH_3 pressures of 100 mbar and 70 Pa, respectively, and a sample temperature of 502 °C. TMG pressures are as indicated (after ref. 15).

Surface diagnostics represent yet another aspect of real-time measurements. Because the connection between bulk and surface properties is relatively weak, the interest here lies primarily in determining growth mechanisms. The difficulties that must be faced are those of sensitivity and selectivity. Penetration depths of light are typically of the order of hundreds of Å, whereas the surface region is typically 1 Å thick. Thus over 99% of a reflected signal originates in the bulk while less than 1% can be attributed to the surface. Optical techniques that have proven useful in surface analysis include LLS, SPA, and RDS/RAS. All rely on symmetry to separate the relatively weak surface contribution from the much stronger bulk component. In LLS the measured property is diffusive scattering, so no signal is detected unless the surface becomes macroscopically rough. This can occur for example if the growth mode changes from layer-by-layer to island formation, or if a pseudomorphic layer becomes so thick that relaxation occurs through the formation of defects.[11] In SPA the quantity of interest is the change in reflectance that results when surface conditions are changed. SPA differs from surface differential reflection (SDR), an earlier, related technique, by using p-polarized light incident at the Brewster or pseudo-Brewster angle to minimize the bulk contribution.[10,12,13] In RDS the quantity measured is the total optical anisotropy of the sample. Because the bulk optical responses of the cubic materials of interest for semiconductor technology are isotropic, the anisotropies observed in an RDS measurement arise primarily from the lower symmetry surface.[14,15] Of the three approaches mentioned, LLS and RDS are true surface spectroscopies that can return information with the surface under steady-state conditions. Present sensitivities of SPA and RDS are significantly better than 0.01 monolayer.

Figure 2 shows an example of RDS applied to determine growth rates, here during epitaxial growth on (001) GaAs by organometallic chemical vapor deposition (OMCVD).[15] These data are the optical equivalent of reflection high energy electron diffraction (RHEED) oscillations commonly used in molecular beam epitaxy (MBE). Because OMCVD is a near-atmospheric-pressure growth technique, electron spectroscopies cannot be used, although it is possible to obtain information about long-range order through X-ray scattering.[19]

Future directions are clear. Real-time optical characterization will become increasingly important not only as a result of further refinements but also with respect to applications to new materials systems. Parallel multiwavelength data acquisition and processing, as demonstrated for example by Killeen et al.[1] in reflectance and Collins et al.[3] in ellipsometry, will be required for controlling growth of multinary systems. The use of optical multichannel detection with SPA and RDS will allow surface reconstructions in OMCVD to be assessed at a glance, as is now done with RHEED in MBE. At the same time, growth chambers will also evolve to meet the needs of optical measurements, specifically by providing optical access to the sample through transparent, strain-free, and deposition-free windows, and by the development of runout-corrected and vibration-free sample mounts and manipulators as recently demonstrated for MBE by Maracas et al.[6] Approaches dealing with lateral inhomogeneity by the use of small spot sizes,[2] imaging,[20] or direct analysis[21] will be developed to deal with the increasingly important phenomenon of selective-area epitaxy.

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