MOLECULAR-BEAM EPITAXY

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The growth in the use of single-crystal, multilayer, III-V semiconductor structures in optoelectronics and high-speed electronics has spurred the development of a variety of methods for precision crystal epitaxy. Among these are beam-epitaxy methods that use as sources either single elements or several combinations of simple chemical compounds. These molecular-beam-epitaxy methods permit the growth of very-high-quality structures with extremely thin layers, almost atomically smooth interfaces, and large and abrupt changes in doping. These growth methods have been the major source of prototype multilayer, III-V semiconductor structures (heterostructures) for basic physics and device studies, and show increasing promise as production methods for such structures.

43

Introduction

During the past two decades, there has been simultaneous interest in growing the single-crystal, multilayer, epitaxial semiconductor composites called *heterostructures*, and in developing devices that use the additional degrees of freedom in device design that the composites engender. These composites were first used almost two decades ago for heterostructure lasers grown by liquid-phase epitaxy (LPE).[1] (Panel 1 defines terms and acronyms used in this paper.)

By the mid-1970s, these lasers—in their simplest form—consisted of a layer of gallium arsenide (GaAs) about 1000Å (angstroms) thick, sandwiched between two 10,000Å layers of aluminum gallium arsenide (AlGaAs), all grown epitaxially on a GaAs substrate crystal. Note that all compositions of AlGaAs have about the same lattice parameter, which eliminates the interface defects that arise from mismatch. This multilayer structure has an abruptly changing composition at interfaces without major crystalline defects. Thus, one can manage light, holes, and electrons through controlled variation of the band structure within the single crystal that results from the composition variation.

Panel 1. Acronyms and Terms

Al_2O_3	alumina
AlGaAs	aluminum gallium arsenide
AlInAs	aluminum indium arsenide
As	arsenic
AsH_3	arsine
Be	beryllium
ESMBE	elemental-source molecular-beam epitaxy
Ga	gallium
GaAs	gallium arsenide
GaInAs	gallium indium arsenide
Ge	germanium
GSMBE	gas-source molecular-beam epitaxy
HBT	heterostructure bipolar transistor
HFET	heterostructure field-effect transistor
HSMBE	hydride-source molecular-beam epitaxy
InGaAs	indium gallium arsenide
InGaAsP	indium gallium arsenide phosphide
InP	indium phosphide
LPE	liquid-phase epitaxy
LSI	large-scale integration
MBE	molecular-beam epitaxy
MOCVD	metal-organic chemical-vapor deposition
MOMBE	metal-organic molecular-beam epitaxy
n-type	material in which electrons carry the current
P	phosphorus
p-type	material in which holes carry the current
PH_3	phosphine
RAM	random-access memory
RHEED	reflection high-energy electron diffraction
Si	silicon
Sn	tin
TEG	triethylgallium

The demonstration of the use of heterostructures for managing photons and electrical carriers provided powerful incentive for using them to study the physics of thin structures and to develop new devices. That incentive has been the driving force for the development of a variety of new epitaxy methods intended to provide greater versatility than available with LPE. Although beam-epitaxy methods can be applied to a variety of crystalline materials, we limit this paper to the growth of heterostructures of the particularly important, III-V compound systems:

- Aluminum gallium arsenide/gallium arsenide

(AlGaAs/GaAs). (This notation is used to indicate this is a multilayer structure with any number of $Al_xGa_{1-x}As$ and GaAs layers, all grown onto a GaAs substrate. The subscripts x and $1-x$ indicate the ratio of Al to Ga. Similar notations are used below, where InGaAs and AlInAs usually refer to the compositions $In_{0.53}Ga_{0.97}As$ and $Al_{0.47}In_{0.53}As$—the compositions that are lattice-matched to InP.)

- Indium gallium arsenide phosphide/indium phosphide (InGaAsP/InP), which includes indium gallium arsenide/indium phosphide (InGaAs/InP).
- Aluminum indium arsenide/gallium indium arsenide (AlInAs/GaInAs) on indium phosphide (InP).

It is reasonable to consider several of the semiconductor epitaxy techniques as a related series, where each member differs in one important aspect from its nearest neighbors. This series, illustrated in Figure 1, ranges from molecular-beam epitaxy (MBE), which uses only elemental sources—at one extreme—to metal-organic chemical-vapor deposition (MOCVD),[1] which uses easily vaporized molecular sources—at the other. It is important to note that the most important way in which the molecular-beam-epitaxy methods cited in Figure 1 differ from the vapor deposition methods is that vapor deposition is done at pressure ranges where movement of molecules is by viscous flow, while in MBE, molecular-flow conditions pertain. Other differences— such as the existence of a boundary layer near the growing surface, and the existence of complex flow patterns that depend on pressure and system geometry in MOCVD, but not MBE—are outgrowths of the difference between viscous flow and molecular flow. In beam-epitaxy methods, the pressure is in the molecular-flow regime, which implies the pressure is low enough that atoms or molecules moving in the growth environment can travel considerable distances without colliding with each other.

For purposes of this discussion, a *molecular beam* may be defined as a stream of atoms or molecules that effuse from a common source under vacuum condi-

44

tions where collisions with each other, or with residual gases, occur less often than collisions with surfaces. That is, the mean distance atoms or molecules travel between collisions with other atoms or molecules (the mean free path) is long compared to the distance the beam travels. Molecular-beam epitaxy is a method for growing epitaxial layers at low pressures by impinging atomic or molecular beams of elements that constitute the layer onto a heated single-crystal surface, where they react.

The original substrate surface is the template for the epitaxial growth. In the semiconductor systems discussed here, the various epitaxial layers have the same crystal structure. That is, they have the same geometric arrangement of the group III and group V atoms. Although often of different composition, the epitaxial layer is really a continuation of the substrate's crystal structure.

Early Beam-Epitaxy Studies. Two precursors to what we now think of as MBE are the studies by Günther[2] and by Davey and Pankey.[3] Günther demonstrated that stoichiometric polycrystalline GaAs films could be grown by vacuum deposition on germanium (Ge) and alumina (Al_2O_3) substrates. The walls of the vacuum system were kept hot enough (150°C) that an ambient pressure of arsenic (As) could be maintained near a substrate by heating an As source oven. A beam of gallium (Ga) atoms impinging on the substrate resulted when a Ga source was heated. Davey and Pankey modified that approach with improved vacuum conditions and a GaAs substrate, and obtained epitaxial GaAs layers.

About the same time, Arthur studied[4,5] the reaction kinetics of beams of Ga and As_2 molecules impinging on heated GaAs surfaces. He observed that, under certain conditions of substrate temperature, all the Ga atoms remained on the surface, but only enough As remained to react with all the Ga to form epitaxial GaAs. Because Arthur used an unheated (rather than hot-wall) system, the As and Ga sources both had to be in "line of sight" of the growing surface.

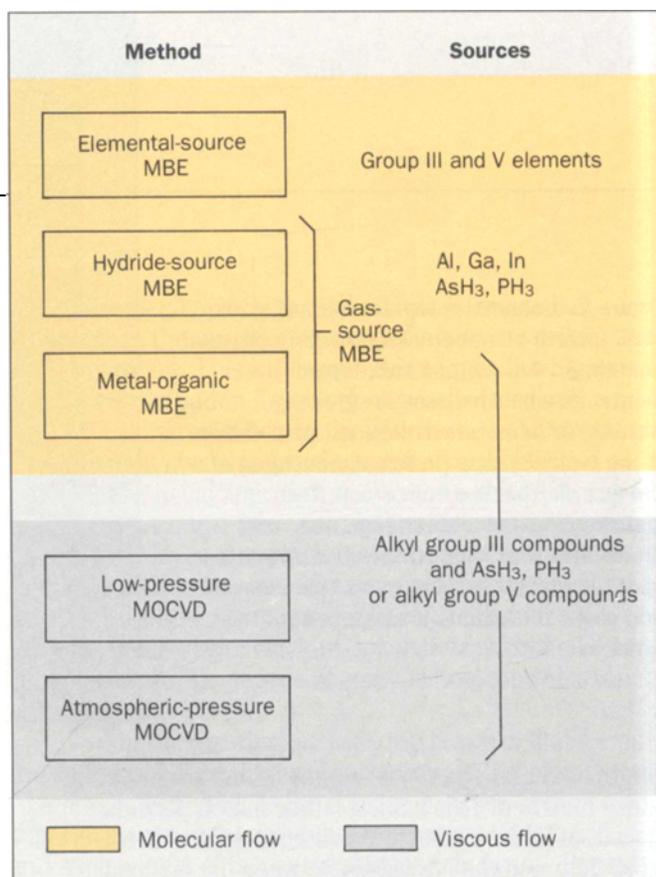Cho transformed the method into one for practi-

Figure 1. Related methods for III-V epitaxy.

cal crystal growth by increasing the source capacities, precisely controlling the temperature of sources, and using improved cryopanels to reduce unwanted impurities. He introduced *in-situ* reflection high-energy electron diffraction (RHEED) as a way to monitor the surface of the growing crystal[6] and study the fundamentals of III-V MBE. By 1971, Cho had produced[7] GaAs epitaxial layers of good enough quality that, for the first time, reasonable electrical and optical properties could be demonstrated with MBE-grown material. In an extensive series of studies that started in 1969 and are still underway, Cho developed MBE into a useful epitaxy method.

MBE-Grown Heterostructures. Cho, Panish, and Hayashi introduced the growth of heterostructures by MBE[8] with the growth of AlGaAs/GaAs in 1970. As did LPE before it, MBE of AlGaAs/GaAs heterostructures takes advantage of the fact that all compositions of Al and Ga in AlGaAs yield material with the same average bond

45

Figure 2. Schematic representation of an MBE growth chamber with effusion cells that contain Al, Ga, As, and two dopant elements, as would be used for growing AlGaAs/GaAs heterostructures. Also shown is the typical shape (in two dimensions) of the flux distribution from a cell. The glancing-angle electron beam, used only with the sample that is not rotated, is diffracted from the crystal surface to provide information about the atomic arrangement of the surface.
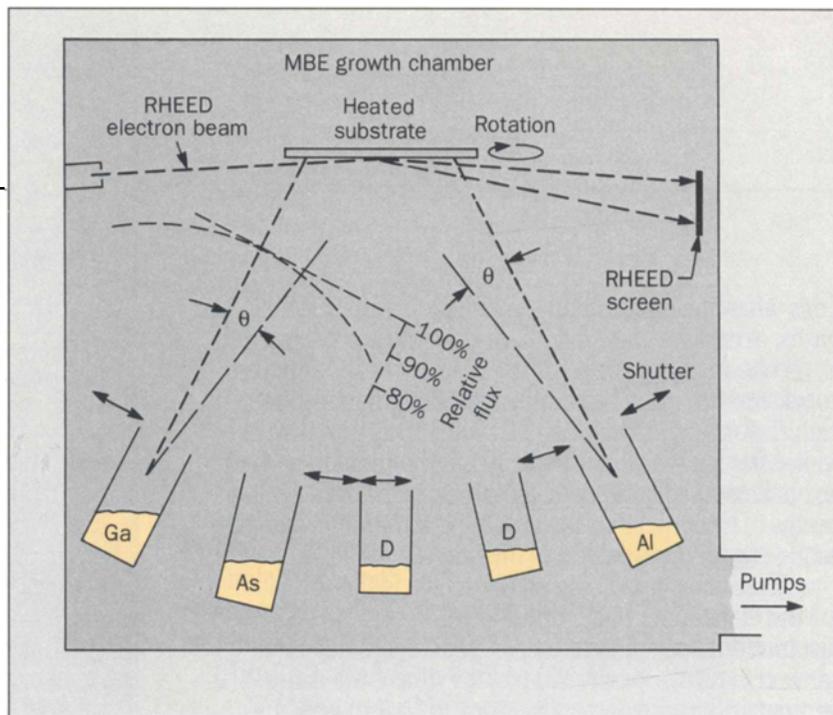
length as GaAs. That is, all compositions are naturally lattice-matched. This natural lattice match permits growth of layered structures without defects in the crystal structure at the interfaces between the epitaxial layers. For heterostructures with different combinations of III-V compounds (such as layers of AlInAs, GaInAs, and InGaAsP on InP substrates), the compositions of the elements in the layers must be precisely adjusted to attain a lattice match for growing materials free of crystalline defects. Nevertheless, the necessary control is possible, and materials combinations without a natural lattice match are being widely studied.

Epitaxy can even be done with non-lattice-matched combinations if the layers are thin enough,[9] often less than 100Å. Such layers are then strained, either positively or negatively, depending on the mismatch. If the layers are thin enough, defects caused by the mismatch are not present. With materials that are highly mismatched, the layers are frequently grown in structures that have alternating strained and unstrained layers and form a so-called *strained-layer superlattice*. Because strain modifies the material's electronic structure, materials combinations can be made that have properties that cannot be achieved without strain.[10] Thus, new applications are possible. The materials combinations,

layer thinness, and interface abruptness requirements often make beam-epitaxy methods the practical primary growth methods for such structures.

Molecular-Beam-Epitaxy Methods

As Figure 1 shows, the variants of MBE differ in the source of the elements used to generate the molecular beams.

The original form of MBE that Cho developed derives its atomic and molecular beams from evaporation of the elements. For convenience, we will refer to it as elemental-source MBE (ESMBE). The other variants of MBE are the gas-source MBE (GSMBE) methods, which are divided into two major categories. For ease of reference, we call them hydride-source MBE (HSMBE) and metal-organic MBE (MOMBE). For these GSMBE methods, simple volatile compounds—rather than the elements—are used as sources for generating the molecular beams.

The GSMBE methods are particularly useful for epitaxy of InP, InGaAs, and InGaAsP on InP single-crystal substrates, where there is no natural lattice match (as occurs in AlGaAs) and precise compositional control is needed to achieve lattice match. The MOMBE version of GSMBE is also seeing increasing interest for its per-

ceived potential for simple scaled-up epitaxy of these InP-based heterostructures and of AlGaAs/GaAs heterostructures, even though such scale-up has not yet been demonstrated.

Elemental-Source Molecular-Beam Epitaxy. The schematic in Figure 2 illustrates the arrangement of an ESMBE system[11,12] for growing AlGaAs/GaAs heterostructures. Evaporation sources exist for each of the elements—Al, Ga, and As plus two dopant elements—that constitute the final layers. The dopants are usually beryllium (Be) for p-type material and tin (Sn) or silicon (Si) for n-type material.

The evaporation sources are chemically inert, cylindrical or conical containers (usually referred to as *effusion cells* or *effusion ovens*) that hold the elements to be evaporated. The containers are heated enough to generate a vapor pressure of the source element within the cell. Effusion cells always have shutters in front for starting and stopping the molecular beam.

In Figure 2, note the schematic representation of a diffracted electron beam for RHEED, which provides all MBE systems with the capability for *in-situ* observation of surface roughness, surface atomic structure, and growth rate on a monolayer-by-monolayer scale. Because vapor pressures vary exponentially with temperature, precise temperature control of the effusion cell is necessary for precise compositional control of the layers. The beam flux of any species that effuses from the cell in the direction normal to the cell's orifice varies with the species' partial pressure in the cell as:

$$F = KP/(MT)^{1/2} \qquad (3)$$

where P is its partial pressure, T the temperature, and M the molecular weight of the species. K is a proportionality constant that includes several fundamental constants and a constant that depends on the cell geometry.

The flux of atoms or molecules in the beam that effuses from typical MBE effusion cells has a distribution in space that falls off with the angle θ from the normal to

the effusion-cell orifice; the fall-off rate is somewhat greater than the cosine of θ, depending on the cell's exact geometry. This is illustrated in Figure 2, where we show the distribution for an effusion cell aimed at the edge of a substrate. Clearly, the distribution of flux for each element over the growing surface is different.

The usual approach to overcome this difficulty is to rotate the sample fast enough that the composition of the growing layer at any point on the surface is the time-averaged flux. In this way, for a sample distance of 12 to 15 cm (centimeters) from the effusion cells, we can achieve AlGaAs layer thicknesses that vary about 0.3 percent/cm. A similar degree of composition control is also obtained.

On the growing surface, the reaction that forms the epitaxial AlGaAs layer, for example, is between Ga or Al atoms, which stick to the surface because of their low vapor pressure at the growth temperature (550 to 650°C), and some atoms of the impinging arsenic beam. Presumably, the Al and Ga atoms have enough mobility on the surface to move to appropriate growth sites. The arsenic arrives at the surface either as As_2 or As_4 molecules, depending on whether a simple effusion cell was used (this yields As_4), or a thermal cracker was added to the cell to dissociate (break apart) the As_4 into As_2. Foxon and Joyce have shown with GaAs[13,14] that the As_2 molecules dissociate on the surface, chemisorb (bind onto the surface), and react with Ga. The As_4 molecules react in a more complex way; and, even when there is excess Ga, a desorbed As_4 flux exists that amounts to half of the impinging flux. The important and net result of all this is: Only enough As atoms stay on the surface to react in a one-to-one ratio with all the Ga and Al, so that control of the gross stoichiometry of the growing layer only requires that there be an excess flux of As.

Hydride-Source Molecular-Beam Epitaxy. For compounds that contain phosphorus (P) and, particularly, both As and P, ESMBE has not shown significant results because it is difficult to achieve the control needed over the ratio of As to P in the flux of molecules that arrive at

47

Figure 3. Schematic representation of the arrangement of effusion sources in (a) HSMBE and (b) MOMBE systems. Note that the hydride crackers produce AsP molecules as well as As$_2$ and P$_2$ when the gases are mixed. D stands for dopant.
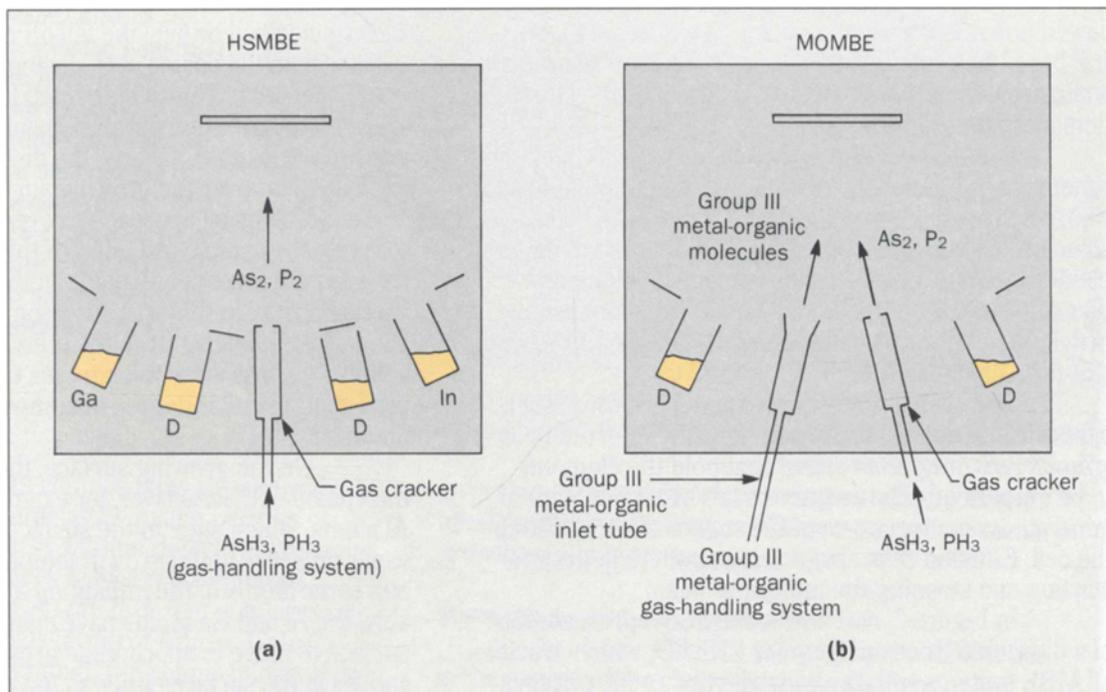
48

the growing semiconductor surface. This difficulty results partly from the exponential dependence of vapor pressure on the temperature of solid As and P, combined with poor temperature uniformity in the large effusion cells needed for those elements. In addition, severe difficulties are generally encountered when an elemental phosphorus source is used. Solid P generally is a mixture of allotropic forms of P, each with a different vapor pressure,[15,16] and control problems result. The species in the phosphorus beam from a solid P source is P$_4$, a molecule that apparently does not readily undergo the reaction that forms the epitaxial compound.

In 1980, the GSMBE group of methods (Figures 3a and 3b) was initiated[17,18] for MBE growth of heterostructures of InGaAs/InP and InGaAsP/InP that had dimensional precision and layer quality comparable to what conventional MBE had achieved with AlGaAs struc-

tures. In the GSMBE version closest to conventional MBE, atomic beams of the group III elements are generated by evaporation of Ga and In from effusion cells. But as Figure 3a shows, a beam that contains As$_2$ and P$_2$ is generated as a result of thermal decomposition of the hydrides, arsine (AsH$_3$) and phosphine (PH$_3$). Thus the suggested name: hydride-source MBE (HSMBE).

We started to replace the solid As and P with hydrides to make it possible to achieve well-controlled beams of phosphorus and phosphorus plus arsenic. In liquid form, AsH$_3$ and PH$_3$ have vapor pressures of about 15 and 35 atm (atmospheres), respectively, at room temperature. These compounds are stored outside the MBE apparatus, and the vapor is delivered into the system by a gas-handling system that uses precision pressure or flow control. This ensures precise control over the beam flux of both As$_2$ and P$_2$ in the MBE system.

Metal-Organic Molecular-Beam Epitaxy. In 1981, simple organometallic compounds—typically group III metal alkyls such as trimethylgallium—were introduced[19] to replace the group III elemental sources. By 1986, Tsang and Campbell had shown that these compounds were useful for epitaxy of high-quality InP and InGaAs layers.[20]

Figure 3b schematically illustrates the growth system used for MOMBE. This system incorporates thermal crackers for AsH_3 and PH_3 as in HSMBE; in the version shown, the dopant elements are still introduced from effusion cells.

An important feature of the method is: The metal-organic molecules are not decomposed *before* the beam is formed but, instead, make up the beam. This is possible because:

- These compounds are volatile enough that they can be admitted directly into the MBE growth chamber from an auxiliary gas-handling system.
- They react on the hot substrate surface to yield the group III element for epitaxy.

A potentially very important feature of MOMBE is that the group III beams are coincident (Figure 3b) because the uncracked metal-organic molecules are mixed in the gas-handling system's manifold and effuse into the MBE system through the same tube. Thus, for MOMBE, the ratios of flux among group III elements and among group V elements that impinge on the growing surface will be constant, even without substrate rotation. This has significant implications for scale-up, because the major geometric constraints imposed by effusion cells are removed.

In the absence of direct studies of the reacting surface, we must infer what we can about the growth chemistry from observations of the growth rate as a function of substrate temperature. Figure 4 shows the results of two interesting sets of studies for the growth of GaAs[21,22] and AlGaAs and InGaAs.[22] The data are for the equivalent GaAs growth rate. In Figure 4, curve (a) is for GaAs grown by MOMBE with beams of triethylgallium (TEG) and As_2. At lower temperatures, the increase in

growth rate with temperature results from the increasing efficiency in decomposition of the TEG to yield Ga for the growth reaction. It has been suggested[21] that the slow decrease above 500°C results from desorption of an intermediate decomposition product that contains Ga.

Curve (b) is for GaAs growth with beams of TEG and As_4. Its similarity to curve (a) implies similar growth chemistry. In curve (b), the abrupt decrease in growth rate above 650°C results from evaporation of Ga atoms from the surface.

Curve (c) is for the equivalent growth rate of GaAs in AlGaAs for conditions like those of curve (b), but here Al is added to the molecular beams. Notice that the slow decrease in growth rate observed in curve (b) is eliminated.
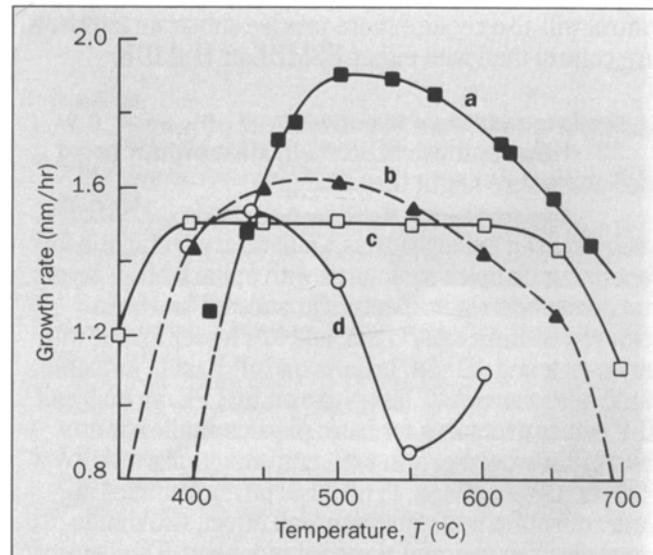

Figure 4. The growth rate of GaAs against temperature during MOMBE growth of: (a) GaAs with TEG and As2; (b) GaAs with TEG and As4; (c) AlGaAs with Al, TEG, and As4; (d) InGaAs with In, TEG, and As4. The data are taken from References 21 and 22.

49

Curve (d) is for the equivalent growth rate of GaAs under conditions similar to those of curve (b), but now In is added to the beam. The slow decrease in equivalent GaAs growth rate with temperature is exaggerated, occurs about 50°C lower, and then reverses above 550°C.

The results in Figure 4 strongly suggest that the loss of intermediate Ga species is inversely related to the chemical reactivity of the growing surface. The surface that contains Al is the most reactive, and the In-containing surface is the least reactive. The turn up of curve (d) above 550°C results from evaporation of In atoms above this temperature, which makes the surface more like GaAs. While we may expect different behavior with different organometallics, these results show that, for the growth of the InP-based heterostructures, compositional control will also require more-precise substrate temperature control than with either ESMBE or HSMBE.

Applications and Future Directions

How are these MBE methods currently being used and where might they lead us?

Elemental-Source Molecular-Beam Epitaxy. ESMBE has been used extensively as a laboratory technique for generating complex structures with epitaxial III-V layers that do not contain P. Spectacular success has been achieved with AlGaAs/GaAs, and to a lesser extent with lattice-matched AlInAs/InGaAs on InP. Except for some of the heterostructure lasers grown by LPE, virtually all III-V heterostructures for basic physics studies and devices have been grown first, and sometimes only, by MBE of AlGaAs/GaAs. [The basic physics studies include: quantum well, quantum Hall effect, two-dimensional electron gas, and resonant tunneling. The devices include: detectors, heterostructure field-effect transistors (HFETs), heterostructure bipolar transistors (HBTs), resonant-tunneling diodes and transistors, intersubband detectors, quantum-well lasers, and superlattice optical modulators.] Recently, ESMBE has been scaled up as a production process[23,24] with multiwafer capability for very

uniform AlGaAs/GaAs structures that have very low defect densities. Successful development of 16-kb (kilobit), static RAM LSI circuits with such scaled-up production has been reported.[24] (RAM is random-access memory, and LSI is large-scale integration.)

ESMBE's major previous success, even without scale-up, has been for the growth of the AlGaAs/GaAs HFET,[25] which required both extreme interface and doping abruptness, a combination that appears to cause difficulty for other epitaxy methods. It is reasonable to expect that, except for materials combinations requiring phosphorus, ESMBE may become the method of choice for some device types that require difficult combinations of materials, doping profile, interface abruptness, and layer thickness. But for III-V structures that require phosphorus and possibly other materials, the potential ease of scale-up with MOMBE may be a decisive factor about what method is used for some applications.

Hydride-Source Molecular-Beam Epitaxy. The original goal of HSMBE—growth of lattice-matched heterostructures in the InGaAsP/InP system, with precision epitaxy comparable to that of MBE AlGaAs/GaAs—has already been achieved. As a result, many of the structures just mentioned for AlGaAs/GaAs have been generated for the first time with InGaAs/InP and InGaAsP/InP. Such achievements are particularly important for devices associated with lightwave communications systems, where the lasers are already InGaAsP/InP heterostructures.

The long-range goal in such studies is to integrate several functions, in addition to light generation, on a single InP chip. A start has been made on superlattice modulators,[26] and the heterostructure bipolar transistor (HBT) is a particularly attractive transistor candidate. As with the AlGaAs/GaAs HFET mentioned above, the optimum $In_{0.53}Ga_{0.47}As/InP$ HBT requires abrupt interfaces and doping profiles, and virtually ideal pn junctions. (A *pn junction* is the region where two layers—one p-type and the other n-type—come into contact.) Here, the doping profile is particularly critical. The $In_{0.53}Ga_{0.47}As$ base is less than 500Å thick and doped p-type to about

50

10^{20} cm^{-3}. Immediately adjacent are n-type layers doped about two to four orders of magnitude lower. Recent demonstrations of the world's fastest bipolar transistors,[27] with frequency response as high as 165 GHz (gigahertz),[28] show that HSMBE structures can meet these stringent requirements.

An added bonus in these studies has been the observation that surface recombination in the InP-based heterostructures is a factor of about 1000 slower than in GaAs. This reduces the effect of surface recombination in small devices that have relatively large ratios of surface to junction area and has allowed devices to be scaled down to submicrometer size with little loss in gain. As a result, it appears that high-speed InP-based HBT circuits will also become important.

Metal-Organic Molecular-Beam Epitaxy. The use of a single, group III source tube to replace the group III effusion cells and the achievement of high-quality InP and InGaAsP layers and heterostructures are particularly important steps in the development of beam-epitaxy methods. As with HSMBE, a variety of InGaAs and InGaAsP/InP heterostructures have been grown for both physics and device studies. It seems clear that the interface abruptness and quality obtained with MOMBE are at least as good as with HSMBE. Qualitative comparisons suggest that good surface morphology is easier to achieve, and lower residual doping (less than 10^{15} cm^{-3}) has been demonstrated.

Few studies of MOMBE of AlGaAs/GaAs heterostructures have been reported, except for Tsang's demonstration[29] of quantum-well lasers with very low threshold current. High-quality AlGaAs with very low residual doping has not been reported. This may be partly because the method is so new that few workers have used it as yet. However, the chemically active nature of the AlGaAs, as suggested by the results summarized in Figure 4, may make epitaxial AlGaAs without carbon incorporation more difficult to achieve than with InGaAsP layers.

A major untapped advantage of MOMBE is: It reduces geometric constraints that complicate scale-up in the other MBE methods, and eliminates the flow nonuniformity that may occur with viscous flow in chemical-vapor deposition (CVD) methods. Although, MOMBE's major accomplishments have been with the InGaAsP/InP system, a major current interest in scale-up appears to be for growing AlGaAs/GaAs HFET structures. At present, all MOMBE studies are done in MBE systems that were designed for ESMBE. It is interesting to speculate that the reason for this is that, lacking a convincing demonstration of sufficiently high-quality AlGaAs with MOMBE, the conservative approach always appears to be to have available the capability for ESMBE. If AlGaAs quality improves or demand for InGaAsP/InP structures increases, this will change. This implies that one of the next advancements in beam-epitaxy methods will be the use of systems designed specifically for MOMBE.

51

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52