THE PRODUCTION OF HIGH QUALITY, III-V COMPOUND SEMICONDUCTOR CRYSTALS

Jim E Clemans, William A. Gault, and Eric M. Monberg

Jim E Clemans *is a senior member of technical staff in the Compound Semiconductor Technology Department at AT&T Engineering Research Center (ERC) in Princeton, New Jersey.* ***William A. Gault*** *is engineering manager of Compound Material Preparation and Wafer Fabrication at AT&T Technology Systems in Reading, Pennsylvania.* ***Eric M. Monberg*** *is a supervisor in the Electronic Materials Research Department at AT&T Bell Laboratories in Holmdel, New Jersey. However, the work reported in this paper was done while Messrs. Gault and Monberg were still at ERC. Mr. Clemans, who joined AT&T in 1977, develops manufacturing processes for growing high quality, gallium arsenide crystals. He has a B.A. in mathematics, and an M.S. and Ph.D. in physics, all from the University of Illinois at Champaign-* (continued on page 98)

A new technique, called the Gault process, has been developed at AT&T Engineering Research Center for growing large, single crystals of gallium arsenide, gallium phosphide, and indium phosphide. It can produce seeded, 50-mm diameter crystals that have lower dislocation densities and more uniform properties at all levels of doping than crystals grown by existing methods. In this paper, we describe the Gault process, which is based on the vertical gradient freeze method. Its features include seeded crystal growth in a boron nitride crucible, with much lower axial and radial thermal gradients than the conventional, liquid-encapsulated Czochralski process. We show that establishing an appropriate phosphorus or arsenic vapor pressure over the melt is an effective method of stoichiometry control. Also, a nearly planar liquid-solid interface has positive implications for radial uniformity and the occurrence of twinning and polycrystalline nucleation.

Compound Semiconductor Applications

At AT&T, III-V compound semiconductors are used for photonic and high-speed electronic applications (Table I). For example, we use gallium arsenide (GaAs), gallium phosphide (GaP), and indium phosphide (InP) in light-emitting diodes (LEDs), lasers, infrared detectors, field effect transistors (FETs), and high-speed, low-noise integrated circuits (ICs). The fundamental properties of each compound, such as band gap and carrier mobility, determine the specific areas for application.

For all these devices, the manufacturing sequence begins with a properly prepared, single-crystal substrate. Because crystal quality relates directly to the final device's performance and manufacturability,

Table I. AT&T Uses for III-V Compound Semiconductors

Device	Area of application		
	GaP	GaAs	InP
Photonic			
LED	Red Yellow Green	0.82 μm, 0.88 μm	1.3 μm, 1.55 μm
Laser	—	0.82 μm	1.3 μm, 1.55 μm
Detector	—	0.82 to 0.88 μm	1.3 to 1.5 μm
Electronic			
FET	—	High speed, low noise	Potential for higher speed circuits (than with GaAs)
IC	—	High speed, low noise	
Other	—	Potential for optical computers	Potential for optical computers

Table II. Physical Properties of Semiconductors

Compound	Melting point (°C)	Vapor pressure (atmospheres)
GaP	1467	35
GaAs	1238	0.9
InP	1062	25
Si	1410	0.000

high-quality crystals are critical to the economical manufacture of these devices.

The compound nature of III-V semiconductors presents crystal growth problems that are not found with elemental semiconductors, like silicon and germanium. At the melting point of the compounds, the Group V constituent (e.g., arsenic or phosphorus) has a large, equilibrium vapor pressure (Table II). If left unconstrained, the Group V element will escape from the melt, altering its composition.

To maintain the melt's stoichiometry, one usually uses either a molten encapsulant, backed by an overpressure of inert gas, or a sealed ampoule, provided with a suitable pressure of the volatile Group V element from a separate source. As Table II shows, the large vapor pressures associated with the phosphides dictate a process that involves a sealed, high-pressure chamber. For GaAs, both high- and low-pressure growth in a sealed quartz ampoule are practical.

Before the Gault process was developed, there were two commercial methods for growing III-V crystals:

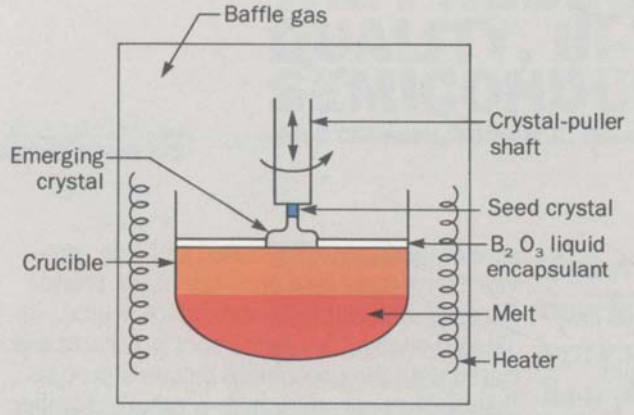
- The liquid-encapsulated Czochralski (LEC) process (Panel 1) is commercially applied to GaAs, GaP, and InP.
- The horizontal Bridgman (HB) process (Panel 2) is commercially applied to GaAs.

The vertical gradient freeze (VGF) method had been applied to GaP only in laboratory environments before the development of the Gault process (Panel 3).

The LEC Process. The liquid-encapsulated Czochralski process is the work horse of III-V crystal growth manufacturing. Growth equipment is commercially available that produce up to 3-inch diameter, round crystals.

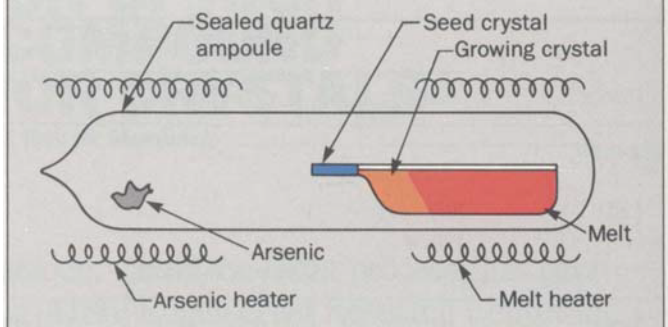
However, the LEC process has several serious drawbacks:

- Crystal growth and later cool-down occur in the presence of a large thermal gradient along the length of the ingot. This *axial* gradient and the resulting rapid cooling create high radial thermal gradients in the crystals, creating thermal stresses that, in turn, cause crystal defects such as dislocations.
- If axial temperature gradients are reduced to lower dislocation levels, diameter control becomes difficult. The lower axial gradient gives a hotter crystal surface and increased decomposition of the crystal surface (above the encapsulant). This causes droplets of indium or gallium to form and run down to the liquid-solid interface and interferes with single-crystal growth.
- Rotating the melt or crystal for diameter control causes



Panel 1: Liquid Encapsulated Czochralski (LEC) Process

A solid crystal is formed as the seed crystal is withdrawn into the cooler region above the melt. To enhance thermal symmetry, both the seed and melt are rotated. The axial temperature gradient is large, in part to maintain a stable diameter. By acting as a diffusion barrier between the baffle gas in the pressure chamber and the volatile, Group V constituent of the compound semiconductor melt, the boric oxide encapsulant prevents the melt's decomposition.



Panel 2: Horizontal Bridgman (HB) Method

Solidification occurs as the seed end of the crucible is moved to a cooler zone by relative motion of the ampoule and the heater assembly. To maintain a partial pressure of arsenic (As) throughout the sealed quartz ampoule, a reservoir of As is heated at the cold end. This partial pressure prevents the decomposition of the gallium arsenide (GaAs) melt.

striae (thin, narrow grooves) in the material.

- No simple method exists for stoichiometry control during the growth run.

Attempts to improve the quality of LEC crystals—through thermal engineering of sophisticated automatic diameter control, magnetic fields to reduce striae, and (for GaAs) arsenic overpressures to reduce decomposition—have met with partial success at the expense of complexity.

For GaAs and InP, other investigators have reduced dislocation densities by adding extra dopant impurities that harden the crystal lattice. But the high levels of strain frozen into the crystal's surface make these crystals more difficult to slice and handle than LEC crystals with lower dopant levels.

In addition, the highly doped InP is nonuniform and has a large, optical absorption coefficient. The high absorption means these crystals are not well suited for either back-illuminated detectors or back-emitting LEDs. Therefore, LEC-grown substrates are a compromise between the lowest dislocation levels and low absorption coefficient.

The HB Process. The horizontal Bridgman process, which has grown low dislocation GaAs crystals, uses a reservoir of arsenic to control the melt composition.

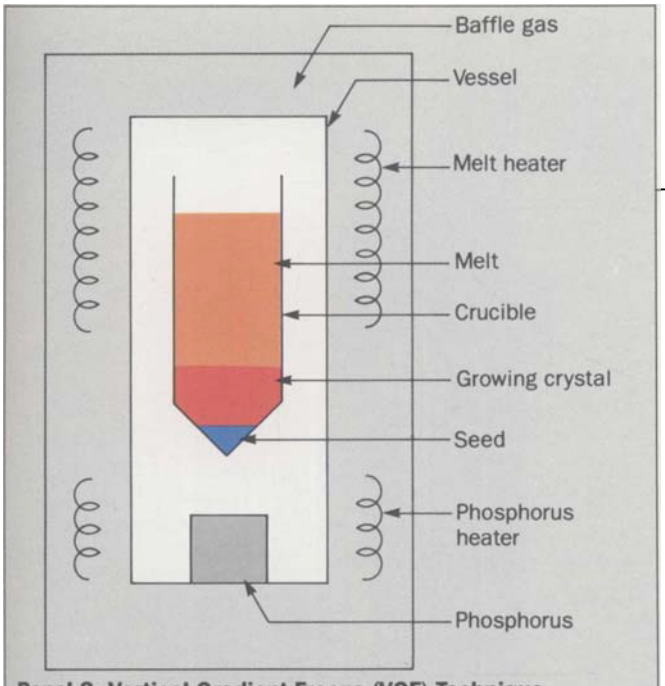
Although this process uses much lower temperature gradients than the LEC method, its application has been limited to GaAs. For any practical size of crystal, the fragile, sealed quartz ampoule may rupture at the large pressures used in growing GaP and InP crystals.

From a manufacturing perspective, the D-shaped wafer that is characteristic of HB crystals leads to handling difficulties and lower process yield. Also, because of the asymmetric, axial thermal environment of the HB furnace, the liquid-solid interface shape is not parallel to the plane of the sliced wafer. This produces nonuniformities in the electrical properties across the wafer.

VGF and Gault Methods. There have been several reports[1,2] of GaP grown by the vertical gradient freeze method. However, until the Gault process was applied, the VGF method had not been developed into a practical, crystal growth process.

The Gault process[3,4] was designed specifically to grow high quality, III-V crystals (GaP, GaAs, and InP) economically in a manufacturing environment. It has the following features:

- The equipment was designed for reproducible and simplified operation. Automatic process control equipment allows unattended growth.



Panel 3: Vertical Gradient Freeze (VGF) Technique

Solidification occurs in an axisymmetric fashion when power to the melt heater is reduced. The liquid-solid interface is in the horizontal plane as it advances vertically while the seed end cools. Very slow cooling is possible because a crucible defines the diameter and the cold-on-the-bottom growth method is not turbulent. A partial pressure from the separately heated, Group V constituent reservoir prevents melt decomposition.

- The thermal configuration incorporates low axial and radial gradients. Also, the gradients can be tailored to optimize growth conditions for each compound.
- When growth is completed, crystal cooling is controlled to minimize the generation of dislocations.
- Only high-purity boron nitride components are used in the hot zone of the growing crystal.
- The growth components are designed to control heat flow and the shape of the liquid-solid interface.
- Analogous to the GaAs HB process, the melt composition is controlled from a reservoir of the Group V element.
- The bulk crystal is nucleated by an oriented, seed crystal that provides reproducible growth direction.

Particular attention was given to safety issues associated with high pressure, electrical components, and chemical hazards.

Crystal Growth Equipment

The most important part of the crystal growth equipment (Figure 1) is a vertical, two-zone heater assembly that is enclosed in a water cooled, pressure vessel. For chemical inertness and cleanliness, stainless steel lines the walls of the vessel. Cooling water is channeled internally between the forging and the stainless steel liner.

A growth vessel inside the heater assembly (Figure 1b) contains elemental phosphorus or arsenic, polycrystalline III-V material. The control heater in the lower zone heats the reservoir of condensed phosphorus or arsenic and generates a vapor in the growth vessel. For stoichiometry control, this vapor of V component provides pressure at the surface of the melt.

The charge heater in the upper zone heats a bottom-seeded, boron nitride crucible that contains a charge of the III-V compound. This zone controls solidification and heat transfer during crystal growth.

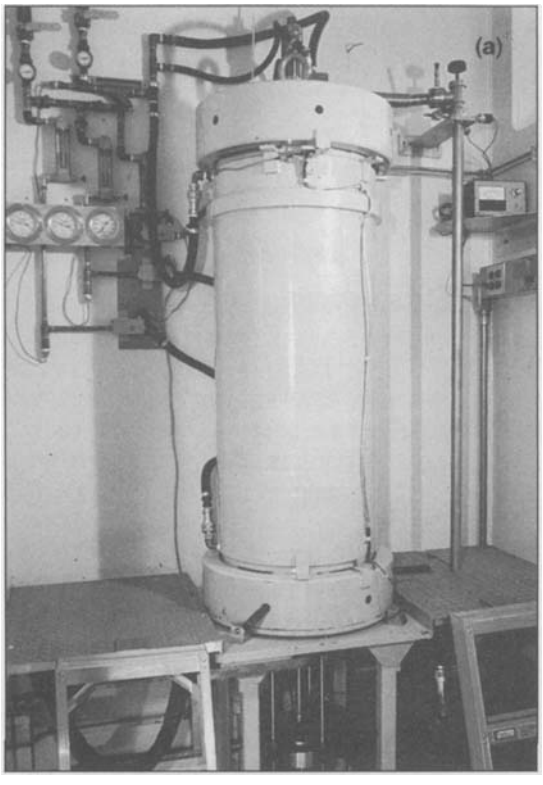
Rotation of the growth vessel averages the equipment's thermal asymmetries. In addition, we can translate the growth vessel (move it up or down) in the heater to adjust the crucible's position in the temperature profile.

Pressure Vessel. The stainless steel lined, pressure vessel was designed for safety and operating convenience. Although designed for 2500-lb/in^2 operation and tested at 3750 lb/in^2, the vessel generally operates no higher than 1000 lb/in^2.

Plug-type end caps, held by locking nuts that are secured by interrupted threads, seal the pressure vessel. Only partial rotation of the cap is required to open or close it.

A specially designed vacuum-pressure valve on the top end cap permits rapid evacuation of the system to 10 μm of mercury (Hg), yet can be safely sealed at high pressures. Located on the bottom end cap are high-pressure seals for a rotation and translation shaft, thermocouples, high-current power leads, and inlet and outlet lines for pressure control of the inert gas.

The Heaters. The upper-zone charge heater is a graphite resistance heater that consists of a shaped, *picket*



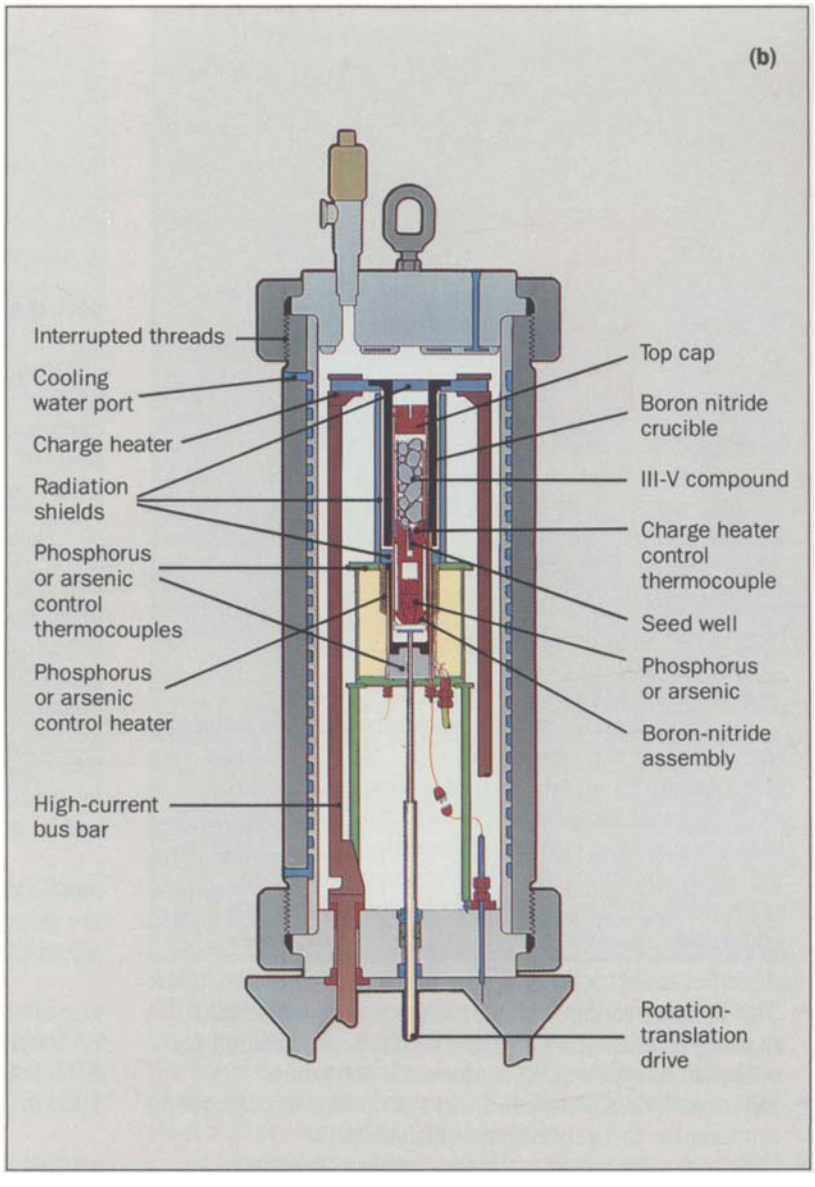


Figure 1. Crystal growth equipment. (a) Gault process, high-pressure vessel. (b) Cross section of high-pressure vessel and internal assembly; for clarity, only one bus bar is shown completely.

fence element, similar to the one used in Czochralski crystal growth. To control resistance and the resulting axially symmetric, power dissipation pattern, the cylindrical heating element is machined to different thicknesses along its length. This provides a way to control temperature gradients in the growth crucible.

The heating element is supported by two high-current bus bars that carry electrical power from a remote low-voltage, high-current power supply.

An insulating radiation shield surrounds the charge heater and extends along its length. A second shield below the heater isolates the low- and high-temperature zones. To improve temperature stability, the junction of the control thermocouple is in the bottom of the charge heater next to the crucible's seed well.

Directly below the charge heater is the lower zone, or control heater for phosphorus or arsenic. The main control thermocouple is housed in the hollow rotation-translation shaft, and the junction is positioned as closely as possible to the bottom of the growth vessel. This is the coldest region in the phosphorus or arsenic heater and the best thermocouple location for vapor pressure control.

Growth Vessel. Figure 2 is a more detailed view of the pyrolytic boron nitride (PBN) growth vessel and its contents. Below the support pedestal is a reservoir for phosphorus or arsenic.

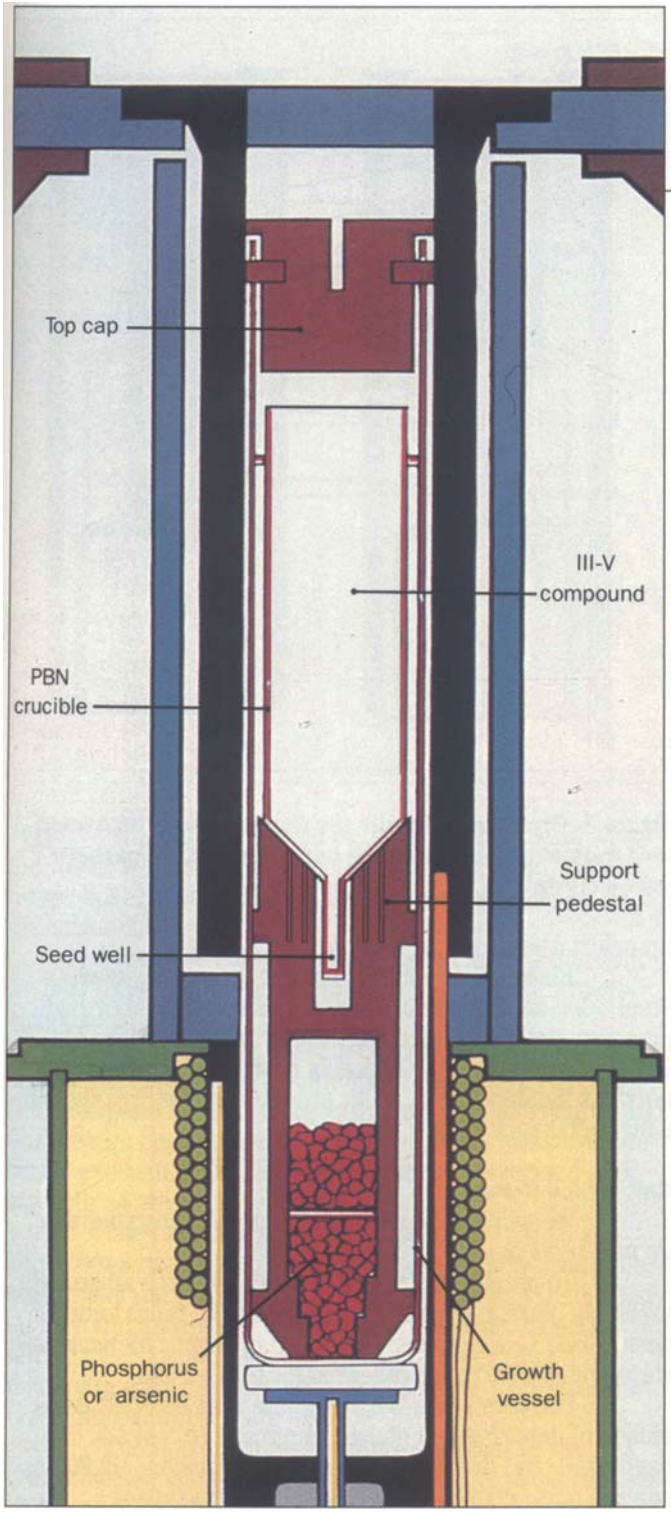


Figure 2. Growth vessel assembly, showing the pyrolytic boron nitride (PBN) crucible.

The support pedestal is a cylinder of hot-pressed boron nitride (HPBN) whose design and thermal conductivity contribute to the desired balance between radial and axial heat loss. For example, several concentric slots are machined around its seed well area. By determining the number and dimensions of the slots, we can alter the thermal geometry and resulting liquid-solid interface shape in the seed well region.

The PBN crucible is nominally 50-mm internal diameter and tapers down to hold a 6.3-mm seed. A cap of HPBN, which is pinned to the top of the PBN growth vessel, confines the phosphorus or arsenic vapor and provides a way to lift the growth vessel into and out of the heater assembly.

During growth, controlled loss of phosphorus or arsenic vapor occurs through the annulus between the top cap and the wall of the growth vessel. However, enough condensed phase, phosphorus or arsenic remains in the reservoir to control melt composition during the growth run.

Thermal Profile. Combinations of charge heater shape, radiation shielding, insulation, and support pedestal slots create the desired axial and radial gradients. These combinations are specific to each crystal compound and the typical operating pressures used.

To measure temperature profiles under growth conditions, a thermocouple is translated automatically along the central axis of the heater zones. A dummy load, which is machined from a solid block of HPBN, takes the place of the crucible that contains the molten and solid semiconductor. The axial thermal gradient measured over the bulk of the HPBN is small, about 10°/cm. It was desirable to increase the gradient in the seed well region to achieve a repeatable position for starting crystal growth.

A goal in material selection and placement of thermally important pieces is to achieve a profile shape that does not change significantly during growth. We can approach this condition through proper balance of convective, conductive, and radiative heat flow over the temperature range of crystal growth. But we must also

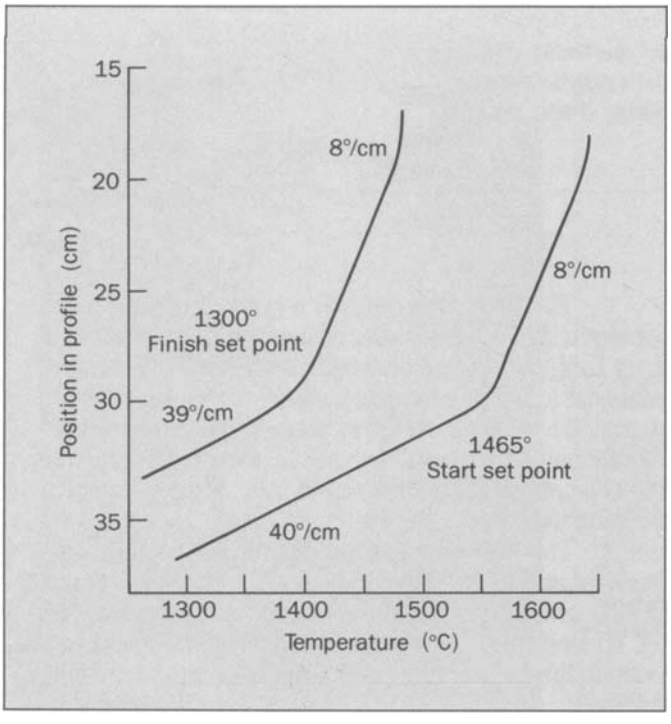


Figure 3. Measured temperature profiles for gallium phosphide (GaP) under simulated growth conditions.

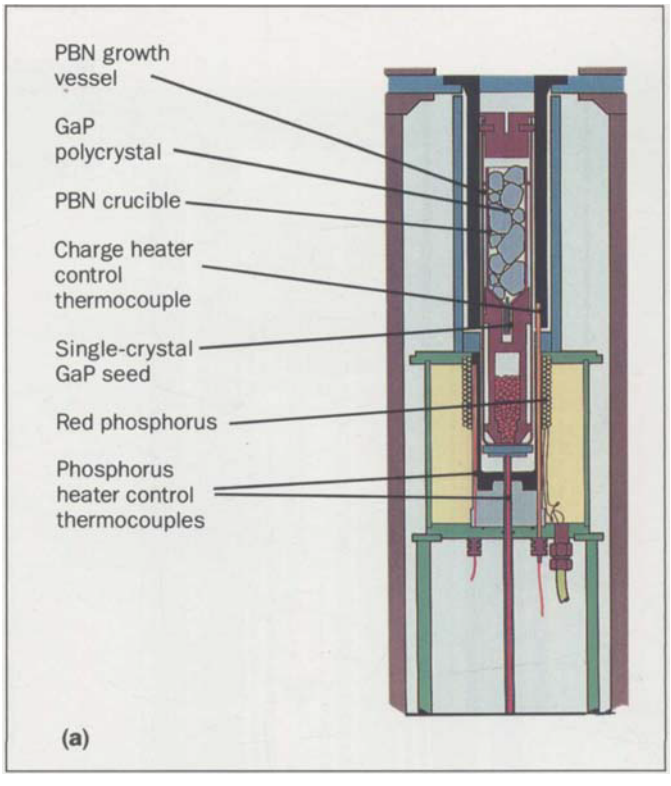


Figure 4. Crystal growth with the Gault process: (a) loaded cold assembly, (b) system just before growth, (c) partially grown crystal.

consider the dynamic reduction in thermal conductivity of the melt-solid system during growth, because the solid has less than half the thermal conductivity of the shrinking melt portion of the charge.

Figure 3 illustrates two axial thermal profiles, measured in the HPBN charge. They correspond to the start and finish conditions for the growth of a 1000g charge of GaP. In the seed region, the measured gradient changes by only 1°C/cm during growth; in the bulk crystal region, it remains unchanged. Experience has shown that these measured gradients are a valuable tool for calculating growth rates in the system.

Safety Features. Safety is a major consideration in the design of the facilities, and all high-pressure components were selected with at least double margins of safety. A comprehensive fail-safe system anticipates serious problems and, in this rare event, safely shuts down the grower.

The growers are housed individually in small rooms, and appropriate exhausting is used when the pressure vessels are opened. For gallium arsenide growth, the special working areas are under negative pressure compared to the surrounding building so that no airborne arsenic particulates can escape.

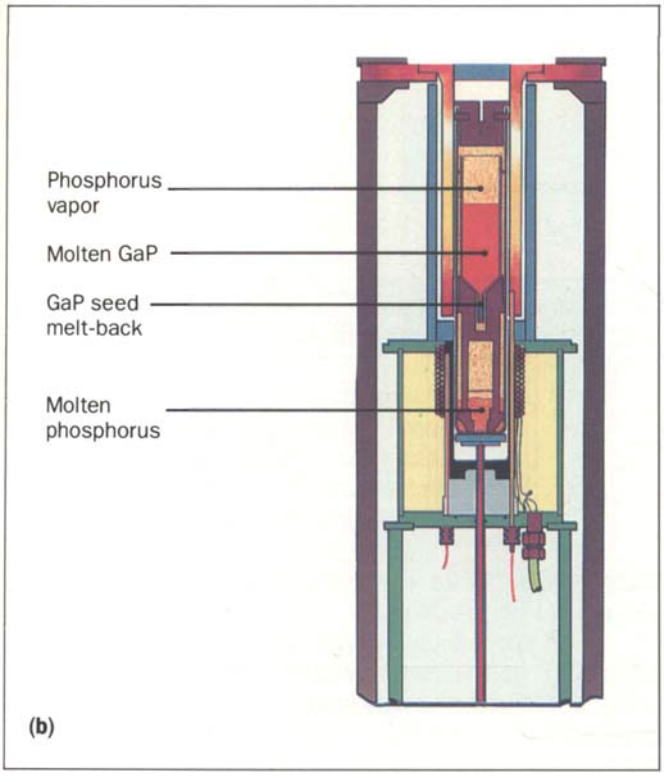
Residual phosphorus condenses on the stainless steel walls of the pressure vessel and is removed by high-pressure steam. For cleaning arsenic from the walls of the GaAs growth system, a specially designed apparatus entrains the deposits in an air stream and captures them in a high-efficiency filter.

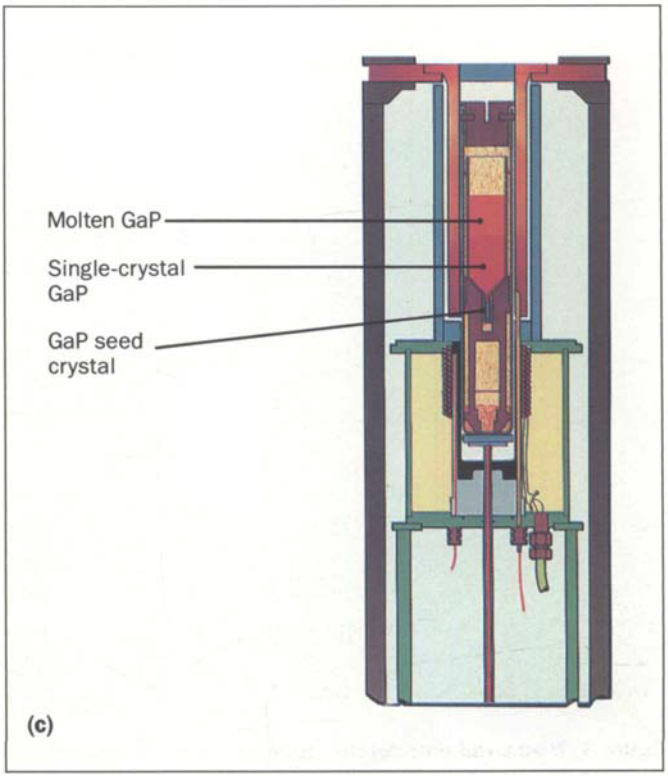
GaP Crystal Growth

We will use GaP as the example to describe the growth sequence.

To prepare for crystal growth, we etch all growth assembly parts in aqua regia and then bake them to dryness at 600°C in a stream of flowing nitrogen. Meticulously clean and dry parts are essential for high yields.

Polycrystalline GaP, with less than one part per million of impurities, is etched to remove excess Ga, degreased, and dried. Red phosphorus, which is 99.999 percent pure, is used as received. Seed crystals are





<111> oriented and etched to fit the crucible seed well; the <111> phosphorus direction faces the melt. We add n-type dopants as either Ga_2Te_3 (gallium telluride) or Ga_2S_3 (gallium sulfide).

When the components are assembled (Figure 2), we load the growth vessel into the high-pressure growth equipment.

The pressure vessel is evacuated to 100 μm of Hg or less, backfilled with a baffle gas of high-purity argon, and evacuated again (Figure 4a). Next, the phosphorus and GaP charge heaters are heated to low levels while the system is evacuated. Finally, the system is pressurized with argon to 54 atm.

The GaP charge heater is heated to a final set-point temperature that will melt the entire polycrystalline charge and the top part of the seed, exposing a fresh seed surface and enhancing subsequent nucleation. Concurrently, the phosphorus control heater is heated at a rate that continually provides phosphorus vapor to control the GaP composition (Figure 4b).

The maximum temperature set point of the charge heater is maintained for two hours to allow the system to reach a steady state. Then, the temperature is slowly reduced, and the melt begins to solidify onto the seed as a single crystal. The crystal grows at a rate of about 3 mm/hr (Figure 4c).

Because of the heat added to melt the polycrystalline GaP, the argon pressure rises from its initial value of 54 atm to about 65 atm before crystal growth starts. It is held at this level during growth.

Once the entire melt has solidified, the crystal is controllably cooled at a rate of 100°C/hr for five hours. This step, along with the low axial temperature gradient, is essential for obtaining low dislocation levels.

Later, when the system has cooled to room temperature, we vent the pressure, and remove and disassemble the PBN growth vessel. The ingot slips free from the PBN crucible, which can then be cleaned and reused.

Most of the growth cycle is automatically controlled. After the system has been pressurized and the heater program has been manually started, the system runs unattended during the typical 40-hour cycle.

Results and Discussion

We used spark source, mass spectrometry to measure the impurity levels in seed and tail slices from

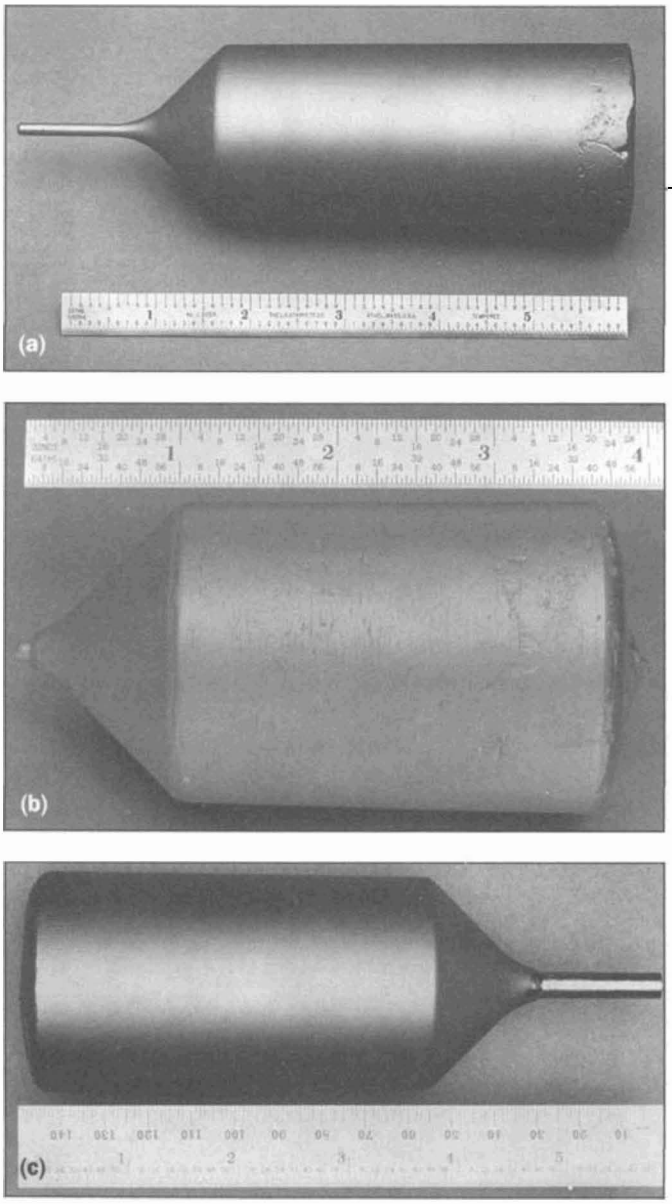


Figure 5. Typical crystals grown by the Gault process: (a) 1300g, <111> gallium phosphide (GaP) single crystal; (b) 750g, <111> indium phosphide (InP) single crystal; (c) 1200g, <100> gallium arsenide (GaAs) single crystal.

five separate GaP crystals. The average values of the species above the detection limit were:

Aluminum	0.4 p/ma
Boron	6 p/ma
Calcium	1.3 p/ma
Magnesium	0.3 p/ma
Silicon	1.8 p/ma

We presume that the elevated boron levels result from growth in PBN crucibles.

A spark source, mass spectrometry study of individual GaP wafers shows that the impurities, including the sulfur or tellurium dopant, were homogeneously distributed from edge to center. This would imply (and will be shown for InP) that the liquid-solid interface is flat.

Figure 5a shows a 1300g, <111> seeded, 50-mm diameter, GaP single crystal that was grown using the Gault process. We have grown both sulfur- and tellurium-doped, n-type crystals.

Because the composition of the melt is actively controlled, the entire melt has been grown into a single crystal. There is no residual gallium, which typically remains with LEC growth. The crystal surface is smooth, and no cracking or spalling of the crystal is visible, which suggests a low strain level.

Figure 5b shows a typical 750g, <111> seeded, 50-mm diameter, sulfur-doped InP single crystal. Again, the ability to control phosphorus pressure during the run eliminates the indium-rich melt that normally remains in LEC growth. Small ridges and steps on the crystal surface replicate the partial delaminations in the inner surface of the PBN growth vessel. Because residual strain is low, these crystals can be sliced with considerably less breakage than LEC crystals.

Similar results were achieved in GaAs growth, and Figure 5c shows a typical 1200g, <100> seeded, 50-mm diameter, silicon-doped crystal.

Figure 6 shows a calculated, normal freezing curve for sulfur-doped GaP that is typical of LEC growth.

Figure 7. Dislocation related, etch pits at three locations on a gallium phosphide (GaP) wafer: (a) edge, (b) 3 mm from the edge, (c) 25-mm diameter core.

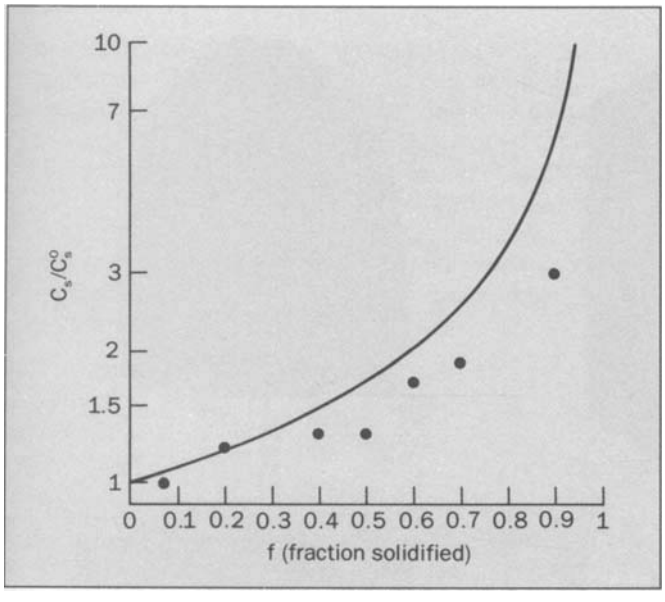


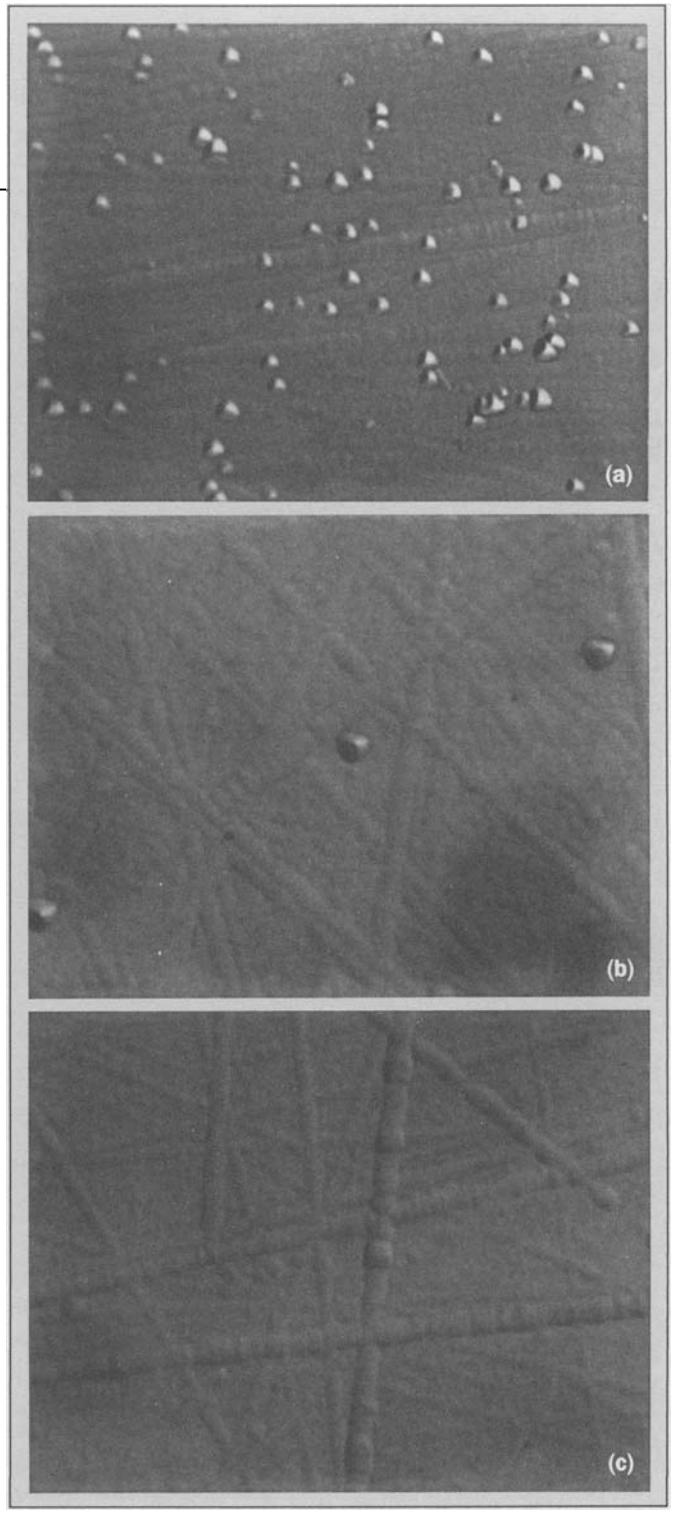
Figure 6. Normal freezing curve for sulfur-doped gallium phosphide (GaP). Also plotted are experimental values for the Gault process.

Also plotted are experimental values for the sulfur concentration in crystals that were grown by the Gault process. These crystals show a more-uniform doping profile.

The experimental values are below the values of the normal freezing curve as a result of the depressed concentration of sulfur in the melt. Because the melt is not encapsulated, the volatile sulfur species can escape from it during the run. The increased uniformity can improve manufacturing yields.

One significant consequence of growth under the low thermal gradients that are typical of the Gault process is the reduction of stress-induced dislocations. This can be seen in photomicrographs (Figure 7) taken in three regions of a 50-mm diameter, <111> GaP wafer.

We polished and etched the wafer. Selectively etched pits reveal edge dislocations and dislocation loops.



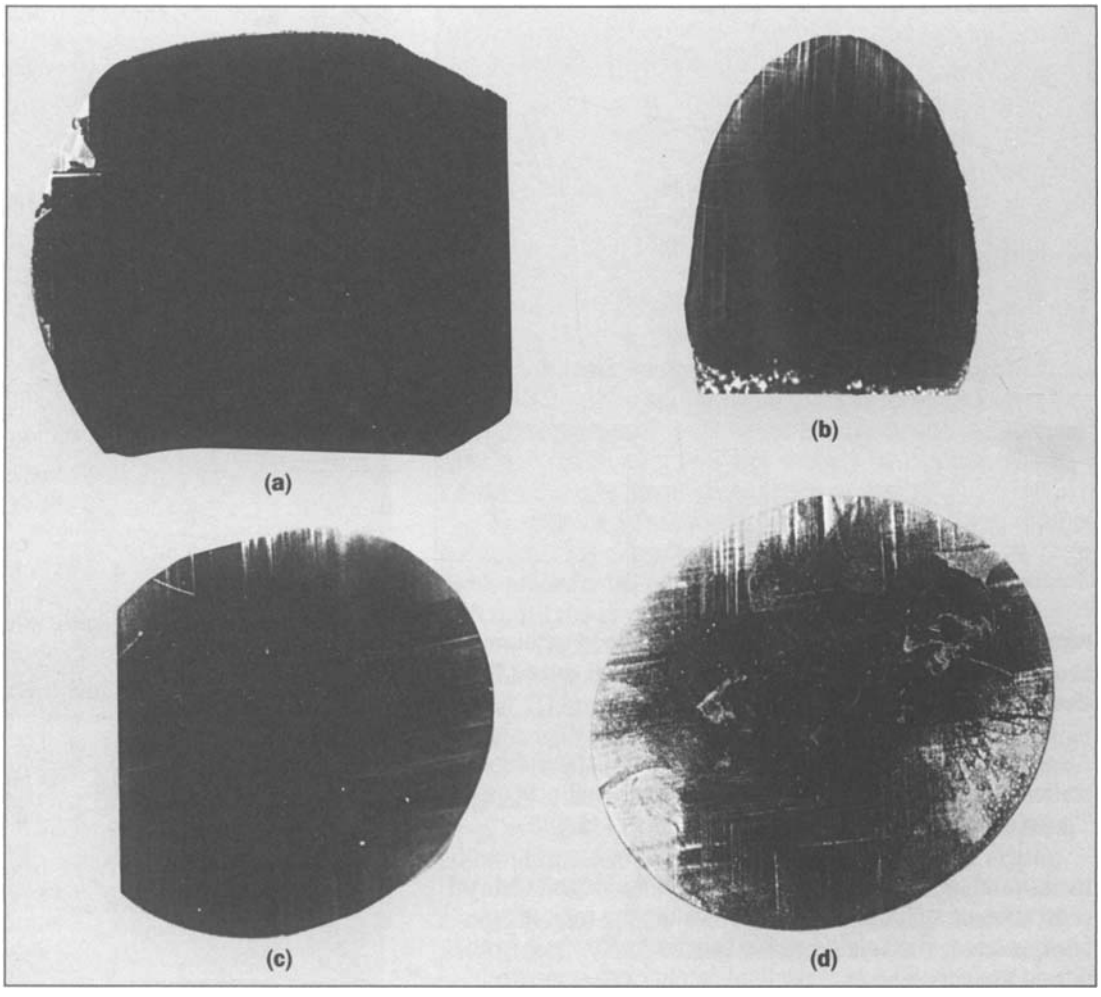


Figure 8. Comparison of indium phosphide (InP) wafers grown with the Gault (a) and LEC (b, c, and d) processes.

At the edge of the wafer (Figure 7a), the dislocation density is about $5 \times 10^3/cm^2$. The density 3 mm from the wafer edge (Figure 7b) is less than $10^2/cm^2$. The area shown in Figure 7c is dislocation free and represents a 25-mm diameter core.

In general, dislocation density in these 50-mm, <111> GaP crystals ranged from 800 to $2000/cm^2$. This is comparable to the best published LEC results[5] and 10 to 100 times better than the values normally reported for large diameter, LEC wafers.

To evaluate the effect of these low-dislocation crystals on LED efficiencies, split lot experiments were conducted using Te:GaP substrates grown by the Gault method and substrates from two commercial LEC sources.

Both sets of substrates were used to grow green-emitting LED chips with liquid phase epitaxy under identical conditions. The resultant chips were etched and individually examined for dislocations. Dislocations have been shown to act as nonradiative recombination centers for electron-hole pairs, which lowers device efficiency.

For the Gault process chips, more than 50 percent had zero dislocations and 70 percent measured less than 700 dislocations/cm^2. All LEC chips had dislocation densities of 3400/cm^2 or more. Following are the average dislocation values for the three sources:

Gault	945/cm^2
LEC-1	69,000/cm^2
LEC-2	24,000/cm^2

Electroluminescent efficiencies were measured for the green LEDs (not encapsulated), when driven at 10 mA. The VGF-based devices were at least 23 percent more efficient than the best LEC-based devices.

InP crystals grown by the Gault process exhibit low dislocation densities, low carrier concentrations, and low absorption coefficients. For 750g, 50-mm diameter InP crystals, there is no significant variation in the dislocation density from seed to tail. Also, the dislocation density does not depend on the sulfur-doping level over the concentration range, 2×10^{17}/cm^3 to 5×10^{18}/cm^3. In this doping range, the dislocation densities for these crystals average 0 to 500/cm^2, as compared to 10^3/cm^2 to 10^5/cm^2 for LEC crystals.

Figure 8 illustrates this difference. Figure 8a is a typical <100> wafer of InP that was grown by the Gault process. The wafer was sliced from a <111> crystal, and then polished and etched to reveal dislocations. Notice the uniformly low level of etch pits and the absence of any slip lines.

Compare this photomicrograph with Figures 8b, c, and d for three <100> wafers of commercially available LEC material. These wafers were cited as having 10^3 to mid 10^4 dislocation density at a sulfur doping level of mid 10^{18}/cm^3. Notice the high levels of slip at the perimeter of the wafers and the nonuniform defect distribution. The seed-to-tail uniformity and the low, InP dislocation levels have a significant economic effect on the manufacture of lasers, LEDs, and detectors for lightwave systems.

As with InP, the thermal conditions of the Gault process permit us to grow high-quality GaAs crystals over a wide range of silicon doping levels. The n-type GaAs crystals have averaged 300 dislocations/cm^2, compared to 30,000/cm^2 for LEC and 3000/cm^2 for HB crystals. These values were achieved over a silicon doping range of 0.2 to 3×10^{18}/cm^3.

In addition, we have grown intentionally undoped GaAs. Our characterization of this material shows it is semi-insulating, with a resistivity greater than 10^7 ohm-cm. The crystals have been annealed at 800°C for 30 minutes; they do not convert to p-type and do not lose their high resistivity.

Summary

The Gault process was designed to address the manufacturing needs for high quality, III-V crystal growth. The process is in use at AT&T's Reading Works in Pennsylvania, where it is used in manufacturing higher quality crystals than possible with other processes. Yields compare favorably with the LEC and HB processes.

Acknowledgments

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Biographies (continued)

Urbana. Mr. Gault is responsible for the manufacture of III-V compound semiconductor materials for lasers, LEDs, and detectors for lightwave systems; for visible LED indicators and illuminators; and for optoisolators. He joined AT&T in 1969, and has a B.S. in chemistry from Juniata College and a Ph.D. in physical chemistry from Iowa State University. Mr. Monberg's group prepares, grows, and characterizes interesting new electronic materials for device applications. He joined AT&T in 1977, and has a B.S. in chemistry from the University of Michigan and an M.S. from the University of California at Los Angeles and a Ph.D. from the University of Michigan, both in physical chemistry.

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