GROWTH OF THICK Ga$_x$Al$_{1-x}$As LAYERS
BY LIQUID-PHASE EPITAXY

by
L. E. Stone, K. Madden, R. W. Haisty

Abstract
A technique is described that is capable of growth of Ga$_x$Al$_{1-x}$As layers of almost uniform composition by liquid-phase epitaxy. Layers of 26 mils thickness have been grown reproducibly when furnace, crucible and geometries were optimized. Composition can be controlled by varying the saturation temperature. Profiles of typical layers, as defined by cathodoluminescence, are compared to usual graded structures.

Texas Instruments, Incorporated
Dallas, Texas
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Background

The growth of Ga$_x$Al$_{1-x}$As layers by use of conventional cooling methods of liquid-phase epitaxy produces a changing AlAs composition due to the depletion of aluminum from the solution. Figure 1 is the phase diagram which indicates that a small Al content in the liquid will produce a large AlAs content in the solid. Figure 2 indicates the temperature dependence (1) in the region of interest and indicates 1% Al in the liquid will produce 30 to 40% AlAs solid at or near 900°C.

Panish and Sumski (2) described the optimum conditions for growth of uniform layers as “use of the largest volume of gallium possible to grow a layer over the shortest temperature span, starting with a liquid which contains no solid.” Similar considerations had established our guidelines. We chose volumes of 100 to 500 grams of gallium, a maximum temperature span of 35 degrees, the region of 960°C (because of solubility consideration) and accepted the premise that slow growth rates would apply.

Experimental

A vertical reactor system is used, similar to early apparatus (3,4), but differing in several significant details. Figure 3 illustrates the reactor system. The glassware is quartz, and the solution is contained in a 40-MM ID high-purity alumina crucible. Solutions normally contain 150 grams of gallium, fill the crucible to 1.5 inches and are used up to 20 times before discarding. Volumes of 500 grams have been used, but show little improvement.

Crucible and substrate holder are supported by high-purity, closed-end alumina tubes, which also house thermocouples.

The substrate holder (Figure 4) is of Poco high-purity graphite. The substrate is supported between threaded graphite rings, on a graphite disc, with the polished and chemically cleaned surface facing up.

Temperature monitoring and control is by Platinel thermocouples housed in the support tubes. Closed-loop temperature control is by MPRY “Thermac” controllers. Programming is automated by a modified high-resolution “Data Trak” programmer.

The furnace is unusual in that it deliberately defines a sharply peaked hot zone near its center, as opposed to the usual “flat as possible” profile. The temperature profile and temperature gradients measured in the gallium solution at various levels within the furnace are shown in Figure 5. Note that gradients of $\Delta T = +25^\circ$C, and $\Delta T = -25^\circ$C (top of solution cooler than bottom) across the 1.5 inch depth of solution are possible. In our experience, a gradient of $\Delta T = 10^\circ$C is near optimum for our crucible geometry, etc.

Gradient Growth Technique

The principle underlying our technique is the transport of ternary material across a thermal gradient. A source of ternary solid is provided at a level in the solution which is approximately $+10^\circ$C hotter than the substrate and within a nominal distance of the substrate. Due to the difference in solubility across the temperature gradient, migration and transport of material occurs and will sustain growth, within certain limits. Growth rate is slow due to the long mean free path of the growth species in the dense liquid. Growth rate and ultimate layer thickness is limited also by growth of random platelets at or near the substrate, which then compete for the ever decreasing available source material. The area factor of these platelets is considered the principal limiting factor.