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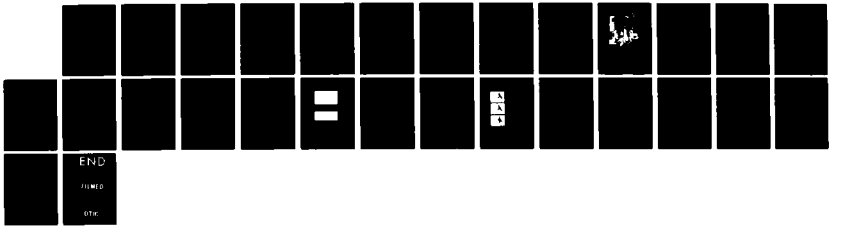

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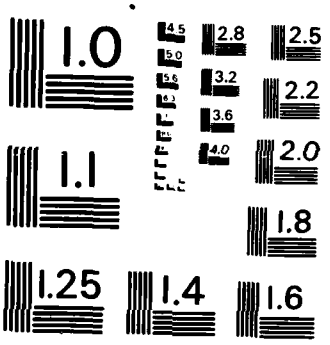

REPORT SD-TR-85-69

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Molecular Beam Epitaxial (MBE) Growth of Gallium Arsenide and Gallium Aluminum Arsenide

H. KANTER
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30 September 1985

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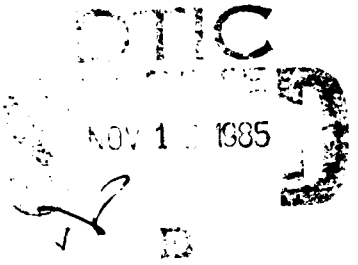

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This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The construction and operation of a molecular beam epitaxial (MBE) system for promoting the epitaxial growth of gallium arsenide (GaAs) and gallium aluminum arsenide (GaAlAs) materials is described. The Aerospace Corporation's Electronics Research Laboratories is studying these materials in support of development of GaAs-based microwave and millimeter-wave devices as well as fast integrated digital circuits.

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PREFACE

The author acknowledges the excellent support provided by Ray Smith and his associates of the Laboratories' machine shop; this was instrumental to the success of this project. Likewise, the assistance of Jerry Wendt in assembly, sample preparation, and evaluation is appreciated.

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FIGURES

TABLE

I. INTRODUCTION

The molecular beam epitaxial (MBE) technique of controlling the growth of thin semiconductor layers was spurred by the evolution of sophisticated device structures that relied on spatial variation of electronic and optic parameters such as bandgap, doping profile, refractive index, and so on. In contrast to vapor-phase or liquid-phase epitaxy, the MBE technique, because of its lower growth rate and precise shutter control, permits abrupt variations on an atomic scale (in the growth direction) and thus the construction of "super lattice" structures that are explored at an increasing pace for their device potential in micro- and millimeter-wave applications, as well as in optoelectronic circuits. The spectrum of devices encompasses high-electron-mobility field-effect transistors (FETs), transistors operating in the ballistic transport regime, impact ionization avalanche transit time (IMPATT) devices of optimized efficiency, quantum well lasers operating with adjustable wavelength, and high-gain photoconductive devices, to name only a few. As a specific example, gallium arsenide (GaAs) FETs constructed from material grown by this technique have lower noise and higher speed in comparison with those made by more traditional methods. Because of the pivotal role material preparation has in modern semiconductor device development, the Aerospace Electronics Research Laboratory (ERL) has acquired this technique for GaAs-related device development efforts.

This report contains a short description of both the MBE system built in-house at ERL and that system's operating procedure. The system is designed for growing GaAs- and aluminum gallium arsenide (AlGaAs)-related semiconductor structures. Future reports will describe material structures and material quality obtained with this system.

II. DESCRIPTION OF THE MBE SYSTEM

The MBE system (Fig. 1) consists of two vacuum chambers constructed of stainless steel in a configuration as shown in Fig. 2a and 2b. Growth occurs in the main chamber, which is equipped with effusion ovens, a mass analyzer, ion gauges for flux and background pressure measurements, a high-energy (20 keV) electron diffraction camera, an ion-pump, a closed helium-cycle cryogenic pump, and extensive liquid-nitrogen-cooled cryo-paneling of about 10 ft^2 in effective area. The eight effusion cells are made of boron nitride (BN), are heated by tantalum (Ta) wires, and are individually shielded by a liquid-nitrogen shroud that also carries the shutter assembly. The total oven assembly (Perkin-Elmer M 400) is mounted on a 13.5-in.-diameter flange and for service can be handled by a single person.

The substrate crystal is mounted on a push rod assembly that allows it to rotate by $\pm \pi/2$ around the rod axis and thus around the sample normal. This is important for the selection of the diffraction pattern when the crystal is in the growth position. The push rod also permits the sample to move between the main chamber and the forechamber, the forechamber being used for sample preparation and sample exchange. With a gimbal, as shown in Fig. 2b, the sample holder can be positioned at a right angle to the rod axis and either in front of the cylindrical Auger analyzer (which includes an e-gun), in front of the load lock for sample exchange, or in a third, unused position, which can be used for a sputter gun. The forechamber is valved off from the main chamber except during crystal growth. (This valve, as well as the isolation valves for the ion pumps and the load lock, uses Viton A O-rings in the valve lid only. All other seals are metal.)

During sample exchange the substrate support, essentially a molybdenum (Mo) disk 1.25 in. in diameter and 0.5 in. thick, is removed from the holder by means of a manipulator and retracted into a third chamber that can be opened to air. The main chamber and the forechamber are never exposed to air during normal operation and have a background pressure in the low, 10E-10 Torr

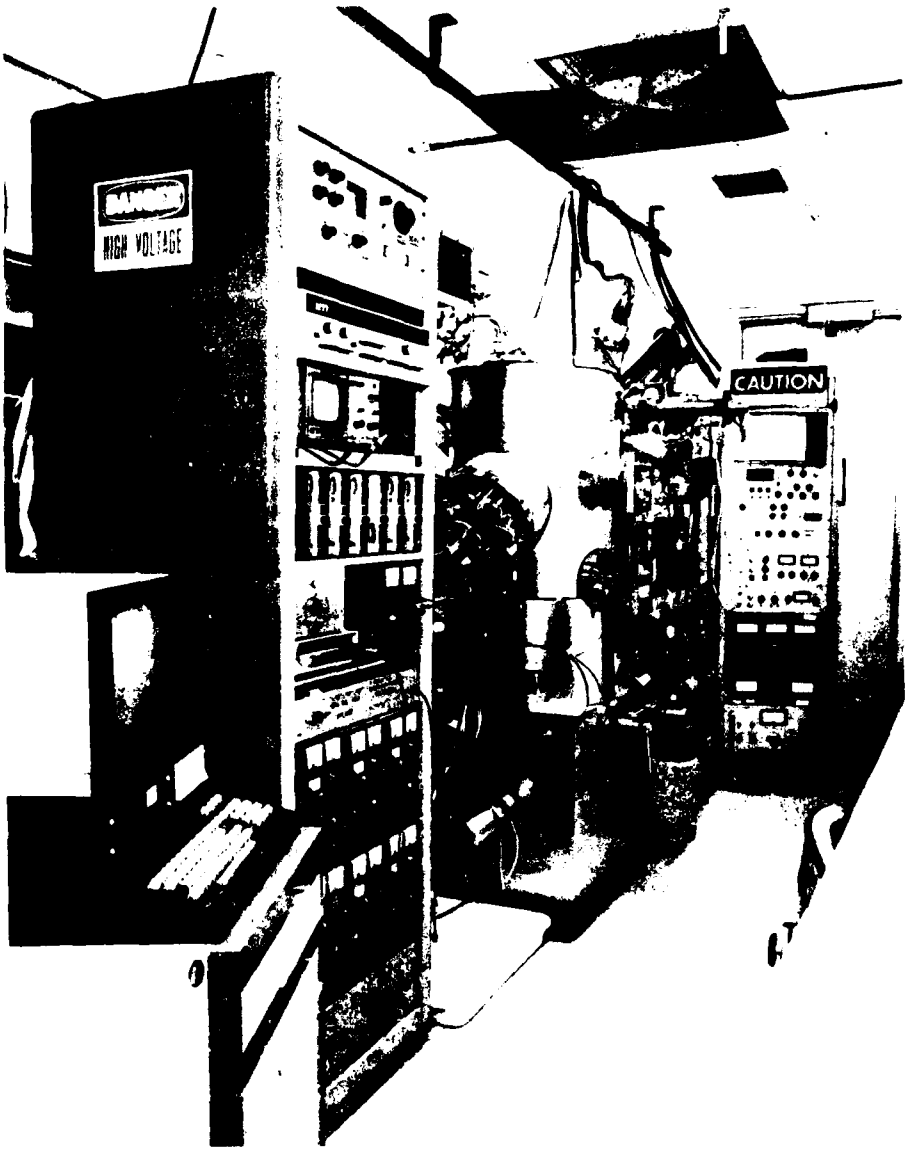


Fig. 1. Photograph of the MBE System. At center is the Perkin-Elmer MBE-400 source flange. The rack at the left contains the HEED supply, mass analyzers, oven-temperature controllers, and power supplies. The oven-temperature and shutter schedule are controlled by the processor in the foreground. The rack at the far end of the room contains the electronics for the Auger analyzer, which is visible at the upper right of the vacuum chamber. [illegible] to the left of the caution sign is the chamber [illegible]

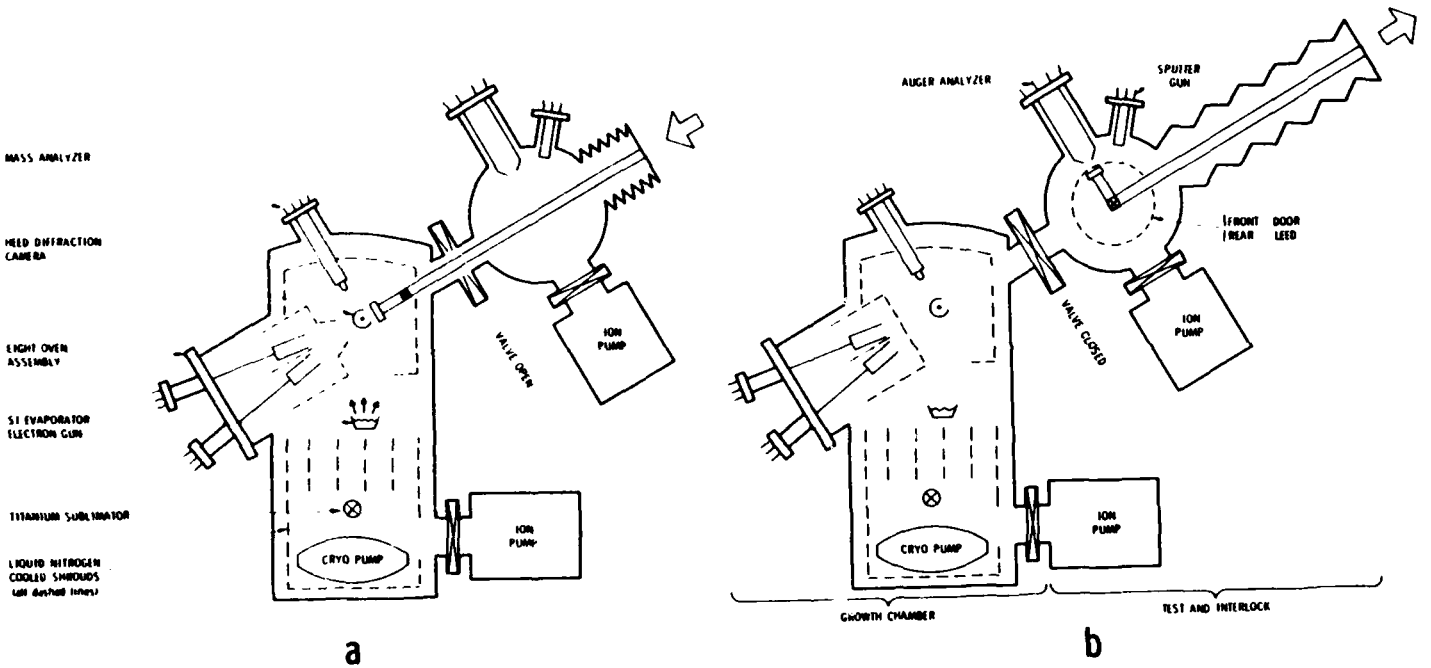


Fig. 2. Schematic of the MBE System. (a) In growth configuration; (b) in analysis configuration. All dashed lines in the main chamber are liquid-nitrogen-cooled surfaces.

region. During sample exchange the forechamber pressure may rise to nearly 10E-8 Torr; it quickly returns to equilibrium after the manipulator has been removed.

III. PREPARATION OF THE WAFERS

A semi-insulating or n-type doped GaAs wafer is used as a substrate for Hall or capacitance versus voltage (C-V) measurements, respectively. The maximum wafer size is 2×2 cm^2. The wafer is degreased in tetrachloroethylene, acetone, and isopropyl alcohol, then etched in a $NH_3(OH):H_2O_2:H_2O$ (1:1:5) solution for 15 min, if a substantial layer is to be removed because of surface damage, or in a concentration of 3:1:25 of the same constituents for 30 sec, if only a light etch is required. The wafer is then rinsed in deionized water for 2 min without being exposed to air. The wafer is left in the water until, usually within 30 min, it is mounted on the Mo disk in a laminar-flow hood. In the mounting procedure the wafer, after being dried in a stream of nitrogen (N_2) gas, is placed on a hot Mo block that is covered with liquid indium (In), with which the back of the wafer is wetted as the wafer is carefully moved back and forth a few times by means of a pair of tweezers. The wafer is then picked up from the block, its back is checked for complete wetting with In, and it is placed on the Mo disk whose top surface is also thinly covered with In. The disk is then removed from the heater stage and cooled. A Ta wire is positioned above the wafer surface to serve as a mask for layer-thickness determination.

The wafer is now ready to be inserted into the chamber. Once in the forechamber it is heated to 300°C so outgassing can commence. A short heating cycle to 500°C removes most of the surface-adsorbed gas without appreciable As loss. The Auger spectrometer checks the surface for carbon contamination. Typical scans are shown in Fig. 3. No carbon peak should be visible in a clean wafer. Preparation procedures and chemicals, including the deionized water, are thus monitored for proper cleanliness.

To remove the surface oxide, the wafer is heated in the forechamber to nearly 600°C while the oxygen (O_2) line of the Auger spectrum is observed. There is no indication of O_2 after less than a minute. However, loss of As from the surface is unavoidable under this condition. Therefore, at present the O_2 is removed in the growth position under an As flux of about 10E+14

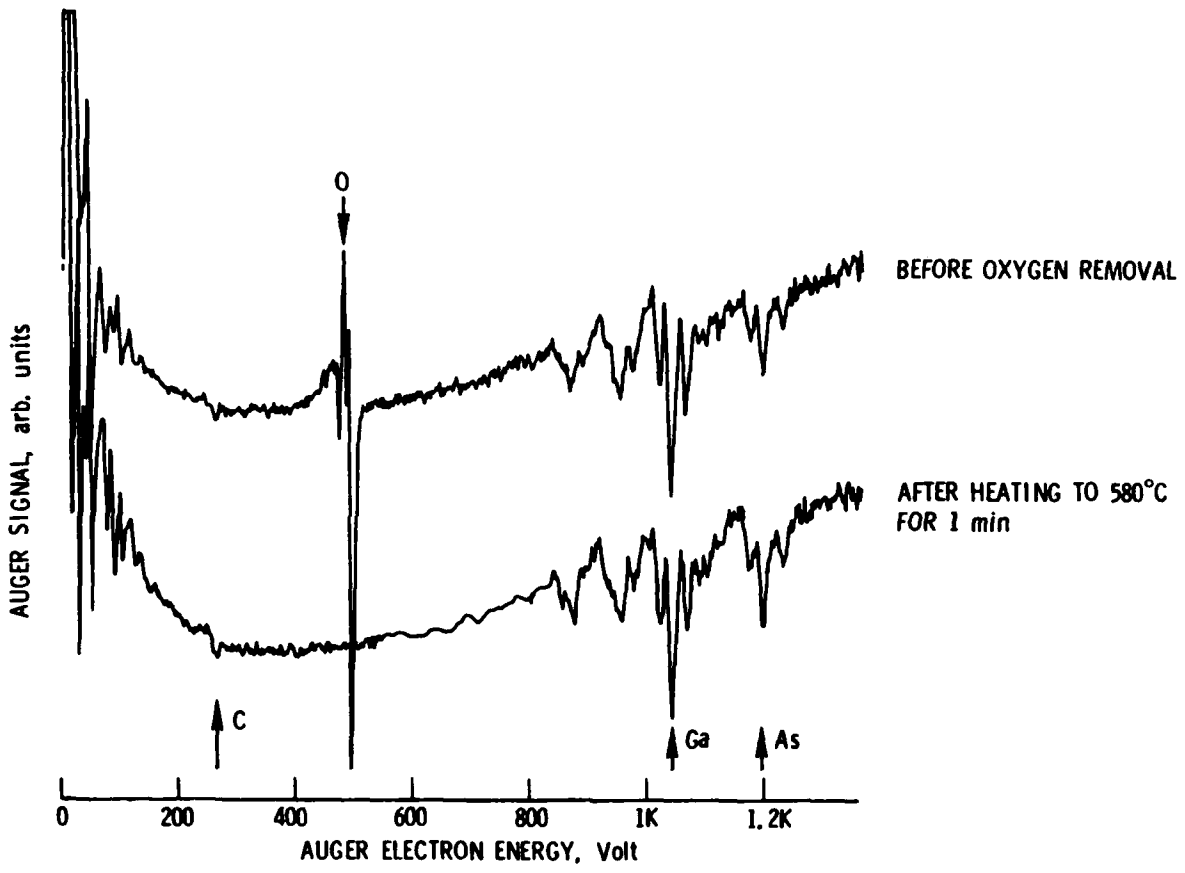


Fig. 3. Auger Spectrum of the Substrate Surface before and after Removal of Oxide Layers in Preparation for Epitaxy.

atm/cm^2 sec. The O_2 removal is monitored on the mass analyzer. This latter procedure has the disadvantage, however, that some O_2, water (H_2O), and carbon monoxide (CO) are introduced into the growth chamber because of surface oxide decay and residual outgassing of the sample. A preferable procedure would involve outgassing and O_2 removal under As pressure in the forechamber.

IV. PREPARATION OF THE SOURCES

All growth materials - gallium (Ga), aluminum (Al), As, silicon (Si), and beryllium (Be) - are evaporated from BN crucibles 2 or 20 cm^3 in size that require periodic recharging. Evaporants, their purity and suppliers are listed in Table 1. Particular care must be taken with Ga, the oxidation of which can seriously affect the quality of the grown GaAs layers. Each charge uses a newly cleaned and vacuum-outgassed crucible into which Ga is placed in solid form in order to reduce its exposure to air; the Ga is supplied packaged in Ar gas as a slightly chilled solid rod, since its melting point is near 30°C. A 0.1% mol fraction of Al is added to the Ga charge and serves as an O_2 getter.

After pumpdown the system is baked to near 180°C until all traces of arsenic oxide have been removed and the partial water pressure falls below 10E-7 Torr. Extensive outgassing of crucibles follows at temperatures somewhat in excess of normal operating temperatures. Once the background pressure of CO has fallen below about 5.10E-11 Torr partial pressure, the system is ready for growth. Care is taken that all ovens operate for some time with shutters closed, to allow for the outgassing that results from a temperature rise when the shutters are closed.

When the ovens idle overnight, their temperatures are held at room temperature for As and at 250°C for all other materials except Ga, whose temperature is held above the solidification point near room temperature in order to minimize corrosion of the metal parts of the oven.

Special attention must be given to the Al oven since it completely wets the walls of the crucible and would cause it to crack when the Al solidifies and cools further. (The temperature coefficients of expansion between the Al and the BN crucible in the radial direction differ by about a factor of two.) In order to preserve crucible integrity in the temperature range of interest, the charge is kept to 1/10 of total crucible volume, the rate of temperature variation is kept below 50°C/min when temperature is below the melting point of Al, and idling temperature is not below 300°C. In each recharging cycle the crucible is checked for cracks.

Table 1: Evaporants, Their Purity and Suppliers

Gallium:	(7N ingot)	Atomergic Chemetals Corp. 100 Fairchild Ave. Plainview, NY 11803
Arsenic:	(6N chunks)	Alfa Products Thiokol/Ventron Div. Danvers, MA 01923
Aluminum:	(6N pellets)	
Indium:	(6N shot)	Gallard Schlesinger Chem. Mfg. Co. 580 Mineola Ave. Carle Place, NY 11514
Beryllium:	(4N flakes)	Varlacoid Chem. Co. 666 S. Front St. Elizabeth, NJ 07202
Silicon:	(Doubly zone-refined pure Si wafers)	

Another peculiarity of the Al charge must be mentioned. At normal operating temperatures near 1200°C, the Al reacts with the BN crucible, giving rise to some N_2 evolution, as evidenced by a mass-28 background of nearly 3.10E-10 Torr (Fig. 4, upper frame).

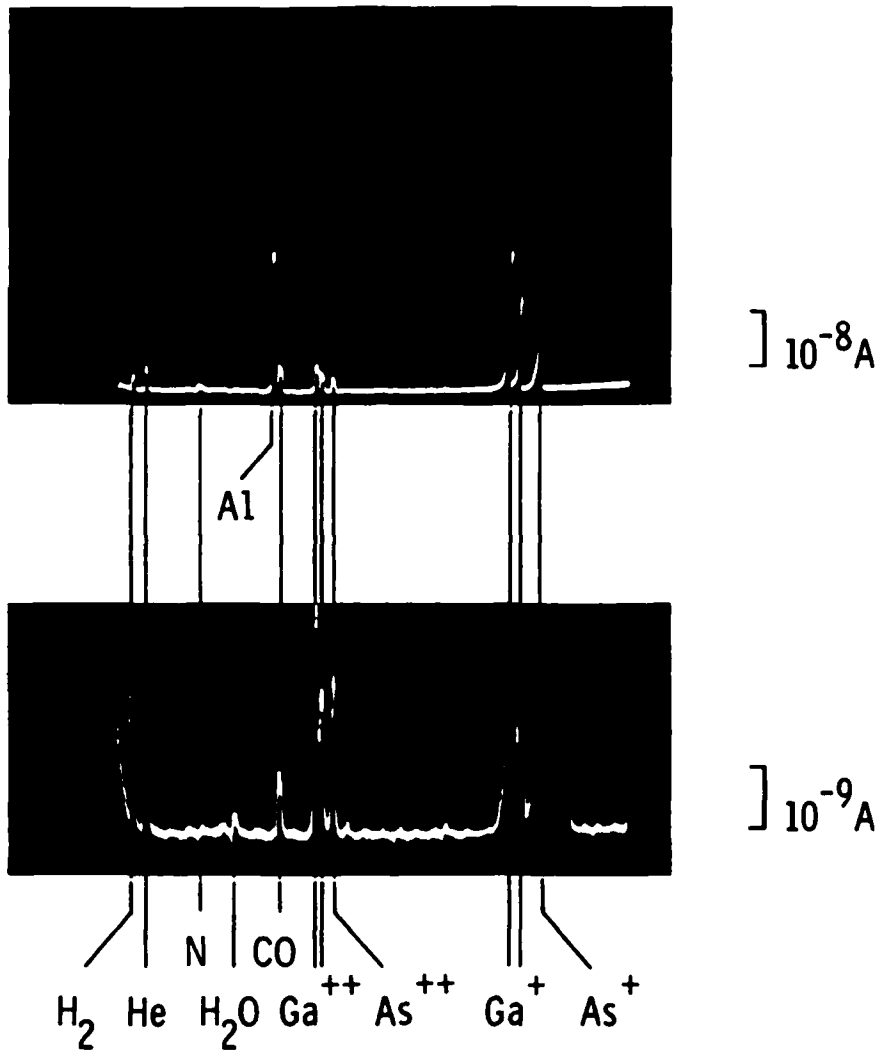


Fig. 4. Mass Analyzer Spectrum. Upper frame: during growth of AlGaAs; the As peak at the right side of the spectrum reaches almost to full scale. Lower frame: during growth of GaAs, at 10 times the sensitivity of the upper frame.

V. EXPITAXIAL FILM GROWTH

Before the wafer is introduced into the growth chamber, the fluxes of the Ga and Al sources are set with the help of an ion gauge positioned at the wafer growth location. A 1-μm/h GaAs growth rate corresponds to a pressure reading of 2.5.10E-7 Torr (Fig. 5). An additional flux of 10E-7 Torr of Al will produce GaAlAs with a ratio of 0.3 Al to 0.7 Ga. The oven dial setting is noted and temperatures are temporarily reduced by about 200°C while the As source is heated and O_2 on the wafer is removed in order to preserve material. When it is time, the Al and Ga ovens are brought back to operating temperature 5 min before the shutter is opened; this is an interval that appears sufficient to stabilize the ovens at the desired flux values.

After flux calibration the As pressure is brought up, and the wafer is moved into the growth position and heated to 600°C so that O_2 is removed. The HEED pattern can now be observed. When the background pressure has dropped to an acceptable level, the Ga shutter is opened and growth begins. Within the first minute, 200 Å of deposition has occurred and the diffraction image has usually improved to its final form. Typical diffraction patterns, shown in Fig. 6, indicate that the surface geometry is reconstructed under As-rich and Ga-rich flux conditions.

The fine tuning of growth conditions depends on sample temperature and Ga/Al-to-As flux ratios and is adjusted by the mass analyzer peak ratios. Typical mass spectra under growth conditions are shown in Fig. 4. While GaAs epitaxy of good stoichiometry is generally accomplished under excess As flux, GaAlAs requires the proper As flux if good-quality films are to be obtained. In practice this means that reconstruction is observed with the diffracted beam when Ga/Al flux is stopped during growth; this contrasts with the situation for GaAs under As-rich conditions, where no change is observed when the Ga flux is stopped. A rough estimate for proper As flux can be made by stopping that flux for 1 sec (during which about 1 atomic layer would be deposited) and observing the return of the diffraction image to normal within a time period of not more than several seconds. Typical time delays observed for the return of the normal diffraction pattern for various Ga/As flux ratios and two sample temperatures are shown in Fig. 7.

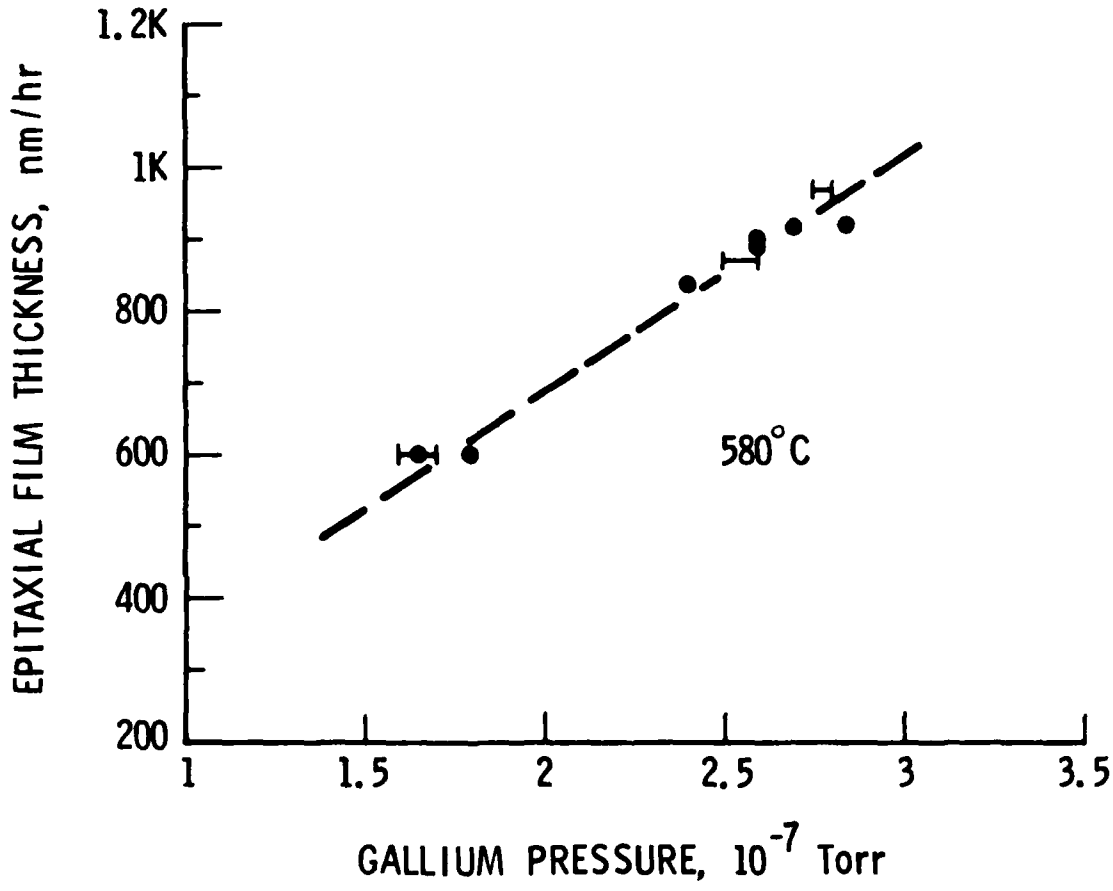
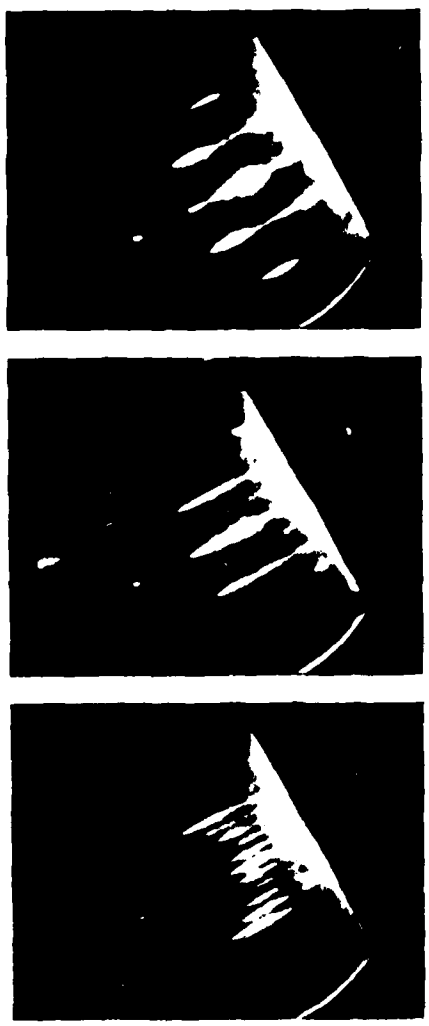


Fig. 5. Flux Calibration: Epitaxial Growth Rate vs. Ga Pressure at the Flux Gauge.



- BEFORE GROWTH
- DURING GROWTH: As-RICH FLUX RATIO
- DURING GROWTH: Ga-RICH FLUX RATIO

Fig. 6. High-Energy Electron Diffraction (HEED) Images for Various Growth Conditions.

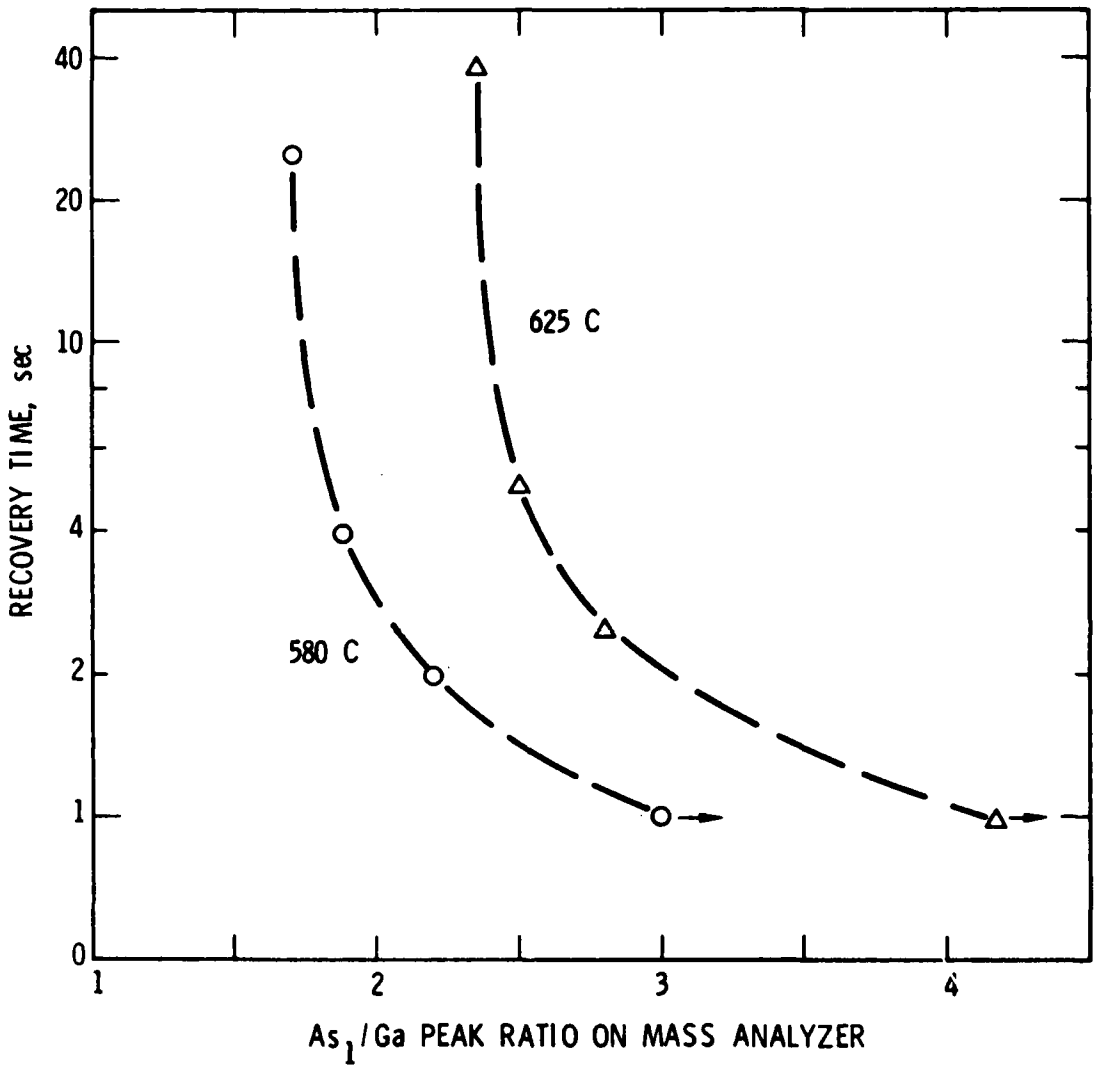


Fig. 7. The Time-to-Recovery of the HEED image, Indicating the Steady-State Growth vs. Flux Ratio after the As Flux Has Been Interrupted for 1 sec (about one atomic layer) for 2 Substrate Temperatures.

The doping of the epitaxial layer is accomplished by the slow evaporation of Si (n-type) and Be (p-type), respectively, with doping density controlled by oven temperature. Typical calibration runs are displayed in Fig. 8a and 8b. Clearly, any doping profile can be obtained by oven temperature variation and shuttering. Since growth is of the order of one atomic layer per second, very abrupt profiles can be obtained with this technique.

After a growth cycle is completed, the wafer is cooled down, retracted to the forechamber, and, when it is near room temperature, removed from the system. Usually a new sample is introduced at this time. Since outgassing of the new sample requires about 1 h, only one sample is processed per working day. With a 2-h extension per working day, two growth cycles per day could be accommodated.

Of interest is layer uniformity. A typical thickness profile is sketched in Fig. 9. Variations within ± 5% are observed. Better uniformity could be obtained if provisions were made to rotate the sample continuously during the growth process.

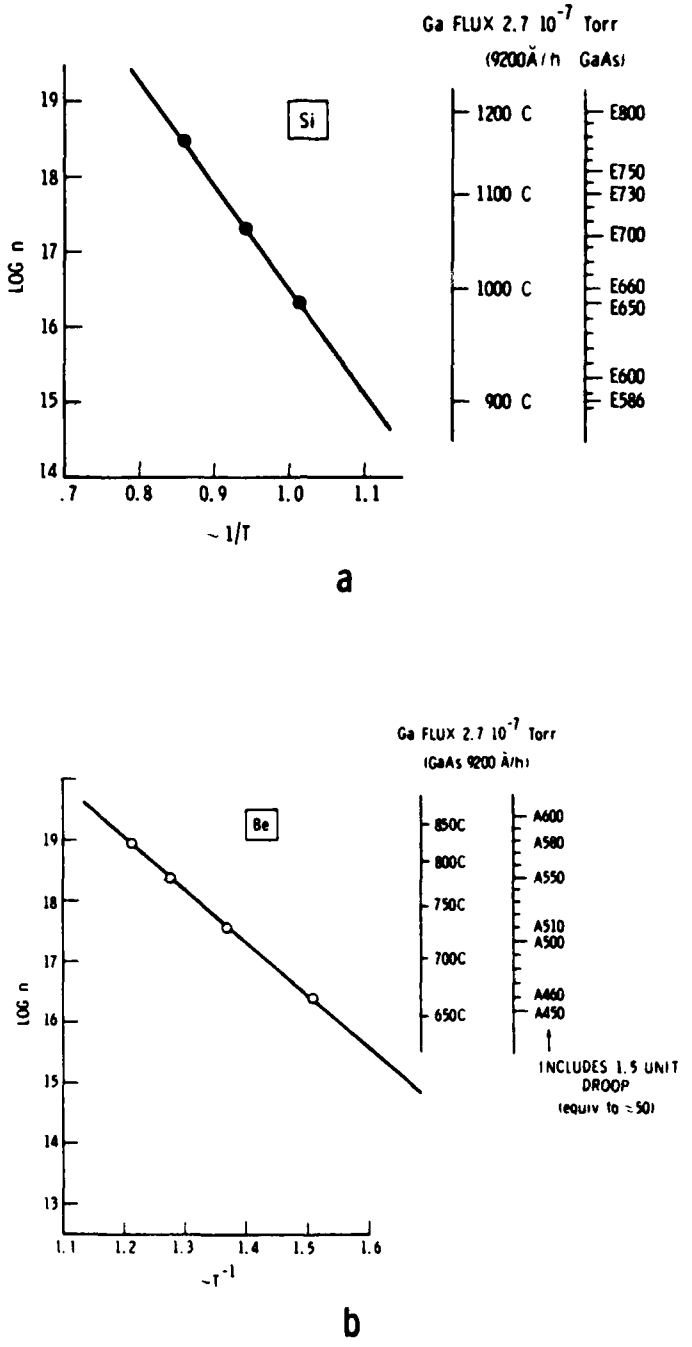


Fig. 8. Calibration of Doping Level vs. Oven Temperature. The scales on the right give the oven temperature and dial setting on the temperature controller: (a) n-dopant Si; (b) p-dopant Be.

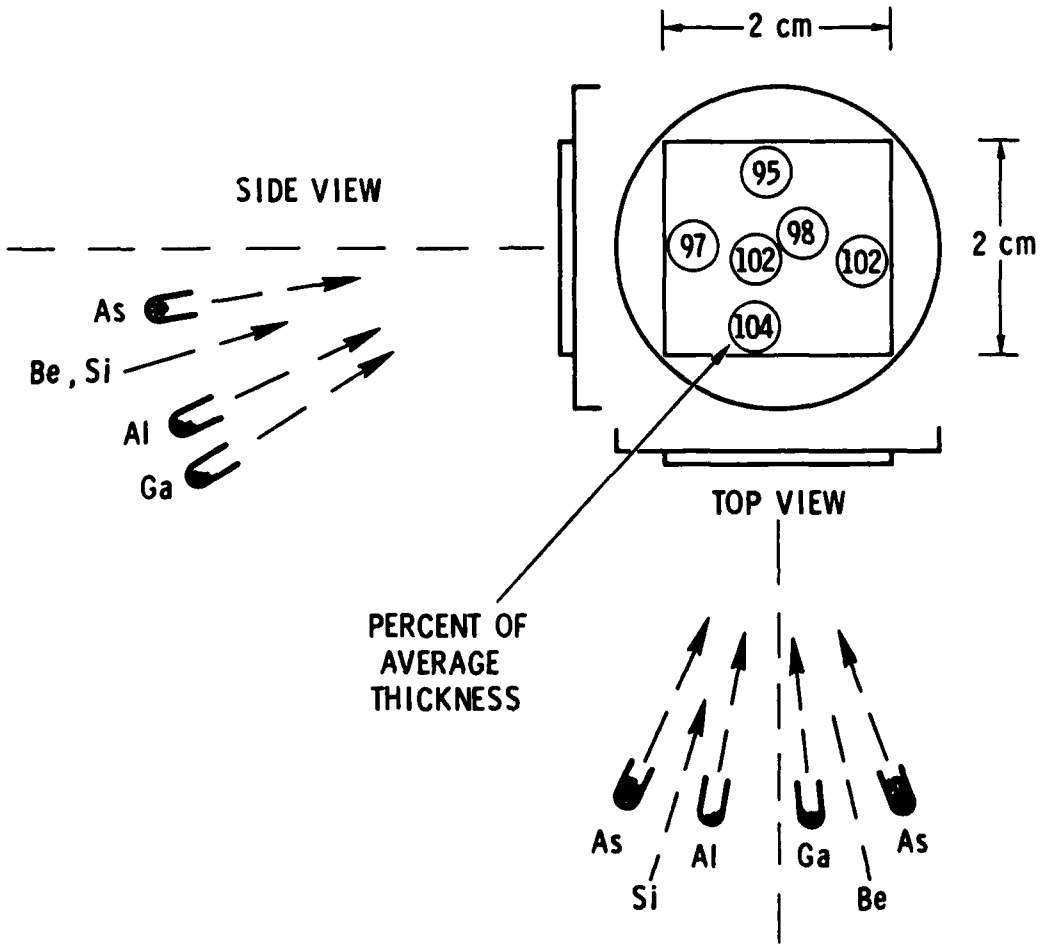


Fig. 9. Thickness Variation and Flux Geometry across the Substrate.

VI. CONCLUSIONS

With this MBE system various material structures of GaAs and GaAlAs suitable for device applications have been made. Thus, FET devices of good dc characteristics have been fabricated whose active layers were grown by this technique.

However, mobility values demonstrating state-of-the-art material quality have not yet been obtained. It is anticipated that quality will improve with growth experience. The quality of optimized material, as well as the performance of devices employing specific MBE-grown structures, will be reported in future publications.

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