Chapter 3

MOLECULAR BEAM EPITAXY AND DEVICE FABRICATION

In the previous chapters we discussed the basic device operation and the small signal and large signal response of a QWITT diode. In this chapter a brief summary of MBE growth of the heterolayers used in this study will be discussed.

Molecular Beam Epitaxy (MBE) is a versatile crystal growth technique that allows precise control of beam fluxes and deposition conditions, and thin films of many material systems have been grown using this technique [67]. All the heterolayers used in this study were grown using a Varian GEN II MBE system. Typically, this system has three metal sources, namely gallium, aluminum and indium, two arsenic sources, and two dopant sources, beryllium and silicon. The source cells are calibrated to determine the growth rate of the epitaxial film as a function of effusion cell temperature. For III-V semiconductors the growth rate of an epitaxial film is determined by the arrival rate of the metal atoms, i.e., the growth rate of GaAs is set by the gallium cell temperature. Fig. 3.1 shows a representative plot of GaAs and AlAs growth rates as a function of cell temperature as measured in-situ by reflection high energy electron diffraction (RHEED). The dopant cells are calibrated by growing bulk layers at various dopant cell temperature settings and then characterizing the carrier concentration and mobility of these bulk samples using temperature dependent Hall measurements. Since stoichiometry control and growth temperature strongly affect the doping characteristics of the epitaxial film, doping cell calibrations are performed for a particular growth temperature and arsenic flux. Fig.

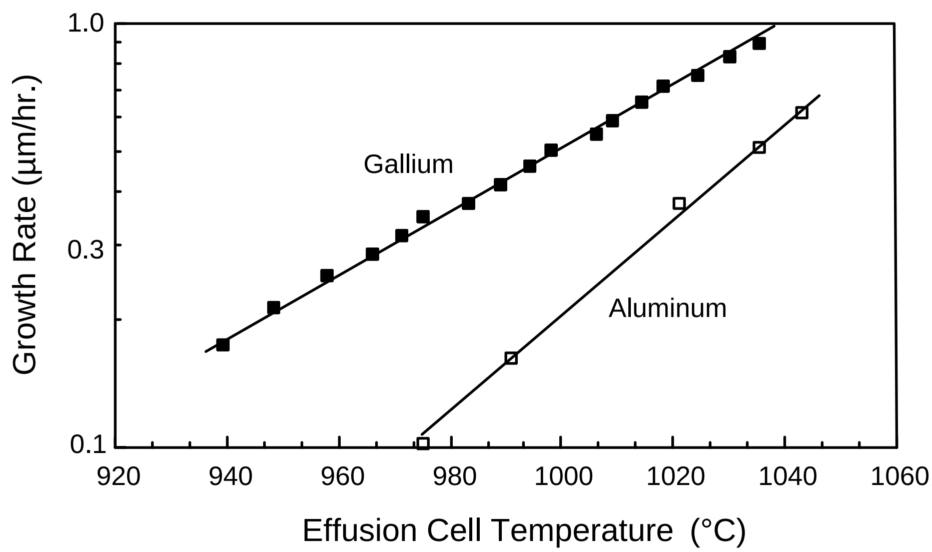

Fig. 3.1: RHEED measurements of the growth rate of GaAs and AlAs as a function of gallium and aluminum effusion cell temperatures.

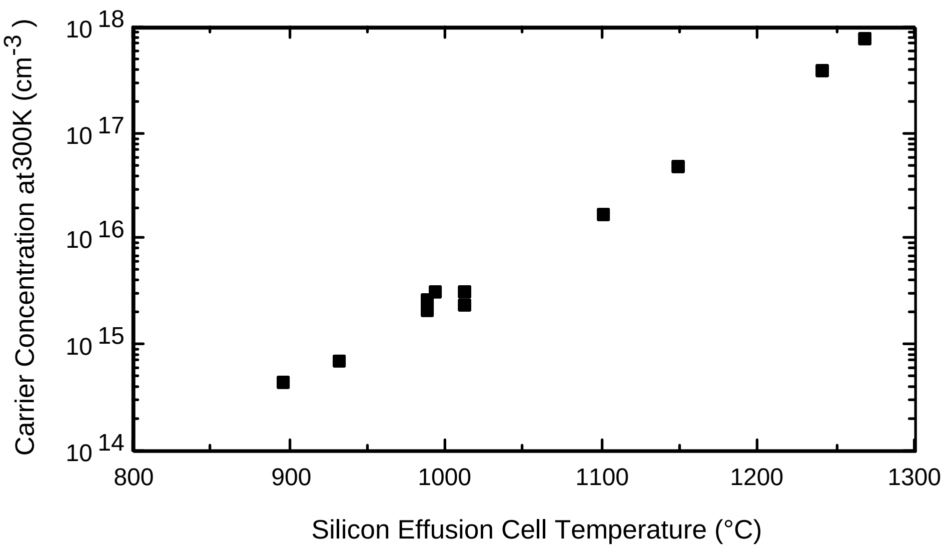

Fig. 3.2: Carrier concentration at 300K, as determined by Hall measurements, for MBE grown silicon doped GaAs layers versus silicon effusion cell temperature.

3.2 shows a representative plot of carrier concentration at 300K as a function of silicon cell temperature for n-type GaAs. The mole fractions of the column III elements in a ternary semiconductor can be controlled by varying the relative growth rate of the metal cells being used. For example, if the growth rates of GaAs and AlAs are known, the aluminum mole fraction in a layer of $Al_xGa_{1-x}As$ can be determined as follows:

$$x = \frac{(\text{Growth rate of } Al_xGa_{1-x}As) - (\text{Growth rate of } GaAs)}{\text{Growth rate of } Al_xGa_{1-x}As} \tag{3.1}$$

Much of the MBE growth performed to grow QWITT structures involved the use of a dimer arsenic cracking source developed in our laboratory [68]. It is well established that dimeric arsenic sources in molecular beam epitaxy (MBE) have numerous advantages over conventional tetrameric arsenic sources. We have developed a refractory, two-zone, large capacity, baffle-free arsenic cracking source for molecular beam epitaxy. The new features of this design include the use of a molybdenum tube to provide efficient cracking, a horizontal sublimator at a right-angle geometry to the cracking section, a baffle-free design, and the use of expanded tantalum heating filaments. Bulk GaAs and GaAs/AlGaAs heterostructures grown using this source exhibit good electrical and optical properties, with clear improvements in electrical behavior when compared to an As_4 source.

One of the most important requirements for successful MBE growth is the ability to produce a stable, reproducible, and uniform molecular beam of each elemental constituent of the thin film by the use of Knudsen or effusion cells [67]. A

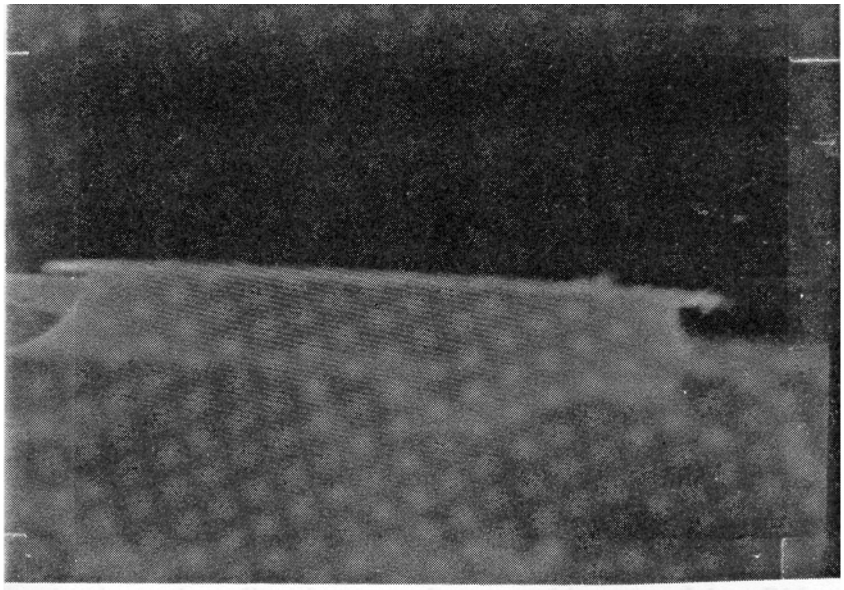

Fig. 3.3: A cross sectional SEM photograph of a quantum well mesa device typically used in this study.

Knudsen cell is ideally an isothermal enclosure with an infinitesimally small exit aperture bounded by vanishingly thin walls [67]. Knudsen effusion cells are critical components in MBE systems and are the basis of nearly all molecular beam generation. The heating element in a Knudsen cell is a wire or foil ribbon noninductively wound, either spirally around the crucible or from end to end, and supported on insulators or inside insulating tubing. In reality, MBE sources are far from ideal Knudsen cells. The exit aperture is not bounded by vanishingly thin walls, and large exit orifices are often employed to achieve enhanced growth rates for the lowest set temperature as well as to improve uniformity of bulk materials and dopants. The conventional cell has a significant radiated heat loss at the opening, resulting in a temperature drop at or near the orifice and causing large thermal gradients along the length of the crucible. We have developed a molecular beam source utilizing single filament construction to generate a reproducible, uniform, and pure beam of elemental molecules [69]. The refractory filament is designed to minimize the thermal gradient between the top and bottom of the pBN crucible that contains the source material (e.g., aluminum).

The sequence of steps typically employed to grow a GaAs/AlAs tunneling structure by conventional MBE in a Varian GEN II system is described below:

<u>Substrate Preparation:</u>

(a) The substrates commonly used are two inch, (100), LEC, n^+- GaAs, silicon doped at 3-5x10^{18} cm^{-3} (Airtron-Litton). These substrates are chemically etched in a 8:1:1 solution of H_2SO_4:H_2O_2:H_2O for 15 minutes and subsequently mounted on molybdenum blocks using indium to obtain a good thermal contact between the block and substrate.

(b) The blocks with GaAs substrates mounted on them are introduced into the load lock chamber and outgassed at 450°C for 15 minutes. The blocks are then transferred into the preparation chamber where they are outgassed individually at 450°C for an hour. The peak pressure in the preparation chamber while a particular block is being outgassed is noted. The outgassed block is transferred into the growth chamber for pre-growth procedures.

Pre-Growth Procedures:

(a) The block is heated to 600°C under an arsenic flux (typically at a BEP of 6-8x10^{-6} torr) to puff oxides off the GaAs surface. The RHEED pattern obtained from the substrate is monitored during the oxide puff off. Initially, the RHEED pattern is cloudy and featureless. As the block is being heated, at around 580°C a distinct clearing of the pattern, that is brought about by the oxide being blown off the surface, is observed. Streaks on the GaAs surface become visible and by rotating the block to align the substrate in the (110) direction a characteristic 2x4 RHEED pattern can be seen.

(b) The substrate temperature is now reduced to 580°C. To facilitate growth rate calibrations, the RHEED pattern is improved further by depositing a few monolayers of n^+-GaAs. An arsenic(As_2)-to-gallium flux ratio of 2:1 is normally used throughout the growth process. This corresponds to an "arsenic stable" [67], 2x4, growth condition. Typical growth rates of 0.2 µm/hr for GaAs and 0.1 µm/hr for AlAs are employed for the quantum well layers. The thicker GaAs layers in the buffer and contact regions are grown at 1 µm/hr to avoid dopant redistribution during growth. The gallium and aluminum effusion cell setpoints corresponding to these growth rates are verified by calibrating the cells using RHEED.

MBE Growth:

(a) A 1 µm n^+-GaAs buffer is grown at 580°C at a growth rate of 1 µm/hr. The gallium cell temperature is reduced to lower the growth rate to 0.2 µm/hr. The substrate temperature is reduced to 550°C (we have subsequently found no significant change in the electrical characteristics of resonant tunneling diodes (i.e., peak current density and peak-to-valley current ratio) by employing growth temperatures between 550°C to 650°C [70]) and the quantum well layers are grown using the computer automated shutter control system. A growth interruption of 3 secs between layers is employed. After the growth of the quantum well regions, a 0.5 µm, n^+-GaAs contact layer is grown at 580°C at 1µm/hr. The RHEED pattern is examined after growth and the characteristic 2x4 pattern should still be clear. The substrate is cooled to about 200°C in an arsenic flux and the block is then transferred back to the preparation chamber. The arsenic flux after growth is noted and compared to the value before growth.

Post-Growth Procedures:

(a) The MBE-grown sample is demounted from the molybdenum block. The epitaxial layers are metallized and subsequently processed using conventional photolithography to form devices using the fabrication sequence described in Appendix B. Fig. 3.3 shows a SEM photograph of a quantum well diode mesa fabricated using the processing sequence described in Appendix B. The undercutting of the GaAs layers from the isotropic GaAs etch produces a gold lip on the edges of the mesa (Fig. 3.3).

A summary of the salient steps involved in the MBE growth and device fabrication of GaAs/AlAs tunneling structures has been described in this chapter.