Chapter 3

Studies on Physical Characteristics, Dislocations, and Electronic Transport Properties.

3.1. Physical and chemical observations.

3.1.1. Microscopic physical observations.

Surface features of LPE-grown epilayers are important to assess the quality of the layers, and are valuable indications for growth-related problems. The most common of these are the followings:

1. **Terrace or ripple patterns** which are associated with misorientation of the substrate surface from low Miller index planes.

2. **Pinnholes** in the epilayer, created by inhibition of nucleation during growth due to the presence of contaminants on the substrate.

3. **Indentations or pits**, caused by either partial filling of pinholes, or replication of etching-related localized imperfections on the substrate surface.

4. **Cusp defects**, thought to be caused by localization of the planar movement of growth steps at some positions by protuberances or depressions on the surface.

5. **Meniscus lines**, created by non-uniform viscous motion of
the growth solution on the surface during layer growth, which is common in layers grown by sliding-boat LPE systems, and

6. Edge growth, caused as a result of increased growth rate round the perimeter of the substrate. The exact reason for such growth is not yet clear. However, a shallow growth melt, and slow growth rates help to reduce this defect.

The grown layers were observed for the aforementioned physical imperfections, under an Olympus microscope fitted with a Nomarski Interference Contrast (NIC) attachment. The layers were found to be uniform, smooth and almost featureless. Photomicrograph of a typical layer is shown in Fig. 3.1.

Thickness of the epilayer was required for determination of the free carrier concentrations by galvanomagnetic measurements. The data was also essential to assess the LPE growth rate. Thickness of each layer was measured from the microscopic observation of the cleaved layer-substrate interface revealed by the Abrahams-Buiocchi (A-B) etchant [3.1]. This etchant is a mixture of a AgNO₃ (0.32gm) solution in water (40ml) and HF (40ml), called solution A, and a CrO₃ (40gm) solution in water (40ml), known as solution B. Photomicrograph of the cleaved section of a layer, revealed by the A-B etchant, is shown in Fig. 3.2.
Fig. 3.1. NIC micrograph of a typical LPE GaAs layer.
Fig. 3.2. Photomicrograph of a cleaved LPE GaAs layer, revealed by the A-B etchant.
3.1.2. Secondary ion mass spectroscopy (SIMS).

The secondary ion mass spectroscopy (SIMS) is a popular technique to determine the impurities present and their distribution in a semiconductor sample. In this technique, the target surface is bombarded with a beam of primary ions like nitrogen plasma having energies of several keV. Atoms and molecules are knocked or sputtered out of the target and may emerge in their ground states, as excited particles, or as positive or negative ions. A fraction of these ions are extracted from the target region and passed into a mass spectrometer where they can be separated according to their mass-to-charge ratio.

The steps between measuring the size of the peaks in a SIMS spectrum and deriving the concentrations of the elements present are not straightforward. The simplest procedure is to compare the peak heights in the sample of unknown composition with the corresponding peaks from a standard sample of known surface composition. In case of GaAs, a pure, single crystal sample is cleaved in situ on a (110) crystal plane where the occupancy of Ga and As atoms are equal. If the unknown sample has a composition only few atomic percent different from that of the stoichiometric compound, then a reliable quantitative analysis is possible.

In the current work, the objective of doing SIMS analysis was to identify the major impurities in the LPE-grown GaAs samples. Another aim was to ascertain the amount of isoelectronic
dopant incorporated in the epilayer. Some typical grown samples were subjected to SIMS experiment, using a standard setup at the Indian Association for the Cultivation of Science, Calcutta. From this analysis, the major donor and acceptor impurities in the layers were identified to be oxygen, and silicon respectively (vide Fig.3.3). Further, it was observed that the amounts of isodopants incorporated were consistent with the respective concentrations of the impurity, calculated using reported values of distribution co-efficients for the isodopant.

3.2. Etch pit density (EPD) measurement.
3.2.1. Introduction to the EPD technique.

A popular technique to assess the dislocation density in an epitaxial layer is the surface method where the point of emergence of a dislocation is revealed. If a crystal containing dislocations is subjected to an environment which removes atoms from the surface, the rate of removal of atoms around the point at which a dislocation emerges at the surface may be different from that for the surrounding matrix. The difference in the rate of removal arises from one or more of a number of properties of the dislocation, viz.

(a) lattice distortion and strain field of dislocation,
(b) geometry of planes associated with a screw dislocation,

so that the reverse process of crystal growth produces a
Fig. 3.3. SIMS data for a typical LPE GaAs:In layer.
(c) concentration of impurity atoms at the dislocation which changes the chemical composition of the material near the dislocation.

If the rate of removal is more rapid around the dislocation, pits are formed at these sites, whereas if it is less rapid, then small hillocks are formed.

3.2.2. EPD measurements done in this work.

As already discussed in Chapter 1, it was shown experimentally that an optimum range of In-doping could reduce dislocation density in VPE-\[3.3\] and MBE-grown \[3.4\] GaAs epilayers. One of the objectives of this work was to do a similar study on LPE-grown GaAs layers.

Here, dislocation density on the surface of the layer was estimated by counting the density of etch pits produced by etching the layers under molten KOH \[3.5\]. About 10gms of KOH pellets were taken in a stainless steel crucible, melted, and held around 350°C. The layer under test, typically, of 1cm x 1cm dimension, was dipped into the molten mass and etched for about 1 minute. After that, the sample was removed from the crucible, washed by DI water, and dried. The etch pits so formed are shown in the photomicrograph of Fig.3.4. These pits were counted under the microscope to get the resultant value of EPD.
Fig. 3. Photomicrograph showing dislocation etch pits formed on a LPE GaAs layer.
3.2.3. Results and discussions.

In the upper part of Fig. 3.5, the EPD measured on a number of samples are plotted with various degrees of In concentration. EPD went below $10^2 \text{cm}^{-2}$ for In-doping concentration of $5 \times 10^{18} \text{cm}^{-3}$. This result is in agreement with that obtained by other workers [3.3]. For large values of In doping concentrations, EPD increased again. We may note that In doping increases the lattice constant of the layer relative to the substrate and the layer gets strained. For high values of In doping density, this strain may be released causing misfit dislocations at the interface, which eventually thread into the epilayer. This was confirmed by microscopic observations of high In-content layers which shows appearance of cross-hatched patterns (Fig. 3.8(a)), typical of mismatched layers [3.6]. This explains the observed increase in EPD for In concentrations exceeding $5 \times 10^{19} \text{cm}^{-3}$. Thus it can be concluded from this study that the beneficial effects of In doping in reducing dislocations in GaAs layers is expected only within an optimum range of the isodopant density.

3.3. Hall measurements.

3.3.1. Introduction to Hall measurements.

The Hall effect measurement is a versatile tool to get useful information on the transport properties of carriers in bulk and epitaxial semiconductors. The Hall co-efficient is
Fig. 3.5. Etch pit density (EPD), Hall mobility ($\mu_H$) and Hall carrier concentration ($n_H$) of GaAs layers grown with different amounts of indium doping.
Fig. 3.6. Photomicrograph of the surface of (a) a heavily In-doped, and (b) a heavily Sb-doped LPE GaAs layer.
inversely proportional to the free carrier concentration in a material where a single type of charge carrier dominates. Although analysis becomes more involved, similar information may be extracted when both electrons and holes are significant. A temperature-dependent Hall measurement is used to provide direct information on the energy positions and concentration of various electrically active impurities present in the material, and the different scattering mechanisms involved. The measurement gives an idea regarding the perfection of the crystal lattice since, at a particular temperature, carrier mobilities are enhanced with increase in lattice perfection. Hall profiling is usually done to get the variation of mobility and density of carriers along the depth of the sample.

In this work, free carrier concentration and Hall mobility of carriers in the epitaxial layers were measured by Hall effect experiments at room temperatures and at 77K. Considering the lamellar geometry of the samples the van der Pauw technique was chosen.

3.3.2. Principles of the technique used.

The van der Pauw-Hall measurements are based on a theorem [3.7] which applies to the measurement of resistivity in a plane uniform lamella with four contacts placed on its edge, as shown in Fig.3.7(a). The sample is required to be uniform in thickness \( h \) and doping density, but it may be of an arbitrary shape. Then
Fig. 3.7(a). Uniform plane lamella with line contacts on its edge representing the ideal geometry for Hall measurements by the van der Pauw method.
van der Pauw's theorem may be expressed in terms of resistances of the form $R_{ABCD}$, given by

$$R_{ABCD} = \frac{V_{AB}}{I_{CD}} \tag{3.1}$$

where, $V_{AB}$ is the voltage measured across contacts $A$ and $B$ when a current $I_{CD}$ flows between $C$ and $D$. It can be shown that $R_{ABCD}$ and $R_{BCDA}$ satisfy the relationship,

$$\exp\left\{-\pi \frac{R_{ABCD} h}{\rho}\right\} + \exp\left\{-\pi \frac{R_{BCDA} h}{\rho}\right\} = 1 \tag{3.2}$$

If $h$, $R_{ABCD}$ and $R_{BCDA}$ are measured, Eqn.(3.2) may be solved for the resistivity $\rho$ of the layer given by

$$\rho = (\pi h / \ln 2) \left\{ \frac{R_{ABCD} + R_{BCDA}}{2} \right\} f(Q) \tag{3.3}$$

where, $Q = R_{ABCD} / R_{BCDA}$, and $f(Q)$ is the van der Pauw asymmetry factor given by

$$\frac{Q - 1}{Q + 1} = \left( f / \ln 2 \right) \cosh^{-1}\left[ 0.5 \exp(\ln 2 / f) \right] \tag{3.4}$$

Values of $f$ are usually obtained from a $f$ vs. $Q$ plot, shown in Fig.3.7(b).

In practice, all the four permutations of the contacts, $R_{ABCD}$, $R_{BCDA}$, $R_{CDAB}$, and $R_{DABC}$ for both the forward and the
Fig. 3.7(b). The van der Pauw asymmetry function $f(Q)$. 
reverse directions of the current were used to calculate $\rho$. The average of these values was taken to be the resistivity of the sample. Care was taken so that individual values of resistances did not differ much.

In order to measure the Hall mobility of carriers in the epilayers, a magnetic field $B_z$ was applied perpendicular to the plane of the sample. The Hall co-efficient is, then, given by

$$R_H = \langle h/B_z \rangle \Delta R_{BDAC}$$

(3.5)

where,

$\Delta R_{BDAC}$ is the change in $R_{BDAC}$ produced by the applied magnetic field, $B_z$. Both the directions of current and magnetic field were reversed, and the current and voltage arms were interchanged to obtain the average value of $R_H$. For samples where electrons are the dominant charge carriers, the Hall mobility $\mu_H$ is simply given by $R_H/\rho$.

3.3.3. System description.

The Hall measurement system consists of the following components:

1. The electromagnet, imported from Newport Inc., USA is capable of generating a dc magnetic field upto 10kGauss across its circular pole pieces of 10cm diameter for a 5cm gap. The electromagnet is water-cooled, and is
powered by a constant current source.

2. The van der Pauw resistivity and Hall voltage measurement unit consists of an IC-controlled constant current generator, fitted with a digital current indicator, and a multi pole rotary switch. This assembly is used to choose different combinations of the current and voltage probes, and reverse the direction of current conveniently during measurements. A Philips PM2525 multifunction digital meter is used to measure the voltage.

3. The Hall sample holder is made of a 30cm long, 3.5cm wide, and 5mm thick perspex strip. Four phosphor-bronze probes are fitted at one end of the strip to provide pressure contacts to the Hall sample. Wires are soldered at the bases of the probes for external electrical connections.

4. A 30cm long glass dewer of 4cm inner diameter is used for Hall measurements at 77K when the sample mounted on the sample holder is immersed in the dewer filled with liquid nitrogen.

3.3.4. Experiments.

Layers grown on semi-insulating substrates, and having n-type unintentional background doping were used for Hall mobility and carrier concentration measurements. Typically, a sample was 5mm square. Small dots of 90% In-10% Sn alloy were
soldered at the four corners of the sample. It was then annealed at 450°C for 10 minutes under hydrogen ambient to form ohmic contacts. For Hall measurements, the sample was mounted on the holder and electrical connections to the constant current source and millivoltmeter were made. In the first part of the experiment, a constant dc current, typically 2.3mA, was passed through two contacts while voltage was measured across the other two contacts. Six readings (V₁ through V₆) were taken for different combinations of the four electrodes (see Fig.3.8) and forward and reverse directions of current. The different combinations of electrodes take care of any possible asymmetry of the sample while reversal of current eliminates errors arising due to thermoelectric effects. In the second part, a constant dc magnetic field of 3.7kGauss, was switched on while voltage was measured across diagonally opposite contacts of the sample. Four voltages (V₇ through V₁₀) were recorded for forward and reverse directions of the magnetic field and the conduction current (see Fig.3.8).

Epilayers were grown on SI substrates, and were nominally n-type, which implied that conduction through the substrate was negligible, and electrons were the dominant charge carriers. So, no complicated processing of the experimental data was required. The Hall carrier concentration, nₜ, and Hall mobility, μₜ, were calculated by using the simplified working formulae.

\[ n_H = 10^{12} \times B I / (1.6 d \Delta V_H) \]  

(3.8)
With $B = 0$,

$V_1 = V_C - V_D$ for $I = I_{AB}$

$V_3 = V_B - V_C$ for $I = I_{DA}$

$V_5 = V_A - V_C$ for $I = I_{BD}$.

With $B = B_2$,

$V_7 = V_A - V_C$ for $I = I_{BD}$.

With $B = -B_2$,

$V_9 = V_A - V_C$ for $I = I_{BD}$.

$V_2 = V_C - V_D$ for $I = -I_{AB}$

$V_4 = V_B - V_C$ for $I = -I_{DA}$

$V_6 = V_A - V_C$ for $I = -I_{BD}$.

$V_8 = V_A - V_C$ for $I = -I_{BD}$.

$V_{10} = V_A - V_C$ for $I = -I_{BD}$.

Fig. 3.8. Schematic of electrical connections used for van der Pauw Hall measurements.
and,

\[ \mu_H = 10^8 \times \frac{\Delta V_H}{(4.532 \times V_{\text{av}} f B)} \]  

(3.7)

where \( n_H \) is in \( \text{cm}^{-3} \), \( B \) is the applied magnetic field in Gauss, \( I \) is conduction current in \( \mu \text{A} \), \( d \) is the layer thickness in \( \mu \text{m} \), \( \Delta V_H \) is the Hall voltage in mV, \( f \) is the van der Pauw asymmetry factor described in Eqn.(3.4), and \( \mu_H \) is the Hall mobility in \( \text{cm}^2 \text{V}^{-1} \text{s}^{-1} \). The quantities \( V_{\text{av}} \) and \( \Delta V_H \) are obtained directly from the experiment as

\[ V_{\text{av}} = \frac{1}{4} (|V_1| + |V_2| + |V_3| + |V_4|) \]  

(3.8)

and

\[ \Delta V_H = \frac{1}{4} (|V_7 - V_5| + |V_8 - V_6| + |V_9 - V_5| + |V_{10} - V_6|) \]  

(3.9)

where all the voltages are in mV. The asymmetry factor \( f \) is a function of the parameter \( Q \) (vide Eqn.(3.4)) which is given by

\[ Q = (|V_1| + |V_2|)/(|V_3| + |V_4|) \]  

(3.10)

or, by the reciprocal of the quantity in the right hand side so that \( Q \geq 1 \).

Measurements were done at room temperatures, and at 77K. For the latter, the sample, mounted on the holder was immersed
slowly in the liquid nitrogen-filled dewer. The dewer was then placed in between the magnetic pole pieces. Then the experiment was done as described earlier.

3.3.5. Results and discussion.

The lower part of Fig. 3.5 shows the results of Hall mobility ($\mu_H$) at 300K and 77K, and Hall carrier concentration ($n_H$) at 300K of GaAs layers grown with different amounts of In-doping. It is seen that the 300K mobility lies in the range, $(8-7) \times 10^3 \text{cm}^2\text{V}^{-1}\text{s}^{-1}$, and the mobility at 77K remains in the range, $(2-3) \times 10^4 \text{cm}^2\text{V}^{-1}\text{s}^{-1}$ while the In doping density varies between $(0.3-5) \times 10^{19} \text{cm}^{-3}$. Also, the trend is similar for 300K and 77K measurements. The Hall carrier concentration at 300K assumes low values in the same magnitudes of isodoping density. It is interesting to note that this was the range of In concentration, which gave the lowest values of dislocation density in our EPD measurement experiment. The 300K results are similar to those of Uddin and Andersson [3.4] who found an improvement in the transport properties of their MBE-grown samples in almost the same range ($\approx 0.1-5) \times 10^{18} \text{cm}^{-3}$) of In-doping. However, they observed peaking of 77K mobility at about $7 \times 10^{18} \text{cm}^{-3}$ of In doping. No such effect is seen here. Beyond $5 \times 10^{19} \text{cm}^{-3}$ of In doping, we see from Fig. 3.5 that Hall mobilities at 300K and 77K fall sharply, while there is a steep rise in the Hall carrier concentration. These observations will be discussed after the
Carrier mobilities in semiconductors are limited by various scattering mechanisms active in the materials. For GaAs, the deformation potential, and the polar optic scattering mechanisms are dominant. With increase in temperature and impurity-content in the material, the effects of ionized impurity scattering become more and more pronounced. The mechanism is particularly important as a means of characterizing material quality. If the Hall mobility and the Hall carrier concentration of a sample are known at a specific temperature, a theoretical model of ionized impurity scattering may be applied to assess the concentrations of ionized impurities and the compensation ratio. Lancefield and co-workers [3.8] made a detailed study on the subject, and prepared a curve which may be used as a look-up table to find out ionized donor and acceptor impurity concentrations, $N_D$ and $N_A$ respectively in LPE GaAs.

In Fig.3.9, values of $N_D$ and $N_A$ in the samples are presented as functions of indium concentration. It is interesting to see from Fig.3.5 and Fig.3.9 that the observed 10-15% rise in mobility upon In-doping is a consequence of the decrease in both background donor and acceptor concentrations. How this reduction actually occurs or why it should be related to In-doping, is not very clear at this stage. It may not be easy to get the answers to these questions since the dopant indium may incorporate additional impurities in the layer [3.9]. However, it is speculated that In atoms occupying Ga-vacancies help to reduce
Fig. 3.9. Donor concentrations ($N_D$) and acceptor concentrations ($N_A$) calculated from the analysis of the Hall measurement data of GaAs doped with indium.
the available cation sites for the group IV impurity atoms, which in turn, leads to a decrease in electrically active donor impurities. Decrease in acceptor concentrations as a consequence of In doping has been confirmed by our photoluminescence experiments and the possible reason for this reduction has been explained. This will be described in Chapter 5.

3.4. Hall measurements of Sb-doped GaAs layers.

Hall measurements, described in the previous section, were done at 300K and 77K on LPE-grown GaAs layers doped with Sb concentration varying in the range, $4 \times 10^{16} \text{cm}^{-3}$ to $2 \times 10^{18} \text{cm}^{-3}$. The results are shown in Fig.3.10. It may be observed that the trend of enhancement of mobility within an optimum range of isodoping is also present in GaAs:Sb samples. However, it is about $(1-5) \times 10^{17} \text{cm}^{-3}$, which is nearly one order of magnitude lower than that for In-doping. Similar to the highly In-doped layers, cross-hatch patterns appear on the layers (vide Fig.3.6(b)) for high Sb-doping densities, indicating lattice mismatch between the layer and the substrate.

3.5. Capacitance-voltage (C-V) measurements.

3.5.1. Introduction to C-V measurements.

In the capacitance-voltage (C-V) technique, small-signal capacitance of the depletion region of a Schottky-barrier or a
Fig. 3.10. Hall mobility ($\mu_H$) as a function of Sb-doping density.
p-n junction diode is studied as a function of the applied reverse bias. This is useful in obtaining extensive information about concentrations and characteristics of electrically active impurities in the epitaxial layer of the semiconductor material on which the diode is fabricated. It is also possible to profile the sample non-destructively by increasing the reverse bias which pushes the edge of the depletion region deeper and deeper into the material. Since the width of the depletion zone bears a definite relation to the applied reverse bias, the spatial dependence of the background doping density can be obtained. However, the ultimate range of such profiling is limited by the reverse breakdown voltage of the device.

In this work, C-V characterization of Schottky-barrier diodes were done to find the variation of the doping density in the epilayers with different amounts of In-doping. Another purpose of such measurements was to cross-correlate the results obtained from the Hall measurements.

3.5.2. Principles of C-V measurements.

It is well-known that the small-signal depletion capacitance for a Schottky-barrier diode is given by,

$$ C = \frac{A(\varepsilon_{s} N_{B})^{1/2}}{2} \left\{ V - \frac{kT}{q} \left[ 1 - \exp\left( \frac{-qV}{kT} \right) \right] \right\}^{-1/2} \left\{ 1 - \exp\left( \frac{-qV}{kT} \right) \right\} $$

(3.11)
where, \( A \) is the diode area, \( q \) is magnitude of electronic charge, \( \varepsilon_s \) is the dielectric constant of the semiconductor, \( N_D \) is the effective donor density at the depletion edge, \( V \) is the total reverse bias applied on the junction, \( k \) is Boltzmann constant, and \( T \) is the diode temperature. At room temperatures and for large bias, \( V \gg kT/q \), which reduces Eqn.(3.11) to

\[
C = A \left( \frac{1}{2} q \varepsilon_s N_D \right)^{1/2} V^{-1/2} \quad (3.12)
\]

Then the net donor density \( N_D \) prevailing at the edge of the depletion region of the diode may be obtained from the slope of the \( 1/C^2 \) vs. \( V \) curve,

\[
N_D = \frac{2}{q \varepsilon_s A^2} \left[ \frac{d}{dV} (1/C^2) \right]^{-1} \quad (3.13)
\]

3.5.3. System description.

The C-V characterization systems consists of a capacitance meter, the test jig, and a desktop computer as a controller of the measurements. The block diagram of the system is given in Fig.3.11. Various components of the system are:

1. The capacitance meter:

A HP 4280A digital C meter/C-V plotter is used to bias the diode, and to measure the small signal capacitance of the
Fig. 3.11. Block diagram of C-V characterization system.
same. A constant dc bias up to ±100V can be applied from the internal bias source of the meter. A 1MHz high frequency voltage of 40mV rms is superimposed on the dc bias to measure the small-signal capacitance using lock-in technique. The meter outputs an internally averaged capacitance reading having a maximum accuracy of 10^-3 pF. The microprocessor-controlled meter is able to provide a staircase sweep bias voltage. Various parameters, like the start and stop voltages, the step height, and the step delay time of the sweep can be programmed manually, or over the HP interface bus (HPIB) with the help of a computer controller.

2. The computer controller:

A HP9000/238 desktop personal computer is used as the controller of the characterization system. The computer, the C meter and a HP 7475A digital plotter are connected via the HPIB. The computer runs in HP-BASIC 4.0 environment, and is used to program the C-meter, trigger the experiment, and acquire data from it. A program, CVDATA has been developed in HP-BASIC for the purpose, which is listed in Appendix A.

3. The test jig:

The test jig used in the C-V experiment was made of a 1/4" thick, 4cm long and 2cm wide copper base plate. Pressure contact to the gold dot was made with the help of a thin, rigid gold wire soldered to a leaf of Be-Cu alloy, fixed to the base
plate, and insulated electrically from the latter. The capacitance of the jig itself was measured to be 2pF.

3.5.4. Experiments.

The C-V experiments involved the following steps.

1. Fabrication of GaAs Schottky barrier diodes:

   Schottky barrier diodes were formed on the layers, grown on semi-insulating (SI), and on \( n^+ (2 \times 10^{18} \text{ cm}^{-3}) \) GaAs substrates. For the SI substrates, ohmic contacts were made in the proximity of the Schottky dots and for the \( n^+ \) substrates, ohmic contacts were formed on the back surface of the substrate. In both cases alloyed indium-tin (In-Sn) contacts were used. After formation of the ohmic contact, the layer surface was thoroughly degreased and slightly etched in \( 5\text{H}_2\text{SO}_4 + 2\text{H}_2\text{O} + 3\text{H}_2\text{O} \) solution to expose a clean semiconductor surface. It was then placed inside a vacuum evaporator and a 1500-2000 Å thick gold film was deposited on the layer under a vacuum of \( 10^{-6} \) Torr through a nickel shadow mask, containing a number of 0.4 mm diameter holes. A number of gold dots of the same diameter were thus deposited on the layer. Each of the dots acted as a Schottky diode. Some of the diodes were directly fabricated on the samples used for Hall measurements, with proximity ohmic contacts in order to get the proper correspondence between the Hall and the C-V data.
2. Measurement of diode area:

Capacitance of a Schottky-barrier diode is directly proportional to the square of the diode area. So, accurate measurement of the diode area is of utmost importance. In order to get the diameter of the diode very precisely, the measurement was done under the Olympus microscope, using a filar eyepiece. The sample was illuminated with polarized light, and observed through an analyzer, which made the gold dot distinct against the background of the epilayer. The diameter of the gold dot was selected by choosing the longest chord, and its length was measured to calculate the diode area.

3. C-V data acquisition:

The diode under test (DUT) was mounted on the test jig, and electrical contacts to the capacitance meter were made. Proper contact to the diode was checked by monitoring the zero-bias capacitance. The capacitance meter was then programmed by running the CVDATA program with the parameters of the reverse bias sweep. The sweep bias was applied to the DUT and the data was recorded in the computer for later analysis. In a typical scan, the sweep was started from a dc reverse bias of 0V, and the reverse bias was increased in steps of 0.5V at an interval of 250ms. The maximum bias to be applied on a particular DUT was determined from the value of conductance of the diode, which was measured by the meter simultaneously. The sweep was stopped as soon as the conductance started increasing. Typically it was 60
always possible to run the sweep up to a reverse bias of 10V.

4. Data plotting and calculations:

The program CVDATA was used to store C-V data in datafiles on floppy disks and to plot $1/C^2$ vs. $V$ data. The background doping density, depletion depth and the built-in potential were calculated using the C-V data by another program, CVLYSIS.BAS (vide Appendix A) when the diode area was supplied.

3.5.5. Results and discussions.

Free carrier concentration values at 300K obtained from the C-V analysis of gold Schottky-barrier diodes are presented in Fig. 3.12 as functions of In-doping concentration. The carrier concentration reduces from $7 \times 10^{18}$ cm$^{-3}$ to $5 \times 10^{15}$ cm$^{-3}$ when the In concentration varies in the range of $(3-7) \times 10^{18}$ cm$^{-3}$, and then increases gradually up to a little over $1 \times 10^{18}$ cm$^{-3}$ as the In doping concentration goes up to $5 \times 10^{19}$ cm$^{-3}$. This behavior agrees with that of the data obtained from the Hall measurements. However, for samples with In-doping above $5 \times 10^{19}$ cm$^{-3}$, the free carrier concentration decreases sharply. This trend is opposite to what we observed from the Hall measurements data (Fig. 3.5). We may explain this by saying that the fall in $\mu_H$, observed in this region, is related to the high density of defects generated from the mismatched interface and not to any actual rise in the carrier concentration. Any quantitative assessment of this effect
Fig. 3.12. 300K background free carrier concentration, $\langle N_D - N_A \rangle$, obtained from the C-V measurements of gold Schottky barrier diodes fabricated on In-doped GaAs layers.
is not easy to perform since dislocation tubes, with all possible orientations relative to the interface, may take part in this kind of scattering. In the van der Pauw technique, $n_H$ is measured as a reciprocal of the Hall voltage, $\Delta V_H$. Any apparent rise in $n_H$ actually corresponds to a fall in the measured value of $\Delta V_H$. The C-V measurement data indicate that the trend in $n_H$ obtained from the Hall measurement is only apparent. The fall in carrier concentration for high values of In doping is expected in view of impurity gettering by high density of misfit dislocations generated.

3.6. Conclusions.

About forty layers were grown to make the study described in this chapter, and to arrive at the final conclusions. The average size of a layer was 1cm x 1cm. EPD and C-V measurements were done at different parts of the same layer to examine homogeneity of the results. It was found that the measured characteristics were similar throughout the layer except at the edges. However, the size of the layer was too small to conduct any such uniformity test for the Hall data. In general, at least three separately grown layers were used for getting the average Hall data corresponding to each value of In-doping concentration and efforts were made to cut the sample, each time, from a new location on the layer.
From EPD, Hall, and C-V measurements conducted on our In-doped materials, it is concluded that there exists an optimum range for In doping where EPD goes to a minimum and the transport properties assume their best values. This range is \((0.3-5) \times 10^{19} \text{ cm}^{-3}\) for the average 8-10\(\mu\)m thick LPE layers grown by us. It is difficult to specify the mechanism involved on the basis of the results obtained. However, it is apparent that reduction of ionized impurity concentrations, enhancement of Hall mobility, and dislocation density reduction are related phenomena. The dopant In atoms are likely to be instrumental in reducing electrically active impurities associated with group III sites, Ga-vacancies, and vacancy-related complexes (vide Chapter 6) in the material. Singh [3.10] has shown theoretically that In atoms have a much higher surface migration rate than Ga atoms. The fast migrating In atoms tend to occupy normally vacant Ga sites in growing crystals. Thus, they lower densities of such vacancy-related defects. These defects may, in turn, be components of dislocations in the material. In fact, several workers have proposed that vacancies condense to generate dislocations during crystal growth [3.11]. Dislocation reduction upon In doping is also explained by the solution hardening model [3.12] whereby \(\text{InAs}_4\) units, due to larger atomic radius of In compared to that of Ga, prevent propagation of dislocations from the substrate into the GaAs epilayer. The rise in Hall mobility may then be attributed to reduced ionized impurity- and dislocation-related scatterings.
Very high values of the isoelectronic dopant have a detrimental effect on the overall properties of the material. As a result of high layer-substrate lattice mismatch, misfit dislocations propagate to the layer, causing a drastic rise in EPD and a corresponding decrease in the carrier mobility. Read proposed that dislocations in semiconductors act as acceptors, and hence in n-type crystals, dislocations capture free electrons around the dislocations lines to form cylindrical space charge regions [3.13]. The Read model may be invoked to explain gettering of impurities at high In doping densities by large density of dislocations. This is reflected in the carrier concentration data obtained from the C-V experiments.

We also carried out Hall mobility and carrier concentration measurements on GaAs layers doped with Sb in the range $4 \times 10^{16}$ cm$^{-3}$ to $2 \times 10^{18}$ cm$^{-3}$. The results show similar trend as observed for In-doped layers. In this case, the optimum range of Sb doping is about $(1-5) \times 10^{17}$ cm$^{-3}$. We shall see from photoluminescence results in Chapter 5 that Sb atoms play a significant role in reducing certain acceptor impurities associated with the group V sites in GaAs. This observation, and the similarity between the behavior of Hall mobility upon In and Sb doping further strengthen the interrelation of isodoping and improvement of material qualities.
References


