CHARACTERIZATION OF STRAINED-LAYER SUPERLATTICES

The tailoring of bandstructures of semiconductor devices to enhance their electronic and optical properties has become, over the past two decades, one of the major driving forces in semiconductor physics. The three major phenomena used for band tailoring are; (i) alloying of two or more semiconductors, (ii) use of heterostructures to cause quantum confinement of formation of superlattice and (iii) use of built-in strain via lattice mismatch epitaxy. Alloying of two materials is one of the oldest techniques to modify and improve the properties of materials. In semiconductors, this is motivated by the sole objective to alter the bandgap of the material to a pre-chosen value; and also to create a material with proper lattice constant to match or mismatch with an available substrate. There are other potential advantages also like improved carrier transport, fabrication of heterostructures etc.

When two semiconductors A and B are mixed via an appropriate growth technique, then the information on the crystalline structure of the lattice and the ordering of the atoms (arrangement of A and B atoms in the alloy) are to be considered very carefully. While in most semiconductors the two or more components of the alloy have the same crystal structure, mixing a diamond lattice material (e.g. Ge) with a zinc-blende material (e.g. GaAs) can lead to some very interesting structures [1]. Figure 3.1 shows the changes in the bandgaps of various material combinations; the solid lines represent direct bandgap regions and the dotted lines the indirect gap regions. The ordering of the atoms in the alloy is also extremely important since the band-structure depends strongly on ordering. The arrangements of the atoms A and B in the alloy may have the following cases:
Fig 3.1: The bandgap range achievable by alloy formation in some III-V compound semiconductors [1].
• All of the A atoms are localized in one region while the B atoms are localized in another region. Such alloys are called phase separated.

• The probability that an atom next to an A-type atom is A is $X$ and the probability that it is B is $(1-X)$. Such alloys are called random alloys.

• The A and B atoms form a well-ordered periodic structure leading to a superlattice.

A few specific cases of these alloys are, (i) GaAs-AlAs alloy which gives AlGaAs system; the most important component of high speed electronic and optoelectronic devices. Nearly all kinds of quantum well structures can be formed utilizing its perfect lattice match with GaAs. It also interestingly exhibits switching of the bandgap from direct to indirect. (ii) InAs-GaAs alloy giving rise to the InGaAs system. These are active components of very high speed electronic and optoelectronic devices. InAs and GaAs have a lattice mismatch of 7%. InGaAs also shows excellent low and high field transport properties and is the one we studied in this work. (iii) HgTe-CdTe giving HgCdTe. HgTe and CdTe are very well lattice matched, do not show miscibility gap and span a bandgap of 0 to 1.5 eV. HgCdTe has a direct bandgap throughout the composition range and is used for very small bandgap device applications. Also, it can be grown on CdTe, ZnTe and even Si and GaAs substrates. (iv) Si-Ge alloy; this alloy allows the heterostructure concepts to be used in Si technology since it can be a component of Si-SiGe structure. Apart from this, these structures don’t find potential applications in the optoelectronic devices. Silicon and germanium have a lattice mismatch of 4% and hence a suitable substrate to grow this alloy on cannot be found easily.

The basis of all the advantages of bandgap tailoring is the charge confinement that is achieved through this process. This is done by growing a thin layer (~ 100 Å) of one semiconductor sandwiched inside a different semiconductor with different bandgap. The electrons (or holes) are then confined in the smaller gap material by the potential barriers arising from the bandgap discontinuity at the interface. As a result, the quantization energy shifts the band edge to a higher energy; the electrons and holes are brought closer and the density of states becomes independent of energy. If the confinement is realized in one dimension, the resulting device is quantum well,
two dimensional confinement gives a quantum wire and three dimensional confinement gives a quantum dot. The present discussion is focused on quantum well devices; the fabrication of these devices by the latest epitaxial growth technologies is relatively simple compared to the processing of quantum wires or dots. The original idea of fabrication of a quantum well structure was proposed by Esaki and Tsu [2].

3.1 Epitaxial Growth

The fabrication of these types of devices became possible only after the major advancements in the technology of epitaxial crystal growth techniques like Molecular Beam Epitaxy (MBE), Metal Organic Chemical Vapour Deposition (MOCVD), Organic Metallic Vapour Phase Epitaxy (OMVPE), Atomic Layer Epitaxy (ALE) and Hot Well Epitaxy (HWE). Epitaxy literally means outer, upon or attached to (Greek epi) + arrangement or order (taxis). The thickness of the layers grown can be controlled to one atom layer at the rate of one micron per minute with the help of these techniques. Much improved crystalline quality, accurate control over substitutional doping and control of interface roughness to less than a couple of monolayers could also be achieved. Thus, by growing materials with different bandgaps, bandgap "tailoring" or "engineering" can be achieved. Therefore epitaxial growth, used to grow layers of elemental and compound semiconductors, as compared to all other crystal growth techniques, which provides better control of purity and perfection as well as doping level, is a well controlled phase transition leading to a single crystalline solid, and is the exclusive growth technique for semiconductor alloys such as $Al_xGa_{1-x}As/GaAs$ and $In_xGa_{1-x}As/GaAs$. The thermodynamics of the phase transition is the first basic tool to be considered carefully. Since this is a dynamic and not an equilibrium process, the kinetics of both the mass transport and of surface processes must also be considered carefully. Defect generation is another problem to be dealt with, since the epitaxial growth is normally at low temperatures and has a high growth rate. In OMVPE, the growth is carried out in a cold wall reactor; the growth is generated by the following reaction

$$Ga(CH_3)(g) + AsH_3(g) \rightarrow GaAs(s) + 3CH_4(g)$$
for trimethyle-Ga and

$$Ga(C_2H_5)_3(g) + AsH_3(g) \rightarrow GaAs(s) + 3C_2H_6(g)$$

for triethyl-Ga. The absence of Cl precludes the etching reaction and simplifies the kinetics by eliminating the possibility of adsorption of chloride species on the surface. The ratio of As to Ga in the input gas stream is greater than one for the most favourable growth conditions. The flow rate is linearly proportional to the gallium flow rate.

Crystalline defects such as grain boundaries, stacking faults, twins and dislocations are known to get generated during the growth process, deteriorating the device properties. These defects have been shown to act as non-radiative recombination centres in many materials [4, 5], and reduce the minority carrier lifetime and growth efficiency in LED [6, 7] apart from degrading the performance of transistors, solar cells, detectors and other devices. Considerable effort has been devoted to the study and elimination of these crystalline defects in semiconductor materials.

All these growth techniques, till recently, concentrated on the use of lattice-matched epitaxy, limiting the associated development of electronic and optoelectronic semiconductor device structures considerably. Wavelengths of laser emission, frequency response and output power of a transistor, all suffered limitations in lattice-matched systems. Strained-Layer Superlattices (SLS) have unique electronic and optoelectronic properties [8]; in these structures the lattice parameter of the deposit is different from that of the substrate which is accommodated by strain in the layers. In lattice-matched epitaxy (fig. 3.2a), the deposit and the substrate have the same lattice parameter; the epilayer atoms getting deposited onto the substrate locate the potential minima corresponding to the substrate lattice sites easily. In Strained-Layer structures, deposit atoms are constrained to the substrate interatomic spacings in the plane of the interface [9], although the lattice parameters of the substrate and the epilayer are different, resulting in a strain in the epilayer and at the interface (fig. 3.2b). The magnitude of this strain directly depends on the composition of the layer and the substrate. This elastic strain energy for a given lattice mismatch increases with the epilayer thickness and beyond a critical thickness, all the strain in the epilayer is relaxed. This process occurs via the introduction of slipped regions into the crystal, bounded by
line defects called the "misfit dislocations". Structures that are strained are termed commensurate or coherent and the strain relaxed materials (fig. 3.2c) are called discommensurate structures. Misfit dislocations are the unwanted defects in the solid which deteriorate the performance of the devices and hence it is important to characterize strain, strain-relieving mechanisms and the limits of strained layer growth. A number of theories exist for the critical layer thickness [5-15] for given structures; the most relevant being the one due to Matthews and BlacJceslee [10, 11, 12].

The critical layer thickness for \( In_{0.1}Ga_{0.9}As \) material lies between 100 A- 300 A. With a view to determine the exact thickness at which tin's system \( In_{0.1}Ga_{0.9}As/GaAs \) SLS relaxes, we have grown a series of such structures and characterized them by ion channeling. Other complimentary characterization techniques like High Resolution XRD and Raman Spectroscopy have also been carried out on these structures and the results are compared. The description of these SLS and also some samples that were obtained earlier is given in the forthcoming section.
Fig 3.2: Schematic illustration of (a) lattice-matched heteroepitaxy, (b) coherently lattice-mismatched heteroepitaxy and (c) relaxed lattice-mismatched heteroepitaxy [9].
3.2 Sample Description

Some of the SLS samples were obtained from the Tata Institute of Fundamental Research, Mumbai, India. These are listed below.

(100) growth direction on GaAs substrate

- 30 alternating layers of $In_{0.6}Ga_{0.4}As$ with thickness 55 Å and GaAs with thickness 225 Å a top cap-layer of GaAs 0.3μ thick and a buffer of 0.6μ.

- 30 alternating layers of $In_{0.5}0.5As$ with thickness 45 Å and GaAs with thickness 225 Å a top cap-layer of GaAs 0.12μ thick and a buffer of 0.6μ.

- 30 alternating layers of $In_{0.6}Ga_{0.4}As$ with thickness 55 Å and GaAs with thickness 900 Å a top cap-layer of GaAs 0.12μ thick and a buffer of 0.6μ.

- 5 alternating layers of GaAs of 200 Å and $In_{0.2}Ga_{0.8}As$ of 70 Å.

Some samples have been grown on the Organo Metallic Vapour Phase Epitaxy (OMVPE) facility at the Solid State Electronics laboratory of Tata Institute of Fundamental Research, Mumbai. All the experiments have been carried out on these samples. The sample specifications are shown below.

Table 3.1: Samples grown at TIFR. Indium content in all the cases is 0.1. All samples grown on GaAs substrate and 5000 Å thick GaAs buffer.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Specification</th>
<th>Thickness (In the same order)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9702</td>
<td>GaAs/InGaAs/GaAs(Buffer)</td>
<td>250 Å / 350 Å /5000 Å</td>
</tr>
<tr>
<td>9706</td>
<td>InGaAs/GaAs(Buffer)</td>
<td>300 Å /5000 Å</td>
</tr>
<tr>
<td>9850</td>
<td>InGaAs/GaAs(Buffer)</td>
<td>100 Å /5000 Å</td>
</tr>
<tr>
<td>9851</td>
<td>InGaAs/GaAs(Buffer)</td>
<td>200 Å /5000 Å</td>
</tr>
<tr>
<td>9853</td>
<td>GaAs/InGaAs/GaAs(Buffer)</td>
<td>300 Å /500 Å /5000 Å</td>
</tr>
<tr>
<td>9854</td>
<td>GaAs/InGaAs/GaAs(Buffer)</td>
<td>300 Å /200 Å /5000 Å</td>
</tr>
</tbody>
</table>
3.3 Rutherford Backscattering Spectroscopy/Channeling

Rutherford backscattering Spectrometry (RBS/channeling) is one technique which can determine the thickness, composition, defect densities, uniformity and strain, and can be applied readily to the superlattices [8, 9, 11].

RBS is based on four basic physical concepts corresponding to specific physical phenomena; each one has a particular capability or limitation of backscattering Spectrometry. These are listed below with brief descriptions.

3.3.1 Basic Physical Concepts

- Energy transfer from a projectile to a target nucleus in an elastic two body collision. This process leads to the concept of kinematic factor and to the capability of mass perception. When a projectile of mass $M_1$ moving with an energy $E_u$ collides elastically with a stationary particle (target) of mass $M_2$, energy will be transferred from the moving to the stationary particle. This interaction is assumed to be elastic with the condition that the projectile energy $E_u$ must be much large than the binding energy of the atom and nuclear reactions and resonances are absent. If $E_s$ is the energy with which the projectile is scattered back, $\theta$ is the scattering angle and $\phi$ the recoil angle, then the kinematic factor $k$ is defined as the ratio of the projectile energy after the collision to that before the collision;

$$k = \frac{E_1}{E_o}$$  \hspace{1cm} (3.53)

The energy and momentum conservation principles give

$$k_{M_2} = \left\{ \frac{[1 - \left(\frac{M_1}{M_2}\right)^2 \sin^2 \theta]^{\frac{1}{2}} + \left(\frac{M_1}{M_2}\right) \cos \theta}{1 + \left(\frac{M_1}{M_2}\right)} \right\}^2 .$$  \hspace{1cm} (3.54)

Thus, with a knowledge of $E_1, E_o$ and hence $k$, the mass of the target can be found out.
The probability that an elastic two-body collision occurs. This leads to the concept of scattering cross-section which in turn leads to quantitative analysis of atomic composition. Suppose a narrow beam of particles impinges on a wide target; a detector is placed at an angle \( \theta \) from the direction of incidence which counts each particle scattered in the differential solid angle direction \( d\Omega \). If \( Q \) is the total number of particles that have impinged on the target and \( dQ \) is the number of particles recorded by the detector, then the differential scattering cross-section \( \frac{d\sigma}{d\Omega} \) is defined as

\[
\frac{d\sigma}{d\Omega} = \frac{1}{Nt} \left( \frac{dQ}{d\Omega} \right) / Q
\]

(3.55)

where \( N \) is the volume density of atoms in the target and \( t \) is its thickness. \( Ni \), therefore is the areal density of the target. The average number of scattering events falling within a "finite" solid angle \( \Omega \) is then given by

\[
\sigma = \frac{1}{\Omega} \int_{\Omega} \left( \frac{d\sigma}{d\Omega} \right) d\Omega
\]

(3.50)

The total number of particles detected or the backscattering yield is written as

\[
Y = \sigma\Omega QNt
\]

(3.57)

i.e.

\[
\begin{pmatrix} \text{number. of} \\ \text{detected particles} \end{pmatrix} = \sigma\Omega \begin{pmatrix} \text{total number of} \\ \text{incident particles} \end{pmatrix} \cdot \begin{pmatrix} \text{number of target} \\ \text{atoms per unit area} \end{pmatrix}
\]

Thus, when \( \sigma, \Omega \) are known and the number of incident and detected particles are counted, the number of atoms per unit area, \( Nt \), can be determined quite accurately.

- **Average energy loss of an atom moving through a dense medium.** This leads to the concept of stopping cross-section and to the capability of depth perception. The likelihood of a Rutherford scattering collision is very less and most often, the projectile penetrates into the target. As it pushes itself through the target, it slows down and its kinetic energy
decreases. The amount of energy lost per unit distance $\Delta r$, which is measured by recording the energy difference before and after the transmission through the target, defines the specific energy loss

\[
\lim_{\Delta r \to 0} \frac{\Delta E}{\Delta x} = \frac{dE}{dx}(E)
\]  (3.58)

The energy at a given depth $x$ below the surface is then given by

\[
E(x) = E_0 - \int_0^x \left(\frac{dE}{dx}\right)dx
\]  (3.59)

$dE/dx$ is a function of energy and not $x$ and so $\int_0^x (dE/dx)dx$ cannot be evaluated without the knowledge of the energy as a function of $x$, $E(x)$. But $E(x)$ is unknown in the equation; the difficulty is resolved by regarding $x$ as a function of $E$, rather than $E$ as a function of $x$. Then,

\[
dx = \frac{dx}{dE}(E)dE
\]  (3.60)

Therefore,

\[
x = \int_E^{E_0} (\frac{dx}{dE})dE = \int_E^{E_0} (\frac{dE}{dx})^{-1}dE
\]  (3.61)

Or

\[
x = (E - E_0)(\frac{dE}{dx})^{-1} |_{E_0}
\]  (3.62)

If $S$ is the target area illuminated by the beam, $N$ the atom density in the target and $\Delta x$ the thickness of the target, then

\[
\frac{dE}{dx} = \frac{SN\Delta x}{S} = N\Delta x.
\]

Therefore $\Delta E$ is set proportional to $N\Delta x$ and the proportionality factor is known as the "Stopping Cross-Section $\xi$".

\[
\xi = \left(\frac{1}{N}\right)\left(\frac{dE}{dx}\right)
\]  (3.63)
• **Statistical fluctuations in the energy loss of an atom moving through a dense medium.**

This process leads to the concept of energy straggling and to a limitation in the ultimate mass and depth resolution of the backscattering spectrometry. An energetic particle that moves through a medium loses its energy via many individual encounters. As a result, identical energetic particles, which all have the same initial velocity, do not have the same energy after passing through a thickness $\Delta x$ of a homogeneous medium; they are no more monoenergetic. This results in fluctuations in the energy loss which places a finite limit for the precision on both the mass and depth perception. For these reasons, it is very important to have quantitative information on the magnitude of energy straggling for any given combination of energy, target material, target thickness and projectile.

When the incident ion beam is directed along a high-symmetry crystal direction, ion channeling occurs [20, 21]. $\chi_{\text{min}}$, which is the ratio of the backscattered particles when aligned (A) to a crystallographic axis to that in the random (R) condition (i.e., $\chi_{\text{min}} = Y_A/Y_R$) is a measure of the crystalline quality of the sample. A $\chi_{\text{min}}$ value of a very good crystalline sample will be around 2-3%. With ion channeling one can determine the tetragonal distortion in the epilayer and consequently the in-plane strain (whether compressive or tensile) can be determined. Therefore, the use of ion channeling in conjunction with RBS provides a measure of the crystalline quality as a function of depth and also allows determination of the strain in epitaxial layered structures. Strain measurements by ion-channeling technique in multi-layered structures is based on the tetragonal distortions induced in the layers.

To align the sample that is cut so that the [001] axis is normal to the incident beam, the axis of rotation of the goniometer is given a tilt of 3° from the incident direction. A "$\phi$" rotation is then given in steps of 1.5° and the backscattered yield is recorded each time. The same procedure is adopted for a tilt of 6°. The position of the most pronounced minima are recorded on polar coordinates. The lines connecting the minima then correspond to {100}, {110} and {111} planes. The intersection of these lines correspond to the [001] axial direction. The coordinates of the point of intersection give the goniometer position that will line up the [001] axis with the beam [21].
The two components of strain are defined as

\[ \epsilon_\perp = \frac{d_L^1 - d_L^2}{d_L^2} \]  
\[ \epsilon_\parallel = \frac{(d_L^1 - d_L^2)}{d_L^1} \]  

where \(d_L^1\) and \(d_L^2\) are the interplanar spacings of the strained layer in the direction perpendicular and parallel to the interface respectively. \(d_L^2\) and \(d_L^1\) are the corresponding values for the free lattice of the epilayer material. The tetragonal distortion in the epilayer \(\epsilon_t\) is related to \(\epsilon_\parallel\) and \(\epsilon_\perp\) as

\[ \epsilon_t = \epsilon_\parallel - \epsilon_\perp = \frac{\Delta \theta}{\sin \theta \cos \theta} \]  

where \(\theta\) is the angle between the positions of [100] and [110] directions where we get minimum yield in the substrate and \(\Delta G\) is the difference in the position of the minimum yield dip of substrate and epilayer.

4.4 High Resolution XRD

High Resolution X-Ray Diffraction (HRXRD) has become an essential and versatile tool to characterize heteroepitaxial structures, and, often complementary to other characterization techniques. XRD is very sensitive to the lattice strain. Point defects are too small to be observed by conventional imaging techniques but they do have appreciable effects on the X-ray intensity profile. With the advent of increasingly perfect single crystals and crystalline layers, a need for refined characterization emerged and HRXRD stands among the most powerful and non-destructive techniques. Under the optimum conditions, HRXRD is capable of detecting strains with a sensitivity of about \(10^{-5}\) [22]. It provides information on the interface structure with monolayer precision.

HRXRD gives a Fourier transform of a crystal volume of typically 1 mm x 1 mm x (5—50) \(\mu m\). The data are X-ray intensity distributed in the vicinity of a reciprocal lattice point (or a Bragg peak), which is integrated over the direction normal to the diffraction plane by a detector wide-open in that direction. A high angular resolution, and thus a high strain sensitivity is achieved by
monochromating and/or collimating the incident X-ray beam, the procedure for which is given in ref. [22].

The relation between the chemical composition and the lattice parameter form the basis for HRXRD. The lattice parameters of the alloys gradually change with the chemical composition and can be approximated by a linear interpolation of the lattice parameters of the binary end members. This relation is known as Vegard's Law [23, 24]. With HRXRD, the lattice parameter and the lattice mismatch can be determined exactly and from this the chemical composition can be deduced. Consider the system of AlGaAs on GaAs. Because of the similar lattice parameters \( c_0(\text{GaAs}) = 5.563 \, \text{Å}, a_0(\text{AlAs}) = 5.661 \, \text{Å} \), the lattice mismatch determination at the Cu-AlGaAs (004) reflection must be accurate to \( 2.7 \times 10^{-5} \) corresponding to an angular width of the layer peak (\( \Delta \Omega \sim 0.001 \)), if the chemical composition is to be determined with an accuracy of 1%.

Another analytical aim of HRXRD is the determination of layer thickness. In measurements of extremely thin single layers, the small reflectivity presents the difficulties, rather than the angular width of the layer peak. Therefore for carrying out HRXRD, one needs to have an angular resolution of at least 0.001° and a dynamical range of intensity.

As stated earlier, HRXRD measures the intensity of scattered X-rays; these are distributed in the neighbourhood of a reciprocal lattice point. \( k_u \) is defined as the incident wave vector, \( k \) as the diffracted wave vector inside the crystal and \( K_0 \) and \( K \) are defined as the incident and diffracted wavevectors outside the crystal, conventionally. So the reciprocal lattice point serves as a reference position in the reciprocal lattice space in describing the distribution of scattered intensity as a function of deviation \( (q) \) of the scattered wave-vector \( K \) from the reciprocal lattice point. The \( q \)-vector lies within the diffraction plane because the scattered intensity distributed in the direction normal to the diffraction plane is integrated by a wide-open detector.

If we define \( \theta_B \) as the angle at which the Bragg reflection takes place, \( (\theta_{BS}, \text{the Bragg angle for the substrate and } \theta_{L}, \text{that for the epilayer}) \), then the Bragg condition is written as

\[
2d \sin \theta_B = \lambda
\]  

(3.67)
Differentiating the above equation, we get

$$\frac{\Delta d}{d} = \epsilon_\perp = -\cot \theta_B \Delta \theta \tag{3.68}$$

For GaAs samples and materials grown on them, (004) (224) (224) (115) and (115) are the directions in which the Bragg equation is satisfied. If $\omega_x$ is the incident angle of the X-ray beam to the substrate and $\omega_y$, that to the epilayer, $\tau_S$ the inclination of the Bragg plane to the substrate and $\tau_L$ that to the epilayer, then $\omega_x = \theta_{BS} + \tau_S$ is called the High Incident angle and $\omega_y = \theta_{BS} - \tau_S$, the Low Incident angle for the substrate. Similarly $\omega_x = \theta_{BL} + \tau_L$ is the High Incident angle and $\omega_y = \theta_{BL} - \tau_L$, the Low Incident angle for the epilayer. We then have [25, 26],

$$\epsilon_\perp = \frac{\cos \tau_S \sin \theta_{BS}}{\cos \tau_L \sin \theta_{BL}} - 1 \tag{3.69}$$

$$\epsilon_\parallel = \frac{\sin \tau_S \sin \theta_{BS}}{\sin \tau_L \sin \theta_{BL}} - 1 \tag{3.70}$$

The tetragonal distortion $\epsilon_f$ is then given by

$$\epsilon_f = \frac{(1 - \nu)\epsilon_\perp + \nu \epsilon_\parallel}{1 + \nu} \tag{3.71}$$

where $\nu$ is the Poisson ratio which is given by

$$\nu = \frac{c_{12}}{c_{11} + c_{12}} \tag{3.72}$$

for the (001) surface and $c_{ij}$ are the elastic constants [22].

Determining $\omega_x, \omega_y, \theta_{BS}$ and $\theta_{BL}$, one can evaluate the strain straightforwardly. If the thickness of the epilayer exceeds the critical thickness, the layer relaxes giving rise to misfit dislocations; the tetragonal symmetry is cancelled and the unit cells of the layer assume a cubic symmetry. In the case of fully strained layer, $\epsilon_\parallel$ is zero and when the layer partially or fully relaxes, $\epsilon_\parallel$ is no longer zero. Thus by carrying out HRXRD, one can establish whether the sample has relaxed or not and also quantify the strain if the system has not relaxed.

### 3.5 Raman Spectroscopy

Raman Spectroscopy also allows one to determine this strain. Constant et al. [27] determined the internal strain in the pseudomorphic $In_{x}Ga_{1-x}As/GaAs$ structures over a wide range of
composition $x$. The quantitative measurement of strain is obtained from the phonon wave number shift between commensurate and incommensurate layers, governed by the formula

$$
\varepsilon_{ph} = \frac{1}{\beta} \frac{\delta \nu}{\nu_o}
$$

where $\beta$ is a constant which depends on the material, growth direction and conditions and phonon type mode. $\nu_o$ is the wave number of the strain relaxed material for the same composition, which is evaluated from figure 3 of ref [27]. This value is compatible to the LO phonon wave number ($\nu$) of In$_x$Ga$_{1-x}$As/GaAs structure with InGaAs thickness 500 Å which is expected to be relaxed. $\delta \nu$ is the wave number shift.

3.6 Experimental

Sample of In$_{0.1}$Ga$_{0.9}$As/GaAs used in this work was grown by Organometallic Vapour Phase Epitaxy (OMVPE) at 100 torr pressure and $640^\circ C$ growth temperature using alkyles trimethyl gallium, trimethyl indium and the hydride arsine. Epilayers are grown on nominal (001) GaAs substrate on a 5000 Å thick epitaxially grown GaAs buffer layer. The V/III ratio used on this growth is about 150.

On sample 9706, RBS and channeling have been carried out by 1.5 MeV and 3 MeV He$^+$ beam at Institute of Physics, Bhubaneswar, at a scattering angle of 150°. The scattering chamber is equipped with a goniometer with five degrees of freedom ($x, y, z, \theta, \phi$). On the goniometer, the 9 precision is of 0.1° and the $\phi$ rotation has a precision of 0.3°. The beam is collimated by a pair of collimators of dia 1.5mm, seperated by a distance of 0.5 meter. The details of this setup are given in [29].

On other samples, RBS and channeling have been carried out at the 2 MeV Van de Graaff accelerator at Indian Institute of Technology, Kanpur. He$^+$ beam of energy 1.2 MeV has been utilized at a scattering angle of 150°. The scattering chamber is equipped with a goniometer with six degrees of freedom; three translational motions ($\pm 10mm$) along the $x$, $y$, and $z$ directions, and three rotational motions namely the 9 rotation ($180^\circ$) around the beam direction (x) with
a precision of $0.0125^\circ$, the $\phi$ rotation ($360^\circ$) around the axis perpendicular to the beam in the vertical plane with a precision of $0.01^\circ$ and the $\Psi$ rotation ($\pm3.5''$) around the axis perpendicular to the beam in the horizontal plane with a precision of $0.007''$.

RBS and channeling have been carried out again on these samples due to some ambiguity in the channeling results of IIT, Kanpur. This work is carried out at Center of Irradiation of Materials, Alabama A & M University, at 3.058 MeV $He^{+}$ beam at a scattering angle of $170^\circ$ and a solid angle of $0.2249\text{ msr}$, the total FWHM is about 35-40 keV. The goniometer of this setup has four degrees of freedom; $x$ (horizontal transversal), $z$ (vertical transversal) with a precision of $0.0025\text{ mm}$ each, $\theta$ rotation (about the $z$-axis) of $(1^\circ$ precision and a $\phi$ rotation (about the beam direction) of $0.001^\circ$ precision. The beam is collimated by a pair of collimators of $1.5\text{ mm}$ and $2\text{ mm}$ respectively.

A brief description of the Philips XTERT Materials Research Diffractometer (MED) at TIFR, Mumbai, is given here.

(i) A series of Pre-aligned Fast Interchangeable X-ray (PREFIX) optical modules for point and line focus applications. Each PREFIX optical module is fitted to a U-shaped mounting bracket which has precisely machined, highly polished surfaces. It also has the ability to rotate ceramic insulated X-ray tube from point to line focus. Because of this feature the system can be changed from a High Resolution or Stress/Texture point focus application configuration into a normal Bragg configuration using the appropriate PREFIX optical modules for line focus applications, in a very less time without losing the alignment. (ii) A PW3040/00 Coasole comprising the X-ray generator, electronics racks with CPU and source controller. The electronics rack contains two position controls to drive the horizontal goniometer, Dual Sealer boards for the detectors and additional multiple DC Motor drive boards to drive the MRD cradle. (iii) A PW 3050/20 horizontal goniometer equipped with two optical disks for Theta and 2Theta. The goniometer is controlled by two position controls (Pos. Con. 2) which are able to drive $9$ and $29$ in steps of $0.0001^\circ$ with a reproducibility of $\pm0.0001^\circ$. (iv) The MRD cradle has five following motorized movements: (a) a phi $\phi$ rotation ($360^\circ$, step size $0.02^\circ$) with a reproducibility of $\pm0.01^\circ$ and a slew speed of $70^\circ$/sec (b) a Psi tilt $\Psi$ ($180^\circ$, step size $0.01^\circ$) with a reproducibility of $\pm0.01^\circ$ and a slew speed of $2^\circ$/sec.
(c) X/Y translation for wafer mapping (±50 mm) with a step size of 0.01 mm and a precision of 10\'; (d) Z translation (±5 mm) with a step size of 0.001 mm and a precision of 1µ; (e) Sample oscillation around any point on the X and Y table perpendicular to the diffraction plane. The cradle can be used as a programmable automatic sample changer for multiple samples that have different heights and shapes. (v) Three PRIMARY primary 4-crystal monochromators and a Ge[220] primary collimator. The Ge-crystals are factory aligned, tilted and set-up in either the Ge[440] or Ge[220] reflection to use the system in the High Resolution mode. The specifications of the assemblies are (a) 4 crystal symmetrically cut Ge[440] setting: Δθ ≈ 5'' (b) 4 crystal symmetrically cut Ge[220] setting: AB ≈ 12'' (c) 4 crystal asymmetrically cut Ge[220] setting: Δθ ≈ 18'' (d) 2 crystal symmetrically cut Ge[220] collimator: Kα1 and Kα2. (vi) A PRIMARY crossed slits collimators with filter slots, used for Texture or Psi-stress measurements. Opening 10 x 10mm continuously, scale 0.02 mm. (vii) A PRIMARY Fixed Divergence Slit Assembly with Masks. A programmable divergence slot assembly is optional. (viii) A PRIMARY Secondary HR-Rocking Curve Assembly with rotatable slit section. (ix) A PRIMARY Secondary HR-Rocking Curve/Triple Axis Optics Assembly with rotatable slit section. (x) A PRIMARY Secondary parallel plate collimator (thin film attachment). (xi) A PRIMARY Secondary Diffracted Beam Optics Assembly with programmable receiving slit. (xii) General Data Collector Windows Software.

Raman spectra are recorded in the backscattering geometry so as to observe the LO vibrations of the singlet type [30], which correspond to the phonons that propagate along the axis of the strained samples [31, 32]. Vertically polarized 514.5 nm argon-ion laser beam of 100 mW power was used to excite the Raman spectra. Scattered light from the samples was collected using a camera lens (Nikkon) and focussing lens. It was dispersed using a double monochromator of model Spex 14018 and detected using a cooled photomultiplier tube of model ITT-FW 130 operated in the photon counting mode. The slit width of the monochromator corresponding to 4.2 cm\(^{-1}\) in terms of FWHM of the instrument resolution function was employed. The scattered light was integrated for 10 seconds and digitally recorded at 0.5 cm\(^{-1}\) wavenumber interval. The optical absorption spectra are recorded using a Chemita 599 UV-VIS spectrophotometer.
3.7 Results and Discussion

RBS spectrum of 1.5 MeV $He^{+}$ on the sample 9706 is shown in figure 3.3. From this random spectrum and the thicknesses of the epilayer are confirmed. The atomic fraction of In is found to be 0.1. The In signal from the epilayer is easily resolvable but the Ga and As signals from epilayer could not be distinguished from those of the buffer. Picraux et al [34] have carried out RBS on $In_{0.19}Ga_{0.81}As$ multilayered (31nm/layer) superlattice. They found that the In fraction (x=0.19) in the alternating layers gives rise to major oscillations and is resolvable upto 16 layers. These oscillations are not seen in the present studies, owing to the lesser composition of indium content [10].

In our study the Ga signal in the $In_{x}Ga_{1-x}As$ is reduced as also the As. The amount of reduction in the yield depends on the crystalline quality of the sample material. For instance, the $\chi_{min}$ of indium for all the samples is around 10% indicating reasonably good crystallinity of the epilayer. Strain measurements by ion-channeling technique in multi-layered structures is based on the tetragonal distortions induced in the layers. These experiments for SLS have been performed in early eighties [11],[35]-[43]. Picraux et al [39] have determined strain in $GaAs_{1-x}Sb_{x}/GaAs$ grown by MBE. We have determined the tetragonal distortion using formula (3.14). $\Delta \theta$, the shift in the minimum yield and the corresponding tetragonal distortion, $\epsilon_{t}$, has been found out from figure 3.4. The results are tabulated in table 3.2. The shifting of the minimum yield towards left with respect to the substrate indicates that the distortion is compressive. This is expected since the lattice parameter of $In_{x}Ga_{1-x}As$ larger than that of $GaAs$. No separation in the minimum yield dip of the substrate and the buffer has been observed.

On samples 9702 and 985 series, RBS/C experiments are carried out at Alabama A & M University. 985 series samples show that the atomic fraction of indium has changed to 0.075 as against 0.1. This is later confirmed by HRXRD. The results of the available data are shown in figures 3.5 - 3.7 and are tabulated in table 3.2.

Channeling at 1.2 MeV on these samples were first carried out at Indian Institute of Technology, Kanpur. The results of this experiment shows that all the samples are relaxed. But High
Resolution XRD and Raman Spectroscopy show a finite strain in all the samples. At these low energies, the critical angle for channeling becomes larger than the angular misalignment [41] and becomes complicated and often ambiguous due to the beam steering effects, in that some of the channeled ions become random in one layer, although aligned in one. This steering effect can also alter the symmetry of angular scan profiles of deep layers [44, 45]. For instance, figure 3.8 shows the angular scan around $<011>$ axis, of sample 9702. The shift in the minima of epilayer from that of substrate is less than 0.1°, giving a tetragonal distortion of $\epsilon_t = 0.17\%$. This indicates that the sample has relaxed, contradicting the experiments of HRXRD and Raman Spectroscopy. It was then decided to carry out channeling at 3 MeV and the samples (other than sample 9706) were subsequently studied at Alabama A & M University; the results of which are reported here.
Fig 3.3: 15 MeV $\text{He}^{++}$ RBS spectrum for a scattering angle of 150° from a single layer of $\text{In}_{0.1}\text{Ga}_{0.9}\text{As}$ on GaAs.

Fig 3.4: Channeling angular scans along the inclined $<110>$ axes from a single 30-nm layer of $\text{In}_{0.1}\text{Ga}_{0.9}\text{As}$ on GaAs.
Fig. 3.5: Channeling angular scan around <100> and <110> axes.

$\Delta \theta = 0.2^\circ$ and $\theta = 45.31^\circ$
Fig. 3.6: Channeling angular scan around <100> and <110> axes.
\[ \Delta \theta = 0.18^\circ \text{ and } \theta = 45.06^\circ \]
Fig. 3.7: Channeling angular scan around $<100>$ and $<110>$ axes.

$\Lambda \theta = 0.2^\circ$ and $\theta = 45.033^\circ$
Figure 3.8: Channeling angular scans along the inclined [110] axes from 25 nm GaAs and 35-nm In$_{0.1}$Ga$_{0.9}$As layer on a GaAs substrate.
X-ray measurements of (004) reflection along four azimuth directions give mean separation between the GaAs Bragg peak and the $In_{0.1}Ga_{0.9}As$ Bragg peak $\Delta \Omega$ such that $\epsilon_\perp$ and $\epsilon_\parallel$ are evaluated straightforwardly. We have measured the asymmetric 224 and 224 reflections also for this sample, from which, $\epsilon_\perp$, $\epsilon_\parallel$ and hence the strain are found out and tabulated in table 3.2. Figures 3.9 - 3.13 show the X-ray spectra of all the samples.

SLS of $In_{x}Ga_{1-x}As$ have been studied by Raman Spectroscopy rigorously [28] and all such studies reveal that InGaAs presents a two mode behaviour; the GaAs-like LO phonon mode and InAs-like LO phonon mode. The GaAs-like LO phonon is more dominant and the TO mode is not observed at all. The wavenumber of InGaAs is shifted from both the LO phonon modes of the bulk GaAs and the GaAs-like LO of the ternary alloy for the same composition. This feature is the superposition of the GaAs LO and the GaAs-like LO of $In_{x}Ga_{1-x}As$ shifted by the efforts of strain in the opposite direction. $\nu_0$ is found to be $288.3 \text{ cm}^{-1}$ and $\nu$ is noted down in figures 3.14 - 3.19 for each sample. ($\beta$ value for the indium content of 10% has been found by least square fit of the values given in table 3 of reference [27]. It comes out to be 1.6602. Strain value is then evaluated using equation (3.20). The results are tabulated in table 3.2 along with those of ion-channeling and HRXRD.

Table 3.2: The values of strain for various samples using Ion Channeling, XRD and Raman Spectroscopy.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Strain ($\epsilon_\perp$, %) By Ion Channeling</th>
<th>Strain ($\epsilon_\parallel$, %) By HRXRD</th>
<th>Strain ($\epsilon_\perp$, %) By Raman Spectroscopy</th>
</tr>
</thead>
<tbody>
<tr>
<td>9702</td>
<td>1</td>
<td>0.96</td>
<td>1</td>
</tr>
<tr>
<td>9850</td>
<td></td>
<td>0.7565</td>
<td></td>
</tr>
<tr>
<td>9706</td>
<td>0.7</td>
<td>1</td>
<td>1.3</td>
</tr>
<tr>
<td>9851</td>
<td>0.7</td>
<td>0.92</td>
<td>0.945</td>
</tr>
<tr>
<td>9853</td>
<td>0.7</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>9854</td>
<td>0.7</td>
<td>0.85</td>
<td>0.9</td>
</tr>
</tbody>
</table>
Fig 3.9: Ω/2Θ scan around (004).
Fig 3.10: $\Omega/2\theta$ scan around (004)
Fig 3.11: $\Omega/2\theta$ scan around (004)
Fig 3.12: $\Omega/2\theta$ scan around (004)
Fig 3.13: $\Omega/2\theta$ scan around (004)
Fig 3.14: Raman Spectrum showing the peak, fitted using the PEAKFIT software.
Fig 3.16: Raman Spectrum. The dashed line shows the fitted curve.
Fig 3.17: Raman Spectrum. The dashed line shows the fitted curve.
Fig 3.19: Raman Spectrum. The dashed line shows the fitted curve.
Fig 3.18: Raman Spectrum. The dashed line shows the fitted curve.
Strain values in such technologically important SLS can also be found out by empirical formula.

\[ \epsilon_\perp = \frac{(a_j - a_\perp)}{a_\perp} \]  \hspace{1cm} (3.74)

and

\[ \epsilon_\parallel = \frac{(a_j - a_\parallel)}{a_\parallel} \]  \hspace{1cm} (3.75)

where \( a_j \) and \( a_j' \) are the lattice constants of the strained layer in the direction perpendicular and parallel to the interface respectively. \( a \) and \( a'' \) are the corresponding values for the free lattice of the epilayer material. The tetragonal distortion in the epilayer \( \epsilon_t \) is related to \( \epsilon_\parallel \) and \( \epsilon_\perp \) as

\[ \epsilon_t = \epsilon_\parallel - \epsilon_\perp \]  \hspace{1cm} (3.76)

These can be calculated by finding out the lattice constants of \( \text{In}_{x}\text{Ga}_{1-x}\text{As} \) using the Vegard's law

\[ a_{\text{In}_x\text{Ga}_{1-x}\text{As}} = xa_{\text{InAs}} + (1-x)a_{\text{GaAs}} \]  \hspace{1cm} (3.77)

and the lattice constants \( a_{\text{GaAs}} = 5.6532 \text{\AA} \) and \( a_{\text{InAs}} = 6.0584 \text{\AA} \), we find \( a_{\text{In}_{0.9}\text{Ga}_{0.1}\text{As}} = 5.6937 \text{\AA} \). \( \epsilon_\parallel \) and \( \epsilon_\perp \) are related to each other by the elastic constants \( c_{11}, c_{12} \) etc as

\[ \epsilon_\parallel = -\frac{c_{11}}{2c_{12}} \epsilon_\perp \]  \hspace{1cm} (3.78)

which again can be found by Vegard's law. Assuming fully strained epilayer, the expected value of \( \epsilon_\perp \) is 0.014 which is higher than the \( \epsilon_\perp \) values obtained from the two measurements described above. The lower \( \epsilon_\perp \) value can occur if the strained layer relaxes. The critical thickness for strain of 0.01 obtained from the Matthews and Blackeslee formulation is close to 200 \text{\AA}. Therefore a partial relaxation of the strain in the epilayer cannot be ruled out at this stage.

The lattice constant of unstrained \( \text{In}_{0.1}\text{Ga}_{0.9} \) will henceforth be named as \( a_{\text{ep}} \) and that of the GaAs as \( a_5 \). According to the definition, the tetragonal distortion that one determines from ion channeling is the change in the lattice parameter from unstrained to strained value. X-ray scattering determines the strain from the lattice parameter the epilayer takes with respect to the substrate lattice parameter.
The two can thus be related as follows:

**Ion Channeling:**

\[ \varepsilon_t = \frac{\Delta \theta}{\sin \theta \cos \theta} = \varepsilon_{\parallel} - \varepsilon_{\perp} \]  \hspace{1cm} (3.79)

\[ \varepsilon_{\parallel} = \frac{a_{\parallel} - a_{\perp}}{a_{\text{epi}}} \]  \hspace{1cm} (3.80)

**XRD:**

\[ \varepsilon_f = \frac{a_{\parallel} - a_{\perp}}{a_S} \]  \hspace{1cm} (3.81)

This \( \varepsilon_f \) is evaluated from the \( \varepsilon_{\perp}^{\text{XRD}} \) that is determined from X-ray measurements as follows [22]

\[ \varepsilon_{\perp} = \frac{a_{\parallel} - a_{\text{epi}}}{a_S} \quad \text{and} \quad \varepsilon_{\parallel} = \frac{a_{\parallel} - a_{\perp}}{a_S} \]  \hspace{1cm} (3.82)

When the epilayer is fully strained,

\[ a_{\parallel} = a_S \quad \text{and so} \quad \varepsilon_{\parallel} = 0 \]  \hspace{1cm} (3.83)

\[ \varepsilon_{xx} = \varepsilon_{yy} = \frac{a_{\parallel} - a_{\text{epi}}}{a_S} = \varepsilon_{\parallel} - \varepsilon_f \]

\[ \varepsilon_{zz} = \frac{a_{\parallel} - a_{\text{epi}}}{a_S} = \varepsilon_{\perp} - \varepsilon_f \]

\[ = -\frac{2\nu}{1 - \nu} \varepsilon_{xx} \]  \hspace{1cm} (3.84)

\[ \therefore \varepsilon_f = \frac{2\nu}{1 + \nu} \varepsilon_{\parallel} + \frac{1 - \nu}{1 + \nu} \varepsilon_{\perp} \]  \hspace{1cm} (3.85)

So, in the case of a fully strained sample, \( \varepsilon_{\parallel} = 0 \) and hence,

\[ \varepsilon_f = \frac{1 - \nu}{1 + \nu} \varepsilon_{\perp} \]

where \( \nu \) is the Poisson ratio = \( c_{12}/c_{11} + c_{12} \).

\[ \therefore \varepsilon_{\perp} = \frac{a_{\parallel} - a_S}{a_S} \]  \hspace{1cm} (3.86)
and thus,

\[ \epsilon_t = -\epsilon_\perp \left( \frac{a_S}{a_{\text{epi}}} \right) \]  

(3.87)

The strain values that are tabulated in table 3.2 can be compared straightforwardly.
References


References


