CHAPTER II
Experimental details

This chapter gives details of different experimental facilities that have been used in this thesis work. Experimental details regarding the sample growth, irradiation, implantation and characterization are described. Sample identifications (IDs) and other nomenclature have also been defined here. This information will be used throughout the thesis. The details of experimental conditions and analysis that have been used in this thesis work are discussed in detail.

2.1 Samples Details

(i) GaN

GaN samples studied in the next (third) chapter are thick layers grown on c-plane sapphire substrates by MOCVD in a rotating disk reactor at Ladex Corporation (Taiwan). The typical growth condition involves a low temperature GaN buffer followed by the growth of GaN film at ~1100 – 1150 °C. The thicknesses of the films were ~2 µm as measured from RBS spectra. These layers are unintentionally doped epilayers. A6 sample is 2 µm thick GaN grown on sapphire substrate. A4 sample is also 2 µm thick GaN on sapphire substrate, but the initial defect densities vary on this sample with respect to A6 sample. RE48 is AlN (50nms) /GaN (2µm) grown on Sapphire. All these samples were grown in typical growth conditions with GaN buffer layers. But in the sample RE48, thin layer of AlN grown to check the nitrogen out gassing on irradiation.

GaN samples used in the Er implantation works (chapter 5) are commercial GaN wafer bought from the TDI Corporation, USA. Typical thickness of the GaN layers is ~5 µms. Small pieces of size 10 mm x 10 mm are cut from the wafers and implantation has been carried out.
The HEMT structure consisted of a 0.1 µm thick AlN buffer layer and 1 µm thick undoped GaN buffer/channel layer then an undoped 50 nm thick Al\textsubscript{0.2}Ga\textsubscript{0.8}N barrier layer. It was grown in a Vacuum Generator V80H (Mark I) MBE system that was originally used for arsenide materials. Conventional effusion cells were used for Ga and Al deposition, and an Applied Epi Uni Bulb RF-plasma source was used for the active N. During the nitride film growth, the chamber was pumped exclusively by a CTI CT-8 cryopump. The base pressure of the system was always kept below 5x10\textsuperscript{-11}Torr. The substrate temperature was monitored by the substrate heater thermocouple that was calibrated to the SiC wafer surface by observing the melting point of aluminum dots on a SiC test sample. The substrate temperature was kept to 835°C for 0.1 µm AlN with a growth rate of 0.2 µm/h. RHEED patterns show that the growth was slightly Al rich. Now the substrate temperature was reduced to 735°C for growth of GaN and AlGaN layers. Growth rate of GaN is maintained at 0.37µm/h. The growth of AlGaN layer was continued under the same conditions by introducing the Al BEP as Al source. The growth process was completed by shutting the Ga and Al sources and turning off the RF plasma. Finally, the substrate temperature was brought down to room temperature. The final structure of the sample (2024) studied in chapter 4 is given by (50nm) Al\textsubscript{0.2}Ga\textsubscript{0.8}N / (1µm) GaN / (0.1 µm) AlN on SI 4H-SiC. The sample was grown on a quarter of a 2" wafer.

2.2 Irradiation and Implantation

All the high energy irradiations were carried out at Inter University Accelerator Center (IUAC), New Delhi high energy accelerator facility (chapters 3 & 4) with 150 MeV Ag ions at fluence of 5x10\textsuperscript{12} ion/cm\textsuperscript{2}. These Ag ions will go to depth of approximately 11.03 µm and 11.13 µm in GaN and substrates respectively, and lose energy at the rate of 24.79 keV/nm in GaN and 22 keV/nm in substrates through electronic interactions For GaN, nuclear energy loss of 150 MeV Ag ions is 9 x10\textsuperscript{-2}keV/nm with Sn / Se ratio as 3.6x10\textsuperscript{-3}. Therefore 150 MeV Ag ions are used to avoid high Sn/Se ratio and possible formation of point defects. We have used these Ag ions based on our previous results on AlGaAs/GaAs samples. To avoid heating of samples a low beam current (0.5 – 2 pnA) was maintained. The samples were oriented at an angle of 5 – 7°
with respect to the beam axis to minimize channeling. The high vacuum (~1x10⁻⁶ mbar) irradiation chamber is shown in Fig.2.1.

Fig 2.1. Irradiation chamber in material science beam line at IUAC, New Delhi

2.2.1 IUAC accelerator facility

![Diagram of IUAC accelerator facility]

Fig 2.2 schematic of the IUAC 15 MV pelletron accelerator
IUAC is the main accelerator facility for the university community in India. It has many facilities, mainly high energy pelletron accelerator and supporting facilities for accelerator based research in many fields like Material science, Nuclear Physics, Accelerator Mass Spectrometry (AMS) etc. Fig. 2.2 shows the schematic diagram of the pelletron accelerator. It uses the Sources of Negative Ions by Cesium Sputtering (SNICS) to produce the negative ions and these negative ions are pre accelerated to 400 keV and injected into the main accelerator. Here, it uses the SF6 gas to insulate the high terminal potential from discharging. This accelerator is basically a Van-de Graff accelerator, where the terminal is kept at centre of the accelerating tube. This high voltage terminal (max 15 MV) is charged by using the metal pellet chain, hence it is called pelletron. The negative ions generated from the source are accelerated to the positive terminal, and these get stripped and become positive ions. As a result of repulsive force between terminal positive ions these ions are again accelerated to high energy. These high energy ions are bent through analyzing magnet where the particular ion species, their charge state and beam energy are selected. Then the switching magnet changes the beams into various beam lines in the beam hall for different experimental facilities. The whole pelletron and beam hall safety is fully computer controlled with all the safety mechanisms.

The acceleration process involves two intermediate steps as described above. In the first step a singly charged negative ion will be accelerated in the field of terminal voltage (V) and will attain an energy V (charge state X V). Then if the particle attains a charge state q after passing through the stripper foil, it will be accelerated once again to energy qV under the same terminal potential. Hence in total the particle attains an energy of (1+q)V. Hence terminal potential is setup based on the required beam energy and available charge states. In general most probable (and most abundant) charge state is chosen so as to have stable beam. Beam current depends on the nature of the ion species charge state, energy, stripper foil and other beam tuning parameters. Gas stripper will provide a stable current. Required current can be obtained through proper beam tuning process.

Material science beam line is used for carrying out Swift Heavy Ion (SHI) based materials research. Several users from different universities work in the area of materials characterization and engineering with SHI at this facility. Fig. 2.3 shows a schematic diagram of the beam line and associated facilities. There are three experimental chambers on this beam line namely High Vacuum (HV) chamber, Ultra High Vacuum (UHV)
chamber and Goniometer (GM) Chamber. All the irradiation reported in this work is carried out in the HV chamber.

Fig 2.3 Schematic diagram of the material science beam line

2.2.2 Er Implantation and annealing

A series of samples consisting of GaN films on Al$_2$O$_3$ substrate have been doped with Er ions through the ion implantation technique, followed by post-implantation annealing. Implantation was carried out at INFN-INFM ion implantation laboratory of INFN-Legnaro National Laboratories, Italy and annealing has been carried out at Department of Physics, University of Padova, Padova, Italy. Er implantations dose and annealing temperature were varied systematically in these experiments. We have prepared three different sets of ErGaN samples containing different doses of Er ions and annealing temperature as follows.
(i) $\text{ErGaNI}$: implantation with $1 \times 10^{14} \text{Er}^+ / \text{cm}^2$ at 190 KeV; 1h annealing in N$_2$ atmosphere at 800°C, 950°C and 1100°C;

(ii) $\text{ErGaNS}$: implantation with $5 \times 10^{14} \text{Er}^+ / \text{cm}^2$ at 190 KeV; 1h annealing in N$_2$ atmosphere at 800°C, 950°C and 1100°C;

(iii) $\text{ErGaN10}$: implantation with $1 \times 10^{15} \text{Er}^+ / \text{cm}^2$ at 190 KeV; 1h annealing in N$_2$ atmosphere at 800°C and 1100°C.

2.3 Characterization techniques

RBS/Channeling measurements on GaN in the third chapter are carried out at the Centre for Irradiation of Materials (CIM), Alabama A&M University, Alabama, USA. The energy dependent RBS/C studies used He ions from 2 MeV to 5 MeV in steps of 0.5 MeV. HRXRD measurements have been recorded at Solid State Physics Laboratory, New Delhi using Philips X pert MRD system with a Cu K$_\alpha$ radiation set in point focus mode. This system has a channel cut, four crystal Ge (022) (Bartels-type) monochromator for Cu K$_\alpha_1$ X-ray beam. AFM (SPA400 of SII Inc, Japan) and optical transmission (JASCO V570 UV-VIS-NIR spectrophotometer) studies were carried out in house. In the chapter 4, RBS/C measurements carried out at IGCAR, Kalpakkam, have been reported. We have carried out channeling angular scans and energy dependence of dechannelling on <1-213> (off normal axis) axis. Energy dependence has been carried out in the range 2.3 to 3.8 MeV by varying in step size of 0.3 MeV. Reciprocal space map on <0002> was measured at Padova using Philips Xpert MRD in triple axis configuration. In this work we have used the in house facility of SPA400 of SII Inc AFM. Photo Luminescence studies in Chapter 5 has been carried out at Department of Physics, University of Padova and the RBS/Channeling lattice site location studies has been carried out at IIT, Kanpur, India. Channeling angular scans are measured on the <0001> and <10-11> axis. Details of the analysis carried out using different experiments are discussed following sections.

2.3.1 RBS/Channeling

RBS is a quantitative and nondestructive method for depth profiling and compositional analysis of materials. It is based on scattering of charged particles (normally He ions) in the central force field of target atoms. A collimated beam of mono energetic ions are
accelerated and bombarded on the sample, which is mounted on a 3 axis goniometer. General schematic diagram of the RBS/C set up is shown in the Fig 2.4. A fraction of ions will backscatter from the samples. The energy spectrum of these backscattered ions is analyzed by using a solid state barrier detector and data acquisition setup. When energetic ions are directed at a small angle $\Psi$ to atomic axis or planes of crystal, these are steered by series of gentle collisions and are channeled into the regions between rows or planes of target atoms. Under the channeling conditions, the bombarding He ions will be backscattered from the first one or two surface monolayers of the material at the same rate as a non-aligned sample, but backscattering from the remaining atoms in the lattice will be drastically reduced since these atoms are shielded from the incident He ions by the atoms on the surface layers. The backscattering yield from a single crystal GaN sample which is aligned along the $<0001>$ axis, will be approximately 1.5% of the backscattering yield from a non-aligned spectrum. Fig 2.4 shows the typical RBS spectrum in aligned and random directions. By measuring the reduction in backscattering yield under channeling conditions, it is possible to quantitatively measure the crystal perfection of a sample [1].

Fig 2.4 Schematic diagram of RBS/Channeling experimental setup and RBS/Channeling spectra
(i) Strain measurements on III Nitrides heterostructures

The quality of epitaxial layers and the strain in such layers or in multilayers can be determined by channeling measurements. Generally these strains are estimated by measuring the channeling angular scans. The sample crystallographic axis or plane is aligned along the beam direction and the angular scans are measured by tilting the sample by 2~3° step by step, in small angular intervals of θ ~ 0.1°. The integrated yield of particular regions of interest, like epilayer and substrates are plotted as yield vs θ. Typical angular scans are shown in the Fig 2.5.

Fig 2.5 RBS/Channeling angular scans and strain measurement
RBS/C has been used extensively for the compositional and structural characterization of InGaN and AlGaN [2-9]. RBS with channeling geometry gives information about both "In" and "Al" concentration and defects density in InGaN and AlGaN samples. The influence of the "In" content on the accumulation of the structural damage in In$_x$Ga$_{1-x}$N films under ion bombardment has been studied using RBS/C [4]. Elastic strain in the In$_{0.18}$Ga$_{0.82}$N on the GaN substrate has been measured by this technique and indicates that the layer is much stiffer along the c-axis direction than in the a-axis direction [7]. Similar work has been done by other groups to measure the elastic strain in the In$_{0.18}$Ga$_{0.82}$N and Al$_{0.28}$Ga$_{0.72}$N [8]. RBS/C has been used to gain insight into compositional depth profile of 75 nm thick InGaN in the growth direction [9]. RBS/C has also been used to study the evolution of the structural defects in Al$_x$Ga$_{1-x}$N films irradiated with KeV heavy ions [2].

(ii) Defect characterization in III Nitrides

The dechannelling analysis is carried out by varying the incident energies and measures the aligned spectrum. The dechannelling parameter is calculated from the normalized back scattering yield of random and aligned spectrum using the formula [15],

$$DP = -\log\left(\frac{1 - \chi_D}{1 - \chi_V}\right)$$

(2.1)

where DP = N$_D$\&sigma;$_D$, N$_D$ is the defect density and \&sigma;$_D$ is the dechannelling cross section, \chi; _D is the minimum yield in the defected crystal and \chi; _V is the minimum yield of the defect free crystal at the same depth. This gives appropriate energy dependence for defect analysis.

The DP shows different energy dependences based on the kinds of defects present. The energy dependence of dechannelling cross section on the incident ion energy (i) for point defects it is E$^{-1/2}$ (ii) for dislocation and dislocation loops, it is E$^{1/2}$ as shown in fig 2.6 and (iii) for stacking faults it is E$^{0}$ i.e. independent of energy.
Fig 2.6 Energy dependence of dechannelling parameter (DP) for dislocations

For point defects,

$$\sigma_D = \frac{\pi Z_1^2 Z_2^2 e^4}{E \psi_e} \propto \frac{1}{\sqrt{E}}$$  \hspace{1cm} (2.2)$$

For dislocations,

$$\sigma_D = K \sqrt{\frac{a b d E}{\alpha Z_1 Z_2 e^2}} \propto \sqrt{E}$$

$$\alpha = \text{const depends on axial or planar and defect type}$$  \hspace{1cm} (2.3)

$$a = \text{THOMAS – FERMI Screening Radius}$$

For dislocation loops,

$$\sigma_D \propto \sqrt{E} \quad \text{for} \quad R > \sigma_D$$

$$\sigma_D \text{ independent of } E \quad \text{for} \quad R < \sigma_D$$  \hspace{1cm} (2.4)

$$\text{Where } R \text{ is loop radius}$$

For stacking faults,

$$\sigma_D \propto \chi_{\text{min}}$$

$$\chi_{\text{min}} = \text{Minimum yield}$$  \hspace{1cm} (2.5)
Based on the model of Quere and Pathak et.al calculations [10,11], the effects of dislocations are modeled as the distortions in the axis or plane. The dechannelling of ions occurs when the channel distortion increases beyond a critical value. The parameter relating the channel distortion to the dislocation parameters is basically the radius of curvature $R$ and is given by,

$$ R = \frac{2 \pi^2 d_o}{b \cos \theta} \quad (2.6) $$

where, $d$ is the distance of affected channel from the dislocation core, $b$ is the burgers vector and $\theta$ is the angle between channel axis and plane perpendicular to the dislocation line. In GaN’s the common type of dislocations observed are: (i) Edge type with $b = 1/3 \langle 11-20 \rangle$ (ii) Screw type with $b = \langle 0001 \rangle$ (iii) Mixed type with $b = 1/3 \langle 11-23 \rangle$, with Dislocation line $\langle 0001 \rangle$& Slip Plane [0001] [12].

![Diagram of GaN crystal structure](image)

**Fig 2.7.** Normal $\langle 0001 \rangle$ and off normal $\langle 1-213 \rangle$ axis of GaNs and the inter atomic distance in respective axis
Fig 2.7 shows inter-atomic distance of Normal and off normal axes in GaN. Our calculation shows that, (i) Normal axis <0001> is not affected by either Edge or Screw Dislocations but it is affected by Mixed dislocations. (ii) Off normal axis <1213> is affected by all the kinds of dislocation i.e. Edge, Screw and Mixed.

To the best of our knowledge, only one dechanneiling study of defects has been carried out by [13] on GaNs. On GaN samples, $\chi_D$ was obtained from the experimental RBS spectra from the GaN layers (i.e. below the surface). In AlGaN heterostructures, $\chi_D$ AlGaN layers are calculated by integrating the counts on the whole layer and GaN $\chi_D$ is calculated from 100 nm just below the AlGaN layers. $\chi_v$ was theoretically calculated to be $\sim$ 3%. DP Versus $E^{0.5}$ plot with linear fit and its slope was used for the calculation of dislocation density,

$$N_D = \frac{\text{Slope} \times \sqrt{E}}{\sigma_D}$$  \hspace{1cm} (2.7)

where $\sigma_D$ is given by [1, 10],

$$\sigma_D = \left( \frac{bdaE}{\alpha Z_1 Z_2 e^2} \right)^{1/2}$$  \hspace{1cm} (2.8)

$\alpha = 12.5, 4.5, 7.5$ for screw, edge, mixed dislocation respectively, the Thomas Fermi screening radius $a = 0.1587$ Å, $d$ is inter planar spacing.

(iii) Lattice site location on III Nitrides

The commonly used application of channeling is to determine the lattice sites of impurity atoms in crystals. The impurity atoms present in the axial or plannar channels are seen by the ion beams. As a result there will be an increase in the $\chi_{\text{min}}$ of the aligned spectra. By comparing the angular scans of different channels, the exact position of the atoms can be deduced. RBS/C method is direct way of determining the atomic position unlike the other techniques. Mossbauer and electron spin resonance, where the electronic information is analyzed to get the atomic positions. In GaN the lattice site studies are made by measuring the angular scans for two directions. The projections of the tetrahedral site projected on these two axial channels are shown in Fig 2.8.
Channeling angular scans show characteristic profiles when solute atoms settle at various positions into the channels [1]. Fig 2.9 shows angular scan of foreign atoms in crystal for different possible sites.

(i) When the solute atoms are in substitutional sites, angular scans dip of solute and host atoms has same width.

(ii) When the solute atom is slightly projected into the channel from substitutional site, then angular scan of solute atom has smaller width than host atoms.

(iii) When the solute atom is moved large into the channel from substitutional site, then angular scan of solute atom has double dip.

(iv) When the solute atom is at center of the channel, then angular scan of solute atom has sharp peak.

(v) For randomly located solute atom, a flat angular scan is observed.

Fig 2.8 projection of Ga and N atoms as seen along $<0001>$ and $<10\bar{1}1>$ axis from the ref [14]
Fig 2.9 Channeling angular scans of foreign atom compared with host material with possible lattice sites
2.3.2 HRXRD

The High Resolution X-Ray Diffraction has become a powerful tool for nondestructive investigation of epitaxial layers. One gets information about the uniformity, thickness, strain, strain relaxation and defects present in the layer. Kinematic and dynamical diffraction theories are used to analyze the diffraction patterns from epilayers and heterostructures. The instrumentation has been developed with double and triple axis spectrometers equipped with multi crystal or channel cut monochromators. Fig 2.10 shows the schematic of the triple crystal diffractometer. Radial scans (ω-20 scan) and rocking curves (ω scan) are commonly used in materials analysis. In radial scans, during the θ scan (detector scan) the angle ω between incident beam and the sample surface changes. Hence the scan runs radial from origin of the reciprocal space. In rocking curves, the detector is fixed and the sample is rotated i.e. ω changes. The accuracy of the diffractometer is achieved through the high precision goniometer and the channel cut monochromators [15].

![Fig 2.10 Schematic diagram of the HRXRD triple axis diffractometer](image)

High resolution X-Ray Diffraction (HRXRD) experiments have been carried out using a Philips Material Research Diffractometer (MRD). The ω–20 scan on (0002) direction are done on all the samples. The experimental radial scans are fitted using Pseudo-Voigt function for integral width (FWHM or β) and Lorentzian weight fraction (f). Lateral correlation and residual strain are calculated from FWHM and ‘f’ using the formulas given below. HRXRD studies are based on the diffraction peak widths. The peak broadening is mainly due to finite size of crystalline domains, non uniform strained
regions near to the lattice defects and contributions from instrumental and thermal broadening. The strain broadening of diffraction peak is proportional to the length of the scattering vector which results in Gaussian broadening, but the size effects have an exponential correlation function leading to Lorentzian broadening. The Peak shape analyses of x-ray diffraction are based on the separation of the Lorentzian and Gaussian broadening using Pseudo-Voigt function. By fitting this function one can get the integral width and Lorentzian weight function. [16-18]

\[
I[\beta L(x) + (1-f)L(x)]
\]

where

\[
L(x) = A \left[ \frac{2}{\pi} \frac{\beta}{(\beta^2 + (x-x_c)^2)} \right] \quad (2.10)
\]

\[
G(x) = \sqrt{\frac{4\ln 2}{\pi} \frac{1}{\beta^2} e^{-\frac{(4\ln 2)(x-x_c)^2}{\beta^2}}} \]

\[
f = \text{Lorentzian shape function}
\]

\[
\beta = \text{Integral width}
\]

\[
x_c = \text{Peak position}
\]

The instrumental and thermal effects have not been considered in the analysis because these are negligible when compared to the defect broadening in GaN. In general, threading dislocations (TDs) observed in GaN are formed due to the large lattice and thermal mismatch between GaN and Sapphire. In GaN samples there are three major type of threading dislocations observed with dislocation line vector along [0001] direction: (i) Pure edge, Burger vector \( b = 1/3[1120] \), (ii) pure screw, Burger vector \( b = [0001] \), and (iii) Mixed type Burger vector \( b = [1210] \). The pure edge TDs give tilt while the pure screw TDs give twist to the grains as proposed in the mosaic model for x-ray analysis of the GaN. Extensive methods are available for quantitative measurement of screw and edge dislocations in GaN using HRXRD and described in detail elsewhere. [19,20]

Broadening in the rocking curves (angular scans or \( \omega \) scans) of the symmetry reflection are only due to the tilt and small correlation lengths parallel to the substrate surface.
Wafer bending or heterogeneous strain does not influence the peak broadening because of the small detector acceptance angle. Broadening in the radial scans ($\omega-2\theta$ scans) of the symmetry reflections are due to the small correlation length normal to the substrate surface and heterogeneous strain along c-axis. The effect of small correlation length and strain broadening can be separated by pseudo Voigt function fit using the different nature of broadening. Correlation length ($L_\perp$) and perpendicular strain ($\varepsilon_\perp$) can be calculated from the value of integral width (FWHM or $\beta$) and Lorentzian function ($f$) by using the formulas given below. [21]

$$L_\perp = \frac{(0.9\lambda)}{[\beta(0.017475+1.500484f-0.534156f^2)\cos\theta]}$$

$$\varepsilon_\perp = \frac{\left(\beta\left[0.1846+0.872692(1-0.998497f)^{1/2}-0.659603f+0.445542f^2\right]\right)}{(4\tan\theta)}$$

(2.11)

Here we have analyzed FWHM, peak shape in the log plots, Lorentzian and Gaussian broadening of x-ray peaks of our samples before and after irradiation.

### 2.3.3 Optical characterization

(i) Photoluminescence (PL)

Photoluminescence (PL) investigation was carried out using an experimental set-up properly dedicated to the study of the NIR PL activity characterizing the Er ions. The excitations giving rise to PL are obtained by means of a cw Ar laser, whose pumping lines at 488nm (maximum laser power: 27mW) and at 476.5nm (maximum laser power: 7.5mW) provide rare earth photo-stimulation. These two lines are used to excite $^4I_{15/2}$ ground state to $^4F_{7/2}$ excited level of Er ion, under resonance and out-of-resonance conditions (i.e. pumping radiation not directly absorbed by the rare earth). The laser beam has a spot diameter of ~1mm and it is modulated by a mechanical chopper (typically at a frequency of 9Hz). Laser line selection is obtained by suitable interferential filters. A
system of lens and mirrors gives the possibility to focus the laser spot with a well-defined incidence angle on the surface sample, as well as to collect and bring the emitted radiation on the entrance slit of a ORIEL single-grating monochromator. This provides the spectral discrimination of the luminescence signal, which is detected by a photomultiplier tube. For these experiments, we have used a HAMAMATSU N2-cooled R5509-72, operating in the 1000-1650nm spectral range. PL emission is acquired with a lock-in amplifier, using the chopper frequency as reference, while data are stored in a PC by dedicated software. Schematic diagram of the PL setup used in this study is given in Fig 2.11.

Time-resolved PL analysis is carried out by switching off the pump beam and by detecting at a fixed wavelength, the PL intensity as a function of time with a transient digitizer (overall time resolution of ~5ms). PL excitation (PLE) measurements have been performed using a 150mW Xe lamp coupled to a monochromator, operating in the 250-800nm range, as pumping source, while the detecting system is the same as described above.

Fig 2.11. Diagram of the PL setup used in this study
(ii) Optical Transmittance

The optical constants of the thin films were calculated using the envelope technique [22]. The spectral transmission characteristics in the wavelength range 190-1500 nm were measured using a JASCO V570 UV-VIS-NIR spectrophotometer.

Optical transmission and reflectance spectra are recorded from 190 nm to 2000 nm wavelength region, keeping standard samples in reference beam. Absorption coefficient is calculated using the relation.

\[ \alpha = \frac{1}{d} \log \left( \frac{1-R}{T} \right) \]  

(2.12)

Band gaps are calculated from \((\alpha E)^2\) vs E plot.

Refractive index is calculated using the relation:

\[ n = \sqrt{N + \sqrt{N^2 - S^2}} \]

where

\[ N = \frac{2 S (T_m - T_m)}{T_m T_m} + \frac{S^2 + 1}{2} \]  

(2.13)

2.3.4 AFM

The microstructures of the deposited films were obtained using AFM. The AFM consists of a microscale cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip of radius of curvature of the order of nanometers. When the tip is brought into the proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law. Depending on the situation, forces that are measured in AFM include mechanical contact force, Van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces, solvation forces etc. Typically, the deflection is measured using a laser spot reflected from the top of the cantilever into an array of photodiodes. The AFM can be operated in a number of modes, depending on
the application. The primary modes of operation are static (contact) mode and dynamic mode.

Fig 2.12. Schematic diagram of atomic force microscope.

In the static mode operation, the static tip deflection is used as a feedback signal. Because the measurement of a static signal is prone to noise and drift, low stiffness cantilevers are used to boost the deflection signal. However, close to the surface of the sample, attractive forces can be quite strong, causing the tip to 'snap-in' to the surface. Thus static mode AFM is almost always done in contact where the overall force is repulsive. Consequently, this technique is typically called 'contact mode'. In contact mode, the force between the tip and the surface is kept constant during the scanning by maintaining a constant deflection through feedback circuitry. In the dynamic mode, the cantilever is externally oscillated at or close to its resonance frequency. The oscillation amplitude, phase and resonance frequency are modified by tip-sample interaction forces. These changes in oscillation with respect to the external reference oscillation provide information about the sample's characteristics. Schemes for dynamic mode operation include frequency modulation and the more common amplitude modulation. In frequency modulation, changes in the oscillation frequency provide information about tip-sample interactions. Frequency can be measured with very high sensitivity and thus the frequency modulation mode allows for the use of very stiff cantilevers. In this work we have used the SPA400 of SII Inc, Japan AFM.
References