CHAPTER 2

Experimental Details: Sample Preparation and Measurements

2.1 Preamble

In this chapter we discuss the details regarding the sample preparation and various characterization techniques adopted to measure the electrical transport properties of the samples. We have performed resistivity, Hall and conductance noise measurements on n-GaAs samples. In order to have most reliable and unambiguous measurements of these properties of the sample, it is of utmost importance that we deposit high quality ohmic contacts on these samples. The methods to prepare ohmic contacts in n-Si and n-GaAs have been discussed. A significant amount of this work is devoted to studies performed on electrical transport properties of Schottky barrier diodes fabricated on n-GaAs and n-Si. The method used to fabricate these Schottky barrier diodes has been elaborated. The various measurement set ups used to characterize above-mentioned properties of the samples have been discussed in detail. The pertinent techniques and set ups which have been developed in house have also been highlighted. Since the major portion of this work is devoted to the study of modifications in transport properties of Si
and GaAs by swift heavy ion irradiation, the salient features of the irradiation facility have also been discussed.

2.2 Sample preparation

2.2.1 Fabrication of ohmic contacts

In order to perform resistivity, Hall and conductance noise measurements on n-GaAs and n-Si, it is highly desirable to deposit suitable ohmic contact metals/alloys over the top of the sample. An ohmic contact to a semiconductor must have the following properties:

- It must have low electric resistance, and its current-voltage characteristic should approximate a straight line going through the origin and extending over the entire range of voltages and currents to which the contact is subjected.
- It must have good mechanical properties. It must adhere firmly, both during formation and subsequent processing, and also in service.
- It must serve purely as a means for getting current into and out of the semiconductor, but play no part in the active processes occurring within the device.

For making ohmic contact on n-Si the aluminium (Al) metal is chosen [1-3]. Before depositing Al on n-Si, the sample is properly cleaned and etched. For removal of organic impurities from the surface of n-Si, it is first dipped in hot (about 70 °C) trichloroethylene (TCE) solvent for 10 minutes. It is then immersed in 1% Hydrofluoric (HF) acid for 1 minute in order to remove native oxide layers from the surface of n-Si. The sample is then rinsed in deionised water (resistivity 18 MΩ-cm) for 5 minutes. After that it is immediately transferred to a diffusion pump operated (having liquid nitrogen trap) high vacuum deposition chamber. The Al is deposited over the top of n-Si by thermal evaporation method. The base pressure of the chamber is about 1×10^-6 mbar. During evaporation the thickness of the Al is monitored using quartz crystal monitor. The photograph of the high vacuum deposition chamber is displayed in Figure (2.1). The typical thickness of the Al contacts is 100 nm. For resistivity, Hall and conductance
Figure 2.1 Photograph of the high vacuum deposition system.
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Noise measurements the four Al contacts are deposited at the corners of the n-Si sample in van der Pauw geometry using a metallic mask. After deposition the sample is taken out of the deposition chamber and put inside a furnace. It is annealed at 525 °C for 25 minutes in argon gas atmosphere for proper alloying of Al with n-Si.

For fabricating ohmic contact on n-GaAs, the alloy used is Au:Ge (88:12). The n-GaAs sample is also cleaned and etched properly before deposition is carried out. The n-GaAs sample is dipped in trichloroethylene, acetone and methanol for 10 minutes in each solvent. This causes the removal of organic impurities from the surface of the sample. Then the sample is dipped in 10% hydrochloric (HCl) acid solution for 1 minute to remove the native oxide layer from the surface. After that it is rinsed with deionised water for 5 minutes. The deposition of Au:Ge (88:12) contacts over top surface of the sample is carried out by thermal evaporation method in a cryo-pump operated very high vacuum chamber having a base pressure of 1×10⁻⁸ mbar. The contacts are deposited at the corners of the sample in van der Pauw geometry using a metallic mask. After deposition of the contacts, the sample is transferred to a furnace and annealed at 430 °C for 5 minutes in argon gas ambient. The ohmic contacts are tested by current-voltage (I-V) measurements and they exhibit good linear behavior and low contact resistance.

2.2.2 Set up for high temperature annealing in inert gas atmosphere for fabrication of ohmic contacts in semiconductors

A set up for annealing the semiconductor samples at high temperatures has been made [4]. For this purpose, a Thermolyne furnace (model 47900) is utilized. This furnace consists of (i) a heating chamber and (ii) a temperature controller. The furnace chamber is heated by two open coil electric resistance heaters and is insulated with ceramic fibre insulation. The temperature controller is located under the furnace chamber. The furnace can provide the temperatures up to 1100 °C and it has temperature stability of ±1 °C. The furnace has a front door from where the samples can be introduced inside it. On the top of the furnace there is a port of 26 mm diameter. An adapter has been
designed and fabricated which fits into this port. Two pipes pass through this adapter one of which serves as the inlet for inert gas (longer one) and the other one as the outlet (shorter one). The length of the longer pipe has been adjusted such that it reaches two to three centimeters above the samples that are kept inside the furnace. This produces flowing inert gas environment in the vicinity of the samples. The outer ends of each pipe are having swage lock ferrule connectors attached to them for gas entry and exit without any leak. The teflon tubes are connected to these ends. One of the teflon tubes from the gas inlet pipe goes to the argon cylinder from where the inert gas can be passed in a controlled manner (regulated by a two stage regulator) inside the furnace. The other teflon tube serves the purpose of throwing out the inert gas to the outside atmosphere. The set up is shown diagrammatically in Figure (2.2). The ohmic contacts on n-Si and n-GaAs have been fabricated utilizing this set up.

![Diagram](image)

**Figure 2.2** Schematic diagram of the set up for high temperature annealing in inert gas atmosphere.
2.2.3 Fabrication of Schottky barrier diodes

When a metal makes intimate contact with a semiconductor, a barrier is formed at the metal-semiconductor interface. This kind of barrier is rectifying in nature and is called Schottky barrier. If we deposit an ohmic contact, which is non-rectifying in nature, on the same semiconductor sample then this complete system is known as Schottky barrier diode. In the present work, several types of Schottky barrier diodes have been fabricated like Ag/n-Si, Au/n-Si, Pt/n-Si, Au/n-GaAs and Ni/n-GaAs. Details of the methodology adopted to fabricate Schottky barrier diodes on n-Si and n-GaAs has been described.

2.2.3.1 Schottky barrier diodes on n-Si

To fabricate a Schottky barrier diode on n-Si wafer, first a back ohmic contact of Al is deposited using the method described in Sub-section 2.2.1. The Al film for ohmic contact is having a thickness of 100 nm. After making the back ohmic contact, the wafer is cleaned again in hot TCE for 10 minutes. Then it is dipped in 1% HF for 1 minute and rinsed in deionised water for 5 minutes. It is then immediately transferred to the high vacuum evaporation chamber. The required metal is deposited on the front polished surface using a metallic mask. The metal contacts deposited are usually 2 mm in diameter. The evaporation of Schottky metal is done in the cryo-pump operated high vacuum chamber. The base pressure during evaporation is $1 \times 10^{-8}$ mbar. For Ag and Au deposition on n-Si, we use thermal evaporation method, while for Pt deposition we use electron-beam evaporation method due to its high melting point. During evaporation the thickness of the metal is monitored by quartz crystal based thickness monitor and the thickness can be controlled within 0.1 Å. The perspective view of a Schottky barrier diode is shown in Figure (2.3 a). The photograph of the cryo-pump operated chamber used for Schottky contact deposition is shown in Figure (2.4).

2.2.3.2 Schottky barrier diodes on n-GaAs

For n-GaAs, two types of Schottky barrier diodes have been fabricated: one on n-GaAs wafers and another on n-GaAs/SI-GaAs epitaxial layers. For diodes fabricated on n-GaAs wafer, first Au:Ge (88:12) back ohmic contact is deposited. Then the sample is
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Figure 2.3 Perspective view of Schottky barrier diode fabricated on (a) n-Si or n-GaAs wafer, and (b) n-GaAs/Si-GaAs epitaxial layer.
Figure 2.4 Photograph of the cryo-pump operated vacuum deposition system.
cleaned by dipping it sequentially in electronic grade TCE, acetone and methanol for 10 minutes in each solvent. After that it is immersed in 10 % HCl for 1 minute for native oxide layer removal. Then the sample is immediately transferred to cryo-pump operated high vacuum chamber where a 2 mm diameter metal contact is deposited on the front polished surface by thermal evaporation method. The base pressure of the chamber is $1 \times 10^{-8}$ mbar. The thickness of the deposited metal is 100 nm and is controlled in the same manner as mentioned earlier. For depositing Schottky contacts on n-GaAs/SI-GaAs epitaxial layers, first a 2 mm diameter Au:Ge (88:12) ohmic contact is deposited on the front polished side. After that the sample goes through usual cleaning and etching procedures. Then the sample is loaded in the high vacuum chamber and a 2 mm diameter metal contact is deposited on the front polished side. The centre-to-centre distance between the ohmic contact and Schottky contact is about 4 mm. The perspective view of this type of Schottky barrier diode is shown in Figure (2.3 b).

### 2.3 Resistivity and Hall measurements

#### 2.3.1 van der Pauw four-point probe method

The resistivity, Hall and conductance or $1/f$ noise measurements have been performed using van der Pauw four-point probe method [3]. In this method two probes carry the current and the other two probes are used for sensing the voltage. The advantage of using four-point probe method is that the effect of contact resistance existing at metal probe/semiconductor contact is eliminated. The four-point probe method was originally proposed by Wenner [5] in 1916 to measure the earth's resistivity. It was used for semiconductor wafer resistivity measurements by Valdes [6] in 1954. In collinear four-point probe method the probes are arranged in-line with equal probe spacing. But for irregularly shaped samples we use van der Pauw method where the probes are located at the corners of the sample. The theoretical foundation of measurements on irregularly shaped samples is based on conformal mapping developed by van der Pauw [7,8]. According to this method the resistivity of a flat sample of arbitrary shape can be
measured without knowing the current pattern, if the following conditions are satisfied:
(i) the contacts are at the circumference of the sample, (ii) the contacts are negligibly small, (iii) the sample is uniformly thick, and (iv) the surface of the sample is singly connected, i.e., the sample does not contain any isolated holes.

Let us consider a flat sample of a conducting material having arbitrary shape, with contacts 1, 2, 3 and 4 along the periphery as depicted in Figure (2.5). According to van der Pauw method the resistivity of the sample is given by

\[ \rho = \frac{\pi}{\ln(2)} \frac{(R_{12,34} + R_{23,41})}{2} F \]

where \( R_{12,34} = V_{34}/I_{12} \). The current \( I_{12} \) enters the sample through contact 1 and leaves through contact 2, and \( V_{34} = V_3 - V_4 \) is the voltage difference between the contacts 3 and 4. \( R_{23,41} \) is also defined in the same manner. The factor \( F \) represents the asymmetry of the contacts and is a function of the ratio \( R_r = R_{12,34}/R_{23,41} \). It satisfies the relation

\[ \frac{R_r - 1}{R_r + 1} = \frac{F}{\ln(2)} \arccosh\left(\frac{\exp[\ln(2)/F]}{2}\right) \]

The dependence of \( F \) on \( R_r \) as calculated from Equation (2.2) is shown in Fig. (2.6). For a symmetrical sample such as a circle or a square, \( R_r = 1 \) and \( F = 1 \).

In case of van der Pauw Hall measurements, the Hall coefficient is given by

\[ R_H = \frac{t \Delta V_{24}}{2BI_{13}} \]

where \( \Delta V_{24} = V_{24}(\text{for } +B) - V_{24}(\text{for } -B) \) with \( I_{13} \) flowing into terminal 1 and out of terminal 3 as shown in Figure (2.5 b). Here \( B \) is the magnetic field. The carrier concentration, \( n \), is calculated from the Hall coefficient as

\[ n = \frac{r}{qR_H} \]

Here \( q \) is the elementary electronic charge and \( r \) is the Hall scattering factor, defined by

\[ r = \frac{<\tau^2>}{<\tau>^2} \]
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Figure 2.5 van der Pauw four-point probe method for measuring (a) resistivity, and (b) Hall coefficient.

Fig 2.6 The van der Pauw correction factor, $F$, as a function of $R_r$. 
Here $\tau$ is the mean time between collisions for the carriers. The scattering factor depends on the scattering mechanisms in the semiconductor and generally lies between 1 and 2. For lattice scattering, $r=3\pi/8=1.18$, for ionized impurity scattering $r=315\pi/512=1.93$, and for neutral impurity scattering $r=1$ [3].

The Hall mobility, $\mu_H$, is given as

$$\mu_H = \frac{|R_H|}{\rho} = |R_H|\sigma$$  \hspace{1cm} (2.6)

The Hall mobility is not the same as conductivity mobility. They are related to each other as

$$\mu_H = r\mu_n, \hspace{1cm} \mu_H = r\mu_p$$  \hspace{1cm} (2.7)

for extrinsic n- and p-type semiconductors.

2.3.2 Experimental set up for resistivity measurements

For measuring resistivity of semiconductor samples, a liquid nitrogen (LN2) dipstick cryostat has been used. In this cryostat one can perform variable temperature resistivity measurements in the temperature range of 80 K to 400 K. The schematic diagram of the cryostat has been shown in Figure (2.7). The sample is placed over thin and circular copper plate. A calibrated PT100 temperature sensor is placed near to the sample on the same copper plate. A 25 $\Omega$ heater is wound at the base of the copper plate. Whole of this sample holder is enclosed by removable brass cup. When this dipstick cryostat is inserted inside the LN2 dewar, this cup kind of enclosure is kept under rough vacuum using a rotary pump (see Figure (2.7)). There are ten electrical connections: four for sample, four for temperature sensor, and two for heater. Two shielded coaxial cables (LEMO cables) serve as the electrical connections for the voltage probes of the sample. These electrical connections travel through the thin SS tube that is about 1 meter in length. The four connections of the sample end as four female BNC connectors, while six other connections terminate in a special D-type male connector. The dc current
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Figure 2.7 Schematic diagram of the cryostat used for resistivity measurements.

through the two probes of the sample is sent using Keithley’s programmable current source (model 220), and the voltage across the other two probes is measured using Keithley’s programmable digital multi-meter (DMM) (model 2001). The temperature of the sample is controlled using a Lakeshore temperature controller (model DRC-93CA). Using this temperature controller the temperature of the sample can be stabilized within 10 mK. This set up is computer controlled with all the instruments interfaced to PC using standard IEEE interfacing. The interfacing program has been written using TESTPOINT software.
2.3.3 Experimental set up for Hall measurements

For performing Hall measurements, a variable temperature Hall cryostat has been fabricated. A schematic diagram of the Hall cryostat is shown in Figure (2.8). The LN$_2$ is poured in a thin SS tube which terminates as a perforated copper block. This whole structure is surrounded by a vacuum shroud in which rough vacuum can be created using a rotary pump. A copper assembly is welded at the end of the copper block. This copper assembly is having a narrow circular neck and then a flat plate type structure which serves as mounting place for the sample. The narrow neck serves as a weak link between copper block and sample holder. A 50 $\Omega$ heater wire is wound around the neck for controlling the temperature of the sample holder. A PT100 temperature sensor is also mounted on the sample holder using GE varnish. In the present set up the temperature of the sample can be varied from 100 K to 300 K. The sample can be introduced in a magnetic field which is perpendicular to the sample plane. The magnetic field of the electromagnet can be varied up to 0.4 T with a pole gap of 40 mm. Arrangement for taking electrical connections from inside the vacuum shroud to outside has been made using a ten pin electrical feed-through. The dc current across two probes is sent using Keithley’s current source and the voltage is measured using Keithley’s digital multi-meter (DMM).

2.4 Conductance or $1/f$ noise measurement set up

The conductance or $1/f$ noise in a sample arises due to the fluctuations in conductivity of the sample. The noise power density of these fluctuations ($S_{\nu}$) varies inversely with respect to frequency ($f$), and hence it is termed as $1/f$ noise. The details about its nature and its relation to various microscopic properties of the material will be discussed in Chapter 3. Here we describe the experimental set up developed and utilized for measuring the conductance or $1/f$ noise in semiconductor samples. The schematic diagram of the experimental set up is shown in Figure (2.9). The conductance noise is measured using standard four-point probe method. The sample is loaded in the cryostat
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Figure 2.8 Schematic diagram of the variable temperature Hall cryostat.
that is shown in Figure (2.7). A dc current is passed through the two probes of the sample using battery-operated dc current source (consisting of a 9 V battery and a variable resistance, \( R_B \)). The voltage developed across the other two probes is fed to a low noise preamplifier (model SR552, Stanford, USA). The preamplifier blocks the dc component of voltage and allows only the ac components to pass through it. The gain of the preamplifier is 100. The amplified ac signal is then fed to a Hewlett Packard’s FFT spectrum analyzer (model HP35665A). The spectrum analyzer displays the spectral power density of voltage fluctuations \( (S_V) \) as a function of frequency \( (f) \). For determining the \( 1/f \) noise with frequency, we take the noise spectra with and without (consisting of preamplifier+thermal noise) current. Then the latter spectrum is subtracted from the former, and we obtain pure \( 1/f \) noise spectrum that is due to conductivity fluctuations inside the sample. The voltage noise sensitivity of the present set up is about \( 10^{-18} \) \( V^2/Hz \). In order to study the temperature variation of noise, the sample is loaded in the cryostat that is shown in Figure (2.7). The temperature is varied between 80 K and 300 K using Lakeshore’s temperature controller. Before taking noise reading at any temperature value, the temperature is stabilized within 10 mK.
2.5 Current-voltage (I-V) and capacitance-voltage (C-V) measurement set up

The current-voltage (I-V) and capacitance-voltage (C-V) measurement set up for doing electrical characterization of Schottky barrier diodes has been tested and installed. The Schottky barrier diode is mounted in the cryostat that is shown in Figure (2.7). For doing I-V measurement, we use Keithley's programmable voltage source (model 230) and picoammeter (model 486). The set up is fully automated with voltage source and picoammeter being interfaced to the computer using standard IEEE interfacing. The interfacing program is written using TESTPOINT software. The temperature dependent I-V characteristics can be recorded by varying the temperature of the SBD mounted in the cryostat using Lakeshore temperature controller.

The C-V measurements are done using Boonton's capacitance-meter (model 7200). The capacitance-meter uses a test signal of 1 MHz having levels of 15, 30, 50, or 100 mV. The maximum measurement range of this instrument is 2000 pF. The whole set up is also fully automated with capacitance-meter being interfaced to computer using standard IEEE interfacing. The interfacing program is written using TESTPOINT software. Here also we can do temperature dependent C-V measurements from 80 K to 300 K in the same way as in case of I-V measurements.

2.6 Pelletron accelerator

The 15 UD 16-MV Pelletron accelerator at Nuclear Science Centre (NSC) [9] belongs to a class of particle accelerators known as tandem Van de Graff accelerator. It is capable of accelerating almost any ion beam from hydrogen to uranium to energies from a few MeV to hundreds of MeV. A schematic diagram depicting the basic principle of acceleration is shown in Figure (2.10). The negative ions are produced and pre-accelerated to about 300 keV by a cesium sputter ion source known as SNICS (Source of Negative Ions by Cesium Sputtering). The ions are mass analyzed by a dipole magnet called injection magnet and are turned in vertically downward direction. The ions then enter the strong electric field inside the accelerator. At the centre of the accelerator tank
there is a terminal shell, which is maintained at high voltage (up to 16 MV). The tank is 26.5 meter long and 5.5 meter in diameter and is filled up with sulphur hexafluroide (SF$_6$) gas at a pressure of about 6-7 bar. The high voltage terminal is about 1.52 meter in diameter and 3.81 meter in height. The negative ions travel through the accelerating tube from the top of the tank to the positive terminal and get accelerated. On reaching the terminal, they pass through a stripper (foil or gas stripper) that strips the ions off their electrons, thus changing them to positive ions.

Figure 2.10 Schematic diagram showing the basic principle of acceleration in pelletron accelerator.
These positive ions are now repelled from the terminal and thus again get accelerated while travelling to the bottom of the accelerator. Thus the same terminal potential is used twice to accelerate the ions. Hence this accelerator is called a tandem Pelletron accelerator. The final energy of the ions emerging from the accelerator is given by

\[ E_f = [E_{decpot} + (1 + q_i)V] \]

(2.8)

where \( E_i \) is the energy of the ion having a charge state \( q_i \) after stripping, \( V \) is the terminal potential in MV and \( E_{decpot} \) is the deck potential of the SNICS source. The ions are now turned in horizontal direction when they enter in a dipole magnet known as analyzer magnet. This magnet works as an energy analyzer and allows the ions with a particular energy (depending upon the dipole magnetic field) to travel in horizontal direction. Then a switching dipole magnet is used to divert the high energy ion beam to the desired experimental station. The whole beam line of the accelerator is in ultra-high vacuum (UHV) condition (vacuum \( \sim 10^{-10} \) mbar). While travelling through accelerator beamline, the ion beam is kept centered and focussed using steering magnets and quadrupole triplet magnets. The beam is visually monitored by beam profile monitors (BPM), and the beam current is measured by means of Faraday cups.

2.7 Materials science beam line

The multi-port switching magnet can direct the ion beam in any of the seven beam lines in the beam hall. Among them one is materials science beam line, which is at 15° to the right with respect to the zero degree beam line. This beam line houses three chambers in succession. First chamber is high vacuum chamber where most of the irradiation experiments are performed. The second chamber is a UHV chamber and it incorporates facilities like \textit{in situ} UHV scanning tunneling microscopy (STM) and residual gas analyzer (RGA). The third chamber is again a high vacuum chamber and it contains \textit{in situ} X-ray reflectivity (XRR) and channeling facilities. A schematic diagram of the materials science beam line depicting the associated facilities is shown in Figure (2.11).
The high vacuum chamber is a cylindrical shaped multiport stainless steel chamber. A view of the high vacuum chamber is shown in Figure (2.12). The vacuum in the chamber can be created either by using a diffusion pump or a cryopump. The typical vacuum during the irradiation experiment is $1 \times 10^{-6}$ mbar. The samples are mounted on a target ladder. It comprises of a thin walled stainless steel tube to the end of which a perforated square copper block is brazed. The samples can be mounted on the four sides of this copper block. In general five samples having dimensions of $1 \times 1$ cm$^2$ can be mounted on each side of the copper block. The target ladder is suspended through a Wilson seal from the top flange of the chamber. This top flange is connected to the chamber through a flexible bellow that can expand up to 11 cm from its minimum position. A stepper motor in conjunction with suitable mechanical assembly is used to control the up and down motion of the ladder. This up and down motion can also be done from the remote data acquisition room using an electronic control system. The sample position can be aligned with respect to the ion beam by first looking at the luminescence of the beam on the quartz crystal, and after that the sample is brought to the position of the quartz. The positions of the quartz and samples are monitored using close circuit television (CCTV) kept in the data acquisition room. The sample temperature can be brought down to LN$_2$ temperature by pouring LN$_2$ from top of the SS tube of target ladder. There is a provision in the ladder for doing variable temperature irradiation studies also. For that there is a copper strip which is mounted on the copper block using four brass screws. A 25 $\Omega$ heater wire is wound at the bottom of the copper strip and a PT100 sensor is mounted in the middle of the strip. With the help of this arrangement the temperature of the strip can be varied from 80 K to 300 K using Lakeshore’s temperature controller. At a time four samples can be mounted on the strip. Three UHV compatible, 10-pin electrical feed-throughs (from MDC) are mounted on the flange of the target ladder. The thin enameled copper wires from the inside of electrical feed-throughs are guided along the SS tube and near the copper block terminate on copper PCB. The electrical connections for the samples for performing in situ measurements can be taken from this PCB. The connections from the outside of
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Figure 2.11 Schematic diagram of the materials science beam line.

Figure 2.12 Photograph of the high vacuum chamber in materials science beam line.
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Electrical feed-throughs are LEMO cables terminating as BNC connectors. The samples mounted on the target ladder are separated from each other by a distance of about 1 cm. The sample to be irradiated is aligned with the ion beam direction. It is magnetically scanned and falls over an area of 1 cm × 1 cm over the sample. The scanning ensures the uniformity of irradiation over the whole area of the sample. A cylindrical SS enclosure surrounds the sample holder and is kept at a negative potential of 120 V. This serves to suppress the secondary electrons coming out of the sample during irradiation. The suppressor has an opening that allows the ion beam to fall on the sample. The electric charge is collected by the target ladder, which is connected electrically to a current integrator (model DANPHYSIK 555) to measure the irradiation fluence. A CCD camera is attached to one of the ports of the chamber and is used for viewing the sample position.

The various in situ techniques developed and installed in high vacuum chamber for electrical characterization of semiconductor materials are described below.

2.7.1 In situ resistivity and conductance noise measurement facility

We have carried out in situ resistivity and conductance noise measurements on MOCVD grown n-GaAs epitaxial layers during irradiation. For performing these measurements the samples are mounted on the copper block of the target ladder. The ohmic contacts are deposited on the top of the sample in van der Pauw geometry. The electrical connections are taken using thin enameled copper wires (SWG 40) from the top of the sample. Thick silver paste blobs are placed over the contacts lest they are degraded during irradiation. The other ends of the wires are soldered to the PCB mounted on the ladder. The corresponding connections come out of the chamber through electrical feed-throughs. For each sample, two probes are connected to the Keithley’s dc current source and the other two probes are given to the input of Keithley’s digital multimeter. First a predetermined fluence is given to the sample and resistivity is measured as described in Section 2.3. In the same way we keep on exposing the sample for different fluences and at each fluence the resistivity is measured. For measuring the
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conductance noise of the sample, it is irradiated with different fluences and after each fluence the spectral power density of noise is recorded using HP’s digital spectrum analyzer. The other details about measurement of conductance noise have already been exemplified in Section 2.4.

2.7.2 In situ Hall measurement set up

The experimental arrangement for performing in situ Hall measurements in the high vacuum (HV) chamber of the materials science beam line has been fabricated and tested. The low temperature target ladder (described in Section 2.7) of the HV chamber was utilized for this purpose. A copper strip was attached below the copper block of the target ladder and the sample was mounted on this copper strip. A Co-Sm permanent magnet, having a uniform magnetic field of 0.2 T, was placed at the bottom of the HV chamber. The resistivity and Hall measurements are performed using van der Pauw technique. The sample is irradiated by swift heavy ions and resistivity of the sample is measured at various fluences. For this, the current through the two contacts on the sample is passed using Keithley’s current source and the voltage across the other two contacts is measured using Keithley’s DMM. Then the sample is introduced inside the uniform magnetic field of permanent magnet and Hall voltage is measured. The carrier concentration and mobility are calculated from resistivity and Hall voltage values using Equations (2.1)-(2.6). The sample is again taken out of the magnetic field and irradiated with next fluence value. Likewise the resistivity and Hall voltage is measured at various fluences. It is to be mentioned here that the temperature of the copper strip can be varied from 100 K to 300 K. For this a manganin wire heater (25 Ω) is wound at the base of the strip. The temperature of the strip can be controlled at any value using Lakeshore temperature controller. Thus in situ Hall measurements can be performed at all the temperatures ranging from 100 to 300 K.

2.7.3 In situ Hall measurement facility in GPSC beam line

Hall measurement facility has been developed and installed in the General Purpose Scattering Chamber (GPSC) beam line for semiconductor characterization [9]. This
beam line is at 45° with respect to the zero degree beam line. Using this facility one can study the mobility and type of charge carriers, their concentration and conductivity type transition in semiconductors. A variable temperature cryostat has been installed in the GPSC beam line to conduct the Hall effect measurements. The variable temperature cryostat is mounted on a specially designed stand and is connected to the GPSC beam line through a vacuum bellow. It is vacuum isolated from the GPSC chamber through a manually operated gate valve. An electromagnet, which can produce a field of 0.40 Tesla at a pole gap of 40 mm is positioned on the stand in such a way that the lower portion of cryostat comes exactly between the pole pieces. Figure (2.13) shows the schematic diagram of the Hall measurement set up installed in the GPSC beam line.

![Schematic diagram of in situ Hall measurement set up.](image)

The sample is mounted on the specially designed copper sample holder in the cryostat. The sample holder has a space of 1.5 cm x 11 cm for mounting the samples and quartz. Electrical connections to the samples are provided by two ten-pin electrical feed-throughs. The provision of twenty electrical connections enables mounting of four
samples at a time for the Hall effect studies. A platinum resistance thermometer (PT100) is used to monitor the temperature. The cryostat design is such that the sample ladder can be moved downward/upward (up to 15 cm) and rotated (upto 360°) to bring the sample exactly between the pole pieces and apply a perpendicular magnetic field which enables one to measure the Hall voltage. In the absence of magnetic field the resistivity of the sample can be measured by passing a constant current and measuring the voltage developed across the voltage contacts.

Rotary pump is used to create rough vacuum inside the cryostat. After rough vacuum creation the gate valve is opened and a vacuum of $2 \times 10^{-6}$ Torr is achieved in the cryostat. Firstly, the beam is adjusted on the quartz crystal and scanned over the desired area for irradiation. After this the sample is irradiated with the appropriate fluence. Then the sample is moved downward to bring it exactly at the center of the pole pieces of the electromagnet and is rotated by 90° for Hall measurements. The resistivity measurements are also made at each fluence in the absence of magnetic field using the van der Pauw method. Then a magnetic field of 0.40 Tesla is applied perpendicular to the sample surface and resultant voltage is measured. After the Hall voltage measurement, the sample is again rotated back by 90° and moved upward to bring it again at the position where further irradiation can be made. After each irradiation with the desired fluence the same procedure is repeated to perform the Hall voltage and resistivity measurements. From the Hall voltage and resistivity data the mobility and carrier concentration are determined.

**2.7.4 In situ I-V and C-V measurement set up**

A facility for performing in situ I-V and C-V measurements is developed in high vacuum (HV) chamber to carry out electrical characterization of Schottky barrier diodes during swift heavy ion irradiation. The electrical connections from Schottky contact and ohmic contact sides are taken using thin enameled copper wires (SWG 45). The ends of the wires are placed over these contacts using colloidal silver paste. For the Schottky contact proper care is taken so that the silver paste covers only a small portion (less than
5%) of the contact area. The Schottky contact is circular in size with a diameter of 2 mm. The electrical connections are taken out of the HV chamber through a G10 board attached to one port of the chamber and it contains ten female LEMO connectors from where the connections can be taken outside. In this way the complete length of the electrical cables from sample to measuring instrument is about 2 meters. This reduces the cable capacitance effect markedly during C-V measurements. The instruments for doing I-V and C-V measurements are kept near the chamber itself. For I-V measurements Keithley’s voltage source and picoammeter are used while for C-V measurements Hewlett Packard’s precision LCR meter (model HP 4284 A) is used. Using this LCR meter the C-V measurements can be taken at various frequencies ranging from 20 Hz to 1 MHz. The C-V measurements at low and high frequencies are used to calculate interface state density at the metal-semiconductor interface as will be described in detail in Chapter 4. This instrument is also interfaced to computer using IEEE interfacing. The program is written in TESTPOINT software. For doing measurements during irradiation, first the Schottky barrier diode is irradiated with a fixed fluence. Then its I-V characteristic is recorded. After that the connections are switched over to LCR meter and C-V characteristics are recorded at various frequencies. This procedure is repeated for various fluences during irradiation.
References


