Chapter 6

Experimental methods and characterization techniques

This chapter deals with the physical route to fabricate the nanopatterned substrate and the basic principle of the experimental techniques used to characterize the ion beam induced morphological, microstructural and compositional modifications of the surfaces.
6.1 Introduction

The experimental works presented in this thesis are based on ion beam nanopatterning which have been performed on commercially available epi-polished Si(100), Ge(100) and GaAs(100) crystal wafers using low energy \( \text{Ar}^+ \) ion sputtering as function of different ion processing parameters. The ion irradiation has been carried out in a low energy ion beam system (M/s Roth & Rau Microsystems GmbH, Germany). The details of the system are extensively discussed in Sec. 6.2 which deal with (i) the design of ion beam equipment and (ii) the working principle of the ion source employed for production and extraction of the ion beam during experiments. The next section is about the characterization techniques which are mainly focused on imaging of ion beam modified surface morphologies. Atomic force microscopy (AFM) has been used as main tool of imaging. In this context, a short discussion on some statistical quantities, extracted from the characterization data (AFM), relevant to this thesis work will be given. The microstructural details and the modification of crystalline structures have been carried out by Transmission electron microscopy (TEM) and the study of the chemical composition of nanostructures have been studied by Energy dispersive X-ray spectrometry (EDS) in TEM.

6.2 Low energy Ion Beam System

6.2.1 Design and facilities of low energy Ion Beam System

In this thesis work, the experiments have been carried out in a high vacuum, broad beam, high current and low energy ion beam system (M/s Roth & Rau Microsystems GmbH, Germany) using ion irradiation on semiconductor surfaces. The photograph of the low Energy ion beam system, installed in SINP, Kolkata, is presented by Fig. 6.1(a). The whole system is operated by computer interfacing through a system
6.2. Low energy Ion Beam System

software IonSys500 [Fig. 6.1(b)]. A schematic layout of the main components of the equipment is shown in Fig. 6.2(a). The whole system consists of mainly two parts (a) Pumping system; and (b) Sputtering chamber where the second one is comprised of (1) discharge chamber; (2) RF ion source; (3) grids; (4) neutralizer; (5) substrate holder; (6) substrate heater, and (7) Faraday cup.

Fig. 6.2(b) & (c) represents the photograph of the sputtering chamber, substrate holder and the temperature rising heaters, whereas Fig. 6.2(d) displays the assembly of the outlet of grid aperture, PBN neutralizer and Faraday cup. The base pressure inside the chamber remains of the order of $10^{-8}$ mbar. Typically, a broad ion beam of diameter $\sim 4$ cm is extracted from an inductively coupled RF discharge ion source equipped with three graphite grid ion optical system (M/s Roth Rau Microsystems GmbH, Germany). During extraction of ion beam, the chamber pressure arises to $10^{-4}$ mbar. The extracted beam has to travel an approximate distance of 33 cm to reach the sample surface. A plasma bridge neutralizer (PBN) is employed to avoid the blowing up of the beam and also for charge neutralization of sample surface. In a PBN, a hot filament is placed in a smaller discharge chamber through which an inert gas (Ar) is supplied. The hot filament emits electrons directed towards the ion
beam. Inside of the sputtering chamber, the substrate holder can be tilted from 0° to 90° with respect to ion beam direction, as well as, it can revolve around its axis up to the speed of 30 rpm. The substrate holder can be cooled by cooling water or optionally by cryo cooler. For later case, the temperature during ion irradiation can be controlled within the range -20°C to 60°C. The optional cryo cooling of the wafer chuck and a He backside heat contact allow a safe processing of temperature sensible wafers. The substrate temperature can also be raised up to 500°C by using a radiation type heater placed at top of the chamber. The ion energy can be varied from 0.3 to 2 keV whereas ion current density can be varied up to 10 mA/cm² with focusing grid system. The ion current density was measured by a Faraday cup.
6.2.2 RF ion source: Details and working principle

Basically, the production of ion beam in present sputtering system needs three key parts: discharge chamber, radio frequency (RF) ion source and grids. Initially, the source gas (here Argon) is introduced into the discharge chamber which is generally made of dielectric material for RF ion sources. Now, the free electrons in the working gas are energized by RF field and these energetic electrons make ionizing collision with other neutral gas atoms. As a result, a large number of ions and additional electrons are produced and hence establish a plasma. Then, grids are used to extract ions from plasma in the form of a beam. The grids are basically electrodes separated from each other by a few mm and has several circular apertures (∼0.5 mm). The closest grid of the discharge chamber and the discharge chamber itself are biased to positive potential with respect to ground. This grid energizes the ions and screened the electrons, thus it’s named as a screen (S) grid. Basically, the positive potential applied to S grid is the beam voltage. The next grid is called accelerating (A) grid as it is biased negatively (accelerating voltage) with respect to ground and this helps to accelerate the ions towards the target. The third one is held at ground potential (0 V) and referred to as the decelerator (or D) grid. Therefore, after passing grid A, the accelerated ions can decelerate while flows through grid D as no positive bias is present there and it exit the grid apertures with ion energy equal to beam voltage. The broad beam results as a combination of individual beamlets corresponding to the individual grid aperture. A schematic of potential diagram across different parts of ion sputtering system during the ion extraction process is represented by Fig. 6.2(e). The ion energy and ion current density can be varied by changing beam voltage and accelerating voltage respectively.
• **Sample preparation and different experimental conditions:**

Commercially available mirror polished Si(100), Ge(100) and GaAs(100) crystal wafers of approximate size 1 cm × 1 cm are cleaned with acetone followed by methanol in an ultrasonic bath for 10 min. Afterwards, the clean and dried samples were irradiated by Ar\(^+\) ion beam for different sputtering conditions. The detail of the sputtering conditions will be stated in their corresponding sections of the experimental results.

### 6.3 Characterization Techniques

#### 6.3.1 Atomic Force Microscopy

Fundamentally, atomic force microscopy or AFM is one member in the family of scanning probe microscopy (SPM) techniques which has grown steadily after the invention of Scanning Tunnelling Microscopy (STM) in early 1980’s by Binnig and Rohrer\(^{145}\) for which they received nobel prize in 1986. In SPM methods, a sharp probe (typically called a tip) is scanned over the surface within a close proximity to monitor the tip-sample interaction. Interactions can be of different types depending on the mechanics of probe, types of sample surfaces and the finite effective range of the interaction. In case of STM, the tunneling current between the metallic tip and conducting surface is the source of interaction between them which is itself a major limitation of this method for the case of insulated surfaces. This limitation led to the invention atomic force microscope in 1986 by Binnig *et al.*\(^{146}\). AFM generates 3D images by scanning the contour of the investigated surface. The image is created by quantifying the interacting forces between the tip and the sample. The advantage of AFM is its ability to produce high resolution images (almost at the level of atomic scale) of the surfaces ranging from conductive to non-conductive, biological to fluid etc.
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- Basic principle of AFM operation:

Figure 6.3: (a) Schematic diagram of the working principle of the AFM head. (b) SEM image of triangular SPM cantilever with probe (tip). (c) A Plot of force as a function of probe-sample separation indicating the different AFM mode region.

The working principle of an AFM is represented by a schematic in Fig. 6.3(a). The central component of an AFM is a cantilever of the order of several microns in length. The tip is supported on an end of the flexible cantilever which has a low spring constant that helps to bend it by very small forces. The magnified image of a cantilever with a tip is shown in Fig. 6.3(b). Usually, the tip and the cantilever is made of silicon(Si) or silicon nitride (Si₃N₄). The tip radius is typically of the order of few tens of nanometer. During scanning, the cantilever progressively moves backward and forward across the surface. Simultaneously, a piezo-electric crystal raises or lowers the cantilever to keep a constant bending of the same. This bending
results due to the force experienced by the tip from the sample. The amount of force between probe and sample depends on the cantilever’s spring constant and the distance between probe and sample surface. According to Hooke’s Law \([147]\), this force can be described by \( F = -kx \), where \( F \) is the force, \( k \) is the spring constant and \( x \) is the cantilever deflection. The cantilever deflection is measured by a “beam bounce” method. A laser beam gets constantly reflected from the top of the cantilever and falls on a position-sensitive photo-detector. This detector measures the bending of the cantilever by calculating the deflection of laser beam from it and generates a 3D map of the surface topography. The force exerted on tip due to sample surface are of different types, viz., van der Waals forces, capillary and adhesive forces and double layer forces. Among these, van der Waals force dominates over others due to short distance between probe and sample. This force can be derived from the Lennard-Jones potential \([148]\) and is given by:

\[
V_{LJ} = \varsigma \left[ \left( \frac{r_m}{r} \right)^{12} - 2 \left( \frac{r_m}{r} \right)^{6} \right]
\]

(6.1)

where, \( \varsigma \) denotes the depth of the potential well and \( r \) denotes the tip and sample distance. The potential will be minimum at a distance \( r_m \) from the surface. The variation of the tip-sample interaction force \( F(r) \) with the distance \( r \) is shown in Fig. 6.3(c). Depending on the region of operation of the tip on this \( F(r) \) vs. \( r \) curve, AFM can be distinguished in different operating modes:

- **Operating modes of AFM:**

  1. Contact AFM = <0.5 nm probe-surface separation.

  2. Tapping mode AFM = 0.5-2 nm probe-surface separation.

  3. Non-contact AFM = 0.1-10 nm probe-surface separation.
Contact mode AFM

In the so-called contact mode of AFM, the tip makes soft ‘physical contact’ with the surface of the sample. When the tip is in range of few angstroms (Å) from the sample surface, it experiences repulsive van der Waals force. In this mode, the tip scans by keeping a constant distance from surface (using the feedback loop) and the variations in tip-sample interaction force reflect the atomic scale topography of the surface. The variation in tip-sample interaction force reflects the atomic scale topography of the surface. This mode is good for rough solid samples and useful for fast scanning required for real-time imaging of surfaces. The disadvantage associated with the mode is it may damage soft samples as the tip remains in very close proximity to the sample surface.

Tapping mode AFM

This mode was developed to achieve high resolution images of sample surface without inducing frictional forces between tip and the sample, so it is known as non-destructive mode of AFM. Here, the tip oscillates up and down at or slightly below its resonance frequency while scanning the surface. The oscillating tip gently taps the sample surface during scanning. When the tip approaches close to the sample surface, various forces like van der Waals forces, dipole-dipole interactions, electrostatic forces, etc., act on the cantilever and this cause a decrease in the amplitude of the oscillation. A feedback positioning unit maintains the constant separation between tip and sample. Thus, the image is obtained by imaging the force of the oscillating contacts of the cantilever tip with the sample surface. This method allows imaging of soft and delicate biological samples but encounters difficulties for liquid samples.
Non-contact mode AFM

Generally, Non-contact mode AFM is used to image the fluid sample surface. In this mode, the tip operates in the non-repulsive region of the $F(r)$ vs. $r$ curve [Fig. 6.3(c)]. During scanning, the probe does not contact the sample surface, but oscillates above the adsorbed fluid layer on the surface at a frequency larger than its resonance frequency. This results in increased sensitivity in comparison with tapping mode AFM, but the resolution of images seems to be poorer. This significant drawback can be removed in ultra high vacuum (UHV), where Non-contact mode AFM gives the best imaging of the sample surface.

- Details of AFM used in present experiments:

The measurements for present thesis work, have been performed using a Multimode scanning probe microscope (SPM) with a Nanoscope IV controller from M/S Veeco Instruments, Inc., USA. It is named as “multimode” because it offers various modes of microscopy, for example: AFM in contact, tapping and non-contact mode, Magnetic Force Microscopy (MFM), Electric Force Microscopy (EFM), Lateral Force Microscopy (LFM), Scanning Tunneling Microscopy (STM) etc. Fig. 6.4 shows the photograph of

![Image](image_url)

Figure 6.4: (a) Photo of the SPM multimode atomic force microscope used in this thesis work installed at SINP, kolkata. (b) A magnified picture of the multimode microscope.
the SPM instrument used in this work. All the AFM measurements of this work are carried out in tapping mode and in air. Images were usually captured with a resolution of 512×512 pixels. This multimode SPM comprises of three primary components: a microscope, a nanoscope controller and a computer with two monitors.

The microscope

![Schematic of the Nanoscope IV multimode scanning probe microscope.](image)

Figure 6.5: *Schematic of the Nanoscope IV multimode scanning probe microscope.*

The microscope is the heart of the SPM and is shown by Fig. 6.5. It contains a multimode head, multimode base and a removable piezoelectric scanner. The head contains the probe, laser and the position sensitive photodiode array. The probe consists of a tip placed at the end of a cantilever. In present experiments, etched silicon Probes of model RTESP7 by Veeco Instruments, Inc., USA are used whose
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radius of curvature is less than 10 nm. These tips are made of Phosphorus (n) doped Si and its front, back and side angles are $15^\circ$, $25^\circ$ and $17.5^\circ$ respectively. With the help of a cantilever holder, the probe is placed inside of the AFM head. The multimode head is positioned above the top of the piezo tube where the sample is placed on. A zoomed view of SPM head is presented by Fig. 6.6(a). The head is attached with a X-Y stage which is used for horizontal movement of the cantilever to locate it at desired position on the sample. Both of the head and the X-Y stage are kinematically mated to the piezo scanner which controls the raster scanning motion of the cantilever. A pair of springs are present at the head to secure it from accidental falling and allows the sample to raised and lowered using the Z translation switch which is located in the multimode base. The base also has a mode selector switch and a digital display. Another X-Y stage is attached with the base and is used to control the position of the video optical microscope. This helps the user to locate the tip at a preferred position on sample. The base display shows a SUM signal which is basically the information related with the photodiode. Fig. 6.6(b) shows the photodiode arrangement in the AFM head. This has four quadrants and the combined signal from all four quadrants results in SUM signal. The differential signal between the top two quadrants (A, B) and bottom two quadrants (C, D) provides a measure of the vertical deflection of the

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cantilever. To get a clear AFM image, the microscope is mounted on an anti-vibration table to suppress the vibrational noise which may arise due to mechanical activities in the environment.

**Nanoscope controller**

This unit provides interfacing among the computer, the scanning system and the probe motion sensor. It supplies the voltage which controls the piezoelectric scanner, accepts the signal from the probe motion sensor and controls the feedback electronics to keep the constant force between the tip and the sample surface.

**Some limitations of AFM:**

In-spite of high resolution 3-dimensional topography imaging on a wide range of material surface (i.e., plastic, metals, semiconductors and soft biological samples, tissues, proteins etc.), there are also some difficulties while scanning with AFM. During tip-sample interaction the tip may get blunt. This points the increase in radius of curvature of the tip which does not reflect the real topography of the examined surface. This is called tip-artifacts. So, special attention was paid during the measurements. For each case, the data have been collected from different position of many samples treated in the same way and also different tips are used for one measurement. This helps to get better statistical results.

- **Some statistical analysis of AFM image:**

One of the most important statistical quantity used to extract from AFM images is the fluctuation of the surface heights around an average height, known as root mean
square (rms) roughness and is given by:

\[ W = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (h(x, y) - \bar{h})^2} \]  

(6.2)

where, \( h(x, y) \) denotes the surface height at point \((x, y)\) and \( \bar{h} \) gives the mean of the surface heights at different points. But, rms roughness cannot extract all the information from sample surface as it deals with the height fluctuations in vertical direction only [13, 152]. The positional correlations between different points which give the surface quantities like feature’s wavelength, homogeneity and lateral correlation length remain unidentified. This can be attained by transforming the height profile in the reciprocal space or Fourier space. The 2D FFT (Fast Fourier Transform) for a square image of dimension \( L \) is given by:

\[ \text{FFT}(k_x, k_y) = \frac{1}{N^2} \sum_{x=1}^{N} \sum_{y=1}^{N} h(x, y)e^{-i2\pi L/N(xk_x+yk_y)} \]  

(6.3)

where, \( N \) is the number of points in the image size \( L \times L \). \( k_x \) and \( k_y \) are the spatial frequency coordinates along \( x \)-axis and \( y \)-axis respectively. The spots in FFT image describe the preferred orientation of structures on surface, whereas its position determines the characteristic spatial frequency i.e., wavelength of the structures. The width of the spot gives the homogeneity and the spatial correlation among the periodicities on the surface. The presence of additional spots in FFT, multiple of the first one, denote the higher lateral ordering of the structures. Higher number of spots indicate the better lateral ordering. However, for better quantitative estimation of these quantities, the two dimensional \( PSD(f_x, f_y) \) function [152, 153] can be considered. A more practical way is to deduce angular averaged \( PSD(f) \) which has the
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form [137]:

\[ \text{PSD}(f) = \frac{1}{N_f} \sum_{n=0}^{N_f} 2D - \text{PSD} = \frac{1}{N_f} \sum_{n=0}^{N_f} (\text{FFT}(k_x, k_y))^2 \]  

(6.4)

where \( N_f \) corresponds to the number of points at constant distance \( f \) which depends on \( L \) and \( N \) where \( (1/N) < f < (N/2L) \) [154]. The first peak position of in PSD gives the wavelength of structures. The lateral ordering of structures can be quantified by estimating lateral correlation length (\( \xi \)) which can be extracted from the FWHM of the first order peak by the relation \( \xi \sim 1/(\text{FWHM}) \). Lateral correlation length scales up to where the spatial correlations are present within the surface.

6.3.2 Transmission Electron Microscopy

A Transmission Electron Microscope or TEM is a powerful equipment that forms detailed images, called “micrographs”, of microscopic objects by passing a beam of electrons through a ultra thin slice of the area of interest [155]. It’s basic principle is same as the light microscope (LM), but TEM uses electron beam to produce images of the object and the magnification is obtained by electromagnetic fields, whereas in light microscope these two purposes are served using light and optical lenses respectively. As electrons have much lower wavelength (0.05 Å) than visible light (0.55 µm), it is possible to get an atomic resolution (10 Å) in TEM which is a thousand times better than a light microscope (0.2 µm).

The configuration of a transmission electron microscope (TEM) is shown schematically in Fig. 6.7. A TEM consists of four major parts: electron source or gun, electromagnetic lens system, sample holder and imaging system. The electron source consists of a cathode and an anode. Generally, a filament of LaB₆ crystal or tungsten acts as cathode which emits electrons when being heated and anode is biased with positive voltage to accelerate the electrons towards the specimen. In modern TEM,
the accelerating potential up to 200-300 kV is possible. After leaving the electron source, the electrons are tightly focused almost in a parallel beam by a condenser which is basically a pair of electromagnetic lenses. These are advantageous due to their ability to change its strength by adjusting the current through it rather than changing the distance between sample and lens. The first lens determines the spot size with which the beam strikes the sample and the second one changes the size of this spot on the sample. Beneath them, a condenser aperture is placed. It is a thin disk or strip of metal with a small circular aperture through it. The aperture size can be adjusted in order to restrict the scattered electrons to fall on the sample which travel at relatively larger angles with respect to the optic axis. The beam from the

Figure 6.7: A schematic diagram of the transmission electron microscope.
condenser aperture strikes the sample and some part of it is transmitted depending upon the thickness and electron transparency of the specimen. The sample is placed at object plane of the objective lens of TEM. The main function of the objective lens is to focus the transmitted electron from the sample into an image. The image lies in the image plane of the objective lens, called the first image plane. Objective lens is the most important one because if there is an error remains during it’s focusing adjustment then it will be magnified by all other lenses afterwards. So, it is necessary to align this lens correctly. The objective lens is immediately followed by the objective aperture (optional). The aperture is placed at the back focal plane of the objective lens for the purpose to block the high angle diffracted electrons and hence to enhance the contrast of the image. An electron diffraction pattern of the investigated sample area, illuminated by incident parallel electron beam, is formed at the focal plane of the objective lens (called back focal plane). The electron diffraction pattern formed at the back focal plane of the objective lens or the image formed at the image plane of the objective lens, finally fall on phosphor screen or charge coupled device (CCD) camera and can be magnified on the viewing screen by the first and second intermediate lens and the projector lens assembly.

There are two modes of performing basic operation of TEM, the diffraction pattern projection and the image projection. In diffraction mode operation, to observe the diffraction pattern on screen, the object plane of the first intermediate lens is set to the back focal plane of the objective lens as diffraction pattern is positioned there. This was discussed in earlier para. The diffraction pattern shows a bright central spot along the optic axis and few other spots due to the diffracted electrons. An aperture of controllable size (called the selected area aperture) is placed at the first image plane to select a particular area from the sample image formed at the first image plane. Now, this selected area acts as a virtual source for the intermediate lens assembly and the electron diffraction from this selected area can be observed in the
viewing screen and is known as selected area diffraction imaging. In imaging mode operation of TEM, the imaging is performed by choosing a spot from the electron diffraction pattern. This can be performed with the help of objective aperture. For sample imaging, the first intermediate lens is adjusted in such a way that the first image plane becomes its object plane. If, one selects the central beam on the optic axis for imaging by the objective aperture then the TEM image is called a bright field image and if any other diffraction spot is chosen, then the image is called a dark field image.

For clear view of image on screen, the sample should be electron transparent that means the sample thickness should be so thin that a major portion of electron beam can pass through the specimen. The penetration power of electron beam through the specimen of any thickness depend upon the energy of the incident beam and on the elemental composition of material. In general, it is necessary to thin a sample below 100 nm for observation in TEM and below 50 nm for high resolution TEM (HRTEM). When electron beam strikes the sample, the ion-sample interaction can produce backscattered or elastically scattered electrons, can also produce inelastically scattered electrons, X-rays, Auger electrons or light etc. The electrons which are transmitted through the sample can be classified into three main categories, namely, unscattered electrons (transmitted beam), elastically scattered electrons (diffracted beam) and inelastically scattered electrons.

Unscattered electrons are basically those incident electrons which exit the sample surface without interaction. The amount of transmitted electrons (unscattered) is inversely proportional to the thickness of the sample and thus, determine the different thickness of sample by the image contrast: the thicker parts of the sample appear darker (For thick sample, few number of electrons can be transmitted and vice versa) while the thinner parts of the sample appear lighter. This simple mass-thickness contrast is exhibited by all specimens, whether amorphous or crystalline.
On the other hand, the elastically scattered electrons are produced when the incident electrons interact with the sample atoms with no loss of energy. These electrons are diffracted by atoms and produce the diffraction patterns which follows the Bragg’s law of diffraction. These diffraction patterns give the atomic spacing between two crystal planes.

In this thesis work, the TEM and HRTEM (High-Resolution Transmission Electron Microscopy), both have been carried out using FEI, Tecnai G² F30, S-Twin microscope operating at 300 kV equipped with a GATAN Orius SC1000B CCD camera. The TEM is equipped with scanning unit and a high-angle annular dark field (HAADF) detector from Fischione (Model 3000).

6.3.3 Energy dispersive X-ray spectrometry (EDS)

Energy dispersive X-ray spectrometry (EDS), sometimes called energy dispersive X-ray analysis, is an analytical technique used for the elemental analysis or chemical characterization of a sample. It makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons. When the electron hit the specimen, it may interact elastically or inelastically with the target atom. In case of inelastic interaction, two basic types of X-rays are produced: Characteristic X-rays and Continuum (Bremsstrahlung) X-rays.

Characteristic X-rays result in the following way: The electron beam knock out an inner shell electron from the specimen atom. As a consequence, a hole is generated there and the atom goes into an excited state. The excited atom then de-excites through a relaxation process which consists of a series of transformations in each of which the vacant orbit is subsequently filled by an electron “jump” from an outer shell. Each such ‘jump’ corresponds to an amount of energy loss specified by the energy difference between the outer-shell electron and the inner-shell electron. For transition of electron from the higher shell, the energy is released in form of X-rays.
The energy of the X-ray gives the characteristic of the specimen atomic number from which it is emitted and thus, called characteristic X-rays. These are named after the two electron shells which are involved in emission of X-rays. The shell with initial vacancy determines the first letter and the second shell, from which an electron jumps to fill that initial vacancy, decides the second letter. For instance, if the initial vacancy occurs in the K shell and the electron drops from the L shell to K shell for filling up the vacancy, the emitted radiation is called a Kα X-ray. If, the electron drops from M shell to K shell and from N shell to K shell, then the emitted X-rays are called Kβ and Kγ respectively. The energy dispersive X-ray microanalyses are usually deal with K, L and M series X-rays.

Continuum (Bremsstrahlung) X-ray results when the beam of electrons interact with the nucleus of the specimen atoms and due to coulomb field between atomic nucleus and electrons, the electrons decelerate and they lose their energy in form of X-rays. The more closer approach of the electron to the nucleus makes the interaction more strong between them. Hence, the electron loses more energy and, consequently, more energetic X-ray photon is emitted. In this case, the primary electrons may lose any amount of energy during interaction, thus the energy distribution of emitted X-ray is continuous and not signifies the characteristic of the specimen atomic number.

The excited atom can also de-excite through a non-radiative transition. In this case, the filling of an inner-shell vacancy of the atom is accompanied by the transition of electron from outer shell. The difference in the corresponding energy loss due to transition is utilized to knock out another electron from the same outer shell or another outer shell rather than emitting the X-rays. This process of electron emission is called Auger electron emission.

In this thesis work, the study on the compositional details of Ar⁺ ion sputtered semiconductor surfaces, Si, Ge and GaAs, have been performed in an energy dispersive spectrometer attached to the TEM (FEI, TECNAI G² F30, S-TWIN microscope).
6.4 Chapter Summary

In this chapter, we have discussed about the instruments used in the experimental work of this thesis. The modification of the sample surfaces have been performed by the broad ion beam in a low energy ion beam equipment. The design and the specifications of the low energy ion beam equipment are discussed in detail. This includes RF ion source, three-grid ion-optics system, the methods of ion beam production and extraction etc. We also give a detail view on characterization techniques used to examine the ion beam modified surfaces. Since, Atomic force microscopy (AFM) is used extensively as a major characterization tool for this thesis work, so it has been discussed in great detail. For microstructural and compositional studies, we have used TEM and EDS respectively which are also elaborately described in later sections.