2.1 Introduction

This chapter explains the details of the various experimental techniques used in the present thesis. It includes techniques for sample preparation, treatment and characterization. The thin film samples were prepared using a radio frequency (RF) magnetron sputtering unit. The as-prepared as well as the flash annealed samples were characterized using various techniques which includes grazing incidence X-ray diffraction (GIXRD), secondary ion mass spectrometry (SIMS), Rutherford backscattering spectrometry (RBS), scanning electron microscopy (SEM) and atomic force microscopy (AFM). The thin film samples were flash annealed in a rapid thermal processor (RTP).

2.2 Thin film sample preparation

The thin films samples were prepared using radio frequency (RF) magnetron sputtering unit, a variant of physical vapour deposition (PVD) technique outlined in chapter 1. This deposition technique makes use of the sputtering process wherein target atoms are ejected from the surface of the target upon energetic particle bombardment. The bombardment set off a collision cascade in the target and sputter-
Fig. 2.1 A schematic diagram showing the working of a RF magnetron sputtering.

-ting results from the backward recoils that reach the surface with energies above the average surface binding energy. All the variant of sputter deposition processes relies on sustained glow discharge plasma created by breakdown of a gas/gaseous mixture at low pressure by the application of an electric field. The discharge in a RF magnetron sputter deposition process is a capacitive discharge as can be seen in Fig. 2.1, where the target (cathode/power electrode) is coupled to the RF generator that produces a RF field at 13.56 MHz frequency through a blocking capacitor. The anode (substrate) is usually grounded. A consequence of this capacitive coupling is the development of self biasing of the target that is negative with respect to the plasma potential necessary to make the average charge current over one RF cycle zero. This provides an effective negative DC voltage offset that is needed to sputter the target (irrespective of its
electrical nature) by the $\text{Ar}^+$ ions. Magnetrons are permanent magnet add-ons so configured with the cathode surface to trap electrons near the cathode and maximize sputtering efficiency by using magnetic and electric fields. It can be used with DC, pulsed DC and RF sputter source. Thus RF magnetron sputtering is a universal technique capable of depositing both conducting as well as non-conducting thin films. Fine details about sputtering and all the variants of sputtering deposition are available in the handbook of thin film deposition by R.F. Bunshah [1]. Details about the deposition unit and the parameters used in depositing the samples for the present thesis work are given below.

The deposition chamber was evacuated to a base pressure of $\sim 5 \times 10^{-6}$ mbar using a combination of rotary and turbo-molecular pump. The sample stage is equipped with heater controlled by a proportional integral derivative (PID) controller and it can heat samples/substrates up to 800 °C. The heater and the thermocouple assembly are hermetically packed in a vacuum compatible metal casing and aluminium foils are wrapped upon the thermocouple leads. These arrangements lead to formation of Faraday’s cages which act as an effective shield against RF interferences. The distance between the substrate and the target $d$ is $\sim 6$ cm. The working pressure $p$ in the chamber is about $1.5 - 4 \times 10^{-3}$ mbar and hence, the $p \times d \sim 0.6$ to 2.4 Pa – cm. The target is 99.9% pure Mo target and sputtering proceeds through plasma created by argon gas feed. The substrates used were $n$-Si (100) and $p$-Si (111) single crystal wafers. The wafers were cleaned by a series of alternating steps involving ultra-sonication and dip rinsing in organic solvents (acetone and ethanol) and deionised water. The silicon substrates were finally dried using hot air blower (from the backside) before they were loaded into the deposition chamber. Initial optimization of the deposition parameters, especially, the sputter power was
performed using the surface roughness \((rms\) roughness) of the as-deposited films as a parameter by depositing films at different sputter powers ranging from 70 W to 200 W. Based on the value of \(rms\) roughness measured by AFM, sputter power of 130 W was chosen for the deposition. The thicknesses of the films were measured by masking a portion of the film and doing a post deposition stylus measurement. Molybdenum thin films with desired thickness were deposited for a particular set of deposition parameters by using the calibrated deposition rate with an error of \(\sim 5\%\). Different sets of films were prepared with different deposition parameters. Different set of Mo thick films were deposited using different deposition parameters for various investigations which makes up the present thesis. 43 nm thin Mo films were deposited on Si substrate to study interface mixing upon \(Ar^+\) ion irradiation and subsequent formation of technologically important molybdenum disilicide (MoSi\(_2\)). Thicker Mo (~ 160 – 200 nm) thin films with varying microstructures were also deposited on Si substrate using different substrate/deposition temperatures (RT to 800 °C) to study issues related to interfacial characterization and diffusion with SIMS. Samples were also prepared to study the effect of deposition temperature on the residual stress in Mo thin films. These studies are discussed in details in later chapters.

2.3 Secondary ion mass spectrometry (SIMS)

SIMS is a versatile surface and near surface micro analytical technique that can perform elemental and isotopic depth profiles, in thin films and bulk materials with high depth resolution. This technique is capable of detecting trace levels of all the elements in the periodic table with varying degrees of sensitivity i.e., in the range of ppm - ppb \((10^{-6} - 10^{-9})\) atomic concentration [2]. The high sensitivity comes from the inherent capability towards trace analysis that is associated with a mass spectrometry technique. The schematic diagram of SIMS is shown in Fig. 2.2 below.
Fig. 2.2 A schematic diagram of a secondary ion mass spectrometer.

The basis of SIMS technique is the use of focused primary ion beam (few kV) as a microprobe to systematically sputter the sample’s surface to eject secondary ions which are then mass analyzed. The primary ion beam (\(O_2^+, O^-\) or \(Cs^+\)) bombardment induces a collision cascade and sputtering process results from the backward recoil. The \(Cs^+\) primary ions are produced by thermionic surface ionization of a Cs containing compound like \(Cs_2CO_3\), while \(O_2^+\) (and \(O^-\)) primary ions are produced by a
cold cathode plasma discharge in a duoplasmatron. The secondary species that are ejected during primary ion bombardment induced sputtering process includes electrons, photons, resputtered primary ions, neutral atoms, charged monatomic/polyatomic (molecule) or clusters with energy ranging from 0 to several hundred eV. A schematic representation of the primary ion interaction with the sample surface is shown in Fig. 2.3.

A fraction of the sputtered atomic species undergoes charge exchange in the near surface environment resulting in the formation of secondary ions, either
positively/negatively charged, which carries characteristics of the sample surface. The basic SIMS equation which relates the secondary ions intensity of species \( m \) in the layer with various fundamental parameters is given by

\[
I_s^m = I_p Y_m \alpha q_m \eta
\]  

(2.1)

where \( I_p, \ Y_m, \ \alpha, \ q_m, \ and \ \eta \) are the primary ion intensity, total sputter yield for species \( m \), ionization probability, fractional concentration of species \( m \) in the surface layer and the instruments transmission efficiency respectively. These secondary ions (most often positive than negative) are extracted by an electrical potential and subsequently analyzed in a mass spectrometer to give the composition of the sample. The ion detectors at the detecting ends include a Faraday cup, electron multiplier, micro-channel plate and resistive anode encoder. The SIMS uses a combination of these detectors to offer a high dynamic range in detection by covering a wide range of count rate from 10 to \( 5 \times 10^9 \) counts per second. Detailed information on the working principle of SIMS and its instrumentation are available in the book by A. Benninghoven [1].

There are several operating modes for carrying out analysis using SIMS:

1. Dynamic SIMS for compositional depth profiling
2. Static SIMS for surface analysis
3. Imaging SIMS
4. Mass spectrum
5. Bar graph
6. High resolution
7. Line scan
Chapter 2

Among the modes listed above, the first three modes of analysis are routinely used. Static SIMS uses low primary ion dose (below $1 \times 10^{12}$ ions/cm$^2$) for qualitative analysis of the top sub-monolayer of the sample to determine surface concentrations of elements and molecules without significantly altering the analyte. Spectrometer with time-of-flight detector is best suited for static SIMS. Spectrometer with magnetic sector like the CAMECA IMS 7F SIMS is more suited for dynamic SIMS analysis of thin films and bulk samples. With dynamic SIMS which involves use of much higher energy primary beam (large current), one can analyze trace impurities and variation in elemental composition with depth from the so called depth profiles [3]. Dynamic SIMS is well suited for studying solid state diffusion, interface mixing and impurities profiles in thin films/multi-layers structures. Imaging SIMS is yet another mode of SIMS. It provides spatially-resolved elemental mapping of the sample or analytes. Imaging can be performed either in scanning ion/ion micro-beam mode ‘or’ direct ion/ion microscope mode depending upon the image that is focused on the detector plane and the detector used.

SIMS measurement forms an important experimental part of this present thesis. All the SIMS measurement were carried out in a CAMECA IMS 7F SIMS with Cs$^+$ primary ions produced by thermionic surface ionization - Cs vapor emanating from a resistively heated reservoir ($\text{Cs}_2\text{CO}_3$) thermally ionizes to Cs$^+$ ions when it is passed through porous tungsten plate heated to 1100 °C. The interface mixing between Mo and Si in Mo/Si bilyaer thin films upon ion beam irradiation has been investigated with SIMS. The issues and artifacts related SIMS depth profiling of polycrystalline materials are investigated using Mo thin films with varied micro-structure. Impurity profiles in Mo thin films sputter deposited at $\sim 4 \times 10^{-3}$ mbar at various temperatures. Studies of homogeneity in ion beam mixed zone using SIMS
ion imaging with a resistive anode encoder and the power of the offline analysis software are also presented.

2.4 Rutherford backscattering spectroscopy

RBS is a nuclear analytical technique widely used for the elemental and compositional analysis of near surface layer of materials. Projectiles with energies between 100 keV to 4 MeV bombard a target and an energy sensitive detector records the energies of the backscattered ions. The scattering in RBS includes both elastic scattering and inelastic scattering and covers nuclear reaction regime as well. The projectile normally uses includes protons (H\(^+\)) or doubly ionized α- particles (He\(^{++}\)) as the Rutherford cross section are better known for these ions, stopping power extensively investigated and incur less damage among other ions of comparable energies. Sometime, Li\(^+\) ions are also used as projectiles.

![A schematic of the experimental set-up for Rutherford backscattering geometry (IBM).](image)

**Fig. 2.4** A schematic of the experimental set-up for Rutherford backscattering geometry (IBM).
A schematic of a typical RBS setup in the IBM geometry is shown in Fig 2.4. In IBM geometry, the incident beam, exit beam and surface normal are in the same plane and they satisfies the condition

$$\alpha + \beta + \theta = 180^\circ.$$  

The simplicity of two-body kinematics can be applied to the backscattering events (with appropriate energy losses consideration). The energies of the backscattered projectile (ions) incident with energy $E_0$ and having mass $M_1$ depend on the mass $M_2$ of the target atoms as

$$E_1 = KE_0$$  

where

$$K = \frac{M_1^2}{(M_1 + M_2)^2} \left\{ \cos \theta \pm \left[ \left( \frac{M_2}{M_1} \right)^2 - \sin^2 \theta \right]^{\frac{1}{2}} \right\}^2$$  

is called the kinematic factor, *defined as the ratio of the projectile’s energy after and before the collision.* Significant amount of momentum (energy) is transferred to the light target atom by the incident projectile. As the mass of the target atoms $M_2$ increases, much reduced momentum is transferred and the energy of the back-scattered ions asymptotically approaches the incident projectile energy. The energy separation is given by

$$\Delta E_1 = E_0 \frac{dK}{dM_2} \Delta M_2$$  

And hence RBS has good mass resolution for lighter elements, but good sensitivity for heavier elements in lighter target and vice versa.
The energy of backscattered ions (as well as yield) is also a function of the depth from which the ions are back-scattered. This occurs because of the energy losses suffered by the ions during inward and outward travels within the target. With the knowledge of the stopping power \( S \), defined as the energy loss (nuclear and electronic collisions) per by unit distance traversed, of the projectile in the material, the measured energy \( E_i \) can be converted into a depth scale \( x \) to give elemental depth profiles or thickness. At low energy (< 100 keV), the nuclear stopping due to collision with target nuclei is important, while electronic stopping gain importance with increasing energy. The stopping power is related to the scattering cross section \( \epsilon \) by the relation

\[
\epsilon = \frac{S}{N} = \frac{1}{N} \frac{dE}{dx}
\]  

(2.5)

where \( N \) is the density of the target material.

The energy loss by the ions in both inward and outward path is governed by the \( S \) or \( \epsilon \) which are characteristics of each material. For a compound consisting of different elements \( i \) with atomic concentrations \( c_i \), Bragg’s rule gives the total scattering cross section as

\[
\epsilon = \sum_i c_i \epsilon_i
\]  

(2.6)

where \( \epsilon_i \) is the scattering cross section of individual element. Fine details about the RBS technique and related mathematical analysis are available in many books [4-5].
A 1.7 MV Tandetron accelerator was used for performing the RBS experiments presented in this thesis. The schematic diagram of the accelerator is shown in Fig. 2.5 and the image of the 1.7 MV tandemron accelerator in Fig. 2.6. The accelerator works on the principle of tandem acceleration i.e. the ions are accelerated twice using the same high voltage applied at the middle of the accelerating tube. Negative ions generated by the source are initially accelerated by the high voltage terminal at the middle of the tube, where a stripper canal filled with nitrogen gas converted them into positive ions which are again accelerated by the same high voltage terminal towards ground potential. The maximum energy of positive ions transformed from negative ions into charge ‘q’ and accelerated by a terminal voltage ‘V’ has the energy given by

\[ E = V (1+q) \] (2.7)

The configuration has another advantage of having both the ion injection system and target at the ground potential. There are two ion sources for beam injection namely (i) a duoplasmatron ion source for production of H\(^+\) and He\(^+\) ions.
and (ii) a SNICS (Source of negative ions by cesium sputtering) sputter source capable of producing negative ions of almost all elements. The beam injection into the accelerator is facilitated by a 90° mass analyzing magnet (resolution ~ 190) for both ion sources. High voltage insulation of the entire accelerating structure is achieved by enclosing it with a pressure vessel filled with SF₆ gas at 6 kg/cm². An ultra high vacuum of 10⁻⁷ mbar is maintained in the accelerating tubes and a turbo-molecular pump has been installed at the high voltage terminal for recirculation of the stripper gas. The high voltage in the range 100 kV to 1.7 MV with a high stability of ± 100 V is generated using a Cockroft-Walton type solid state power supply. The accelerated ions are focused by an electrostatic quadruple lens before a high energy switching magnet analyzes the energy of the beam and switch it to the experimental ports.

**Fig. 2.6 The 1.7 MV tandetron accelerator.**
located at ± 10° and ± 30° angular positions. An implantation beam line with a beam sweep system, neutral trap, beam profile monitor and retractable slits for beam collimation has been installed at the 10° port of the switching magnet. An UHV compatible irradiation cum ion beam analysis chamber (Fig. 2.7) has been installed at the end of the beam line. The RBS measurements work presented in this thesis were performed in this chamber.

And the computer simulation program known as SIMNRA develop by M. Mayer [6] for analyzing RBS spectra was used to model the compositional layer of acquired RBS spectra for various samples.

Fig. 2.7 The ion beam analysis chamber.
2.5 Grazing incidence X-ray diffraction

X-ray diffraction experiments for fairly thick films and bulk samples are generally performed in the symmetric Bragg-Brentano (BB) diffraction geometry. The BB geometry is highly penetrating (10 – 100 μm) [7] and is most often not useful for analyzing thin to very thin films (nm range) because of the correspondingly low diffraction volume within the thin film that results in a very low peak-to-noise ratio and poor structural information about the film [8]. When an X-ray diffraction experiment is performed in an asymmetric geometry employing a low fixed grazing incidence, it is referred to as GIXRD. A schematic of grazing incidence X-ray diffraction (GIXRD) configuration is shown in Fig. 2.8.

Fig. 2.8 A schematic diagram of the experimental configuration for grazing incidence X-ray diffraction. $K_0$, $K$ and $Q$ are the incident wave vector, scattered wave vector and the scattering wave vector respectively.
As in the symmetric (BB) case, the configuration is still coplanar with wave vectors $K_o$, $K$ and $S_3$ all lying in the same plane. However unlike in BB geometry, the incident angle is kept fixed at a low value near critical angle and the detector scans the $2\theta$ circle. The crystalline planes scattering the X-rays are no longer the plane parallel to the sample surface, but planes perpendicular to the Q vector as shown in Fig. 2.8.

The advantage of using a low and fixed incidence angle is that it limits the penetration of the X-ray beam and maximizes the diffraction volume within the thin films by increasing the path length as well as foot prints of the X-ray beam within the thin film specimen as shown in Fig. 2.9. The path length of the X-ray beam within a film of thickness $t$ becomes $l = t / \sin \alpha$ which can be multiple times of the actual thickness of the film and the foot print (of the X-ray beam with width $d$) = $d / \sin \alpha$.

The penetration depth of X-ray in a material having a linear absorption coefficient $\mu$ is given by the equation

$$\tau = \frac{\sin \alpha \sin \beta}{\mu (\sin \alpha + \sin \beta)} \quad (2.8)$$

![Diagram of X-ray path length and foot print in GIXRD.](image)

**Fig. 2.9** X-ray path length and foot print in GIXRD.
where $\alpha$, $\beta$ are the incident exit angle of the X-ray respectively. The essence of this depth is that about 63 % of the diffracted beam originates from depth $\tau$ below the sample surface.

In GIXRD, the penetration depth varies strongly for values of $\alpha$ near the critical angle according to the above expression, but if $\alpha$ is not too close to the critical angle i.e., if $\theta_{hkl}$ is not in the vicinity of 0 or 90°, it can be approximated to the expression

$$\tau = \frac{\sin \alpha}{\mu}$$

(2.9)

A reliable choice of $\alpha$ is guided by the criteria that the average path length

$$l = \frac{1}{\mu}$$

(2.10)

The X-ray diffraction data are not just limited to structural information such as the crystalline phases and sizes. A well equipped lab source X-ray diffractometer can give information regarding the full stress tensor, strain and textures. Synchrotron X-ray sources can still give more information including that of amorphous materials. The XRD measurements presented in this thesis were performed in two diffractometer systems namely: - (a) Stoe GmbH diffractometer and (b) Bruker Discover D8 Diffractometer. The STOE diffractometer works with Cu k$_{\alpha}$ ($\lambda = 1.5406$ Å) produced by a tube source and experiments were performed in the parallel beam grazing incidence geometry with sample rotation. The phase characterization of the ion beam irradiated Mo/Si samples were performed in the Stoe diffractometer. The Bruker D8 diffractometer is a four circle diffractometer and is equipped with a 6 kW rated rotating Cu anode generator that produces pure Cu k$_{\alpha}$ ($\lambda = 1.5406$ Å) with intensity that
is much higher than the conventional tube source diffractometer. The rotating anode
generator is operated at 4.5 kW and 100 mA current in a vacuum of 1 x 10^{-7} mbar.
The Eulerian cradle (i.e., goniometer) in the D8 diffractometer allows the sample to
the tilted and rotated along any specific orientation using \( \chi \) and \( \varphi \) axis making it well
suited for residual stress and texture measurements. These are in addition to routine
diffraction investigation for structure analysis. Hence, the involved measurements for
stress analysis were performed in the Bruker diffractometer as specified in the
respective chapters that is to follow. The stress measurements were performed in the
asymmetric parallel beam geometry with a 0.7° angle of incidence following a
modified \( \sin^2 \Psi \) method of analysis. The \( \chi \) axis was used to vary the sample tilt angle
\( \Psi \). Measurements were also made with detector \((2\theta)\) scan in an extended range from
30° to 145° to record all measurable reflections. These extended 20 scans form the
basis of residual stress analysis using multiple \( hkl \) reflections.

2.6 Ion beam irradiation - 150 kV gaseous ion implanter

Materials undergo significant atomic rearrangement when irradiated with
energetic ions. The phenomenon is referred to as \textit{ion beam mixing} if the atomic
intermixing and alloying due to ion irradiation occurs at the interface separating two
materials eg.: the interface of binary or multi-layer thin film systems [9]. The ion
beam irradiation of the Mo/Si thin films in the present thesis were performed in J-15
gaseous ion accelerator facility. A schematic diagram illustrating the layout of the ion
irradiation facility is shown in Fig. 2.10 and the 150 kV accelerator used for the
purpose is shown in Fig. 2.11. The accelerator is an electrostatic accelerator and it
consists of a radio frequency (RF, 100 MHz) gaseous ion source. The ions are then
extracted from the source and accelerated along the accelerating tubes to the required
Fig. 2.10 A schematic diagram of the experimental set-up for J 15 150 kV gaseous ion accelerator.

Fig. 2.11 J 15 150 kV gaseous ion accelerator.
voltage in the range 10 – 150 kV. A Cockfoft – Walton voltage generator produce the required high power voltage (tuneable 10 – 150 kV). The ions are mass analyzed by a 45\(^\circ\) bending electromagnet with appropriate magnetic field. The accelerated mass analyzed ion beam is collimated and focused using an electrostatic quadropole lens, and allowed to impinge on the sample placed inside an irradiation chamber. The irradiation chamber as well as the accelerating tube are maintained at ultra high vacuum (~10\(^{-7}\) mbar) using a turbo-molecular pump. A liquid nitrogen trap is used for trapping organic contaminants to maintain oil free vacuum.

The beam current and ion dose (ions/cm\(^2\)) were measured using a current integrator. To facilitate beam current measurements, the sample stage is made out of a block of copper which is insulated from the rest of the chamber. A secondary electron trap (Faraday cup) is used to eliminate error in beam current measurement resulting from emission of secondary electrons form the target. A 40 V potential is used to suppress the secondary electrons from escaping the Faraday cup (thin metallic cylinder). A low beam current 0.5 \(\mu\)A was maintained during the irradiation to avoid the beam heating of the sample during irradiation. An X-Y beam rastering was used over an area of 1 cm\(^2\) for uniform irradiation of the sample. All the irradiations were carried out at room temperature. The gaseous ions used for the irradiation work of this thesis are Ar\(^+\) ions and the energy of the ions were 110 keV.

2.7 Scanning electron microscopy

The SEM is a microscope that uses a focused beam of electrons to scan the surface of a sample. The major advantages of SEM over ordinary microscope are the large depth of field and a much higher resolution (down to few nm) which provides a clear 3D image of the topographical features of the scanned sample surface. The
maximum magnification and hence resolution is decided by the minimum diameter of
the focused electron beam. Resolution down to 1 nm is achievable with SEM
equipped with field emission (FE) electron gun compared to ~ 10 nm that can be
achieved with normal SEM with thermionic electron source. The interaction of these
impinging electrons with the atoms of the sample surface produces various secondary
signals. These secondary species gives information on sample’s elemental
composition and topography based on whether characteristics X-rays or backscattered
and secondary electrons are detected. Detection of back-scattered electrons (BSE) can
tell about the distribution of mean atomic number of the sample as BSE signals are
atomic number dependent (bright regions associated with heavy elements).
Combining BSE with characteristic X-ray spectrometry, one can get elemental
distribution of different elements present in the sample. X-ray spectrometry alone can
perform elemental analysis of all elements down to atomic number 5 (boron) in a
scanned area few mm wide and deep. The most commonly detected signal is that of
secondary electrons emitted by surface atoms excited by the probing electron beam.
The number of secondary electrons is a function of angle between the probing beam
and the emitting surface, and hence it contains information about the topographical
features of the surface.

In the present thesis, a Gemini make FE-SEM was used for imaging the
microscopic or morphological features of the Mo films deposited at different
temperatures. Cross sectional scanning micrographs were acquired to study the micro-
structural evolution of Mo films.
2.8 Atomic force microscopy

Atomic force microscopy is one of the most commonly used scanning probe microscope for the visualization of surface micro-topographies ranging from atomic structures to features extending to few tens of micrometers. The AFM works for both conducting as well as non-conducting samples and in this regard has an edge over scanning tunnelling microscope which only works on conducting samples. The operating principle of an AFM is depicted in Fig. 2.12. It illustrates the working principle of the laser beam deflection method. The paths of the laser beam both ‘directed onto’ and ‘reflected from (as a result of change of angle)’ the cantilever head changes as the cantilever bends. This deviation is detected by the position sensitive photodiode detector. The cantilever bends because of the force between the tip and the sample. This force, $F$, can be calculated using the following equation

$$F = k s$$

(2.11)

where ‘s’ is the distance by which the cantilever bends and ‘$k$’ is its spring constant.

It can operate in several modes [10]:

(a) Contact mode

In this mode, the tip is in mechanical contact with the sample surface. Typical force exerted on the sample surface by the tip ranges from ~ 0.1 μN to 1μN. In this mode, the repulsive part of the Van der Waals force from the surface balances the force applied predominantly on the tip by the bending cantilever. This mode is well suited for acquiring well resolved micro-topography of hard crystalline sample and surfaces, but not suitable for soft surfaces because of the associated elastic and plastic deformation.
Fig. 2.12 A schematic illustration of the working principle of an atomic force microscope.

(b) Non-contact mode

In this mode, the cantilever oscillates close to its resonance frequency near the sample surface (5-10 nm) so that the force on the tip arises predominantly from the attractive part of the Van der Waals force field. The magnitude of force is very small ~ 1 pN. This mode is truly non-invasive and suitable for soft and biological samples. However, the resolution in non-contact mode is not as good as in contact mode.

(c) Tapping mode

In tapping mode, the cantilever oscillates at resonance frequency very close to the sample surface and the tip comes into contact with the sample’s surface
momentarily during each oscillation. Since it is a combination of both the contact and non-contact modes, it is sometime called semi-contact mode. The magnitude of vertical force on the tips in this mode remains same with that of contact mode; however with lesser interaction between the sample and the tip as a result of tapping, it enables one to scan soft surfaces with better resolution than non-contact mode. The lateral friction and drag forces present in the contact mode are also overcome in this mode.

In the thesis, AFM micro-topographic scans were exclusively performed in the tapping mode to study the surface topography of different as-deposited Mo films deposited under different deposition parameter. It was also used to study the topographic evolution of the SIMS crater in various Mo films. The acquired topographic micrographs were used to determine roughnesses of the surfaces. The roughness parameter used in the present thesis is the de facto standard root mean square (rms) roughness – the surface heights relative to least square fitted line profile and it is expressed as

\[
R_q = \sqrt{\frac{1}{L} \int_0^L Z^2(x)dx} = \sqrt{\frac{1}{L} \sum_{i=1}^{N_L} Z_i^2(x)}
\]  

(2.12)

2.9 Rapid thermal processor

Rapid thermal processor is an annealing set-up normally used in semiconductor device fabrication and microelectronics technology for flash heating of silicon wafers to high temperature (up to ~ 1200 °C). The heating duration is short ranging from few seconds to several minutes in contrast to conventional furnace annealing. Because of the rapid and short heating durations, it is used in many
applications like dopant activation annealing in silicon wafers, annealing out defects introduced by ion irradiation, contact alloying or metallization in integrated circuits, crystallization, rapid thermal oxidation, nitridation and thermal diffusion. In the present thesis, I have made use of a Jipelec JetFisrt 100C for flash annealing as-deposited and ion irradiated Mo/Si thin film samples. The main component of a RTP is the cold-wall reactor chamber and the heating equipment - an array of 12 tubular infrared lamps heating a 4 inch Si wafer on which samples are placed. The unit is equipped with both K-type thermocouple and pyrometer for temperature measurement and the control is through a PID temperature controller. The reactor chamber can be pumped down to $1 \times 10^{-3}$ mbar enabling vacuum annealing. There are two purge lines for performing annealing in different flowing gaseous ambient. The annealing cycle is executed through programmable logic controller (PLC) and PC interface with user defined recipes and parameters.

![Jipelec JetFirst 100C rapid thermal processor](image)

**Fig. 2.13** A Jipelec JetFirst 100C rapid thermal processor [11]
2.10 Summary

In this chapter, details of all the experimental techniques which include techniques for thin film deposition, treatment and characterization, used for carrying out the work of the present thesis were discussed. The techniques are RF magnetron sputtering (PVD), SIMS, RBS, GIXRD, SEM, AFM, RTP and ion beam irradiation facility. The accelerators used for RBS and ion irradiation are also discussed.
Chapter 2

References


