Chapter 1

1 Introduction

1.1 Ion beam interaction with surface

Applications of ion beam during different stages of fabrication of micro and macro devices are increasingly becoming popular[1]. Possibility of mask less fabrication process is one of the greatest advantage that ion beam offers as compared to other lithographic techniques. Apart from that, there are different ion beam processes that are useful for surface modification during device fabrication. Since these processes depend on energy, mass and direction of the ion and the properties of the target, the processes can be designed in such a way as to have an accurately controlled program of device fabrication. The most important physical effects of incident ions on the substrate are: sputtering of neutrals, (this effect enables substrate milling), electron emission and ionization of substrate atoms (this effect enables imaging, but causes charging of the sample), displacement of atoms in the solid (induced damage) and emission of phonons (heating). Another most important effect is chemical interactions of incident ions with the target where chemical bonds are broken and molecules are dissociated (this effect is exploited during deposition). A broad ion beam can cause any of these effects depending on the above mentioned parameters. But if the ion beam is focused to a fine size, then the effects occurred will be highly localized. Such a focused ion beam (FIB) becomes a powerful tool to cause any of these effects on a minutely small area. Improvement in the technology and performance of FIB has opened up new possibilities in the field of device manufacturing [2]. The four major FIB processes that can be used for modifying surfaces and its applications are explained below [2].
1.1.1 Milling

The removal of sample material is achieved by using a high ion current beam. The energy of the incoming ions will be deposited on the surface bound atoms and the result is a physical sputtering of sample material, as illustrated schematically in Figure 1-1 (a). This is a useful damage process that happens on the surface with fine control and precision. By scanning the beam over the substrate, any arbitrary shape can be etched. Efficiency of removal of target material is generally represented by sputtering yield which is the ratio of number of particle ejected from the surface to the number of incident ions. Typical sputtering yield varies depending on the materials, incident angle and energy. However, these numbers cannot be used directly to calculate the etch rate, because, etching (milling) rate depends on the current density, scanning style, re-deposition of sputtered material. Milling requires high beam current, of the order of 10s of nA and above.

1.1.2 Deposition

FIB enables the localized maskless deposition of both metallic and insulating materials. The principle is similar to chemical vapor deposition (CVD) but assisted by ion beam. The main difference is the better resolution but lower deposition rate of FIB. The deposition process is illustrated in Figure 1-1 (b); the precursor gases are sprayed on the surface by a fine nozzle, where they are adsorbed while the incident ion beam continuously decomposes the adsorbed precursor gases. Then the volatile reaction products desorb from the surface and are removed through the vacuum system, while the desired reaction products remain fixed on the surface as a thin film. The smallest features that could be deposited are of the order of 100nm (lateral dimension) and thickness is about 10nm. Aspect ratios between 5 and 10 are obtained, at a typical deposition rate of $0.05\mu m^3s^{-1}[3]$. Here the current needed is very small, of the order of few pA to few nA.

1.1.3 Implantation

Figure 1-1(c) sows the process of implantation which occurs at higher incident ion energy than for the process of milling where the Bragg peak happens at much deeper inside and thus
the incoming ion gets trapped inside. Here energy deposited by the ion on the surface is very less compared to the surface binding energy and thus the target atoms do not get sputtered away. Instead the material gets modified by the presence of deeply implanted incoming ions. This process is useful for doping, inducing defects, oxidation, nitriding etc.

1.1.4 Imaging

As illustrated in Figure 1-1(d), during FIB imaging, the finely focused ion beam is raster scanned over a substrate, and secondary particles such as neutral atoms, ions and electrons emitted from the sample. As they leave the sample, the electrons or ions are collected on a biased charge particle detector such as Micro Channel Plate detector. The detector bias is a positive or a negative voltage for collecting secondary electrons or secondary ions respectively. The secondary ions that are emitted can be used for secondary ion mass spectroscopy (SIMS) of the target material. By raster scanning the ion beam, the spatial distribution of various elements on the sample can be determined. By keeping the beam fixed on the sample, the distribution of elements along the depth also can be determined. During FIB operations, a large numbers of secondary electrons leave the sample and the secondary electron yield is a function of the material composition as well as topography of the surface. Using this material and the angular dependency, high resolution images of the surface can be easily obtained. Emission of electrons from surface may cause sample charging problem especially on insulating sample materials. To prevent positive surface charges from building up, the substrate can be flooded with electrons from a separate electron source. The system thus prevents damage due to electrostatic discharge and it enables imaging of non-conducting materials such as glass (which is often used in micro systems). Imaging requires very small ion current in order to minimize the damage to the surface.
1.2 A brief history and description of conventional FIB system

The idea of “manipulating atoms one by one, the way we want” was initially imagined and advocated by the great scientist R P Feynman while addressing the American Physical society in 1959 where he delivered the famous speech “There is plenty room at the bottom”[5]. His lecture was an invitation to a new field of physics and there he fantasized that “a source of ions, sent through the microscope lenses in reverse, could be focused to very small size”. Thereafter, the first successful focused ion beam (FIB) was developed by Levi-Setti in 1974 [6]. It was based on field emission technology and in 1975, Orloff and Swanson et al. developed FIB using gas field ionization source (GFIS) [7]. Four years later, in 1979 the first LMIS based FIB was built by Seliger et al and they reported submicron sized gallium ion beam with current density of 1.5 A/cm²[8]. Since then many companies have established their FIB system in the market having various specifications and features. FIB with 4.5 nm resolution with current range between 1 pA
to a few nA is available as commercial product [9]. These machines have Ga LMIS as the ion source with a guaranteed life time of about 1000 hrs. There are also dual beam systems available from leading manufacturers with combined FIB and SEM [10]. This gives the possibility of viewing the FIB processes online with utmost clarity using the SEM. This enables to explore a new way of fast sample preparation, 3D nano analysis, TEM, EBSD & atom probe sample preparation or structural modification of sample surfaces at the nanometer scale and has increased the applicability of the system. Though these machines are very useful for processes at nm level, but their performance comes down when the material removal volume is high due to their very low available current at the focused spot. The latest innovation among the commercial FIB is the introduction of Plasma based ion sources in place of LMIS. These machines generate much higher currents at the focal plane compared to LMIS-FIB. Two such products are in market which are launched as recently as second half of 2012, one is Vion from M/S FEI and another one is iFIB by M/S Orsay Physics [11][12]. iFIB is based on ECR plasma, whereas Vion Plasma focused ion beam has ICP as the ion source and generates Xe ion beam. The minimum resolution available from Vion is 25 nm with a current range of 1.5 pA - 1.3 µA. With over 1 µA of beam current, it can remove material much faster (>20X) than FIBs based on liquid metal ion sources (LMISs), while still preserving excellent milling precision and imaging resolution at low beam currents. iFIB can produce maximum current of 2.5 µA of ions of various gases. There is some activity going on in the Seoul National University in development of ICP and ECR ion sources for FIB applications [13]. It is also worth mentioning that microwave ion source based FIB activity is going in Indian Institute of Kanpur [14].

1.3 Typical LMIS-FIB system

A conventional LMIS-FIB system consists of an ion column, a working chamber, a vacuum system, a gas system and a workstation that provides the user interface [15][2][16]. A schematic diagram of an FIB ion column is shown in Figure 1-2. The structure of the column is similar to that of a scanning electron microscope, except that the FIB system employs a gallium ion (Ga⁺) beam instead of an electron beam and in many FIBs, an ExB mass analyzer is
incorporated when alloy samples are used as source of liquid metal. The pressure inside the column is maintained at typically $1 \times 10^{-7}$ mbar. The ion beam is generated from a Liquid Metal Ion Source (LMIS) by the application of a strong electric field. This electric field causes the emission of positively charged ions from a liquid gallium cone, which is formed on the tip of a tungsten needle. A typical extraction voltage is 7000 V and is further accelerated to 10 – 50 KeV. The extraction current under normal operating condition is ~2 μA. After a first refinement through the spray aperture, the ion beam is condensed in the first electrostatic lens. The upper octupole then adjusts the beam stigmatism. The ion beam energy is typically between 10 and 50 keV, with beam current varying between 1 pA and 10 nA. Using the variable aperture mechanism, the beam current can be adjusted over four decades, allowing both a fine beam for high-resolution imaging on sensitive samples and a larger beam for fast and coarse milling. Typically, seven values of beam current can be selected. Their exact values depend on the machine type and the users preferences.

Figure 1-2 Schematic of conventional GaLMIS-FIB [17]
The samples that are to be treated by a FIB are mounted on a motorized five-axis stage, inside the work chamber. Under normal operating conditions, inside this stainless-steel chamber a vacuum in the low $10^{-6}$ to $10^{-7}$ mbar range is maintained. Loading and unloading of the samples is usually performed through a load-lock, in order to preserve the vacuum inside the work chamber as much as possible. It typically takes a few minutes to load or unload a sample.

1.4 A review of ion sources developed for FIB

Liquid Metal Ion Source (LMIS) and Gas Field Ion Source (GFIS) are the two ion sources that produce ion beams with normalized brightness of $10^9$ and $10^{11}$ Am$^{-2}$sr$^{-1}$eV$^{-1}$, with a few pA to 100 nA of ion current respectively at the final focal spot [18]. LMIS is generally used in FIB though there were efforts to employ GFIS. The cooling requirement needed for GFIS and the very low current extracted has made GFIS less popular. But both the ion sources have very low virtual source which is reflected in their high brightness and hence the probe size is also very small. Brightness is 5 nm for LMIS and 0.28 nm for GFIS. With increase in the ion emission current in GFIS and LMIS, due to coulomb repulsion, there is a rapid increase in the divergence and the ion energy spread. Increased divergence and ion energy spread contribute to spherical and chromatic aberrations, thereby increasing the final focused spot size. High brightness of these field emission type ion sources is predominantly due to high current densities ($10^9$ and $10^{11}$ A/m$^2$ for LMIS and GFIS respectively) emitting from very small area of a few nm$^2$. Their emission currents are typically 2 - 5 μA. Major drawback of GFIS is that it produces very low current on final focal spot and that of LMIS is that it produces ion beams of few metallic elements only causing contamination on the milled surfaces due to implantation of metallic ions. Using these low current ion beams, it takes very long time, may be several hours to drill holes in a few micron thick metallic layers in microcircuits. As the Liquid metal ion source emits ions from a very sharp point, the current density at the emission region is very high. At the emission tip, due to high current density, there exists a very strong coulombs repulsion (space charge effect) and cause the repulsion among the ions. This in effect causes the trajectory displacement and results into the wide divergence and low angular current density at the source itself. Since LMIS has low angular current density, it is essential to employ larger
apertures to achieve larger probe currents on the target. About 10 nA and above, due to large size of the apertures, the spherical aberrations become dominant and the probe size grows rapidly. State of the art Ga LMIS from FEI shows that up to 10 nA, the spot size grows as $I^{0.25}$ and beyond this, spot size grows as $I^{1.5}$, where $I$ is the current in the probe as shown by Smith et al [19]. To overcome these major drawbacks of field emission type ion sources, recently a new approach of producing FIB of variety of gaseous elements from plasma based ion sources has emerged. For FIB applications, among various types of plasma based ion sources, Duoplasmatron, Penning Ion Gauge (PIG) ion sources, compact ECRIS (as in iFIB by M/S Orsay Physics) and Inductive Coupled Plasma (ICP) ion sources are the most popular ones. ICP based ion source has distinct advantage of not having filaments and also that they can produce ion beams of even reactive gases over long periods. In these ion sources, the plasma surface at the emission region can be manipulated by adjusting the gas pressure, power, and the electrode geometry in such a way that the ions are extracted with optimum perveance. At optimum perveance, divergence from aberrations can be minimized which is much larger than source emittance-limited divergence. In plasma based ion sources, emission area is large and hence the current density is lower as compared to that of LMIS or GFIS. However they have significantly higher angular current densities, typically 3 order more than that of LMIS. This high angular current density is an important factor in generating high current FIB and can achieve high milling rates.

Several works have been reported on the design of ICP based ion source with high brightness and high angular current density. Guharay et al. have reported 5 A/cm² ion beam on target with normalized brightness of $10^3$ Am⁻²sr⁻¹eV⁻¹ for Argon ions from PIG ion source [20] not much work is reported further e on utilizing PIG for FIB applications. ICP based ion sources for FIB applications have been developed by Mordyk et al. and Q ji et al.[21][22]. Smith et al. have reported activities on development of ICP based ion source based FIB. They have achieved submicron diameter ion beams of Ar and Xe. They have reported the brightness is of $5100$ Am⁻²sr⁻¹eV⁻¹ and $9000$ Am⁻²sr⁻¹eV⁻¹ with the current densities of 76 mA/cm² and 118 mA/cm² for Ar and Xe ion beams respectively [23]. In their work, Smith et al. have compared the performance of ICP (with Xe ions) and LMIS (Ga ions) with more than 100 nA current
focused on to the target, ICP based FIB system is found to produce smaller spot size than that of LMIS based system. It is interesting to note that the ion source designed for FIB applications by Smith et al does not make use of any magnets to confine the plasma and could achieve high milling rates [24]. However, in such plasma sources, there is a partial plasma confinement due to the magnetic field produced by the RF current in the antenna. Conventional ICP sources that are used in semiconductor processing, utilize multicusp magnetic fields to produce high density plasma with low plasma potential, uniformly over large area of wafers. In case of ICP based ion source for FIB applications, the creation of uniform plasma density by using magnetic field is not an important factor since the ion beam is extracted from a very small aperture in plasma electrode. ICP based ion sources producing low energy spread and high brightness ion beams with high angular current density of various species, in combination with focusing column will open more possibilities and advancement for fabrication of micro devices.

1.5 Aim of this thesis

One of the main drawbacks of LMIS- FIB machines is that contamination is inevitable due to the use of liquid-metal ions, so a noble-gas ion source is very much desired. Ga ions not only change the electrical properties but can also affect the mechanical, optical and magnetic properties of the devices. Gallium staining due to deposition of gallium ions in the quartz substrate during FIB repair of a photo-mask is another important issue that limits the use of FIB machines. The scope of FIB machines can be enhanced significantly by the use suitable noble-gas ion source.

The main benefits of using Plasma ion source as a replacement of LMIS are:

1. Large angular current density, ie, high process speed
2. Ion energy spread is independent of extracted current
3. Availability of variety of gaseous species which will help in selecting the beam for the particular process. (Xe, Kr Ar for machining, O for oxidation, N for nitriding, Proton for imaging etc)
4. Capability of rapid switching among the ion species is fast and easier
5. Stable current and long life
6. No contamination

But the main drawback of the plasma based ion source is the large virtual source size. This can cause two difficulties. Firstly, the source brightness is low as compared to LMIS. Secondly, in order to obtain a submicron probe size, a large demagnification of the source is required and hence demands a complex focusing column. Since plasma ion source has many benefits over LMIS, it is worth carrying out research to develop a new focused heavy ion beam system with a plasma based ion source.

The scope of the thesis is the development of ion source, extraction of the ion beam, characterizing and optimization of ion beam parameters with emphasis on achieving high current focused heavy ion beam making it suitable system to achieve high speed micromachining capability. With extensive experimental studies on all the main parameters of the ion beam, improvement in performance of the ion beam parameters is achieved. Further experiments of high speed patterning on Si, milling of high aspect ratio apertures in thin and thick metal foils, milling extremely hard cutting tools et prove that the system has capabilities to mill large volumes with at least two orders faster rate than the conventional systems.

This thesis is arranged in 9 chapters including the Introduction as chapter 1 presenting brief of FIB technology, current research activities in this field.

Chapter 2 explains about the Plasma based ion source which is selected as an alternative for LIMS. This also explains some of the innovations carried out to improve the performance of the ion source to produce high current ion beam with suitable parameters for achieving high current FIB.

Chapter 3 explains about the design of the extraction system. As being the most crucial part of any ion source, a stress is given to describe the features of the ion beam extractor that are important in generating collimated beam. Design of the extraction is carried out using ion beam extraction simulation tool IGUN by Dr R Becker, and a briefly discussed in this chapter. Detailed experiments in extraction of gaseous ion beam from the source are also explained here.

Chapter 4, 5 and 6 includes some of the important part of this thesis which contain the beam diagnostics carried out for characterizing the ion beam and optimizing the ion source
parameters. These three chapters describe the most important parameters that affect the current and size of the ion beam at the focal plane. Chapter 4 describes about ion energy spread of the ion source and the many methods followed to reduce it to a possible minimum. Measurement and characterization of emittance of the ion beam, which determines the focusability of the beam, is illustrated in Chapter 5. Measurement of brightness and the relation between emittance and brightness of the ion beam is also explained in this chapter. To have a high current at the final focal plane, the angular current density of the beam has to be high, so that even with introduction of beam limiting apertures in the column to reduce the divergence angle, the current passing through the aperture will be high enough to generate high throughput. Chapter 6 explains the measurement of angular current density which is found to be 3 orders higher than that of the LMIS. A further improvement in the angular current density by 30% is achieved by modification of the ion source and the details are presented in Chapter 6.

It is also seen that lower RF power could produce ion beams heavier elements with higher angular current density which is an added advantage. The milling experiments on thick metal foils were carried out using very low RF power and Xe ion beam.

Chapter 7 describes the details of the design and construction of the focusing column. This chapter also presents an account of the detailed experiments on focusing the ion beam. Both argon and xenon ion beams were used in all the experiments described in the thesis and very encouraging results were obtained. 4.12 µA of xenon beam is obtained at the target with 29 µm beam dia. This is highest current density ever reported at this beam dimensions. Minimum beam size obtained was 2 µm with a current of 80 nA.

In order to prove the high current capability of the system, several experiments were carried out for measuring the milling rate on different metallic foils and patterning capabilities on Si and the details are described in Chapter 8. Highest recorded milling rate on one of the high melting point metal Ta is 191 µm³/s with 10 keV xenon beam. A few possible applications of the system are also explained. The milling rates on copper were found to be more than 700 µm³/s, which is a very interesting result.

All the results are summarized in Chapter 9 and also few future possibilities in the improvement of the system and applications are presented.