Design, Development and Performance of UHV Chamber for in-situ Magneto-optic Kerr Effect and Resistivity Measurements

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Abstract. An UHV chamber is designed and developed for preparing thin films and multilayers of various elements, alloys and compounds using electron beam evaporation (EB) and ion beam sputtering (IBS) techniques. The developed chamber is equipped with magneto-optic Kerr effect (MOKE) and resistivity techniques in order to perform in-situ magnetic and transport properties measurements of ultra thin films. The calibration and performance of the developed chamber with deposition techniques is evaluated by depositing films of Fe and Si on float glass substrates. Systematic illustration of the versality of the UHV system has been done by performing in-situ MOKE and resistivity measurements simultaneously during the growth of Co film (from a fraction of a nm to 17 nm) on float glass (surface roughness 0.5 nm) and sheet glass (surface roughness 1.6 nm) substrate using electron beam evaporation. It is found that the substrate with higher roughness display maximum coercive field at Co thickness of 3.0 nm, while in case of smoother substrate the maximum coercive field is observed at 1.3 nm thick film.

1. Introduction
Thin films and multilayers have emerged as an important class of nanostructured materials with immense possibility of tailoring their properties in order to achieve the desired functionality. Recent technological advances in thin-film deposition techniques have made it possible to tailor materials having layer or bilayer thickness in the range of a few nanometers to generate the desired physical properties with applications in magnetism [1], neutron [2], and soft x-ray optics [3] etc. In all these applications, a precise control of layer thickness, and good interface quality in terms of sharpness and smoothness of the interface, play a crucial role in determining the quality of the final device. The preparation of thin films is therefore of importance in both fundamental research and practical applications. A large number of techniques have been developed for deposition of thin films [4-6], whereas electron beam evaporation [5] and sputtering [6] are most commonly used techniques for thin film deposition. It is found that in general, the surface roughness of the film deposited by IBS is significantly smaller as compared to the films deposited by e-beam evaporation [6]. Furthermore, the grain size of the sputtered crystalline films is significant refined as compared to the films deposited by e-beam evaporation. Advantage of the evaporation process is that, it is carried out in clean ultra high vacuum conditions (~ 10^-9 mbar) thereby depositing a film with minimum contamination. Since the mean free path of the evaporated atom is very large compared to the chamber dimension under ultra high vacuum conditions, the distance between the evaporation source and the substrate can be kept large enough to have uniform deposition over a large substrate area. The choice of the technique depends on the specific goals and type of the material used [5,6]. In-situ characterization during the film growth or after film deposition are equally important, specially in case of the ultra thin films.
because the exposure of ultra thin film to ambient can result in oxidation of the films and thus modification in its properties [7]. Furthermore ex-situ measurements, as a function of film thickness involve growing a series of samples of varying film thickness. In contrast by continuously monitoring the properties with film thickness in-situ, a complete thickness dependence curve can be obtained during growth of a single sample. In-situ method thus makes it practical to vary growth parameters, such as substrate temperature, pressure in chamber and film growth rate.

The present work describes the design and fabrication of a versatile UHV system for thin film deposition using EB and IBS techniques. The main motivation behind developing the present system is to fabricate good quality thin films and characterization of these films for their magnetic as well as transport properties using MOKE and resistivity method in-situ. We have briefly presented our in-situ MOKE and resistivity experiments conducted simultaneously during the Co film growth.

2. Design and description of UHV chamber

In order to design UHV chamber the following considerations have been taken into account for fabricating good quality thin films and multilayers; (a) good quality UHV compatible materials for construction (b) sufficient space for deposition and characterization facilities (c) good control over layer thickness and uniformity over required substrate area (d) good quality and sharp interface (e) minimum contamination and stresses in the layer etc.

SS304L material is used in fabrication of the chamber. It has a diameter of 300 mm and a height of 550 mm. The designed chamber has number of ports to accommodate the various components; electron gun, ion beam gun, masking arrangement for deposition techniques, substrate holder and sample heating arrangement, quartz crystal thickness monitor, windows for in-situ MOKE, electrical and motion feed-throughs, an UHV gauge, residual gas analyzer (RGA) and vacuum pumps.

![Diagram of UHV chamber setup](image)

**Figure 1.** (Online coloured) A pictorial view for arrangement of the (a) ion beam sputtering system (b) electron beam evaporation system in the UHV chamber. For visualisation of the inner view all ports of the chamber are not shown.
Chamber is designed to equip with two deposition techniques, IBS and EB. At a time only one technique can be used for the deposition. Arrangement for both with the chamber is shown in the figure 1a and 1b. One 100 CFF port is kept parallel to the horizontal cross section of the chamber for the target holder. The target holder (a rectangular block) is mounted on a rotary motion feed through and can hold four targets at a time (figure 1a). One 100 CFF port kept at 45° with respect to the target plane, which is focused to the center of the target. Same port (which is destined for the target) is also used for electron gun in case of the EB deposition (figure 1b). The top flange of 250 CFF, houses the substrate holder and a view port. One port is also provided in the plane of the substrate for viewing the film or substrate during deposition. Some additional angle ports of 35 CFF are also provided at 45° with respect to the substrate. Two of them are used for in-situ MOKE measurements. Two ports 63 CFF and 100 CFF are attached at 180° to each other at the level of the substrate, which will be utilized in future for in-situ reflection high-energy electron diffraction (RHEED) measurements.

The chamber has a volume of about 60 lt and surface area exposed to vacuum of 9000 cm². Considering the gas load due to the surface area, a rough estimate of time required for evacuating complete chamber to a pressure of ~10⁻⁷ mbar with a 500 l/s turbo molecular pump is ~3h, whereas the time required to pump out the gas load due to the volume of the chamber is negligible. Thus, the main gas load is due to out-gassing from the internal surfaces of the chamber and it minimized by employing proper cleaning and polishing procedures. The leak detection in the chamber is carried out using helium leak detector (ALCATEL, Model ASM 110) and total leak was found to be less then ~5x10⁻¹⁰ mbar l/s. After installing all the port, the chamber was evacuated by a TMP of 500 l/s capacity (VARIAN 551, TV) backed by a oil free diaphragm pumping. A compact cold cathode gauge (Model: IKR 270, Pfeiffer controller) has been used for vacuum measurements in a pressure range of 5x10⁻¹¹ to 1x10⁻² mbar. The ultimate vacuum obtained in the chamber is 9x10⁻¹⁰ mbar after backing the chamber at 200°C for 18 h.

Ion source used in the present system is a 3 cm diameter Kaufman type hot-cathode grided source (common wealth scientific corporation). For precise gas flow, a mass flow controller (MFC) [model: MKS 1179A] with an accuracy of 1% is used, which can operated in the range of 1-100 cm³/min with most of the commonly used sputtering gases [ Ar, N₂, O₂, etc.]. In general, Ar gas flow is used for thin film deposition using IBS, whereas N₂ and Ar gas mixture was used in a controlled ratio in some special cases (for nitride film etc.). After introducing the flow of the sputtering gas (4 sccm) in the chamber, the pump can be operated at a constant pumping speed in order to maintain a pressure of 1x10⁻⁴ to 5x10⁻⁴ mbar. An e-gun (TELEMARK Model: No.-528) of 3kW, having three crucibles is used to deposit thin films in case of electron evaporation method. The distance between the e-gun and substrate holder is about 35 cm, which provide film thickness uniformity of 1% over 3 cm diameter of the substrate. According to our requirements this distance can be varied by moving the substrate holder up and down (± 5cm). In order to monitor the thickness of the film during deposition RF shielded thickness monitor is used and therefore can also be used for in-situ thickness monitoring during ion-beam sputtering.

3. In-situ magnetic and transport properties measurements

MOKE and four probe resistivity techniques are used to study magnetic and transport properties of the magnetic ultra thin films in-situ. For this purpose a UHV compatible Helmholtz coils (HCs) has been designed and fabricated (not shown) for applying variable magnetic field along the plane of the film. A maximum of ~180 Oe can be produced using 3A current with 21 V power supply. For in-situ MOKE measurements, a polarized He-Ne laser light (λ= 632.8 nm) is used. It passes through a photo elastic modulator (PEM) and enters into the UHV chamber through a window. The light reflects back from the magnetized sample placed between the Helmholtz coil and come out from the chamber through another glass window. Finally, it enters into the photo diode detector though an analyser whose polarization axis is nearly crossed with that of the polarizer axis. The change in the intensity of the laser light in the detector is proportional to the magnetization of the sample, the output of the photo diode detector vs. the applied field (H) yields the hysteresis loop of the sample. Four Pt
(1 mm × 3 mm) contacts are deposited on bare substrate and four fine copper wires are attached with this using silver paste in order to perform resistivity measurements. The sheet resistance of the sample is obtained using as the relation: \( R_s = k \frac{V}{I} \); where \( I \) is the constant current through the sample, \( V \) is the voltage developed and \( k \) is a correction factor [10]. The more information about the set up is given in reference [11].

4. Calibration and performance of the deposition facilities

The calibration and performance of the developed chamber with deposition techniques (IBS & EB) is evaluated in the following stages; i) thickness uniformity and maximum thickness regions ii) calibration of the thickness monitor, iii) deposition rate, precise thickness control and pressure have been optimized by depositing various metals. Only calibration using ion beam sputtering technique by depositing Fe and Si film has been shown here.

![Block diagram of the top view of the chamber, showing the positions of the float glass substrate and position of the crystal thickness monitor.](image)

**Figure 2.** Block diagram of the top view of the chamber, showing the positions of the float glass substrate and position of the crystal thickness monitor.

In order to find out the region of maximum thickness homogeneity at the substrate, (110x15) mm long stripe of float glass is loaded (figure 2). Iron is sputtered for 15 min using ion beam of energy 1000 eV in Ar gas atmosphere. After deposition the sample is taken out from the chamber and XRR measurements from different regions along the length of the sample have been performed. The XRR patterns were fitted with Parratt’s formalism [12] in order to get the thickness of the film (figure 3a). The thickness obtained from the fitting has been plotted as a function of distance from one corner of the strip to other (figure 3b). 30 mm region of the sample at the center has been found uniform with thickness variation of less than 1%. Average thickness in this region is 26.5 nm and the thickness obtained from the thickness monitor is 23.7 nm. Thus actual thickness is 1.1 times larger as compare to that obtained by crystal monitor, this may be because of the position of the crystal monitor, which is off from the center of the substrate. The thickness monitor is calibrated for further depositions.

To check the stability of the deposition rate, Si films were deposited from the most uniform region of the chamber (obtained from the Fe deposition). A mask with a rectangular cut of 30 mm x
20 mm has been fixed just below the substrate holder such that the cut on the mask comes just at the uniform thickness region. The arrangement of the mask and substrate is given in the figure 4a.

![Figure 3](image1.png)

**Figure 3.** (a) Fitted X-reflectivity patterns and (b) Fe film as a function of distance (d) along the length of the sample

A silicon film has been deposited for total 23 min duration at different regions (for 5 min, 7 min and 10 min) of the substrate by moving the substrate holder. The thickness obtained from XRR measurements are written in front of the corresponding XRR pattern (figure. 4b). It may be noted that this deposition rate is constant over a period of time in all three regions. The thicknesses during deposition were also continuously monitored by quartz crystal monitor and found consistent with the XRR measurements. Similar calibrations were also performed using electron beam evaporation (not shown here). Such a detailed evaluation of the performance of the deposition system has enabled us to deposit thin films and multilayers with thickness control of the order of 0.1 nm. Such calibrations are required for easy adaptability to carry out ultra thin film related in-situ research works.

![Figure 4](image2.png)

**Figure 4.** (a) Schematic of the arrangement during the film deposition form the most uniform area (b) XRR patterns along with the Parratt’s fitting for 5 min, 7 min and 10 min sputtered Si film
5. In-situ study of Co films

5.1 In-situ MOKE as a function of Co film growth

The sensitivity and performance of the in-situ MOKE is tested by performing hysteresis loop measurements of Co film as a function of thickness in UHV chamber with the vacuum of \(~2\times10^{-8}\) mbar. The hysteresis loop and resistivity measurements were done with simultaneous deposition of Co material on float glass substrate with film growth using electron beam evaporation. The rate of deposition was kept very slow (0.02 nm/min) and continuously monitored by quartz crystal thickness monitor. The in-situ MOKE loops were collected during film growth in the longitudinal geometry using Helmholtz coils with a maximum field of 120 Oe. Figure 5 shows the development of the MOKE hysteresis loops as a function of increasing film thickness. A significant MOKE signal appears at a thickness of 0.7 nm, indicating the onset of the ferromagnetism in Co film after 0.7 nm. The absence of any hysteresis loop up to 0.5 nm indicates that the film may be in super-paramagnetic phase up to this thickness. The observed loop at ~0.7 nm film thickness shows that the sensitivity of the in-situ MOKE set-up is very good.

Figure 5. In situ hysteresis loops (longitudinal MOKE) of the Co film as function of thickness. Only few representative loops are presented.

5.2 Effect of roughness on magnetic and transport properties

In order to study the substrate roughness effect on magnetic and transport properties, MOKE and resistivity measurements have been performed on two substrates (one by one), float glass (FG) and sheet glass (SG) having surface roughness, 0.5 nm and 1.6 nm respectively, as a function of film thickness (from a fraction of a nanometre to 17 nm). Electron beam evaporation method has been
used to prepare Co film on both substrates. Figure 6 shows the sheet resistance of Co film as a function of film thickness for both substrates. Although the trends of the sheet resistance with film thickness for the both samples are same, but it is observed that the substrate roughness plays a very important role in determining the percolation threshold (drastic decrease in the sheet resistance) and conductivity characteristics of the film. Percolation of the Co/SG film occurs at a higher film thickness (~1.4 nm) as compare to the Co/FG (~0.5 nm). The percolation in case of the Co/SG sample at the higher coverage of the Co film indicates that the islands start to connect at the higher thickness. In case of the Co/FG sample smooth surface is the cause for the percolation at the lower thickness. Slow decrease in the resistance (for Co/FG sample) after the percolation and even up to the 3.0 nm thickness show that the roughness influences the morphology of the film up to the 3.0 nm. Evolution of coercivity (Hc) with Co coverage on the both substrate (FG and SG) is illustrated in figure 7. The most noticeable difference is the critical thickness at which the hysteresis with finite width (onset of the ferromagnetism) has been observed. In case of the Co/SG sample the onset of the ferromagnetism start with the Co thickness ~1.8 nm whereas it was 0.7 nm in case of the Co/FG sample. Loops are also sheared (not shown) in shape even up to the higher thickness of the Co coverage as compared to the Co/FG sample. This may be attributed to the domain structure for higher roughness sample, which may made-up of large irregular domains with perpendicular up and down magnetization. The coercivity displays a sharp increase with increase in Co thickness and reaches maximum coercive fields for film thickness ~1.4 nm and ~3.8 nm for Co/FG and Co/SG, respectively. The Co film with smoother substrate becomes continuous at lower film thickness.

![Figure 6](image1.png)  
**Figure 6.** Variation of the $R_{Co}$ as a function of Co film thickness for FG and SG substrates

![Figure 7](image2.png)  
**Figure 7.** Variation of the $H_c$ as a function of the Co film thickness for FG and SG substrates

The coercivity is higher in case of Co/SG sample even up to the 16.0 nm coverage of the Co film. Although, it is observed that the coercivity is very sensitive to the roughness in ultralow thickness range up to ~5.0 nm. Co film with higher roughness displays higher value of the coercivity $H_c$ ~32 Oe at 3.8 nm whereas smoother film shows maximum $H_c$~ 27 Oe at 14.0 nm. As we have seen in the resistance measurements these thickness 14.0 nm and 32.0 nm are those at with the films are becoming continuous.

In conclusion, a UHV chamber has been designed and developed to prepare good quality thin film and multilayers using IBS and EB deposition techniques. The chamber is also equipped with MOKE and resistivity measurement facilities to study magnetic and transport properties of ultra thin film in-situ, since the exposure of ultra thin film to ambient can result in oxidation and thus modification of magnetic properties. A systematic illustration of the versatility of the set-up has been done by depositing pure cobalt film ranging from fraction of nanometre to several nanometres and performing
in-situ measurements during deposition. In order to show the role of interface roughness on the magnetic and transport properties of cobalt film, a combined in-situ study of both magnetic and magneto-transport as a function of the film thickness have been done. The percolation threshold for rough film is observed at the higher coverage of the Co film, which indicates that the islands start to connect at the higher thickness for rough substrate. Co film with higher roughness displays higher value of the coercivity, $H_c \approx 32$ Oe at 3.8 nm whereas smoother film shows maximum $H_c \approx 27$ Oe at 14.0 nm.

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