Electron beam deposition system for X-ray multilayer mirrors

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Abstract. We have developed a multi source electron beam evaporation system to prepare high quality X-ray multilayer mirrors. The system has three electron guns mounted in an ultra high vacuum chamber. The deposition system is evacuated by a turbo molecular pump and two sputter ion pumps. A movable masking system is mounted to deposit several kinds of multilayers. First results on niobium–carbon X-ray multilayers are presented.

Keywords. Electron beam evaporation; X-ray multilayer; thin films; ultra high vacuum; XUV optics.

1. Introduction

In the X-ray and vacuum ultra violet (XUV) regime grazing incidence optics is extensively used, as normal incidence reflectivity, R , of all materials decreases rapidly with decreasing wavelength λ ($R \propto \lambda^4$) (Spiller *et al* 1980). This optics suffers from disadvantages of aberration in the image quality and poor collection efficiency. Progress in thin film deposition techniques has led to the development of multilayer XUV optics which can operate at higher angles of incidence. Multilayer coatings for XUV optics consist of stacks of thin films composed of materials with alternatively high and low scattering power having period in the range 10 to 100 Å. The development of these optical elements have found wide application in a variety of fields including synchrotron radiation instrumentation, plasma diagnostics, soft X-ray spectrometers and X-ray astronomy (Denz 1986; Verhoeven *et al* 1989; Barbee 1990; Yamashita 1995).

Multilayer optical elements are fabricated by any technique in which thin films can be formed by means of 'atom by atom process'. Physical vapour deposition techniques are generally used for the fabrication of X-ray multilayer mirrors. Electron beam deposition (Spiller and Golub 1989; Yamashita *et al* 1989), sputtering (Barbee 1981; Sella *et al* 1986; Hasan *et al* 1992) and laser ablation (Gaponov *et al* 1985) are widely used in the fabrication of X-ray multilayers. In this paper we describe the construction and performance of an ultra high vacuum electron beam (e-b) evaporation system developed by us for X-ray multilayer fabrication. Our choice of the various features incorporated in the system are discussed with respect to our objective to obtain well defined interfaces and high reflectivity XUV mirrors. During the design and fabrication of the system significant effort was made to use the hardware developed indigenously or available in the local market and only very few major components were imported. First results obtained on niobium–carbon multilayers prepared using this system are presented.

2. Electron beam evaporation system

Three e-b evaporation sources have been incorporated in an ultra high vacuum deposition chamber evacuated by a turbo molecular pump and two sputter ion pumps.

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Figure 1. Schematic diagram of electron beam evaporation system. (1. Substrate mounting flange, 2. sample masking arrangement flange, 3. sputter ion pump connecting flange, 4. turbo molecular pump connecting flange, 5. sample masking arrangement, 6. liquid nitrogen substrate cooling arrangement, 7. view ports for substrate and mask, 8. view ports for electron guns, 9. electron guns, 10. flanges for water and electrical feedthrough for electron guns, 11. electron gun masking flange, 12. Viton o-ring flange, 13. bottom shell and 14. top shell.)

Two quartz crystal monitors and a quadrupole mass analyzer have been installed to monitor the thin film thickness and deposition rate. A substrate holder which can be cooled to liquid nitrogen temperature has been installed. A movable masking system has been mounted just below the substrate to deposit several kinds of multilayers

Figure 2. Photograph of the electron beam evaporation system. The top shell is mounted on the stand and is connected to a turbo molecular pump and two sputter ion pumps.

without venting the system. The schematic diagram of the electron beam evaporation system is shown in figure 1. The system consists of various sub-systems which are described below.

2.1 *Ultra high vacuum chamber*

Ultra high vacuum (UHV) chamber, made of austenitic stainless steel AISI 304 grade, has been fabricated in our workshop. The chamber has length 1150 mm, diameter 512 mm and wall thickness 4 mm. The chamber is divided into two shells, connected to each other through a flange with a vacuum degassed Viton O ring. This is the only place where Viton has been used, as the two shells have to be separated for changing the e-b gun targets and substrate. All other ports have conflat flanges with OFHC copper gaskets fabricated locally. Various side ports have been welded along the circumference through which various sub systems are connected. All the side ports were helium leak checked to leak rate of 1.0×10^{-10} std cc/sec before TIG welding on to the main chamber.

Figure 2 shows the complete assembly with various sub systems. The top shell of the main chamber is fixed rigidly to the stand whereas the bottom shell is a demountable type. The Viton O ring flange can be baked to a temperature of 150°C while the other parts of the chamber can be baked to a temperature of 300°C. The baking is done using heating tapes. To reduce degassing from the chamber during evaporation cooling water lines are brazed on the chamber walls.

The bottom shell of length 450 mm, houses three electron guns, water line feed-throughs, high voltage feedthroughs and mechanical shutters. A port has been provided for cryo pumping if required.

The top shell of length 700 mm houses the substrate holder, a movable sample masking arrangement, two quartz crystal monitors, quadruple mass analyzer, temperature monitors for substrate and ambient temperature in the chamber, leak valve, vacuum pumps and vacuum gauges. This shell has two view ports for viewing the complete upper shell and two view ports for viewing the electron guns. To avoid coating on the e-b guns view ports, shutters are provided through two rotary feedthroughs. Provision has been made for mounting ion guns for ion assisted deposition and ion beam sputtering. Two diagonally opposite ports have been provided to connect turbo molecular pump and sputter ion pump respectively. The substrate is loaded from the top of the chamber. A movable sample masking arrangement is mounted just below the sample.

The electron beam evaporation chamber is pumped by a 450 l/sec turbo molecular pump bought commercially and two sputter ion pumps of 270 l/sec developed at our centre. The vacuum pumps are isolated from the chamber through two bellows sealed gate valves developed at our Centre. Provision has also been kept for mounting a titanium sublimation pump. The pumping speed available to the chamber is maximized by providing a opening of 152 diameter and reducing, to a minimum, the length of the connecting pipes. The sputter ion pumps are not exposed to air during venting of the main chamber. The vacuum in the chamber is monitored by cold and hot cathode gauges developed at our centre. Another measure of the vacuum in the chamber is provided by the sputter ion pump current.

For constant monitoring of partial pressure of various gases before and during deposition, a quadrupole mass analyzer of 300 AMU has been mounted in the chamber. This unit is also used for leak detection and for monitoring deposition rate.

2.2 *Electron guns*

Three ultra high vacuum compatible e-b guns of 3 kW, with separate power supplies, are mounted on the lower shell of the vacuum chamber. The photograph of the lower shell with the electron guns mounted is shown in figure 3. The electron guns are mounted on an equilateral triangle with a distance between the substrate and the electron gun of 900 mm. This large distance is essential to maintain film thickness uniformity of $\pm 1\%$ in multilayers for XUV application. Our choice to mount three separate e-b guns instead of a multi crucible single gun has been based on several technical considerations. For deposition at 10^{-9} mbar, the electron gun targets have to be degassed just below the deposition temperature for long periods of time (e.g. ten to twelve hours in case of carbon). With separate guns, masks can be put on the e-b guns and the targets can be degassed simultaneously. Furthermore, deposition rate and thermal drift in the quartz crystal monitor changes when the e-b gun target temperature is changed. These problems can be overcome in multigun system by feeding constant power to the guns and controlling the deposition with the help of mechanical shutters on the electron guns. The choice for three guns instead of two guns normally mounted in e-b deposition systems for X-ray multilayer has been due to the following consideration: Strong adhesion of the multilayers to the substrate is essential. Normally thin buffer layers, viz. chromium, nickel, of a few tens of Å are needed between the substrate and multilayer to increase the adhesion (Yamashita *et al* 1992). Over coating of multilayers with thin film of other material is required for some application requiring

Figure 3. Photograph of the inner portion of the lower shell showing 1. electron guns, 2. electrical feedthroughs and 3. water feedthroughs.

increase in grazing incidence reflectivity (Lodha *et al* 1994). In numerous cases the three-layer structure provides an enhancement of reflectivity at the first Bragg peak compared to the best classical solution using two materials (Boher *et al* 1990).

The e-b guns were procured from the Indian market (M/s Hind High Vacuum, Bangalore) and have been made ultra high vacuum compatible, as they were designed by the supplier to operate at 10^{-6} mbar pressure. The guns have 180 degree magnetic bending using ALNICO magnets. The magnets supplied had very rough surface. The grinding of the magnets was done to increase the surface finish and re-magnetization of the magnets was done. Iron pole pieces were coated with copper to reduce degassing. All stainless steel fasteners were used. The waterline feedthroughs mounted on 35 CF, were vacuum brazed to the electron guns. The electrical power to the guns is fed by 12 kV/150 amp feedthroughs mounted on 18 CF flange. Breakdown was observed in these feedthroughs due to high voltage radio frequency spikes generated by thyristor switching in the electron gun power supply. This problem was circumvented by providing an additional epoxy casted length between ground and high voltage point.

2.3 Substrate holder

The substrate is loaded from the top through a CF 150 flange. The maximum substrate size which can be accommodated through this port is 150 mm diameter. For depositing the multilayer at liquid nitrogen temperature, the substrate holder was fabricated from a 100 mm diameter OFHC copper block which can be filled with liquid nitrogen. This substrate also acts as a cryo pump. Multiple substrates can be mounted on this sample holder. The substrate temperature is monitored using a thermocouple feedthrough.

A movable substrate masking is mounted just below the substrate holder. At present a commercially available linear motion feedthrough of 50 mm stroke is used. We will be replacing it with a linear motion feedthrough of 160 mm stroke. This feedthrough has been designed and is under fabrication.

In the existing system we have fixed position for substrate and moving shutter. This is technically a much easier solution compared to fixed shutter and moving substrate. The evaporation sources are also fixed in the existing design. When using fixed substrate and sources, the constant angle of incidence of the vapour can cause shadowing effect leading to increase in interfacial roughness. We plan to remove this problem by mounting the substrate on a tilted platform and rotating the substrate holder using a rotary motion feedthrough. Also we plan to have a substrate holder which can be heated up to 600°C.

To view the substrate and substrate masking arrangement, two CF 100 view ports are provided which makes an angle of 120 degrees with the substrate plane. These ports can also be used for real time soft X-ray reflectivity measurements during deposition.

2.4 Thickness and deposition rate controller

Thickness and deposition rates are monitored using two 6 MHz quartz crystal micro balances mounted on two sides of the substrate holder. The control units which are used for these monitors are from the local supplier (M/s Hind High Vacuum, Bangalore). This unit is not suitable for monitoring thickness of a few Å with the desired accuracy of 0.1 Å. Further the thermal drift in frequency of the quartz crystal is fairly large which cannot be corrected in this commercial thickness monitor. But fortunately the oscillator electronics of this thickness monitor was very stable and so the frequency change in the quartz crystal monitor could be monitored to an accuracy of 0.1 Hz using a 10 MHz stable frequency counter. For monitoring thickness up to 0.1 Å, the frequency of the quartz crystal is monitored. From the change in the frequency and the rate of decrease of frequency, the thickness and deposition rate are calculated. The increase in frequency observed due to thermal load to the quartz crystal was used to correct for thickness error due to thermal load. A good control of thickness is evident by the results presented for niobium carbon multilayer prepared using this system. A secondary measure of the thickness during deposition is obtained from a quadrupole mass analyzer that has been installed in the system.

3. X-ray multilayer preparation

Niobium/carbon (Nb/C) multilayer system has been reported as a useful combination for near normal incidence imaging optics for use at wavelengths λ in the "water window" ($2.4 \leq \lambda \leq 4.4$ nm) (Xu and Evan 1990). Nb/C multilayer was prepared on float glass substrate and characterized using X-ray reflectivity measurements at Cu K α (1.54 Å). Base pressure of 10^{-9} mbar was obtained after baking the chamber at 300°C for 12 h. Individual evaporation targets were degassed, with masks on the e-b guns and the substrate.

We were able to measure carbon thickness of 0.5 Å, but the absolute values of carbon thickness varied by 10% from the desired thickness value because of the thermal frequency drift in the quartz crystal monitor during the deposition and sputtering of

Figure 4. Reflectivity profile of Nb/C multilayer measured at Cu $K\alpha$ (1.54 Å) along with the calculated profile.

graphite used for deposition of carbon. The niobium deposition rate was highly controlled in the electron beam deposition system. Deposition rate was 0.1 Å/sec and the periodicity of the multilayer (i.e. thickness of niobium plus the thickness of carbon) was controlled within 0.5 Å. The multilayer period was 50 Å, with nine layer pairs and the thickness ratio of niobium to carbon was 1:2. The multilayers were prepared with the substrate at liquid nitrogen temperature, as it has been reported for Pt based X-ray multilayers that higher reflectivity is obtained for multilayers prepared at liquid nitrogen temperature than those prepared at room temperature (Lodha *et al* 1996).

4. Multilayer characterization

X-ray reflectivity (XR) measurements were carried out on Siemens D5000 diffractometer at 1.54 Å (Lodha *et al* 1996). The calculated XR profile was obtained by the successive application of Fresnel equation at each interface. Each layered region was characterized by optical constant δ , β that define the refractive index $\tilde{n}$ ($= 1 - \delta - i\beta$) and the thickness of the layer, as obtained from the quartz crystal monitor. A computer program was developed to calculate the reflectivity of N stratified homogeneous media as a function of angle for a given incident wavelength or as a function of wavelength for a fixed incidence angle. The best fit of the calculated XR with the measured XR was obtained by interactively changing the multiplication factor for niobium and carbon thickness as obtained from the quartz crystal monitor. The average bilayer period is derived by this fitting procedure.

Measured and calculated reflectivity of niobium–carbon multilayer is shown in figure 4. The positions of the Bragg peaks give an independent measure of the bilayer periodicity of the multilayer. Absolute incident intensity measurement was not possible in the present XR setup as the sample could not be moved out of the beam just before the reflectivity measurement. The intensity normalization was done with respect to the grazing incidence reflectivity. Due to this the absolute peak reflectivity may be overestimated.

X-ray diffraction (XRD) measurements were performed at constant incidence angle of 2 degree. No diffraction peaks of niobium and carbon were observed suggesting multilayer films to be amorphous. Similar results have been reported for Mo based X-ray multilayers prepared using a UHV electron beam deposition system.

5. Conclusions

A three source ultra high vacuum electron beam deposition system was developed. The first results on Nb/C multilayer showed that the system can be used for the development of X-ray multilayer optics. This system is good for undertaking research work on the development of X-ray multilayer optics as small quantities of various material combinations can be tried. The criteria for choosing a particular material combination are dictated by a variety of physical, chemical and metallurgical properties. Most of the time it is difficult to find the rule of thumb since these properties are interdependent in a complex pattern. The thickness uniformity of evaporated multilayers over length of few centimeters is usually better compared to sputtered multilayers. Real time characterization of deposited thin films is possible in the e-b evaporation systems, allowing correction for thickness errors during fabrication.

During deposition with e-b evaporation, the evaporated material has thermal energy (0.2 to 0.4 eV) while in sputtering the material has energy of few eV. Thus sputtered multilayers usually have lower strain and larger density than evaporated layers. This drawback of thermal deposition can be removed by doing an ion assisted thermal deposition. We plan to incorporate ion assisted deposition in the existing system.

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