MULTILAYER X-RAY OPTICS

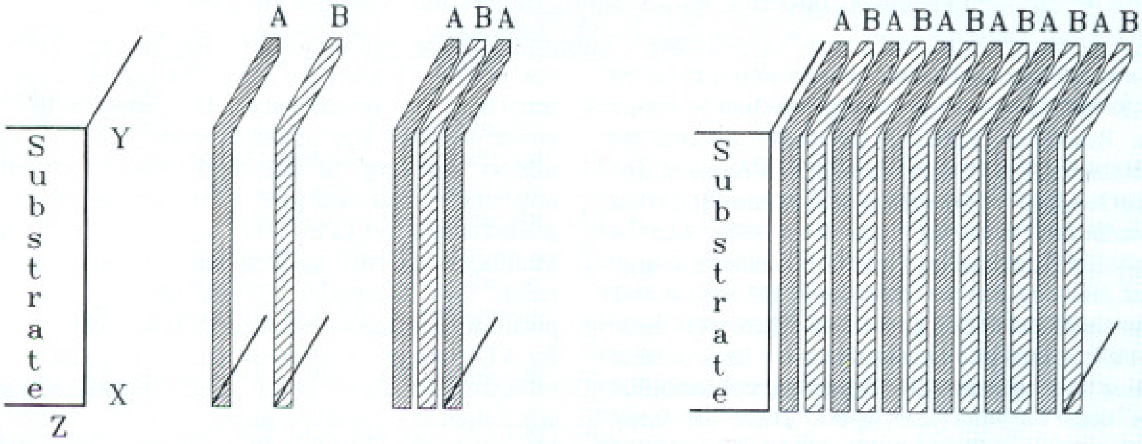
Over the last two decades there have been significant technological advances in materials science and deposition techniques through which it has been possible to extend the visible light optical technology to shorter wavelengths at one end and hard x-ray technology to longer wavelengths on the other. These extensions have led to a significant development of x-ray and vacuum ultra violet (XUV) optics in the wavelength region of 10 to 500 Å.

Wavelengths in XUV region are two orders of magnitude away from visible light and x-rays. Shorter wavelengths promise higher resolutions but at the cost of correspondingly tighter fabrication tolerances. Further because of high absorption of XUV radiation one needs to use reflective elements. As the refractive index is very close to unity for all the elements, reflecting optics has to be used at extreme grazing incidence. On the other hand, high reflectivity at normal/near normal incidence can be achieved by using multilayer XUV coatings which consist of a stack of alternating layers of materials with high and low atomic numbers. Multiple reflections from the multilayer structure are added coherently to increase the efficiency of reflection.

X-ray multilayer structures require the capability to deposit uniform, ultra thin (~10 Å), alternating films of high and low Z materials, which have super smooth surfaces of few rms roughness, with sharp interfaces and no interdiffusion between the layers. The basic requirements of the x-ray multilayer structure are shown below. As is clear from the figure, fabrication of these structures requires proper understanding of substrate surface, individual layers, interfaces and finally multilayer structures for a particular application.

Multilayer XUV optics has found wide applications in synchrotron radiation beam lines, materials science, astronomy, x-ray lasers, x-ray lithography and plasma diagnostics, etc. Illustrated on the next page are the application of multilayer x-ray optics to a variety of fields. It has a basic technology input from ultra precision machining, thin film deposition, surface, material and interface characterization techniques and precision optics. Because of stringent requirements by the industry in x-ray lithography, medical engineering and micro mechanics, there is a strong overlap of interest in this multi-disciplinary technology developed.

Centre for Advanced Technology (CAT) Indore has an ongoing program of high technology research and development in the fields of synchrotron radiation, high power lasers, materials science, biology and industrial applications. All these areas of research would need an input of x-ray optics and measurement in general and multilayer x-ray optics in particular. We discuss some applications of multilayer optics research based on synchrotron radiation sources.



A:High Atomic Number Layer e.g.W,pt,Ni...

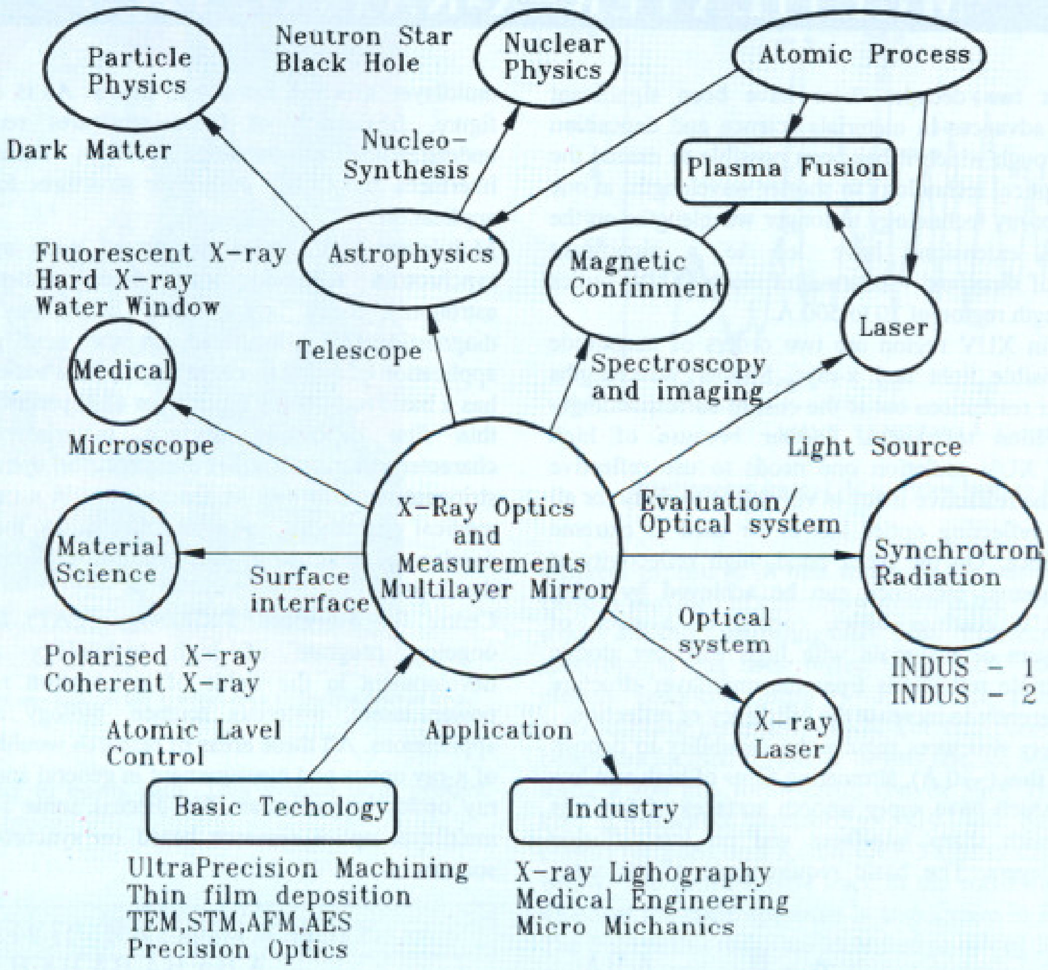
A:Low Atomic Number Layer e.g.C,Si,B...

Substrate
Flat ?
Smooth ?
Clean ?
Optical constant ?

Individual Layers
Flat ?
Smooth ?
Composition ?
optical Constant ?
Arial Concentration ?
structure ?
1. Amorphous
2. Random poly crytalline
3. Textured polycrystaline
4. single crystal
Thickness effect ?

Interface
Flat ?
Smooth ?
Compositionally abrupt ?
A/B and B/A interfaces equivalent ?
contaminated ?
Coherent ?

Multilayer
Ideal:
Flat
Smooth
abrupt
Clean
Uncontaminated
Uniform
Non Ideal:
Imperfections as listed



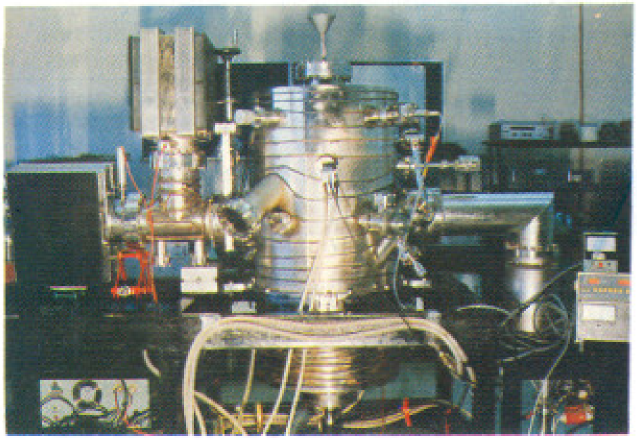
Multilayer mirrors find application in synchrotron radiation beam lines to extend the use of Bragg reflection to longer wavelength. Resolution values $\lambda/\Delta\lambda$ of 50 to 100 are routinely obtained. If much higher resolution is required, one has to deposit hundreds of layer pairs with minimal thickness errors. Contribution from all these layer pairs can be obtained only if the penetration depth of the incident energy is significant implying that the thickness of the high atomic number layer should be as thin as possible. Multilayers made of two similar low absorption materials have a large number of contributing layers and thus give higher spectral resolution without the need for ultra thin layers. There are many experimental techniques like scattering which do not need high spectral resolution but need high intensity. Multilayer structures find use in these types of experiments on synchrotron radiation beam lines. For high resolution monochromator application at larger angles of incidence multilayer gratings are being developed which combine high reflectivity with high spectral resolution of the grating.

In the third generation high brilliance synchrotron radiation sources there is a necessity to protect the first optical element from very high white flux. Damages of first optical element due to high power densities have been observed for present synchrotron sources. These optical elements have to be protected by pre-monochromators to maintain the high brilliance through the optical system. To overcome this problem multilayer mirrors are being used. Periodic multilayers with 2d of a few tens of Å and grazing angles of one to a few degrees are efficient x-ray reflectors acting as small band pass system and can be tailored for handling high power densities. Size of these mirrors is much smaller than mirrors operating in the total external reflection. For obtaining a larger band pass in the high energy region, depth graded multilayers called x-ray supper mirrors can be used.

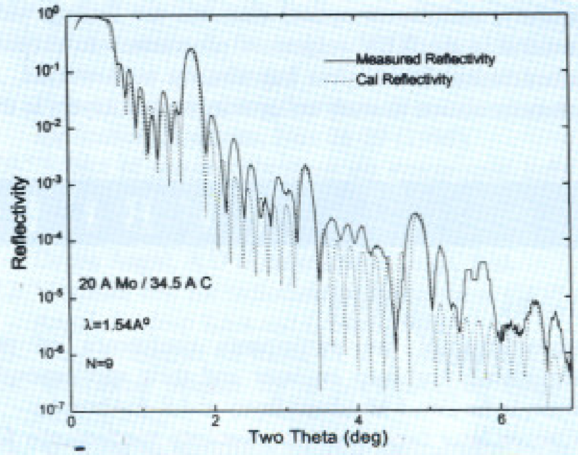
Multilayers are also used as linear polarizor in the VUV range. This is possible because the angle for which σ-polarization reflection is canceled, is very close to 45 degree for VUV region. Multilayers can be fabricated with good reflectivity at these angles. These polarizors have wide application in VUV ellipsometry.

High reflection efficiency of multilayers is used to an advantage in soft x-ray fluorescence analysis. Figured multilayer optics is currently being used in scanning electron micro probes to obtain soft x-ray images. Multilayers find application in XUV standing wave techniques to study atoms or layers on substrate. Radio theraphic imaging of soft biological tissue is another example where multilayers find application. The undesirable background can be easily reduced by multilayer filtering. This reduces the radiation damage significantly.

The activity of research and development of multilayer x-ray optics at CAT was initiated in 1991 under the synchrotron radiation utilization program. The goal is to study the physics of multilayers and develop multilayer x-ray optical systems for a variety of applications. In the next part of this article we



Electron beam evaporation system



Reflectivity of Mo/C multilayers

briefly discuss the existing status of development and research activities being carried out in this direction.

Thermal evaporation, electron beam evaporation and sputter deposition are the most widely used methods for fabricating XUV multilayers. In CAT, for the fabrication of XUV multilayers, an ultra high vacuum electron beam evaporation system has been developed (shown above). . The choice for the development of this system rather than a sputter deposition system was based on the initial requirement to fabricate multilayers using various material combinations. Using this system XUV multilayers are fabricated in an ultra high vacuum condition (10^{-9} mbar). The system has three ultra high vacuum compatible 3 kW electron guns with substrate to sample distance of 900 mm. The substrate temperature can be varied from liquid nitrogen temperature to room temperature. The thickness and deposition rate are monitored using quartz crystal monitor. The system has been used for the fabrication of multilayers with a variety of material combinations.

The criteria for choosing a particular material combination are dictated by a variety of physical, chemical and metallurgical properties. There are many combinations of high Z materials like Mo, Ni, Pt etc. and low Z materials like C, Si, B etc. Among these, metal carbon system is a promising combination for synchrotron radiation application. We present some work carried out in CAT in metal-carbon multilayer system.

As an extension of the earlier work on Pt/C system, thermally induced structural modifications in Pt/C, Mo/C and Ni/C x-ray multilayers were studied. This study has been done to investigate the thermal load which the multilayers can be subjected to before undergoing any structural modifications. Thermal stability of these multilayers has been studied under vacuum annealing using x-ray reflectivity (XR) and x-ray diffraction (XRD) techniques. Typical x-ray reflectivity data for a Mo/C system is shown on top right side of this page.

Any thin film deposited on a substrate offers two interfaces namely air-film and film substrate. Therefore, the amplitudes of x-rays reflected from these two interfaces will interfere with each other. Hence the intensity of reflected x-rays from such interfaces will show maxima and minima or oscillations known as Keissig oscillation as a function of angle of incidence. For x-rays since the refractive index is close to unity (slightly less than 1) the refracted wave will bend away from the normal. Above a particular angle for a given film-substrate combination, the x-rays will have total reflection. This is called critical angle. In a plot between reflectivity and angle of incidence of x-rays from a film-substrate combination, the critical angle gives the density of the film. Period of Keissig oscillations gives thickness of the layer and amplitude determines the roughness.

Theoretical modeling of the multilayers was done by successive application of Fresnels equations at each interface. Each layered region was characterised by the optical constants. For calculation of optical constants, tabulated atomic scattering factors f1 and f2 and the bulk density of materials were used. The thickness values of metal and carbon for each stack obtained from the quartz crystal monitor were taken as initial estimates of thickness of individual layers. A computer programme has been developed to calculate the reflectivity of N stratified homogeneous media. The best fit of the stimulated XR with experimental XR was obtained by interactively changing the thickness ratio, interfacial roughness, surface roughness and density of the film.

Pt/C and Ni/C multilayers prepared by electron beam evaporation methods have been studied for thermal stability under isothermal and isochronal annealing up to 500 ^{0}C using XR and XRD techniques. For Pt/C, it has been observed that the multilayer is stable up to 450 ^{0}C but there was a monotonic increase in the bilayer spacing of Pt/C which was 50 Å accompanied by the gradual increase in the crystallite size and grain texture. At 500 ^{0}C, multilayer reflections of Pt/C vanish, platinum crystallites grow abruptly and there is a strong texture of platinum in the [220] plane. These results are in agreement with the reported work on the stability of the sputtered deposited Pt/C multilayers exposed to intense synchrotron radiation. This indicates that the thermal stability does not depend on the method of preparation of multilayers.

A similar study performed on the Ni/C multilayer system shows that this system is stable up to 350 ^{0}C. In addition, there is an enhancement in the reflectivity on annealing multilayer at 100 ^{0}C.

Cover photograph shows the details of the electron guns mounted inside the UHV chamber.

In summary, multilayers optical elements are finding wide applications in the XUV region. A programme to prepare and characterise these layers has already been started. A reflectometer station to study reflectivity of multilayers in the wavelength range 40-1000 Å on a toroidal grating based monochromater beam line on Indus-1 is being fabricated.

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