Growth of Multilayer Optics for Synchrotron Radiation Sources

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Abstract: W/B\textsubscript{4}C multilayers have been deposited using magnetron sputtering with period ranging from 3.5 nm to 1.6 nm and layer pairs up to 300 to be used as soft x-ray/hard x-ray monochromators and soft x-ray polarizers. Normal incidence mirrors have been developed to be used in 12.5-13.5 nm using Mo/Si with reflectivity of ~67% at 13 nm. In addition, high thermal stability NbC/Si multilayers in this wavelength range have been developed. The reflectivity of these mirrors is comparable with Mo/Si but have much higher thermal stability compared to Mo/Si. We have also deposited thermally stable C/B\textsubscript{4}C, low Z/low Z multilayer. These mirrors can be used as polarizer and high resolution soft x-ray monochromators.

Keywords: Synchrotron radiation; Multilayer, X-ray reflectivity, X-ray diffraction, X-ray optics

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1. Introduction

Multilayers with short periods are of interest due to their widespread use as radiation-stable dispersive optical elements [1], polarizers for synchrotron radiation, dispersive elements for X-ray diagnostics of high-temperature plasmas, normal incidence reflectors for soft X-ray microscopy in the water window (2 to 4 nm), X-ray astronomy. Recently developed free electron laser (FEL) sources like Stanford, Desy, Sacia and Fermi@elettra[2] generate X-ray pulses of very high intensity induce radiation damage in optical components [3, 4]. The emerging technology requires improved optical components and therefore numerous research works are being carried out to find high stability and high reflecting x-ray multilayer mirrors [5].

We are fabricating high reflectance mirrors for developing soft x-ray Schwarzschild microscope, polarimeter for soft x-ray polarization experiments, and hard x-ray dispersive and focusing devices for Indus-2. Development of short period multilayers is a pre-requisite for the above mentioned goal.

To meet the above objectives we have developed various multilayers to be used in soft x-ray and hard x-ray region. In this paper we report fabrication and characterisation of normal incidence Mo/Si multilayer mirror operating at 12.4 nm, high thermal stability NbC/Si as a possible replacement of Mo/Si mirror and thermally stable C/B\textsubscript{4}C multilayer mirrors to be used as polarizer and high resolution soft x-ray monochromators. We also report fabrication of high reflectivity short period W/B\textsubscript{4}C multilayer mirrors of periods varying from 3.5 nm to 1.5 nm. We also discuss fabrication of large area (300mm X 100 mm) Mo/Si multilayer mirrors These multilayer mirrors have been
fabricated using a custom built large area DC/RF magnetron sputtering system and an in-house assembled Ion beam sputtering setup.

2. Experimental details

Mo/Si and W/B$_4$C multilayers were fabricated using magnetron sputtering system which has both DC and RF compatibility [6]. There are two rectangular cathodes of size 500 mm × 100 mm each. The sputtering process is in a horizontal configuration. The base pressure in the main chamber and in the load lock system were 1×10$^{-8}$ mbar and 8×10$^{-8}$ mbar, respectively. Load lock chamber contains a RF ion etcher for substrate cleaning. Substrate moves linearly over sputter sources with variable speed. DC power is used for sputtering of Mo, W and RF power is used for Si, B$_4$C. NbC/Si and C/B$_4$C multilayers were deposited on silicon substrate using ion beam sputtering system. Film thickness was controlled by keeping the deposition time fixed for each layer.

Characterization of all the samples was done by x-ray reflectivity (XRR) technique, using Bruker D8-Discover system consisting of Θ - Θ goniometer with Cu target. The system had incident beam parabolic mirror along with two bounce Ge (220) monochromator to give parallel beam. Standard alignment procedures were done to align both axes within 0.002°. The soft x-ray reflectivity measurements were performed at Indus-1 reflectivity beamline [7].

3. Results

After optimizing process parameters by fabricating smaller number of layer pairs, Mo/Si MLs were fabricated with larger number of layer pairs and characterized using hard x-ray reflectivity, thickness error of ~ 0.03% per layer is obtained from higher order peak broadening. The lateral variation of the periodicity is controlled within 0.05 nm over the 300×100 mm$^2$ as shown in Figure 1. Figure 2 shows the angle versus reflectivity scan of ML with d=6.8 nm and N=65 layer pairs at a wavelength of 12.7 nm. The ML has 63% peak reflectivity at an incidence angle of 71°. In inset, wavelength versus reflectivity scan measured at a Bragg angle of 72.5° is presented. The performance of the structure is comparable with the best achieved without any barrier layer.

We have fabricated C/B$_4$C multilayer for high resolution application near the boron and carbon edge. These multilayers have been tested for thermal stability. The results show that the character of multilayer structure is preserved even after 800 °C annealing. Raman spectroscopy indicates graphitization of carbon layer with increasing annealing temperature. Figure 3 shows Reflectivity of C/B$_4$C multilayer sample measured at various annealing temperature near Boron K edge (6.56 nm) in inset, wavelength versus reflectivity scan measured at a Bragg angle of 72.5° is presented. The performance of the structure is comparable with the best achieved without any barrier layer.

Recently, we have proposed that NbC /Si multilayer could be a good replacement of Mo/Si to be
Fig. 3. Reflectivity of C/B₄C multilayer sample measured at various annealing temperature near Boron K edge (6.56 nm).

used below Si Ledge (100 eV) [8]. This combination is thermally stable and has similar reflectivity as Mo/Si. A 30 layer pair sample of NbC/Si with d= 7.04 nm has given a peak reflectivity of 42.45% at 13.0 nm wavelength as shown in Fig. 4. The reflectivity performance of the same multilayer measured after 600°C annealing for 1h is also shown in the figure. After the annealing due to contraction in multilayer period changes from 7.04 nm to 6.94 nm the Bragg peak shifts towards the lower wavelength side. The multilayer reflectivity is reduced by a small amount from 42.45% to 40%. Reflective of the as deposited mirror is comparable with Mo/Si and after annealing it better than Mo/Si. No signature of silicide is seen at the interface even after annealing as is the case with Mo/Si multilayers.

W/B₄C material combination has been identified as a promising candidate for making X-ray multilayer structure at ultra short period [9]. We had deposited W/B₄C multilayers using magnetron sputtering with period ranging from 3.5 nm to 1.6 nm. Detailed structural characterization was done

Fig. 4. Measured and fitted soft x-ray reflectivity spectra of as deposited and 600 °C annealed sample is shown.

Fig 5. Variation of X-ray reflectivity with multilayer period measured at 8.05 keV

Fig 6. Measured x-ray reflectivities of W/B₄C multilayers with variable bi-layer number at Cu-Kα radiation (8.05 keV).
using hard x-ray reflectivity, grazing incidence x-ray diffraction and cross sectional transmission electron microscopy We analysed the influence of period on the reflectivity of multilayer Figure 5. It was found out that the minimum thickness of W and B$_4$C which can be deposited as continuous layer were 0.9 nm and 1 nm. It was observed that reflectivity of the multilayers with period less that 2 nm drops rapidly from ideal structure. This indicates that 2 nm period is the lowest thickness at which good quality mirror can be deposited. The evolution of interface roughness with increasing bi-layer number, N was also investigated by fabricating multilayer with period 2 nm and bi-layer numbers 20, 40, 100, 200 and 300. It was observed that interfaces were becoming smoother with increasing N and diffused component was reducing with increase in no of layer pairs. Reflectivity of first Bragg peak for sample with 100,200 ad 300 layer pair sample is shown in figure 6. The 300 layer pair, 2 nm period sample has shown 1.2% energy resolution at 8 keV and 60% reflectivity. The achieved reflectivity and resolution are comparable with the best multilayers deposited by magnetron sputtering [10].

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