Characterization of Surface Layer in Mo/Si Multilayer Using Total Electron Yield and X-ray Reflectivity Techniques

Mamta Sinha1,2* and Mohammed H Modi1

1Indus Synchrotrons Utilization Division, Raja Ramanna Centre for Advanced Technology, Indore 452013, India
2Birla Institute of Technology, Mesra, Ranchi-853012, India

Abstract

In x-ray multilayer, the thickness of top layer plays an important role in determining its reflectivity performance. In experimentally grown multilayer, the top layer parameters are found significantly different from those of underneath layers due to growth related issues and contamination effect. The calculations suggest that for top layer characterization the sensitivity of reflectivity technique depends on layer material. Considering the top layer of silicon the first Bragg peak reflectivity of Mo/Si multilayer changes by 2-3% while change in top layer thickness by a factor of two and more. In case of SiO2 as a top layer material the 1st Bragg peak reflectivity changes by 13%. The analysis of total electron yield (TEY) data reveals that the technique can be used to probe 2-4 Å variation in top layer thickness. The both technique-reflectivity and TEY, together gives an complete information of multilayer structural parameters.

Keywords: Soft X-ray; X-ray reflectivity; Total electron yield; Multilayers; Beam line

Introduction

Multilayers have found wide applications in soft X-ray/extreme ultra violet (EUV) lithography, soft X-ray microscopy, synchrotron beamlines and many other technological areas. Structural periodicity ‘d’ of a multilayer can be tailored according to choice of wavelength and therefore its applicability has spread in different regime of wavelengths and angles. Structural parameters play a key role in determining multilayer performances. Well defined periodic structure give rise to multiple Bragg peaks along with Keissig fringes. In the soft x-ray region near Si L-edge the Mo/Si multilayer is considered as one of the efficient mirrors [1]. The reported reflectivity for Mo/Si multilayer is 74% near 130 Å wavelength region for near normal incidence angle.

Due to the reaction with ambient and contamination effect, it is found that the values of top most layer thickness, roughness, and density parameters deviate significantly from those of underneath layer values in the experimentally grown multilayers. If the top layer consists of a low Z material then the calculations suggest that the change in reflectivity of Mo/Si multilayer is about 2-5% with 100 Å and more change in top most layer thickness. In some cases a capping layer on top of multilayer stack is deposited e.g. SiO2, C, SiC, Pd, Rh, and in such cases the change in Bragg peak reflectivity may go up to 20% with top most layer thickness variation [2]. The top layer plays a crucial role in defining the multilayer performance. Its proper characterization is required.

X-ray reflectivity (XRR) technique is used to analyze in depth structural details of a multilayer stack. The technique gives information about thickness, interface roughness and density of a layer structure. The total thickness of a multilayer stack give rises the interference fringes called Keissig fringes. The periodic arrangement of a layer stack gives the Bragg reflection peaks. Any thickness variation in top layer causes a change in amplitude of Keissig fringes which many a times lies under the error bar of source stability. Therefore XRR technique is less sensitive to top layer parameters especially in case of multilayer samples.

Ejima et al. have reported that the TEY technique is more powerful in analyzing a surface layer [3]. In a TEY method, electrons emitting from the surface are measured. Pepper proposed a TEY formalism for thin films and multilayer. Ejima extended this formalism for multilayer analysis [4]. The top layer thickness dependence of TEY technique has been reported using a Mo/Si multilayer [4].

In the present work, structural analysis of a Mo/Si multilayer is carried out using soft X-ray reflectivity (SXR) and TEY method. This comparative study suggests that the SXR method is less sensitive in comparison to that of TEY method. Details of our TEY and SXR analysis of a Mo/Si multilayer is discussed.

Experimental Details

Mo/Si multilayer of 20 layer pair with a periodicity of 66.9 Å and Γ=0.64 is deposited by ion beam sputtering technique on silicon substrate. Angle dependent reflectivity and TEY measurements were carried out for 124.8 Å wavelength at Indus-1 synchrotron source using reflectivity beamline. TEY signal was detected by measuring surface current using Keithley 6514 electrometer. The experimental data of TEY were fitted using MATLAB code based on Ejima model7.

Results and Discussions

SXR data analysis

In order to understand the effect of thickness of top most layer on multilayer performance a detailed calculations are carried out by assuming a multilayer stack terminated by top most layer of different materials. The results of such calculations are shown in the Figure 1 where reflectivity of the 1st Bragg peak is plotted as a function of the thickness of the top most layer in the Mo/Si multilayer (Mo-26 Å/Si-40 Å) ×20. In this calculations, two cases are considered a) top layer of Si and b) top layer of SiO2. From the figure, it is obvious that the first...
Bragg peak reflectivity changes from 57.8% to 55% upon change in the thickness of top Si layer from 0 to 140 Å. If the top layer is assumed to be of SiO₂ then this change in 1st Bragg peak reflectivity is slightly higher as ~13%. This suggests that the effect of top layer thickness on 1st Bragg peak reflectivity depends on top layer materials.

In order to experimentally investigate the effect of top layer thickness on multilayer performance and the sensitivity of SXR technique to the top layer a Mo/Si multilayer (Mo-26 Å/Si-40 Å) × 20 was used and SXR and TEY measurements were performed. The measured and fitted soft x-ray reflectivity curves using 124.8 Å wavelength of this multilayer are shown in the Figure 2. The SXR analyses suggest that the thickness of Si layer is 40 Å and Mo layer is 26 Å. Si-on-Mo interface has roughness in 7-9 Å range whereas for Mo-on-Si interface it is in 10-12 Å range. The two interfaces in Mo/Si multilayer are known to exhibit asymmetric interface behaviour [5]. During the fit of the experimental data the thickness of the top most silicon layer is taken as 40 Å. It is obvious from the Figure 2 that the calculated data are in good agreement with the experiment in the Bragg peak region but it shows a significant deviation in near critical angle region. In order to get a best fit the thickness of the top layer was varied and found a good agreement with the experimental data for the top layer as Si-26 Å + SiO₂-14 Å. In Figure 2, different curves are generated by varying the SiO₂ layer thickness. It comes out that the SXR pattern marginally changes in the near critical angle region upon change in the SiO₂ layer thickness in 10-20 Å range.

It comes out that the SXR sensitivity to top layer thickness is relatively less, of course it is dependent upon layer material.

**TEY data analysis**

To further investigate the multilayer structure a TEY vs. angle spectra of Mo/Si multilayer was measured simultaneously along with the reflectivity data. To fit the TEY spectra a two layer model is used where initial 19 layer pair of Mo/Si multilayer (Mo 26 Å/Si 40 Å)×19 was taken as similar to that one used for obtaining the fit of SXR data. A large mismatch between measured and calculated TEY spectra is found which suggest that the fit parameters obtained from SXR analysis does not hold true for TEY case (Figure 3).

The reason for this difference might be due to the attenuation length. For soft X-rays, TEY intensity is due to integration of high energy photoelectron and low energy secondary electrons produced in cascade, that have a broad kinetic energy distribution peak below ~5 eV [6]. Due to the shape of universal escape depth descape curve (~L) in which descape is minimum near 1 keV and increases exponentially going towards lower energy, so the TEY signal is dominated by low energy electrons in cascade.

As the TEY spectra do not get fitted using the fit parameters obtained from SXR analysis, certain changes in the optical parameters of constituent layers was tried. By changing the optical constants of Si and Mo by factor of 2 the agreement between experimental and calculated data improved. In this refinement process the thickness of topmost layer comprising 14 Å SiO₂ + 26 Å Si was also varied. A best was obtained for 23 Å SiO₂ + 17 Å Si. The fitted graph is shown in Figure 3. Other fit parameters used in TEY analysis are attenuation length 4.5 Å for Mo and 8 Å for Si layer. Transmission rate for all interface are 0.3 except for the top layer. The thickness of Si layer is found as 40 Å and 25.3 Å for molybdenum. Roughness values are 12.1 Å and 7.3 Å for silicon and molybdenum respectively. Topmost SiO₂ layer thickness and the roughness value are found to be 23 Å and 5 Å respectively (Table 1).

|      |  SXR  |  TEY  |
|------|-------|-------|
| d    | Delta | Beta  |
|       |       |       |
| SiO₂ | 14Å   | 0.0096 0.0161 | 23 Å 0.0139 0.0096 |
| Si    | -0.0076 0.0032 | 17 Å -0.0144 0.0022 |
| Mo    | 0.0435 0.0029 | 25.3 Å 0.0292 0.0052 |
| Si    | -0.007 0.0032 | 40 Å -0.0144 0.0022 |

Table 1: Structural and optical parameters obtained from the SXR and TEY fit at 124.8 Å wavelength.
To understand the TEY sensitivity to top layer thickness, different curves are generated by varying the SiO$_2$ layer (topmost layer) thickness. It is found that the shape of standing field pattern generated near 80 deg position changes along with a change in its angular position (Figure 3). This suggests that the TEY is more sensitive to top layer thickness variation than reflectivity technique.

Due to the small attenuation length of electrons, with very low energy below 5 eV for soft x-ray region, only electrons of near-surface of Mo/Si multilayer would be able to reach sample surface. This would result in the TEY intensity where the electrons of near-surface region contributes more. The optical constants and structural properties obtained for near-surface region by TEY can be considered to be more accurate than that of SXR technique, depicting the surface sensitivity of TEY technique.

Conclusions

Structural and optical characterization of Mo/Si soft x-ray multilayer is carried out using SXR and TEY technique. SXR analysis suggests that the multilayer Bragg peak reflectivity changes significantly with top layer parameters, if the top layer is of different materials. The SXR technique is found less sensitive for top layer thickness characterization. The calculation carried out using Ejima model suggests that the TEY method is more sensitive for top layer characterizations.

References

1. Takenaka H, Kawamura T, Haga T, Kinoshita T, Ishii Y (1995) Evaluation of Large-Area Mo/Si Multilayer Soft X-Ray Mirrors Fabricated by RF Magnetron Sputtering. Jpn J Appl Phys 34: 5027.
2. Singh M, Braat J JM (2001) Capping layers for extreme-ultraviolet multilayer interference coatings. Optics Letter 26: 259-261.
3. Ejima T, Harada T, Yamazaki A (2006) Phase change observation in reflection multilayers by total electron yield and reflection spectra. Appl Phys Lett 89: 897-899.
4. Ejima T (2003) Formulæ of Total Electron Yield for Multilayers: Extension of Pepper's Method. Jpn J Appl Phys 42: 6459-6466.
5. Modi MH, Lodha GS, Nayak M, Sinha AK, Nandedkaret RV (2003) Determination of layer structure in Mo/Si multilayers using soft X-ray reflectivity. Physica B 325: 272-280.
6. Ruosi A, Raisch C, Verna A, Werner R, Davidson BA, et al. (2013) Electron sampling depth and saturation effects in perovskite films investigated by soft X-ray absorption spectroscopy. Materials Science 90: 125120.