Investigation of ZrC/Al interfaces in a Al/ZrC/Al/W waveguide-like structure by soft X-ray reflectivity technique

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Soft x-ray reflectivity study of ZrC/Al interfaces by making a Al/ZrC/Al/W waveguide structure

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Abstract

ZrC/Al multilayer is found suitable for soft x-ray/EUV region near the Al L absorption edge. Intermixing of Al at the interfaces is a serious problem in order to achieve the calculated reflectivity performances from an experimentally grown multilayer. In this study our aim is to investigate the ZrC/Al interfaces by making a waveguide structure as Al/ZrC/Al/W. We used soft x-ray reflectivity (SXR) technique to study the x-ray waveguide structure composed of 4 layers on Si substrate. Structural parameters of the stacks, density, thickness and roughness of the layers, are determined through fitting of SXR data.

Introduction

In extreme ultraviolet (EUV) region (30 – 150 eV), a mirror composed of a single layer cannot reflect significant amount of radiation at normal incidence, so it is quite challenging to develop reflective elements with enhanced reflectivity. Multilayer mirrors comprised of alternating layers of low Z/high Z elements which can form stable and smooth interfaces came into existence as a solution to this problem[1]. The atomic numbers (Z) of these materials is alternating in order to enhance scattering length density contrast. The reflected intensity is enhanced by N^2 times in comparison to that of thin film mirror composed of single layer. Since resonance and absorption limitations of most atoms are in the similar EUV range, no effective reflector exists in the literature.

In the recent years, Al–based reflective multilayers are widely used in the extreme ultraviolet (EUV) spectral region[2,3,4]. Al has been found a good spacer layer in the multilayer combination below the Al L- absorption edge (~17 nm) owing its low absorption[4]. Further Al-based multilayers pose severe problem of inter-diffusion and non-homogeneous crystallization of Al etc[4]. In recent years, Zr/Al multilayer has shown enhanced performance in the EUV region and found suitable in solar satellite instruments in EUV region[3]. In Zr/Al multilayers, interfacial roughness is a serious issue and is not found to be constant throughout the multilayer period. Further, it has been reported that the optical performance of Zr/Al multilayer is limited from variable interfacial structure[3]. These Zr/Al multilayers are found to be thermally unstable after 200°C. In a recent study, optical constant contrast between ZrC and Al suggests that ZrC/Al multilayers are better than Zr/Al multilayers[5]. However the performance of actual ZrC/Al interfaces has not been investigated in terms of intermixing.

In the present study we have investigated ZrC/Al interfaces by making an x-ray waveguide of 4 layers Al/ ZrC/ Al/ W on Si substrate. In x-ray waveguide, the electric field of a radiation can be confined inside a layer through the generation of x-ray standing waves. The guiding layer is generally made of a light material surrounded by two layers made of heavy materials. Waveguides can be used to generate a beam of nanometre size or to study a material placed at the maximum of the electric field in order to benefit from a selective and efficient excitation. Sample has been prepared using ion beam sputtering technique. Soft x-ray reflectivity (SXR) measurements are carried out at 6 and 7 nm incident wavelengths at Reflectivity beamline of Indus1-1 synchrotron radiation source.

Experimental Procedure

An x-ray waveguide composed of 4 layers: Al (7nm) / ZrC (5nm) / Al (15nm) / W (50nm) has been prepared on a polished Si (100) wafer using ion beam sputtering technique. Prior to deposition, a base pressure of 3×10^{-5} Pa was achieved. Deposition has been carried out under argon ambient at constant pressure of 6×10^{-2} Pa. Commercially available sputtering target of ZrC, Al and W (99.99% purity) has been used for deposition. Optimized parameters, 1 kV beam voltage and a gas flow rate of 3 standard cubic centimetres per minute for better rms roughness, were used for the deposition.

SXR measurements have been carried out using reflectivity beamline[6]. The experimental station is a UHV reflectometer consists of single axis goniometer to accomplish θ-2θ scans. A soft x-ray silicon photodiode detector (International Radiation Detector Inc.) is used for measuring beam intensity. The reflectivity as a function of the glancing angle scan has been performed at 6 and 7 nm incident wavelengths. SXR data has been analysed using Parratt formalism[7]. Effect of surface roughness has been taken into account using Névot-Croce model[8]. A nonlinear least square refinement routine based on genetic algorithm has been used to refine fitting parameters[9].

Results and discussion

Fig. 1 shows the schematic layout of the waveguide structure used for the present SXR analysis. The indicated thicknesses are the aimed ones during the deposition process. Here ZrC layer of 5 nm thickness is sandwiched in between two Al layers of 7 and 15 nm thickness respectively and whole stack is on 50 nm W layer on Si (100) substrate. The actual thicknesses have been determined by SXR analysis and are discussed in following section.

To obtain more reliable structural parameters, SXR measurements have been carried out at two different wavelengths and same parameters (thicknesses and roughness) are used at both wavelengths for fitting. So measurements are carried out at 6 and 7 nm (below and above Zr M4 edge 6.63 nm). Scattering length density (SLD) (ρ) and imaginary part of scattering length density [Im(ρ)] are calculated from delta (δ) and beta (β), the real and imaginary parts of the optical index respectively, obtained from CXRO website[10]. These calculated ρ and Im(ρ) are used as initial parameters for the data analysis. Fig. 2 and Fig. 3 represents the measured and fitted soft x-ray reflectivity data for the stack at 6 and 7 nm incident wavelengths respectively.

To fit the SXR data, we tried with 4 layers model Al / ZrC / Al / W on Si substrate and varied the structural parameters like thickness, roughness, for the stack, but were unable to match the calculation with the experimental results. Then we introduced in the simulation a native oxide layer of SiO_2 layer on Si substrate and a top surface layer formed due to contamination/oxidation. The best fit for both 6 and 7 nm SXR data were obtained with six layers model: surface layer (6.8nm) / Al (7.9nm) / ZrC (8nm) / Al (15nm) / W (5.7nm) / SiO2 (2.4nm) on Si substrate. For surface layer, ρ and Im (ρ) for Al_2O_3 are taken as initial input. For Si substrate bulk values are used and kept fixed during the fitting procedure.

Fit parameters, thickness, roughness, ρ and Im (ρ), obtained from the SXR analyses are summarized in Table 1. For comparison, bulk values for ρ and Im(ρ) obtained from CXRO website are given in bracket. Fig. 4 shows the SLD profile obtained from fit parameters of SXR data along with the SLD profile of the bulk values for 6 and 7 nm incident wavelengths respectively. To calculate the SLD profile using bulk values, thickness and roughness given in Table 1 are used. Bulk SLD profile in Fig. 4 suggests that density contrast for ZrC is significantly different for 6nm (below Zr M4 edge) and 7 nm (above edge). SLD for W is found to be slightly lower than the bulk value. In Fig. 4 it is clear that the SLD for surface layer is remarkably lower that the Al_2O_3 values, which suggests that the surface layer is not native Al oxide (Al_2O_3) but it is a low density layer formed due to reaction of surface with ambient. Further, ρ and Im(ρ) for ZrC are found higher than the bulk values at both 6 and 7 nm incident wavelengths. This indicates the possibility of intermixing of ZrC/Al interfaces or it may be because of existence of Zr M4 edge at 6.63 nm wavelength. Roughness values are found slightly high (0.5nm to 2.49 nm) and are listed in Table 1 for each interface. Further investigations on intermixing of the stack are going on using other conventional techniques.

Conclusions

In the present study, a waveguide structure of 4 layers Al/ ZrC/ Al/ W on Si substrate. Structural investigations are carried out using SXR technique. Detailed analysis of SXR data using 6 layers model revealed the thickness of the stack to be surface layer (6.8nm) / Al (7.9nm) / ZrC (8nm) / Al (15nm) /W (5.7nm) / SiO_2 (2.4nm) on Si substrate. Roughness of the interfaces is found to be in 0.5nm to 2.49 nm range. SLD for all layers was found to be less than bulk SLD, while for ZrC SLD was found higher than bulk values. This higher value may attribute to the intermixing among the ZrC/Al interfaces.

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Fig. 1 Schematic layout of the waveguide structure sample used for the SXR analysis.

Fig. 2 Measured and fitted SXR data at 6 nm incident wavelength.

Fig. 3 Measured and fitted SXR data at 7 nm incident wavelength.

Fig. 4 Scattering length density (SLD) profile obtained from the fit parameters at 6 nm and 7 nm incident wavelengths.

Table 1: Fit parameters of the stack obtained from the SXR analysis. Values in bracket correspond to bulk values for SLD ρ and Imaginary part of the SLD Im (ρ) obtained from CXRO website. For surface layer bulk values corresponds to Al_2O_3.

	Thickness (nm)	**Roughness (nm)**	**ρ 6 nm**	**Im ρ 6 nm**	**ρ 7 nm**	**Im ρ 7 nm**
Surface layer	6.8	1.89	1.07e-5 (2.64e-5)	9.38e-06 (1.23e-5)	9.89e-6 (2.38e-5)	6.00e-06 (1.49e-5)
Al	7.9	0.63	1.17e-5 (1.73e-5)	1.42e-05 (1.32e-5)	1.14e-5 (1.40e-5)	9.14e-06 (1.59e-5)
ZrC	8	2.45	8.08e-6 (5.12e-6)	1.33e-05 (7.74e-6)	1.15e-5 (1.09e-5)	8.99e-06 (2.47e.6)
Al	15	2.14	1.10e-5 (1.73e-5)	1.31e-05 (1.32e-5)	1.147e-5 (1.40e-5)	8.22e-06 (1.59e-5)
W	57	2.49	2.35e-5 (2.46e-5)	2.52e-05 (3.10e-5)	2.00e-5 (2.19e-5)	1.21e-05 (2.11e-5)
SiO2	2.4	0.96	1.37e-5 (1.37e-5)	6.16e-06 (6.75e-6)	1.285e-5 (1.29e-5)	7.98e-06 (7.67e-6)
Si Substrate	----	0.5	1.26e-5 (1.26e-5)	1.23e-5 (1.23e-5)	1.08e-5 (1.08e-5)	1.35e-5 (1.35e-5)