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Interface analysis of Mg/Sc and Sc/Mg bilayers using X-ray reflectivity

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1 ABSTRACT

Mg/Sc and Sc/Mg bilayers were studied to investigate their interfaces. Time-of-flight secondary ion mass spectroscopy and grazing incidence x-ray reflectivity are combined to determine the realistic structure of the bilayer stack: thickness, refractive index, and roughness of layers and interlayers respectively. In addition, soft x-ray reflectivity measurements provide sensitive information about the chemical environment of scandium, when the incident photon energy is tuned in the vicinity of the Sc L-absorption edge (~ 400 eV). Both Mg-on-Sc and Sc-on-Mg interfaces have high roughness (~1 nm) and a MgSc compound interlayer but are shown to be asymmetric.

Keywords: Grazing incidence x-ray reflectivity, Interfaces, Soft x-ray reflectivity, Time-of-flight secondary ion mass spectroscopy.

1. Introduction

The water window range, located between the absorption edges of carbon (285 eV) and oxygen (530 eV), is particularly suited for imaging biological samples with high optical contrast. Within this range also falls the nitrogen K absorption edge (395 eV) with characteristic $K\alpha$ emission at 393 eV. This emission band is quite intriguing for the design and application of multilayer mirrors as optical components for spectroscopic purposes [1]. Crystal spectrometers when equipped with suitable periodic multilayers designed with periods ranging from few to some tens of nanometers can diffract nitrogen K emission band, which is otherwise impossible to achieve with a crystal owing to the long wavelength to disperse [2–4].

Different Mg/Sc multilayers with and without Co barrier layer have been previously studied for application in this spectral range [5]. Out of those systems, Sc/Co/Mg/Co multilayer displayed promising results with an expected reflectivity of 32% in the nitrogen K edge energy range. However, it was found that strong interdiffusion takes place at the Mg-on-Sc and Sc-on-Co interfaces, leading to limited reflective properties [5]. Given in figure 1(a) and b) is the simulation of a perfect (without roughness or diffusion) $[Mg(3nm)/Sc(3nm)]_{x25}$ and $[Sc(3nm)/Mg(3nm)/ZrC(1nm)]_{x25}$ periodic multilayer systems with more than 50% reflectance at the N K absorption edge of 395 eV. It is thus evident from the simulation that such systems can pose as ideal candidates for application in the N K emission range. However, in reality such perfect systems cannot be achieved as the interdiffusion at interfaces, formation of rough surfaces and even in some cases presence of buried layers hinder the quality and overall optical performance of any multilayer system. Thus, it is a prerequisite to describe precisely the interlayers and have information about the interactions at the surface of the stack, in-between each layer and at the bottom layer and the substrate. Consequently, we now aim to study the bilayer systems made of Mg and Sc to investigate the behaviour of Mg-on-Sc and Sc-on-Mg interface separately.

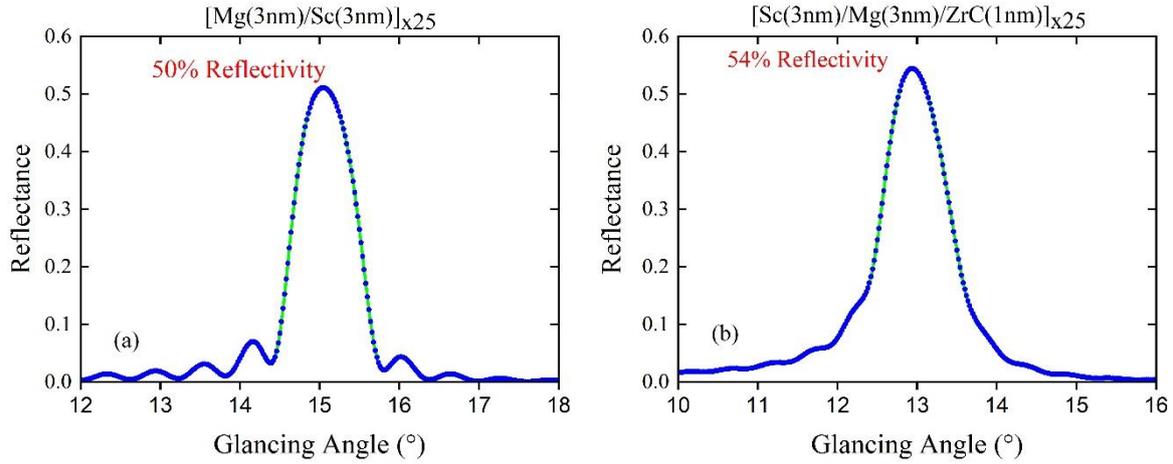


Figure - 1 (a) and (b) show the simulated reflectance calculated for $[Mg(3nm)/Sc(3nm)]_{x25}$ and $[Sc(3nm)/Mg(3nm)/ZrC(1nm)]_{x25}$ multilayer at the incident photon energy of N K edge 395 eV.

Grazing incidence hard x-ray reflectivity (GIXRR), a non-destructive tool, is used to probe the two bilayer structures to deduce structural parameters: thickness, roughness and density of layers and interlayers. In GIXRR, the optical contrast is not sufficient to identify the small compositional gradients at the interface for thin films of low atomic numbers [6,7]. Whereas, in the soft x-ray region, near absorption edges, with a marginal change in the composition, the atomic scattering factors vary drastically, in turn enhancing the optical index contrast [8,9]. In this study, soft x-ray reflectivity (SXR) is employed to record the reflectivity curves at photon energies around the Sc $L_{2,3}$ absorption edge. Furthermore, time of flight secondary ion mass spectroscopy (ToF-SIMS) analysis was also done to understand the nature of different layers and interlayers formed. It gives the distribution of layers of inside the stack and provides information of the possible compounds formed as a result of intermixing between the layers.

2 Experimental

2.1 Sample Preparation

$[Mg(20nm)/Sc(20nm)]/Si$ substrate and $[Sc(20nm)/Mg(20nm)]/ZrC(1.5nm)/Si$ substrate (labelled as Mg/Sc and Sc/Mg respectively in the following) samples were fabricated using ion beam sputtering. Commercially available sputtering targets of Mg, Sc and ZrC with 99.5% purity were used. Deposition chamber was evacuated to 10^{-5} Pa before sputtering. The targets were sputtered by 1 keV Ar ions using Kauffman ion source with 3 sccm flow. Working pressure was maintained in ~ 0.1 Pa range during the deposition. The sputtering targets were pre-sputtered for ~ 10 min to remove surface contamination. Samples were then deposited at a typical deposition rate of 0.5 - 0.7 nm/min for Mg, Sc and ZrC.

2.2 Characterization

Grazing incidence reflectivity measurements using hard x-rays were performed using a table top Bruker D-8 reflectometer equipped with a θ - 2θ goniometer and a Cu $K\alpha$ source (8048 eV). The reflectivity in the soft x-ray region was carried out at the soft x-ray reflectivity beamline at Indus-2 synchrotron source. The beamline operates in the energy region of 100-1500 eV with moderate resolving power ($E/\Delta E \approx 1000$ -6000) and high photon flux of 10^9 - 10^{11} photons/s [10]. The beamline operates under ultra-high vacuum ($< 3 \times 10^{-7}$ Pa). The reflectometer is kept in a high vacuum experimental chamber equipped with a two-axis goniometer and a linear translation stage for sample movement. The reflected beam is detected using a Si photodiode connected to a Keithley electrometer. SXR measurement was done for the [Sc/Mg]/ZrC bilayer by tuning the energy of the incident beam around the Sc L-absorption edge, at energies varying from 370 to 420 eV. The data obtained was then fitted using IMD, a program for modelling optical properties of thin films and multilayers, based on Parratt recursive formalism to obtain the structural and optical parameters of the stack: thickness, roughness, density and real and imaginary parts of the optical index of the layers and interlayers [11,12]. It utilizes nonlinear least squares curve fitting technique based on χ^2 minimization method to fit the reflectivity curves.

To highlight the morphology of interfaces for bilayers time-of-flight secondary ion mass spectroscopy (ToF-SIMS) measurements were performed. The analysis was done using Bi^+ ion gun operating at 30 keV and ~ 2.5 - 3.0 pA. The depth profiling was made with a O_2^+ ion gun operating at 500 eV, ~ 100 nA over an analysis of $100 \mu\text{m} \times 100 \mu\text{m}$. Only positive ions were recorded. ToF-SIMS profiles are recorded by measuring the intensity of each incident ion of a given mass as a function of the sputtering time. It must be noted that the time scale is not relative thickness of each layer and that the sputtering yields of two elements is not related to the difference of concentration of these elements [13].

GIXRR fit of the reflectivity data can result in various possible fitted structures when we take into account different thickness, composition and density of constituting layers and interlayers. Hence, we first use ToF-SIMS to determine a priori information on possible structure, by measuring the distribution of elements as a function of sputter time inside the bilayer stacks. Then, the possible structure obtained from the ToF-SIMS analysis is used to fit the measured GIXRR curves for both bilayers.

In the x-ray region, complex refractive index for matter is given by $n = 1 - \delta + i\beta$, where δ (dispersion coefficient) and β (absorption coefficient) are the optical constants. The SXR curves measured around the Sc $L_{2,3}$ -absorption edge (400 eV), with energies ranging from 370 to 420 eV, are fitted and the values of optical constants δ and β are determined. The calculated values of δ and β for Sc are then plotted as a function of incident energy across the absorption edge. Using the fit values of film thickness, real part of refractive index (δ), and roughness, a depth dependent δ -profile is calculated and plotted as a function of the depth. In case of hard x-ray analyses, the δ -profiles are more analogous to mass density profiles. However, in case of soft x-rays the presence of different edges affects δ value significantly which then does not vary directly with mass density.

3 Results and discussion

Figure 2 shows the ToF-SIMS profiles of Mg^+ , Sc^+ , Si^+ ions as well as C^+ ions obtained on the Mg/Sc bilayer. For sake of clarity, the regions corresponding to Mg and Sc layers and Si substrate are delimited by vertical lines. The progression of Mg and Sc profiles suggests that Mg is diffusing towards the Sc layer, resulting in an interlayer formation. The small increase, between the Sc layer and the substrate could be indicative of an interlayer. Even if there exists no direct relation between the sputtering time and the depth, it takes about four more time to sputter the Sc layer than the Mg one. Knowing that the ratio of the sputtering yields of Mg to Sc by oxygen ion of 1 keV is about 0.5 (it should be the same order of magnitude for 0.5 keV ions), we expect the Sc layer to be at least twice thicker than the layer containing Mg atoms. Carbon is only present at the surface owing to atmospheric contamination.

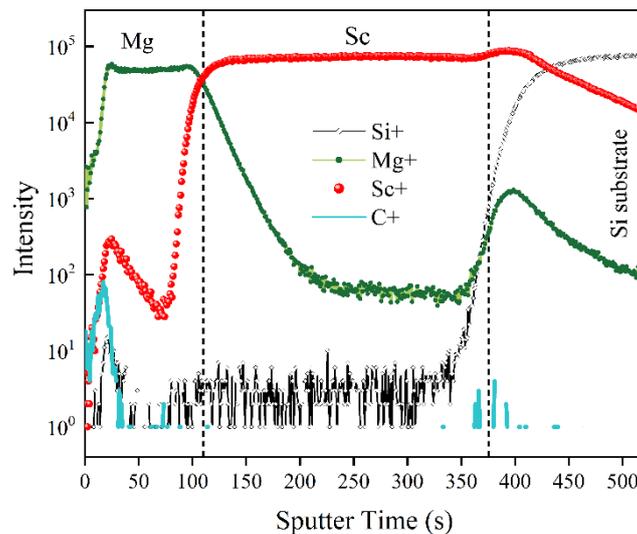


Figure 2 - ToF-SIMS profile for Mg/Sc Bilayer.

Now that we have an idea of possible structure of the Mg/Sc bilayer, this information is implemented to fit the measured GIXRR curve. Figure 3(a) shows measured as well as fitted GIXRR data of the Mg/Sc bilayer. The best fit was obtained using a four-layer model consisting of a top layer due to surface oxidation (MgO), an MgSc layer, the nominal Sc layer and an interlayer at the scandium and silicon substrate interface. Table 1 collects the structural parameters of the stack deduced from the fit. From these parameters, the δ -profile is plotted in Figure 3(b). The thickness of the layers containing Mg atoms (4.5 nm) is much lower than the aimed thickness (20 nm), whereas the Sc thickness (17 nm) is a little lower than the aimed one (20 nm). This could come from a problem in the calibration of the Mg and Sc deposition rates or of a low sticking coefficient of magnesium atoms on scandium.

The formation of the interface layer on Mg-on-Sc interface can be understood by the value of the mixing enthalpy of Mg and Sc. It is found that the maximum values of enthalpy calculated from semi-empirical Miedena model [14] for Mg-Sc is -5 kJ/mol. This negative value means that the interface Mg-on-Sc is prone to interdiffusion and a mixed region is likely to be formed as a result of diffusion of Mg atoms into the Sc layer during the deposition. The Sc layer is mostly uniform with a density close to that of the bulk. An increase in the δ -profile is seen at the Sc-on-Si interface. The calculated delta value of the interlayer (16.76×10^{-6}), Table 1, suggests that the interlayer formed at the Sc-on-Si interface could either be an oxide of scandium, Sc_xO_y (10.50×10^{-6}) or a scandium silicide ($ScSi$ 10.50×10^{-6} ; Sc_3Si_5 10.69×10^{-6}). But since the fitted δ value of the interlayer is higher than those of Sc_xO_y , $ScSi$ and Sc_3Si_5 , we suggest that the interlayer is rather a mix of $ScSi$ interlayer with the presence of O atoms resulting into a higher density [15,16]. The source of oxygen is the native oxide layer naturally formed of top of the silicon substrate.

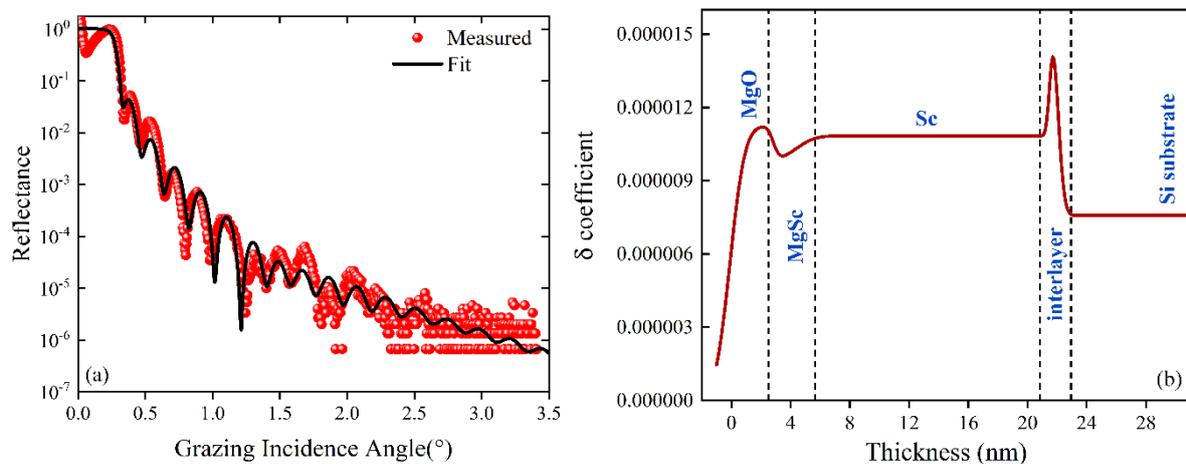


Figure 3 - For the Mg/Sc bilayer: (a) experimental and fitted GIXRR curves; (b) in-depth δ -profile deduced from the structure used to fit the GIXRR curve.

Table 1 - Structural and optical parameters of the Mg/Sc bilayer, where z and σ are the thickness and roughness of each layer respectively; δ and β are the dispersion and absorption coefficients. The thicknesses and roughness's are given ± 0.1 nm.

Layer	z (nm)	σ (nm)	δ ($\times 10^{-6}$)	β ($\times 10^{-7}$)
MgO	2.93	0.80	11.24	1.23
MgSc	1.54	0.32	9.79	3.95
Sc	17.15	1.00	10.84	6.76
Interlayer	0.51	0.20	16.76	5.95
Si substrate	-	0.35	7.58	1.73

Figure 4, shows the ToF-SIMS profile of Mg^+ , Sc^+ , Zr^+ , Si^+ ions as well as C^+ ions of Sc/Mg bilayer where the different layers are separated delimited by vertical dotted lines. As in the Mg/Sc sample the diffuse transition of the profiles between the Sc and Mg layers suggests the existence of an interlayer at the Sc-on-Mg interface. Owing to the very small thickness of the ZrC layer, it is difficult to know if the shoulder observed on the Mg profile around 360 s could evidence the presence of an interlayer between Mg and ZrC layers or is due a transient sputtering regime. Carbon is only present at the surface owing to atmospheric contamination and is not enough intense to be observed from the ZrC layer.

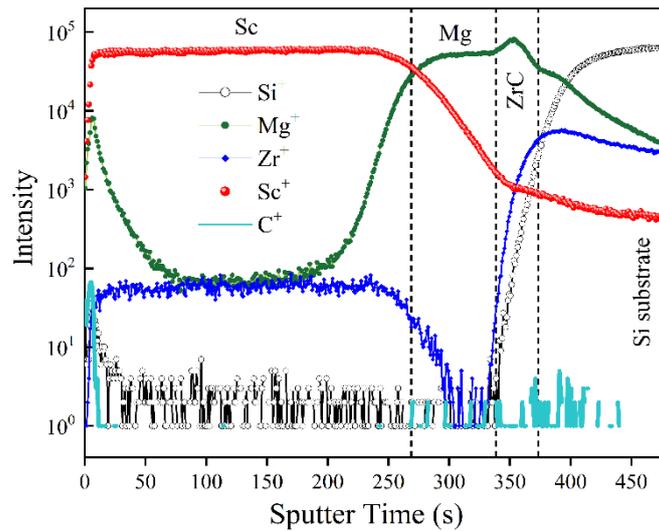


Figure 4 - ToF-SIMS profile of Sc/Mg bilayer.

Similarly, here, the ToF-SIMS analysis result is applied to find a structure suitable to fit the Sc/Mg GIXRR curve. The fitted and measured curves are shown in Figure 5(a). The fitted

structure is a six-layer model, that is to say in addition to the nominal layers of Sc, Mg and ZrC, a top layer due to surface oxidation (Sc_2O_3), an interfacial layer (MgSc) at the Sc-on-Mg interface and a native oxide layer (SiO_2) on top of the silicon substrate have to be introduced. SiO_2 layer is added due to possible oxidation of the Si substrate. Thickness and roughness of different layers along with their δ and β values for each layer as extracted from the GIXRR analysis are given in Table 2. The fitted optical constants for each of the nominal layers are close to their bulk densities. The delta δ -profile deduced from the chosen structure of the parameters of Table 2 is shown in Figure 5(b).

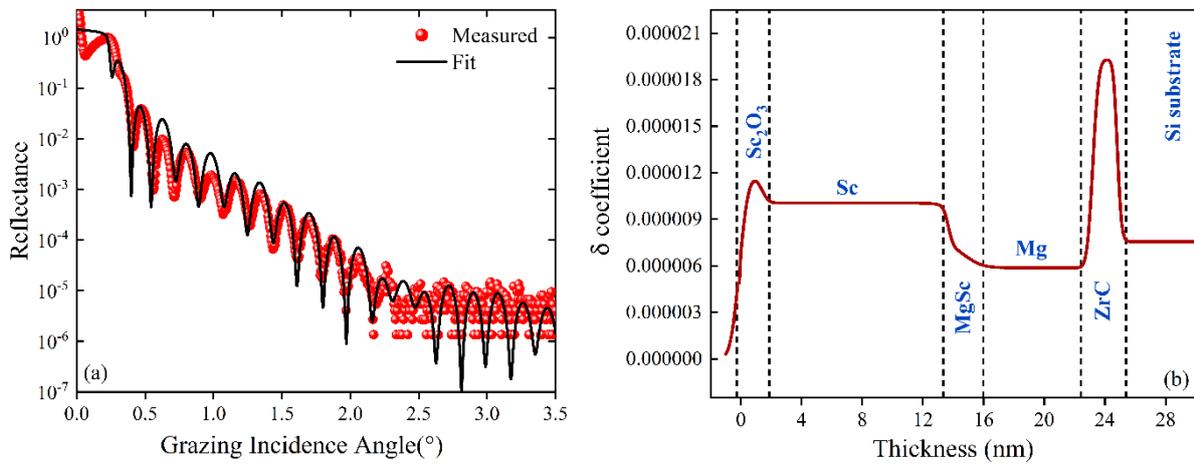


Figure 5 - For the Sc/Mg bilayer: (a) experimental and fitted GIXRR curves; (b) in-depth δ -profile deduced from the structure used to fit the GIXRR curve.

Table 2 - Structural and optical parameters of the Sc/Mg bilayer, where z is thickness of each layer, σ being the roughness of each layer δ and β are the dispersion and absorption coefficients. The thicknesses and roughness's are given ± 0.1 nm.

Layer	z (nm)	σ (nm)	δ ($\times 10^{-6}$)	β ($\times 10^{-7}$)	δ ($\times 10^{-6}$) (Bulk)	β ($\times 10^{-7}$) (Bulk)
Sc_2O_3	1.47	0.46	11.81	5.85	11.99	5.95
Sc	12.3	0.36	10.05	7.52	9.10	6.85
MgSc	0.94	0.25	7.67	4.09	7.67	4.09
Mg	8.59	1.00	5.88	0.87	5.58	0.83
ZrC	1.6	0.30	19.29	9.64	19.16	9.57
SiO_2	0.24	0.20	8.71	1.13	7.13	0.92
Si substrate	-	0.25	7.58	1.73	7.58	1.73

As for the previous sample, the thickness of layers containing Mg atoms as well as the layers containing Sc atoms are smaller than the aimed designed ones. On the contrary the ZrC layer thickness is close to the aimed one. The large thickness of the Sc layer prevents the underneath Mg layer from oxidation. The thickness of the MgSc interlayer is limited to 0.9 nm in the Sc/Mg bilayer while it is 1.5 nm in the Mg/Sc bilayer. This shows that Mg-on-Sc and Sc-on-Mg interfaces are asymmetric. A shoulder of native oxide of SiO₂ is expected to be observed at the bottom of ZrC profile, but since the fitted thickness of SiO₂ is very small (0.24 nm) it is difficult to see SiO₂ on the δ -profile.

Near the absorption edge, refractive index is sensitive to the chemical environment, so any change in local environment of the atom will be reciprocated by the change in its optical parameters [7,17,18]. Thus, SXR technique can give elemental sensitive information, by tuning the energy of x-rays to the absorption edges of Sc [19,20]. The SXR fitted and measured curves for Sc/Mg bilayer, as function of incident angle at different photon energies around the Sc L absorption edge ranging from 370 to 420 eV are shown in Figure 6.

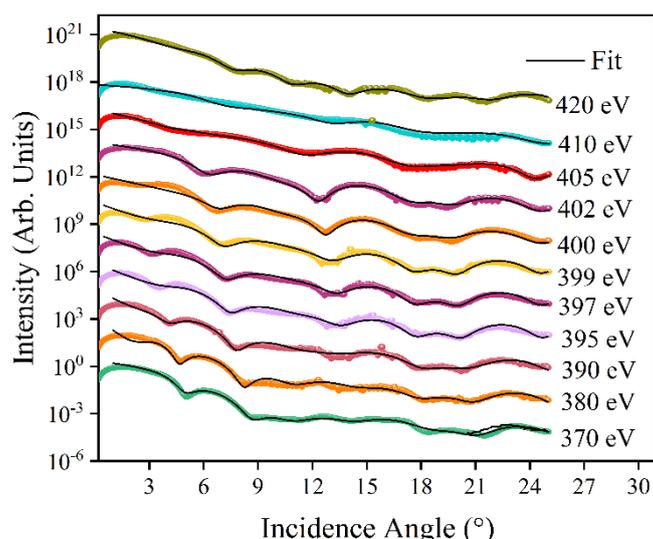


Figure 6 – Measured (color lines) and fitted (black solid lines) angle-dependent SXR curves at energies across the Sc L_{2,3} absorption edge.

The Reflectivity curve measured for the Sc/Mg bilayer at 8048eV is fitted to determine the complete structure of the stack, i.e. the thickness, roughness and density of each layer and interlayer formed. The SXR curves measured around the Sc L-edge ranging from 370 to 420 eV are then fitted using the structure deduced from the former analysis. The curves are fitted only by changing the refractive index i.e; δ (imaginary part) and β (real part). If we introduce other parameters such as the thickness or roughness, the fitted structure will change for each measured energy. Hence, it will no longer be possible to draw a fair comparison on the variation

of refractive index at different photon energies around the absorption edge. The calculated δ and β values of the Sc layer are plotted in Figure 7 (a) and (b) in comparison to the tabulated values as and CXRO values obtained from online database [21,22]. Close to the Sc edge (400 eV) the anomalous behaviour is evidenced by the negative values of the δ parameter. The β parameter is low before the edge and then increases sharply owing the empty Sc d states. A scale difference for both δ and β curves is observed in comparison with CXRO database at Sc- $L_{2,3}$ edge. At lower energies below the absorption edge, the δ values of Sc are similar to those of CXRO.

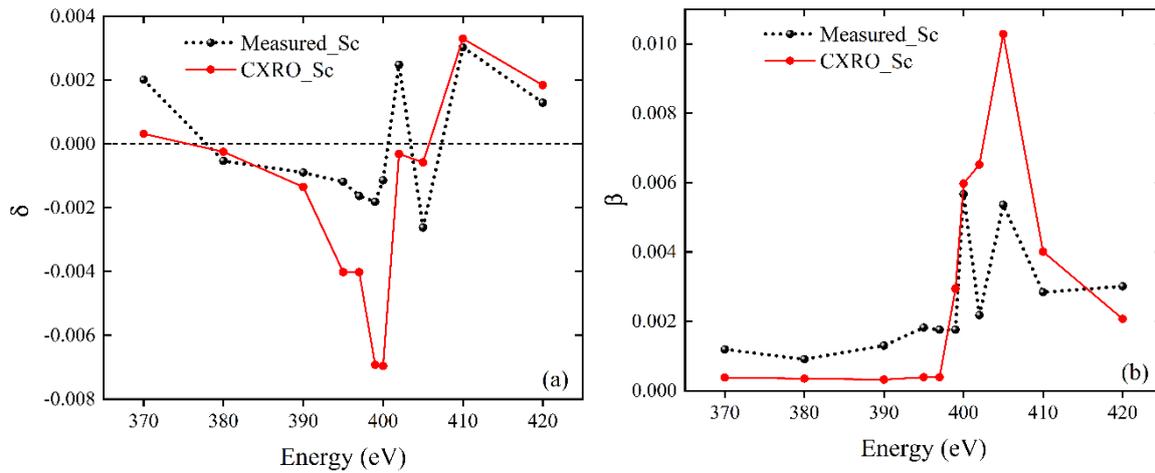


Figure 7 - Dispersion (δ) (a) and absorption (β) (b) coefficients of Sc layer for photon energies in the vicinity of the Sc $L_{2,3}$ edge extracted from the fit of SXR curves and compared to CXRO tabulated values.

4 Conclusion

Mg/Sc and Sc/Mg bilayers were characterized by GIXRR and SXR measurements and fitting. ToF-SIMS was useful as a first step in the analysis to limit the number of possible structures used in the process of fitting reflectivity curves. Both samples are oxidized at their surface, the magnesium oxide being about twice thick as the scandium oxide. An intermixing between the Mg and Sc layers takes place with an asymmetric behaviour between the Sc-on-Mg and Mg-on-Sc interfaces. This confirms the need to insert barrier layers at the interfaces of Mg/Sc stacks to develop periodic multilayers as efficient dispersive elements for the water window range.

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List of figure captions:

1. Figure 1 - (a) and (b) show the simulated reflectance calculated for $[\text{Mg}(3\text{nm})/\text{Sc}(3\text{nm})]_{\times 25}$ and $[\text{Sc}(3\text{nm})/\text{Mg}(3\text{nm})/\text{ZrC}(1\text{nm})]_{\times 25}$ multilayer at the incident photon energy of N K absorption edge at 395 eV.
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3. Figure 3 - For the Mg/Sc bilayer: (a) experimental and fitted GIXRR curves; (b) in-depth δ -profile deduced from the structure used to fit the GIXRR curve.
4. Figure 4 - ToF-SIMS profile of Sc/Mg bilayer.
5. Figure 5 - For the Sc/Mg bilayer: (a) experimental and fitted GIXRR curves; (b) in-depth δ -profile deduced from the structure used to fit the GIXRR curve.
6. Figure 6 – Measured (color lines) and fitted (black solid lines) angle-dependent SXR curves at energies across the Sc $L_{2,3}$ absorption edge.
7. Figure 7 - Dispersion (δ) (a) and absorption (β) (b) coefficients of Sc layer for photon energies in the vicinity of the Sc $L_{2,3}$ edge extracted from the fit of SXR curves and compared to CXRO tabulated values.

List of table captions:

1. Table 3 - Structural and optical parameters of the Mg/Sc bilayer, where z and σ are the thickness and roughness of each layer respectively; δ and β are the dispersion and absorption coefficients. The thicknesses and roughness's are given ± 0.1 nm.
2. Table 4 - Structural and optical parameters of the Sc/Mg bilayer, where z is thickness of each layer, σ being the roughness of each layer δ and β are the dispersion and absorption coefficients. The thicknesses and roughness's are given ± 0.1 nm.