A.6: Study of off-stoichiometric aluminum oxide thin film using soft x-ray reflectivity technique

Soft x-ray reflectivity (SXR) is an important non-destructive tool to probe thickness, surface / interface roughness and optical index profile in thin film structures. In the soft x-ray region, the contrast in optical constants is sufficiently high for thin films of low atomic number elements to identify the small compositional gradients at the interfaces. Near the absorption edge, the energy-dependent atomic scattering factor gives a resonant behaviour and offers an opportunity to enhance the optical index contrast among low atomic number materials. The resonant behaviour can be observed by tuning the energy of x-rays to the absorption edges of specific elements under the investigation. Resonant soft x-ray reflectivity can be used for the characterization of interlayer composition in Low Z/Low Z thin film structures. In the present study, a sputter deposited aluminium oxide thin film is used to determine optical index profile in the 60-200 Å wavelengths region using angle dependent reflectance experiments. Reflectance measurements are performed using soft x-ray reflectivity beamline at Indus synchrotron source. Optical index profile derived over extended wavelength region is used to obtain depth graded compositional details of the alumina thin film.

In Fig. A.6.1, the experimentally obtained δ values of 240 Å thick aluminum oxide thin film are shown by filled circles. The δ values are obtained by best fit of the reflectivity vs angle curves for different incident wavelengths. In the SXR analysis it is assumed that the alumina film is comprised of a three layer structure i.e. an interfacial layer near the Si substrate, a principal aluminum oxide layer and a top surface layer arising due to interaction of the surface with the ambient. Grazing incidence x-ray reflectivity (GIXR) measurements at = 1.54Å wavelength as shown in Fig A.6.2 are used to determine the thickness of interfacial layer, principal aluminum oxide layer and top surface layer as 30 Å, 240 Å, and 18 Å respectively. In the inset of the figure, schematic of the layer model used for reflectivity data analysis is shown.

The XPS measurements are carried out near O 1s and Al 2s core level to confirm the off stoichiometric nature of the aluminium oxide film. The Al 2s and O 1s core level spectra suggest that the AlO_x and Al_2O_3 phases are present. The O/Al ratio as calculated from the peaks corresponding to Al_2O_3 phase is found to be 1.52. Similarly, the value of x is calculated from the ratio of the area corresponding to the AlO_x peak in O 1s and Al 2s core level and is found to be 1.97.

Optical index values as obtained from the analysis of R vs θ curves at different photon energies are shown in Fig. A.6.1. For the sake of the comparison the optical index profile of Al_2O_3 are plotted along with optical index profile of $AlO_{1.6}$. From the figure it is evident that the experimental profile lies in between that of the Al_2O_3 and $AlO_{1.6}$ profiles. For the optical modeling we have considered the film to be comprised of two phases as $AlO_{1.6}$ and Al_2O_3. To obtain a best fit for experimental profile the fractional composition of these two phases are considered and it is found that for 50% of AlO_x (x=1.6) and Al_2O_3 contribution the experimental profile gets a best fit. This exercise has been carried out for various AlO_x + Al_2O_3 combination but the best fit is found for $AlO_{1.6}$+ Al_2O_3 combination

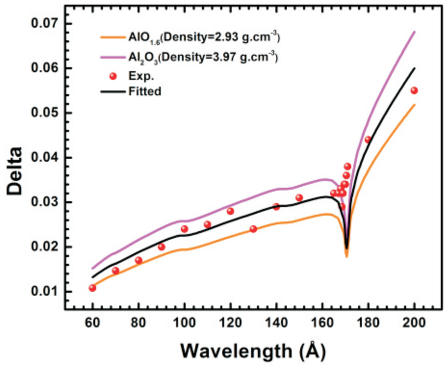

Fig. A.6.1: Optical index profile (delta) for AlO_x (x=1.6), Al_2O_3 are calculated for 2.93 g/cm³ and 3.97 g/cm³ density respectively and compared with the experimentally measured profile of aluminum oxide thin film. The combination of 50% AlO_x (x=1.6) + 50% of Al_2O_3 gives best fit to the experimental data.

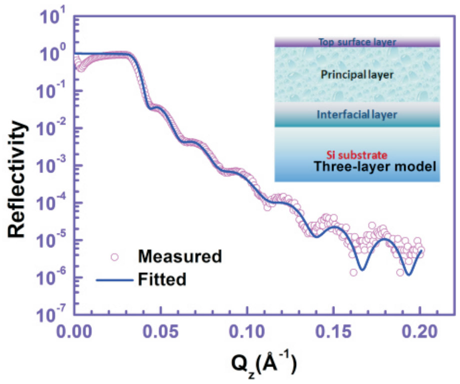

Fig. A.6.2: Measured (open circles) and fitted (continuous line) GIXR spectra of aluminium oxide thin film is shown. Schematic of three-layer model used for the fitting is also shown.

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