A.17: Micro-structural analysis of nanolayered materials at BL-16 beamline

X-ray reflectivity (XRR) is a well-established non-destructive tool for the micro-structural characterization of thin layered materials. The XRR technique allows a user to evaluate micro-structural parameters such as thickness, the surface and inter-face roughness, and density variation in the thin film medium extending from few nanometers to some hundreds of nanometers, with angstrom resolution. The XRR technique however has the limitation that it does not provide any element specific information.

X-ray fluorescence (XRF) spectrometry is another non-destructive technique widely used for the trace elements analysis. By measuring x-ray fluorescence intensities from a thin film structure at grazing incidence angles, the elemental depth sensitivity inside the thin film medium can be enlarged to nanometer regime. The XRR technique, in combination with the grazing incidence XRF (GIXRF) measurements, has shown to be a very powerful probe for the non-destructive micro-structural evaluation of the thin layered materials. The combined analysis approach offers determination of consistent micro-structural parameters such as thickness, roughness, density variations, and interface diffusion, for a thin film system with improved accuracies.

The BL-16 beamline on the Indus-2 synchrotron facility was initially set up with a primary aim of trace elements analysis and micro-fluorescence mapping applications of environmental, archaeological, biomedical, and material science specimens involving heavy metal speciation and their localization.

Considering the numerous advantages of the synchrotron radiation (SR) based advanced spectroscopy methods in the area of surface condensed matter physics, and to meet the requirements of BL-16 beamline users of Indian universities and various research laboratories, an in-house developed x-ray reflectometer station has been set up on the BL-16 beamline, with capabilities to perform simultaneous XRR and GIXRF measurements of the thin layered structures. The combined XRR-GIXRF analysis feature of the BL-16 reflectometer offers a novel capability to perform GIXRF assisted depth resolved x-ray absorption fine structure (XAFS) studies to investigate the chemical state and electronic structure of the thin nano-layered materials. The new XRR-GIXRF experimental station has been equipped in a manner that allows BL-16 users to perform XRR and/or GIXRF measurements of the thin layered materials independently with a minimal setup time and without affecting operations of other installed beamline experimental stations.

Figures A.17.1 and A.17.2 show x-ray reflectivity and GIXRF measurements for a few representative multilayer structures; trilayer (C/W/SiO_2/Si substrate) and a W/B_4C periodic multilayer (number of periods: 15) structure using the BL-16 x-ray reflectometer station. A good agreement was realized between measured and theoretical computed results.

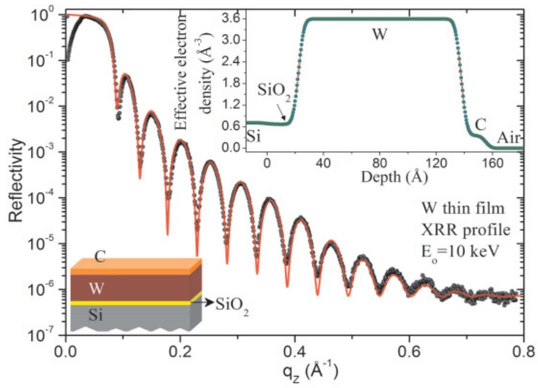

Fig. A.17.1: XRR profile of the W thin film structure measured at 10 keV x-ray energy. In the inset, computed effective electron density profile of the W layer medium is shown. A schematic diagram of the W thin film on a Si substrate is also shown.

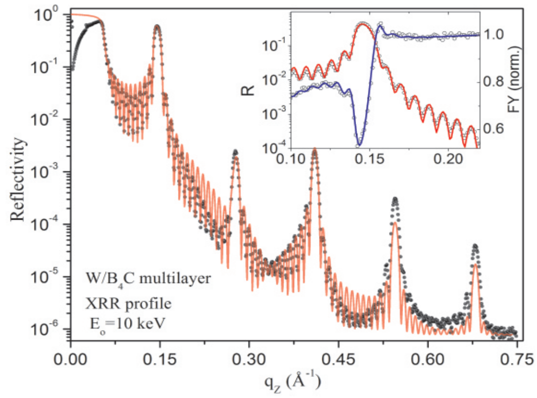

Fig. A.17.2: Measured XRR pattern of a W/B_4C multilayer structure at 10 keV x-ray energy using BL-16 reflectometer station. The inset shows combined XRR-GIXRF measurements carried out for the same multilayer structure at 12 keV x-ray energy.

To summarize, a grazing incidence x-ray reflectometer station has been designed, installed, and tested successfully for use on the BL-16 microfocus beamline of Indus-2 and is available for the users' applications. For more details, please refer to *G. Das et al. Rev. Sci. Instrum. 86, 055102, (2015)*.

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