Application Report XRD 33

D8 DISCOVER Plus

● X-Ray Reflectometry on Thin Films and Superlattices

The D8 DISCOVER Plus equipped with the ATLAS™ goniometer and the high-efficiency turbo X-ray source (TXS-HE) is a diffraction solution designed to meet current and future analytical needs in research and production. In this report, its capabilities for the analysis of thin films using X-ray reflectometry are presented.

The physical properties of surfaces, ultra-thin films and multilayer coatings are of paramount importance in modern technology. X-ray reflectivity (XRR) is a unique analysis technique for the non-destructive and calibration-free investigation of the structural properties of thin films down to the sub-nanometer scale. These properties include layer thickness, roughness, mass density and chemical composition regardless whether the films are amorphous, liquid, polycrystalline or epitaxial.

For detailed XRR analysis of thin film samples, the reflected intensity needs to be measured over the largest possible angular range to achieve the optimum spatial resolution in the density profile. To maximize the accessible range, the instrumental setup requires a high-intensity X-ray beam, reduced air-scattering and a detector with high dynamic range and low background noise.

Innovation with Integrity

A D8 DISCOVER Plus with Cu TXS-HE was used to perform XRR measurements. The diffractometer was configured with a Goebel mirror, 0.2 mm slit and an Eulerian cradle using a vacuum chuck with integral knife-edge collimator to minimize air scatter. The secondary side consisted of an antiscattering-slit and a LYNXEYE XE detector. Two thin-film samples were measured.

The first sample was a glass substrate with a 15 nm film of RuO_2. Data were collected from 0 - 14° 2θ. The total measurement range was split into subregions with variable step size and counting time to reduce the overall measurement time. Total measurement time was less than 15 minutes due to the high intensity beam from the TXS-HE and covered more than 9 order of magnitude in intensity. The XRR data were analyzed using DIFFRAC. XRR. A direct Fourier analysis of the measurement estimated the thickness of the RuO_2 film to be 15.8 nm. With this starting value and the assumption of ultra-thin layers beneath and on top of the RuO_2 film, a three layer model was created and fit to the measured data (see Figure 1). The obtained sample model is presented in Table 1.

The second sample consisted of 20 repetitions of a (WSi_2/C) bilayer deposited on a Si substrate. The reflectivity was measured up to 20° 2θ with a step size of 0.01°. As before, the measurement range was performed with variable counting time to account for the strong decay of the XRR signal. The complete data set was measured in about 20 minutes - spanning over more than 8 orders of magnitude in intensity. For the analysis of the WSi_2/C superlattice, the Fourier transformation directly displayed an average period of the WSi_2/C bilayer to be ≈11.2 nm. Imposing this constraint to the sample model and adding a native SiO_2 layer at the substrate resulted in a good fit to the data (Figure 2). Minimal asymmetry of the superlattice peaks at high angles indicates that there are no significant variations of the superlattice period – an evidence of the high quality of the deposition process. The thickness of the WSi_2 and the C layers was determined with a precision better than 0.01 nm. This precision is achievable because of the enhanced measurement range accessible with the high intensity of the TXS-HE.
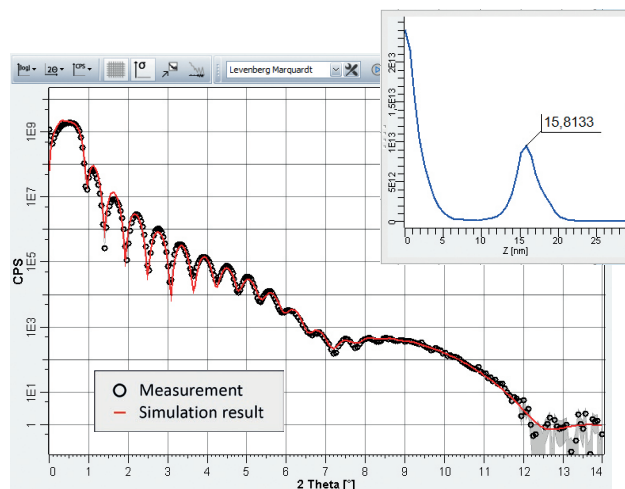

Figure 1: Push button FFT thickness estimation and model based fitting results of the RuO_2 film using DIFFRAC.XRR.

Layer	Material	Thickness [nm]	Density [g/cm³]	Roughness [nm]
Layer	RuO_2	1,77	1,28	0,193
Layer	RuO_2	14,85	6,97	0,288
Layer	SiO_2	0,68	2,65	0,453
Substrate	Si	∞	2,32	1,078

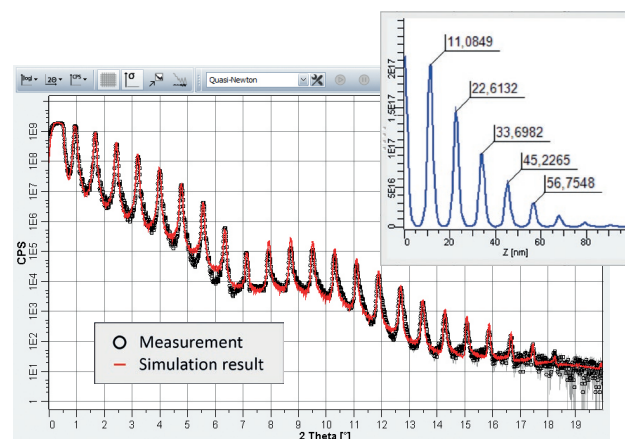Table 1: Sample structure of the RuO_2/Si sample as obtained by DIFFRAC.XRR.

Figure 2: Push button FFT thickness estimation and model based fitting results of the superlattice sample using DIFFRAC.XRR.

Repetition	Material	Thickness [nm]	Density [g/cm³]	Roughness [nm]
10	C	10,010	2,21	0,277
10	WSi_2	1,148	15,02	0,153
1	SiO_2	2,923	1,95	0,198
1	Si	∞	2,32	0,302

Table 2: Sample structure of the superlattice sample as obtained by DIFFRAC.XRR.

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