X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD X-ray Reflectometry

Application Report 45

X-rays have a wavelength on the order of a fraction of a nanometer, making them very useful for the interferential analysis of films in the sub-nanometer to 100 - nanometer range. In an X-ray Reflectometry (XRR) experiment the x-ray beam illuminates the sample surface at a very low angle of incidence. At angles near zero, the beam is totally reflected by the surface resulting in a plateau of signal. From a material-specific angle, the incident beam penetrates the surface. This value is known as the critical angle and is proportional to the $\sqrt{\rho}$ of the material. Once the beam penetrates, internal layer-interfaces are illuminated, resulting in additional critical angle effects and reflections. The internal reflections as well as the surface reflection produce an interference pattern in the specular signal. Fringe spacing is related to the thickness of layers in the coating while signal persistence is related to the roughness of the interfacial layers.

In this application note we demonstrate XRR analysis with the D6 PHASER on a thin tungsten film.

For XRR, the recommended configuration is the advanced primary optic to create a thin, parallel beam, and the universal stage for adjustment of the sample surface normal and positioning in the center of the instrument. Samples can be placed on the spring stage holder or held fast with the vacuum finger. Any LYNXEYE Family detector used in high count rate mode is sufficient for analysis. The telescopic slit shaft allows positioning of a slit close to the sample, increasing resolution while also lowering background.

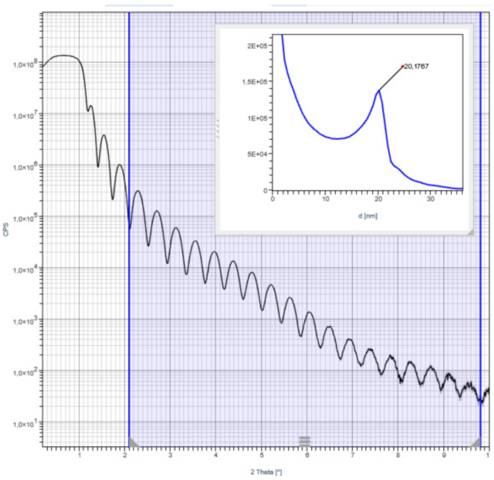


Figure 1

FFT analysis in DIFFRAC.XRR of the W thin film.

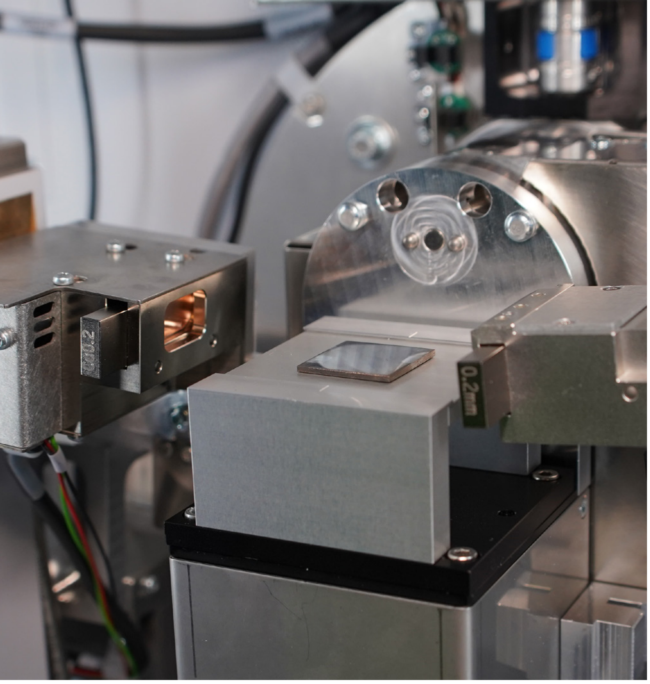
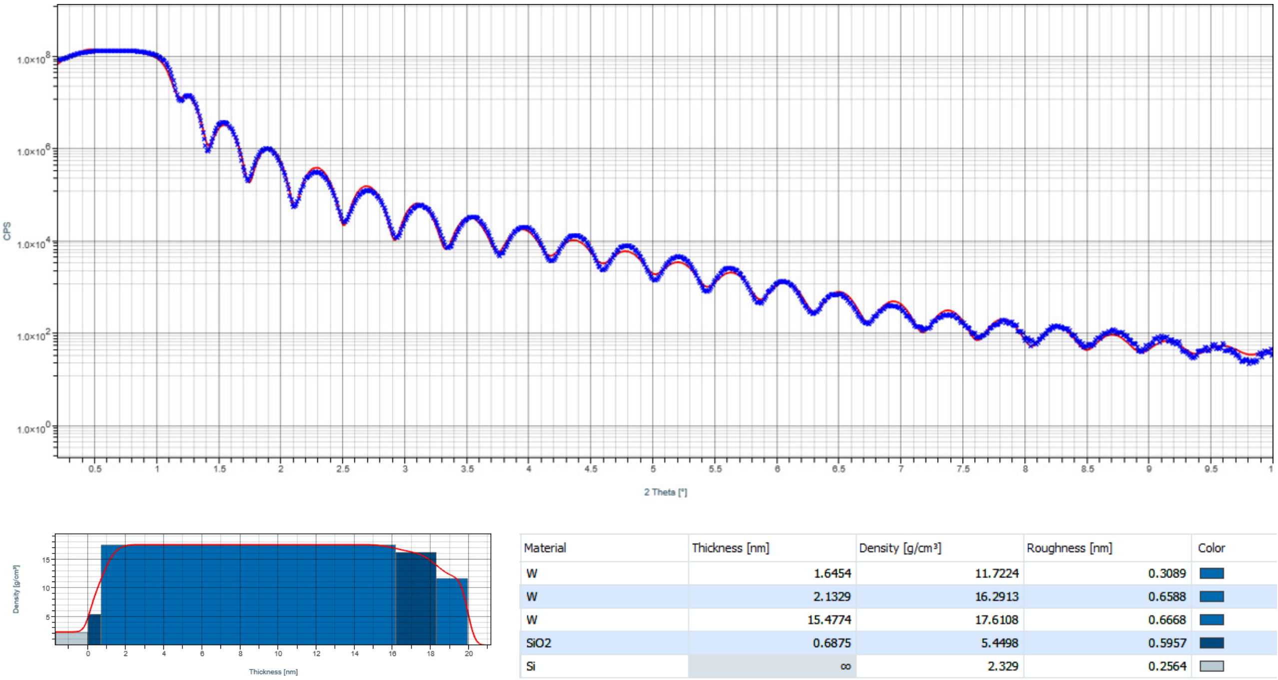


Figure 2
Measurement geometry for X-ray reflectometry in the D6 PHASER.

Measurements are carried out over short angular ranges with 0.2, 0.1, and no copper absorber. The use of absorbers is required due to the large dynamic range of the measurement. Signal in the D6 PHASER is significantly enhanced compared to conventional XRD instruments by the small measurement circle giving long fringe persistence and measurement time on the order of minutes.

The measured data are imported into DIFFRAC.XRR where they are merged into a single data set. A quick estimate of the film thickness can be found using the FFT tool. This analysis transforms the scale of the data yielding peaks at thicknesses corresponding to the periodicity of the fringe pattern (Figure 1). In this case, a single peak at 21.2 nm is observed.
To dig deeper into the data, fitting can be performed (Figure 2). The sample is quickly constructed using the materials database, then regression against the data is performed. Here, it was found that in addition to the substrate and film, interfacial layers exist between the Si and W and at the W surface.

The D6 PHASER and DIFFRAC.XRR make a powerful pair for tackling thin film analysis.



Material	Thickness [nm]	Density [g/cm³]	Roughness [nm]	Color
W	1.6454	11.7224	0.3089	🟦
W	2.1329	16.2913	0.6588	🟦
W	15.4774	17.6108	0.6668	🟦
SiO2	0.6875	5.4498	0.5957	🟦
Si	∞	2.329	0.2564	⬜

Figure 3
Fitting analysis of the W film data in DIFFRAC.XRR. The sample was constructed using the materials database then regression was performed. Additional layers were added to fit the density profile of the film.

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