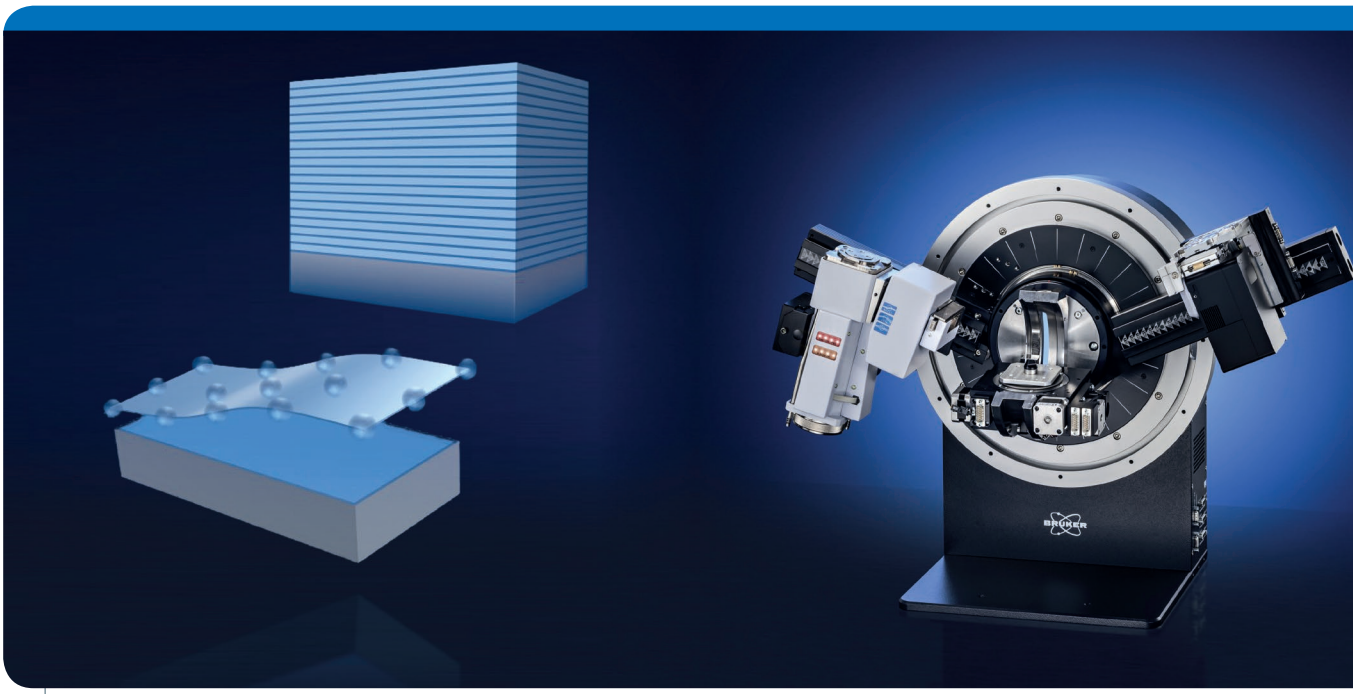


Application Note XRD 619

X-Ray Reflectometry with the EIGER2

- Investigation of a Ta coating on Si and a GaN/AlN superlattice

X-ray Reflectometry (XRR) is a fast, non-destructive way to measure thickness, roughness and density of thin film coatings, multilayers and superlattices. This information can be extracted from the specular interference pattern and allows characterization of crystalline and amorphous films. In this report we examine two typical examples: a Ta coating on Si and a complex GaN/AlN superlattice.

Report Summary

- Absorber-free data collection with EIGER2 allows regression of fine details in layer structures
- A Ta coating on Si was characterized revealing multiple interfacial oxide and contamination layers
- Analysis of a GaN/AlN superlattice revealed subtle variations in sub-period layer density and thickness

All materials exhibit total reflection of X-rays incident below a certain critical angle, giving a reflected intensity nearly as great as the direct beam. As the incident angle is increased when measuring XRR, penetration into the material causes the reflected intensity to drop precipitously. XRR data can therefore span many orders of magnitude. To avoid detector saturation, data is typically acquired as several consecutive scans using absorbers of varying thicknesses; however, with the EIGER2 R 500K the entire scan may be obtained in a single range without absorbers, recording accurate intensities over the full dynamic range.

Experimental

Data were collected on a D8 DISCOVER diffractometer equipped with a Cu sealed tube (40 kV/40 mA), Göbel mirror, fixed primary and secondary slits, Centric Eulerian Cradle, and EIGER2 R 500K detector operating in 0D mode.

Figure 1 shows the improvement in statistical error that can be achieved by collecting XRR data in a single scan (blue) as opposed to breaking the scan into pieces using different absorbers and then merging (red). Both curves, with and without absorbers, show the same overall intensities due to exceptional linearity of the EIGER2 over a large dynamic range.

Evaporated Ta coating on Si

The first sample to be measured was a Ta coating on Si, prepared by evaporation in vacuum. An $\omega/2\theta$ coupled scan was performed from 0.1° to 12° 2θ, with a 0.01° increment and a total scan time of 10 min. Primary and secondary slits were 0.2 mm, and the detector aperture was 0.225 mm.

Data were brought into DIFFRAC.XRR for fitting. In addition to Ta and Si, native oxide layers SiO_2 and Ta_2O_5 as well as a surface layer of amorphous C were identified. An excellent fit was obtained by not only accounting for the Ta film and oxide layers, but also the asymmetric interfacial density profiles. The resulting Ta film thickness was 8.7 nm, with a 2.8 nm Ta_2O_3 native oxide layer on top. The fit result and depth profile are shown in Figure 2.

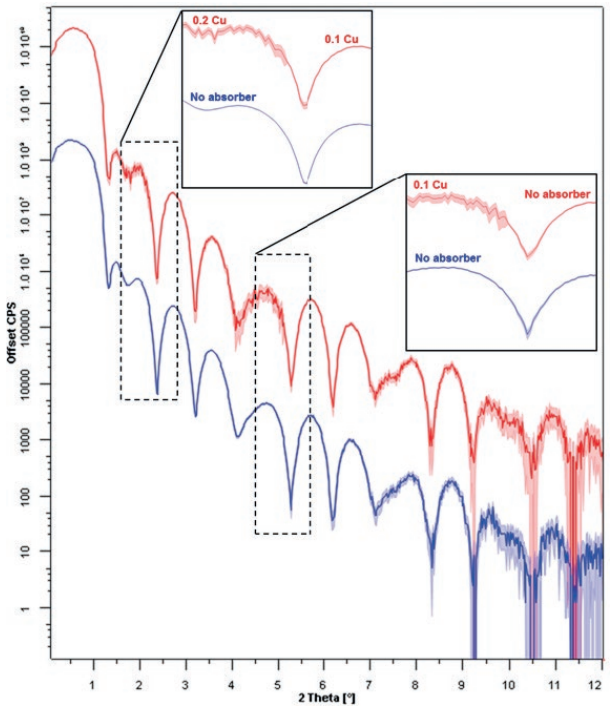


Figure 1. Comparison of XRR data taken with absorbers (red) and in a single shot with no absorbers (blue), Shading corresponds to the statistical error. (Inset) Absorber transition ranges. Scans offset for clarity.

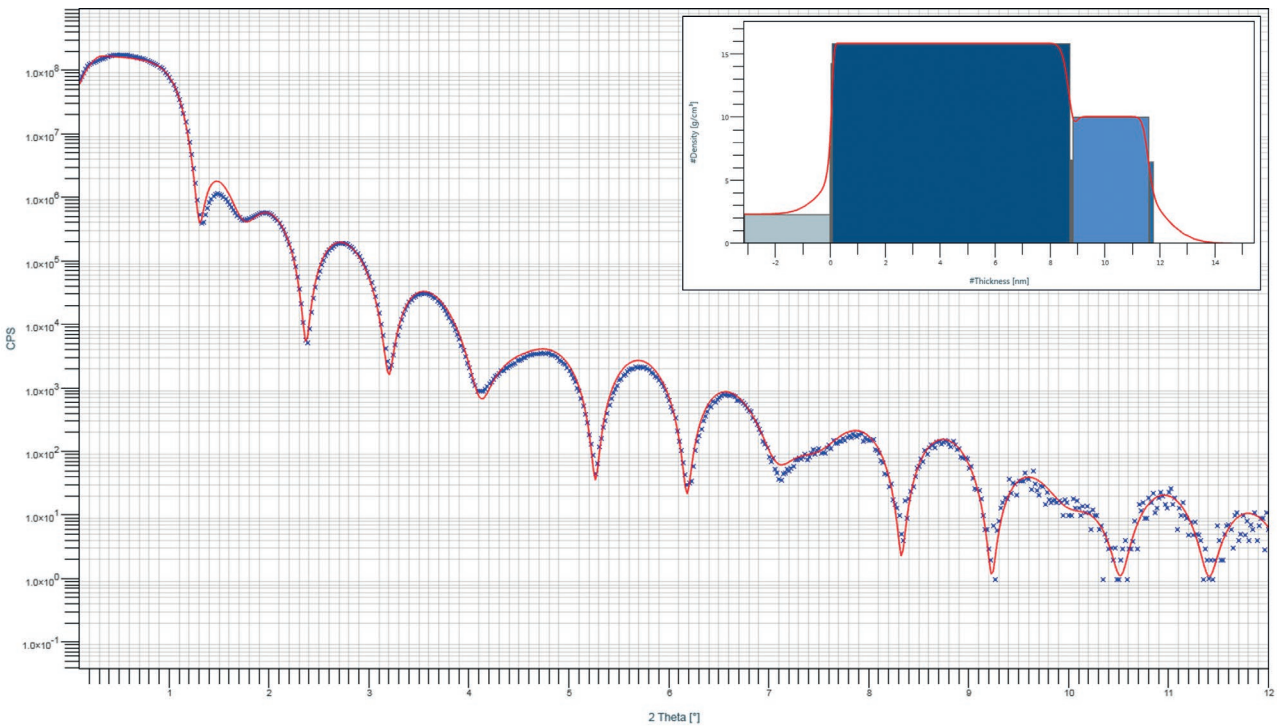


Figure 2. XRR pattern of a Ta coating on Si (black) and model fit (blue). (Inset) Density depth profile showing native SiO_2, Ta_2O_5 and amorphous C layers in addition to metallic Ta.

GaN/AlN Superlattice on Al_2O_3

The second sample was a 20-layer GaN/AlN superlattice, prepared by MBE on a c-plane sapphire substrate. An $\omega/2\theta$ coupled scan was performed from 0.1° to 15° 2θ, with a 0.005° increment. A scan time of 24 hr was chosen in order to better resolve the fine structure and high angle oscillations. Instrument resolution was minimized through the use of 0.05 mm primary and secondary slits and a detector aperture of 0.075 mm.

Data were again brought into DIFFRAC.XRR for fitting. The initial superlattice model was assumed to be uniform, with 20 8 nm periods of AlN and GaN layers constrained to the same thickness, density and roughness. This simple model resulted in a period of 8.1 nm, with AlN and GaN layer thickness of 6.8 nm and 1.3 nm and densities of 3.2 g/cm^3 and 6.8 g/cm^3, respectively. It was found that the quality of the fit could be improved by a factor of 6 by expanding the superlattice and allowing the thickness, density and roughness of each layer to vary independently. The fit result is shown in Figure 3 with the density depth profile shown in the inset. A systematic increase in density of the GaN layers is observed ranging from 6.0 to 7.3 g/cm^3 while a random change from 3.2 to 3.6 g/cm^3 is seen in the thick AlN layers. In contrast, the thickness was relatively uniform, varying from 1.2 to 1.3 nm for the GaN layers and 6.8 to 6.9 nm for the AlN layers. Roughness was found to systematically decrease from the substrate to surface, ranging from 0.4 nm to 0.3 nm in the GaN and 0.4 nm to 0.2 nm in the AlN. The systematic change in density of the GaN layers in addition to change in roughness may indicate a need for tighter control of the deposition conditions, particularly for the GaN layer.

Conclusion

XRR was performed on two different samples and the data were fit using DIFFRAC.XRR. In the first sample, asymmetric interfacial density profiles were observed for both the Si substrate, Ta coating and native oxides, as well as a surface carbon layer likely resulting from long term storage in atmosphere. In the second sample, fitting the GaN/AlN superlattice revealed a systematic variation in density in the GaN layers and a trend toward reduced roughness from the substrate to surface of the film stack. The large dynamic range of the EIGER2 resulted in consistent statistics throughout the measurement allowing regression of these fine details in the layer structures.

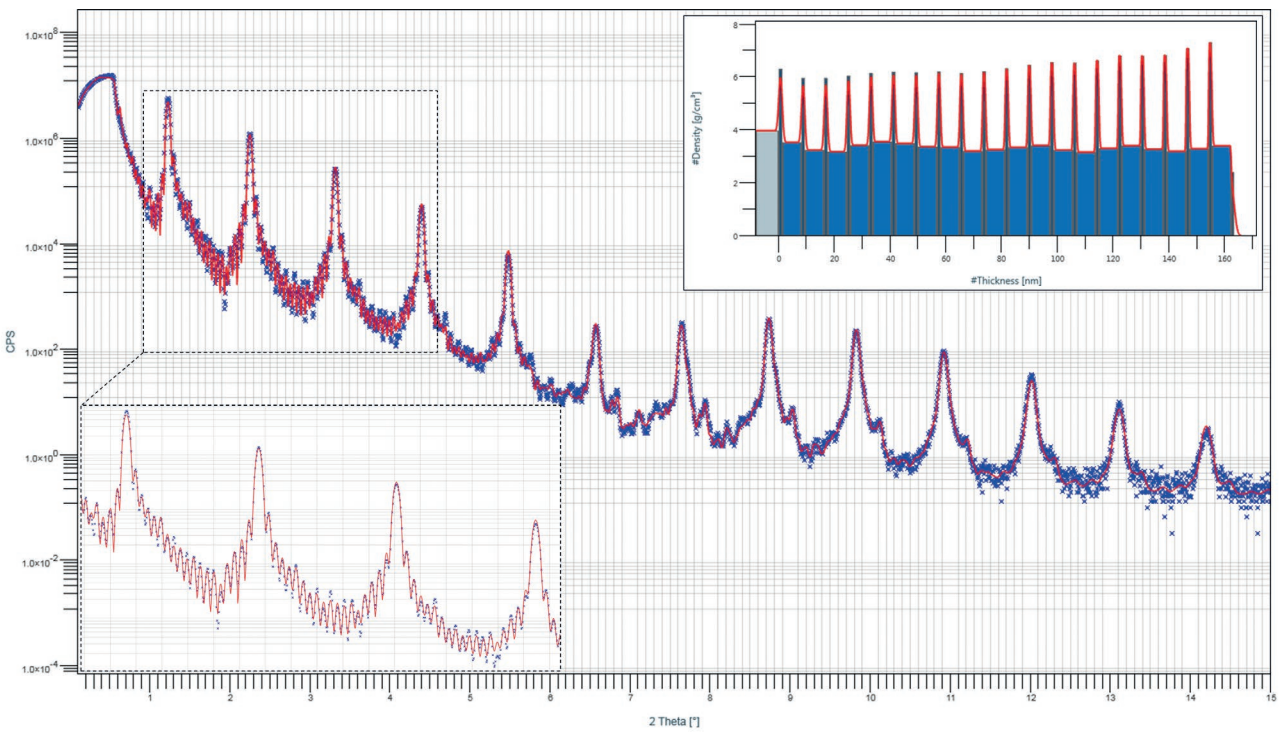
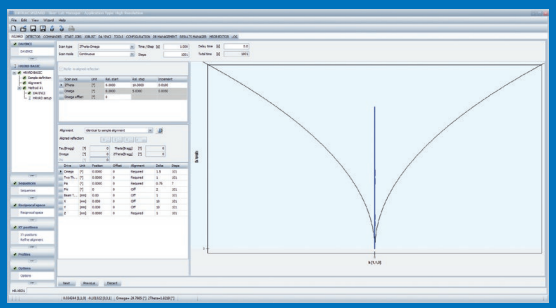


Figure 3. X-ray reflectometry pattern of an AlN/GaN superlattice (black) and model fit (blue). (Inset) Density depth profile showing 20 superlattice periods, with thickness and density variation.

DIFFRAC.SUITE Workflow for X-Ray Reflectometry

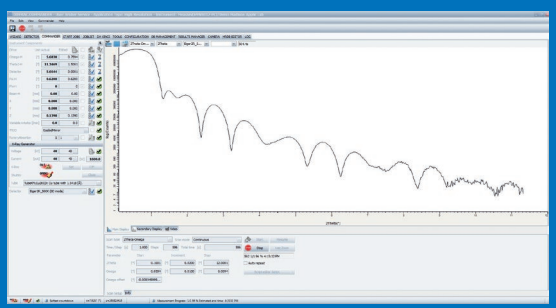
PLAN in DIFFRAC.WIZARD

- Create sample definition using customizable material database
- Graphical interface for instrument component settings including optics, slit sizes and detector opening
- Measure at multiple points with mapping



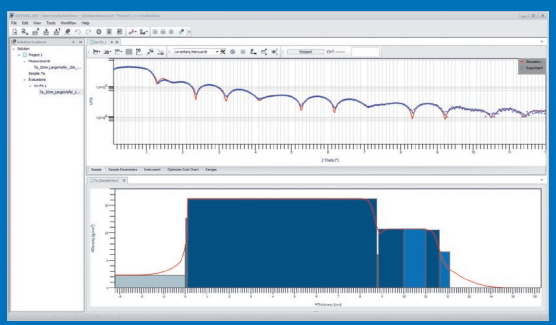
MEASURE in DIFFRAC.COMMANDER

- Direct measurement control or launch predefined experiment methods
- Automatic sample alignment with easy-to-use script editor
- Real-time data monitoring



ANALYZE in DIFFRAC.XRR

- Estimate layer thickness using FFT
- Model complex data using a flexible sample editor including material and sample database support
- Multiple available fitting algorithms
- Clear, concise report generation



Bruker AXS GmbH
info.baxs@bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/xrddetectors

www.bruker.com