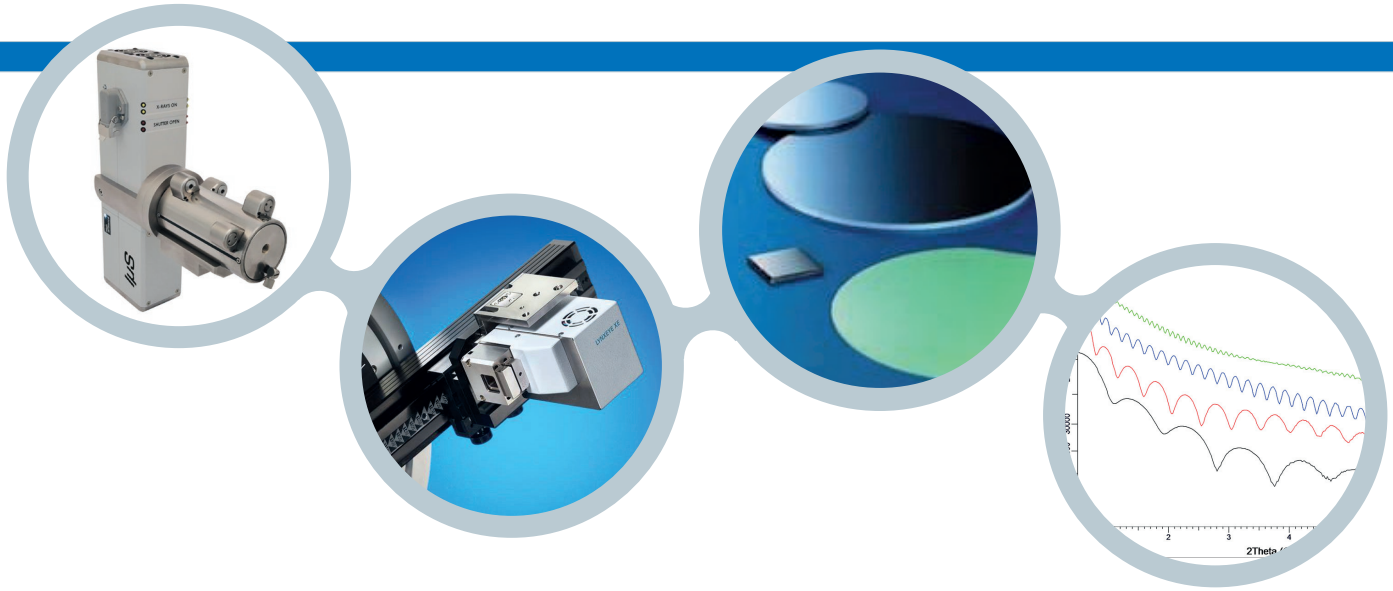

Application Note XRD 603

D8 DISCOVER with IμS$^{High\ Brilliance}$ Microfocus Source
Application: X-Ray Reflectometry

Introduction

The D8 DISCOVER combined with the INCOATEC IμS$^{High\ Brilliance}$ Microfocus source is an innovative x-ray diffraction (XRD) solution that is uniquely suited for multipurpose modern materials research characterization. In this report, we present the capabilities of this system in an X-Ray Reflectometry (XRR) configuration to analyze thin films and coatings to determine thickness, density and roughness of a single or a sequence of layers.

In this report, measurements of the NIST Standard Reference Material 2000 (NIST SRM 2000) will be shown along with analysis in DIFFRAC.LEPTOS via modeless Fast Fourier Transform (FFT) and full pattern modeling. Additionally, analysis of a sequence of epitaxial $SrTiO_3$ on Si films with thickness ranging from 8 nm to 100 nm via FFT will be shown and the model based analysis of several ultra-thin 3 unit cell (1.1 nm) and 4 unit cell (1.5 nm) epitaxial $LaAlO_3$ on $SrTiO_3$ films will be shown.

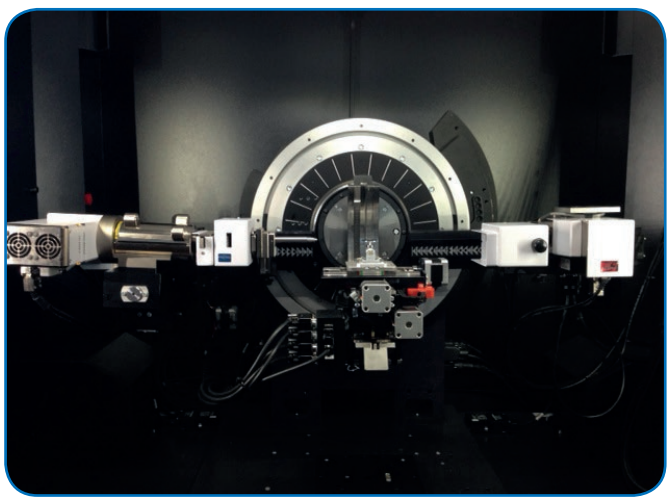

Figure 1: D8 DISCOVER with IμS$^{High\ Brilliance}$ configured for X-Ray Reflectometry

X-Ray Reflectometry

The configuration used for X-Ray Reflectometry is shown in Figure 1 and detailed in Table 1. The IμS$^{\text{High Brilliance}}$ and integrated MONTEL-P optic produces a very brilliant primary beam with 1 mm diameter spot size. Typically a 0.2 mm linear slit is used after the MONTEL-P optic, resulting in a probe that is 0.2 mm by 1.0 mm and a beam footprint on the sample of 20 mm x 1 mm at 1° 2θ and 2 mm x 1 mm at 10° 2θ. An automatic attenuator is used to ensure the response of the detector is linear through the entire measurement which spans 7 orders of magnitude in intensity. Since the primary beam is well collimated in the both the equatorial and axial directions, axial soller slits are not needed to reduce diffuse scatter from the background. The LYNXEYE XE detector can be used in a 0D configuration for alignment of the sample. For collection of the XRR measurement, the detector was used in a 1D mode, with opening of 7 channels (75 μm per channel, .525 mm total opening), or 0.09° 2θ, a value optimized to two times the incident beam divergence of 0.045° θ. Using the detector in this method results in the same resolution achieved with a 0.2 mm detector slit with a conventional 0D detector, but with the count rate of a 0.5 mm detector slit.

Source	IμS$^{\text{High Brilliance}}$ Microfocus (Cu)
Optics	Montel-P
Divergence slit	0.2 mm
Stage	Centric Eulerian Cradle (CEC)
Secondary Optic	Motorized Detector Slit (0.5 mm opening) and Rotary Absorber
Detector	LYNXEYE XE

Table 1: Typical XRR instrument instrument setup for the D8 DISCOVER with IμS$^{\text{High Brilliance}}$

Measurement and Analysis of NIST SRM 2000

The NIST standard reference material 2000 (NIST SRM 2000) consists of a 25 mm x 25 mm x 0.725 mm (001) oriented Si substrate with an epitaxial 50 nm $Si_{0.85}Ge_{0.15}$ film and 25 nm Si capping layer. Figure 2 shows a coupled scan of SRM 2000, measured from 0.3° to 7.5° 2θ, with step size of 0.02° and time of 5 second per step resulting in a total scan time of 30 minutes. There are three meaningful distances present in the NIST SRM 2000 (50 nm $Si_{0.85}Ge_{0.15}$ layer, the 25 nm Si capping layer and the 75 nm total film thickness) resulting in a scan with a triplicate pattern of fringes. The simplest method for analyzing X-Ray Reflectometry data in DIFFRAC.LEPTOS is to perform a fast Fourier transform (FFT) of the data which requires no prerequisite knowledge of the sample structure. The FFT of the data in Figure 2 is shown in Figure 3. Three prominent peaks are seen in the result at 25.2 nm, 49.5 nm and 74.5 nm, which are in good agreement with the description of the NIST SRM 2000 standard. A more thorough method of analysis in DIFFRAC.LEPTOS is to create a model of the sample using basic information about the film structure and refining the model against the data. Figure 4 contains the result of the model based refinement. The upper window shows the fit of the data with the blue model curve closely following the black data curve. The lower left window contains the refined layer parameters, including the thickness, density and roughness of each layer. The lower right window shows a plot of the density as a function of depth in the sample with the blocks representing the nominal thickness and density, and the blue line indicating the effect of roughness.

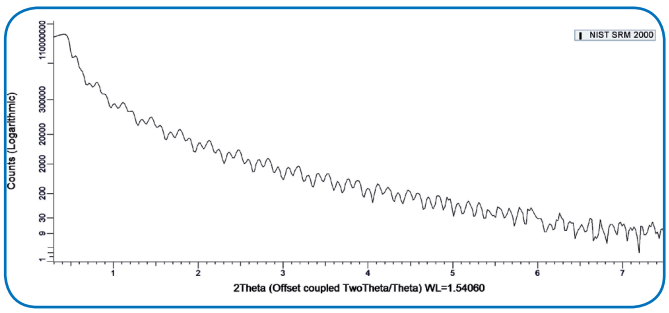

Figure 2: XRR measurement of NIST SRM 2000 with the D8 DISCOVER with IμS$^{\text{High Brilliance}}$ and LYNXEYE XE.

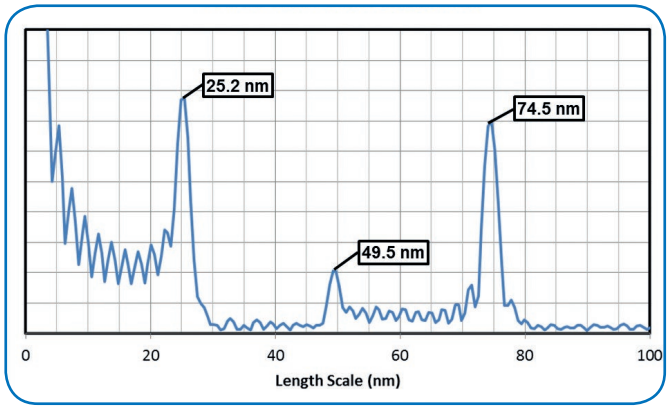

Figure 3: Fast Fourier Transform analysis of NIST SRM 2000 in DIFFRAC.LEPTOS.

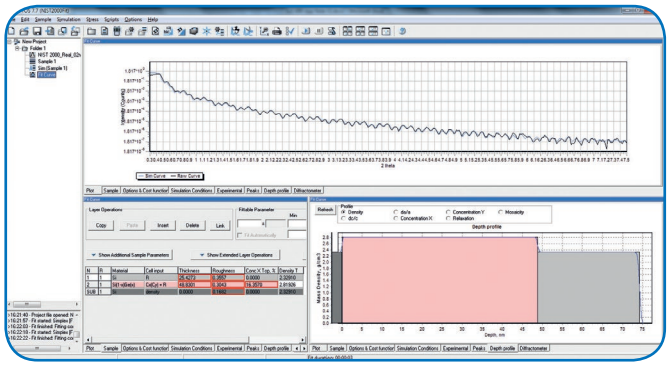

Figure 4: Model based analysis of NIST SRM 2000 in DIFFRAC.LEPTOS

Measurement and FFT Analysis of $SrTiO_3$ on Si

Modern thin film deposition equipment often includes the ability to quantify the atomic flux incident upon the sample with quartz crystal oscillators, but this does not always represent the true amount of material deposited during a run. X-Ray Reflectometry is a simple method for determining the absolute film thickness. A series of films of $SrTiO_3$ on Si were grown via molecular beam epitaxy (MBE) with anticipated thicknesses of 8 nm, 16 nm, 40 nm and 100 nm. The samples were approximately 1 cm x 1 cm in size. Figure 5 contains X-Ray Reflectometry curves measured from 0.5° to 10° 2θ, with step size of 0.02° and time of 5 second per step resulting in a total scan time of 40 minutes. In this case, only the film thickness is needed, so a simple FFT analysis is performed, with the result shown in Figure 6.

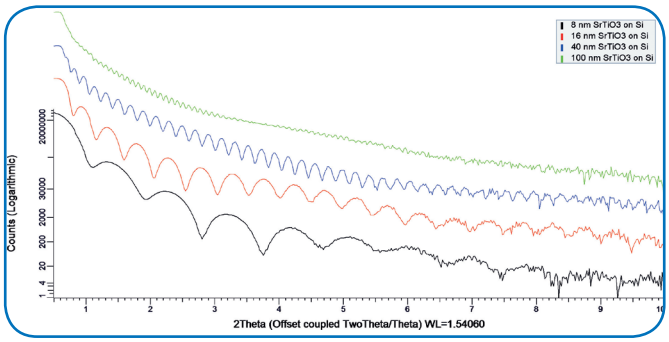

Figure 5: XRR measurement of several $SrTiO_3$ films on Si with the D8 DISCOVER with IμS$^{High\ Brilliance}$ and LYNXEYE XE.

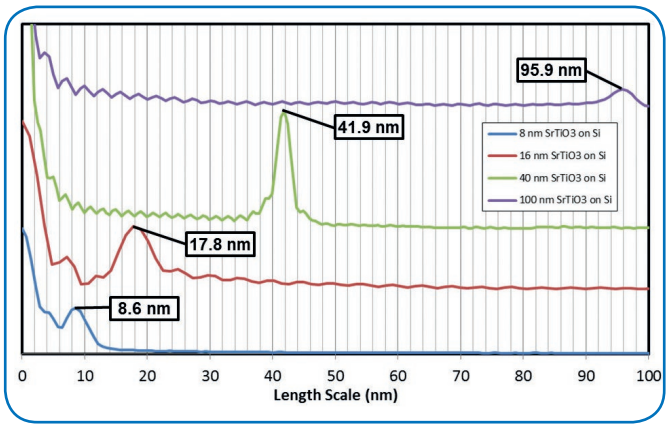

Figure 6: Fast Fourier Transform analysis of $SrTiO_3$ on Si films of various thicknesses in DIFFRAC.LEPTOS

Measurement and Model Based Analysis of $LaAlO_3$ on $SrTiO_3$

$LaAlO_3$ is a very intriguing material. When three unit cells are deposited on $SrTiO_3$ substrate, the material is an insulator. When a fourth unit cell is added, the material becomes conductive. This effect is the result of a two dimensional electron gas being created at the film-substrate interface. When the film is created, the substrate size is fairly small (5 mm x 5 mm) to minimize cost and to maximize film uniformity. The ultra thin film thickness and small sample size makes analysis with a conventional tool challenging. By utilizing the D8 DISCOVER with IμS$^{\text{High Brilliance}}$ and MONTEL-P optic, the beam is focused where it is most important, on the sample, minimizing background and maximizing signal. Figure 7 contains X-Ray Reflectometry curves measured on 5 mm x 5 mm samples of 3 and 4 unit cell $LaAlO_3$ on $SrTiO_3$ from 0.3° to 10° 2θ, with step size of 0.02° and time of 5 second per step resulting in a total scan time of 40 minutes. There is a visual difference between the 3 and 4 unit cell samples, with the 4 unit cell data showing a shift of the fringes to lower angle, consistent with behavior of a thicker layer. Due to the complex nature of the interaction of the $LaAlO_3$ film with the $SrTiO_3$ substrate, the full model approach in DIFFRAC.LEPTOS is used. The resulting fits, shown in Figures 8 and 9, show a significant difference, not only in the film thickness, but also the interaction layer within the substrate. DIFFRAC.LEPTOS not only is capable of modeling the film and interaction layer thickness, but is also capable of fitting the roughness as variation in the density of the interaction layer.

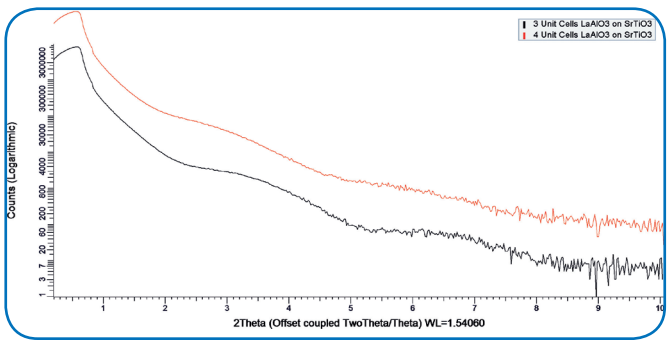

Figure 7: XRR measurement of 3 and 4 unit cell (1.1 nm and 1.5 nm respectively) thick $LaAlO_3$ films on $SrTiO_3$ with the D8 DISCOVER with IμSHigh Brilliance and LYNXEYE XE.

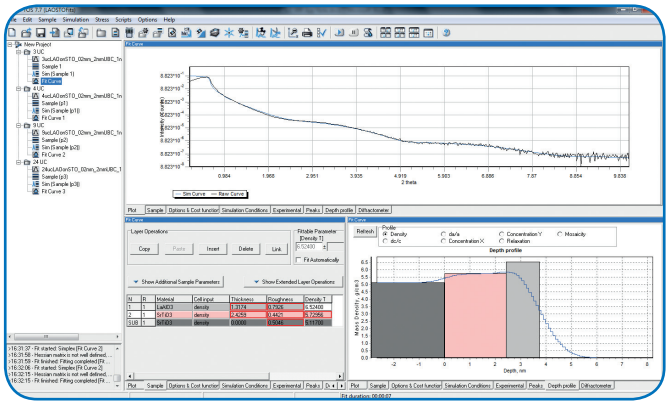

Figure 8: Model based analysis of 3 unit cells $LaAlO_3$ on $SrTiO_3$ in DIFFRAC.LEPTOS

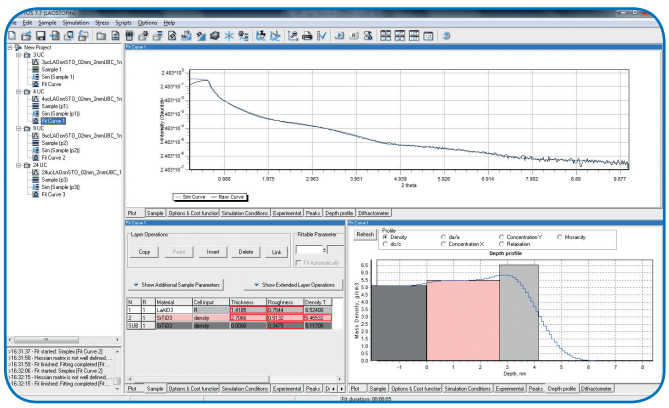

Figure 9: Model based analysis of 4 unit cells $LaAlO_3$ on $SrTiO_3$ in DIFFRAC.LEPTOS

Conclusion

The D8 DISCOVER with IμS$^{\text{High Brilliance}}$ and DIFFRAC.LEPTOS were used to collect and analyze X-Ray Reflectometry data collected from a variety of thin films ranging in thickness from 1 nm to 100 nm. The IμS$^{\text{High Brilliance}}$ combined with MONTEL-P optic creates a beam with extremely high brilliance and low divergence, resulting in an ideal configuration for measuring samples as small as several mm on a side with exceptional signal to background. Using the LYNXEYE XE in a scanning 1D configuration reduces data collection time by a factor of 2 while maintaining the same resolution when compared to the conventional slit based 0D setup. DIFFRAC.LEPTOS is a very powerful tool for the analysis of X-Ray Reflectometry data, whether it be through the modeless fast fourier transform method or the full model based approach.

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