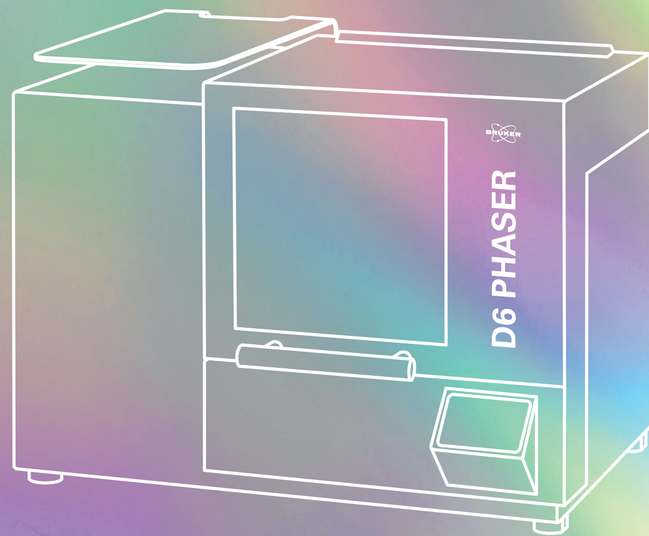

X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD Grazing Incidence Diffraction

Application Report 46

Grazing incidence diffraction (GID) is a method for studying polycrystalline thin films. These samples usually show very weak diffraction signal arising from the thin film in Bragg-Brentano symmetric diffraction because the X-rays pass through the thin film into the underlying substrate, which dominates the resulting signal due to its larger scattering volume.

If, however, instead of the divergent beam path of the Bragg-Brentano geometry, a thin parallel beam is directed at the sample surface so that it penetrates only the thin film but not the underlying substrate, the resulting signal from the film is dominant. By optimizing the angle of incidence of the parallel beam, it is possible to decouple the scattering signal of the layer from that of the substrate (Figure 1).

Double slit collimation is used to produce a parallel beam in the D6 PHASER. The line focus of the tube combined with a tightly closed primary divergence slit is sufficient to collimate a suitably parallel beam. Alternatively, a combination of a tightly closed variable primary slit and a second pluggable slit can be used. By using the universal stage (Figure 2), the angle of incidence of the parallel beam with respect to the sample surface can be held fixed while a detector scan is performed. An equatorial Soller collimator is used on the secondary side. It limits the angular resolution and the flux at the detector. The resulting data can be used for phase identification, as well as residual stress analysis and its depth variation controlled by the angle of incidence.

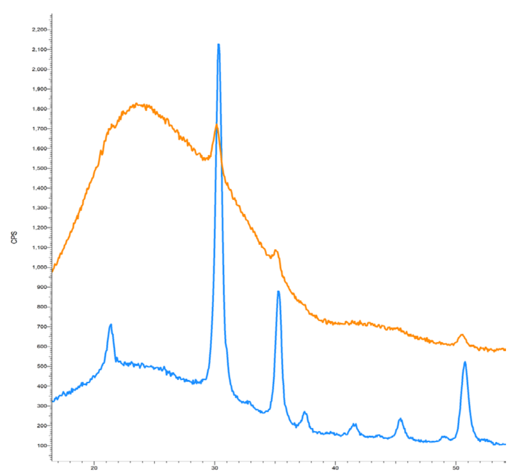

Figure 1
DIFFRAC.EVA view of a 20 nm indium tin oxide (ITO) layer deposited on a glass substrate. The symmetric theta/theta scan (orange) shows a signal dominated by the glass substrate, while the ITO signal is nicely emphasized in the 0.3° fixed angle GID scan (blue).

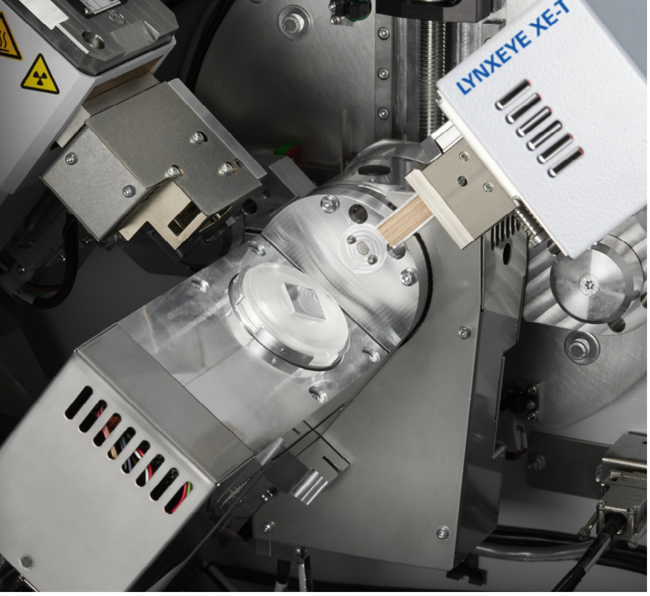

Figure 2
D6 PHASER equipped for GID with the variable divergence slit module, universal stage and a secondary side equatorial Soller collimator mounted on the LYNXEYE XE-T detector.

In asymmetric geometries such as GID, unlike symmetric geometries, the diffraction planes do not have the same orientation during data acquisition. The normal to the planes is close to the surface normal at low diffraction angles and approaches the in-plane direction at high diffraction angles. This allows analysis of the lattice deformation due to the strain exerted on the film by the substrate. Note, that the observation and accuracy of the residual stress depends strongly on the ability of the diffractometer to acquire data up to high angles.

Figure 3. shows the evaluation of a GID scan performed on a 20 nm tungsten film and acquired in 40 min up to 140°. The thickness of the layer was also accurately measured with the D6 PHASER, as reported elsewhere (see Application Report 45). Tungsten is being considered as a replacement for copper as a metallization material in semiconductor devices with film thicknesses of less than tens of nanometers. Depending on the growth conditions, such confinement can result in significant residual stresses that can lead to undesirable effects (e.g., delamination). It is therefore critical to estimate the stress, which in some cases can be more than a tenth of a GPa.

The evaluation is based on a decomposition of the entire powder pattern using a cubic Im-3m space group phase and additional angular shifts related to (i) the refraction effect of X-rays at the sample surface and (ii) lattice strain inducing an angular dependent peak shift expressed by elastic theory (multiple sin2ψ(hkl) method). The refinement is performed in DIFFRAC.TOPAS and shows a significant compressive residual stress in the tungsten layer of -2.4 GPa.
The D6 PHASER and DIFFRAC.TOPAS are a great combination to go beyond qualitative phase analysis and provide quantitative structural information in thin film analysis.

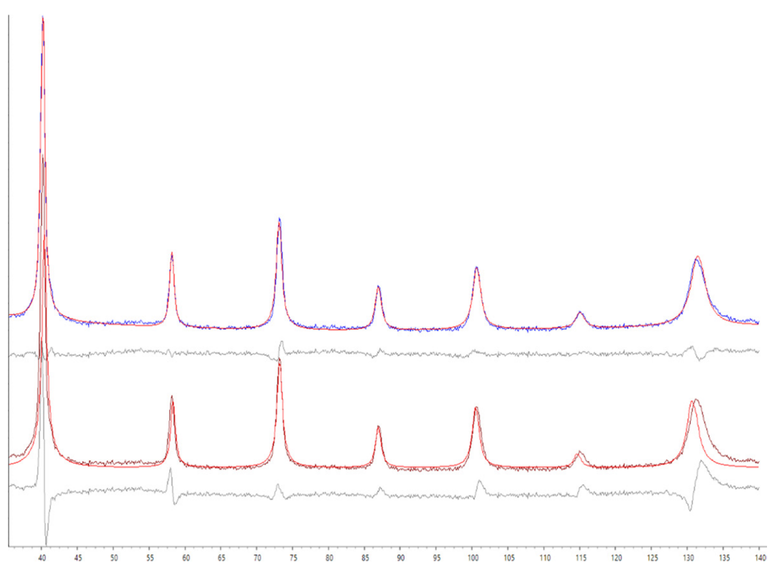

Figure 3
GID data from a 20 nm tungsten layer refined with DIFFRAC.TOPAS. The addition of residual stress in the model for the W phase (top pattern) significantly improves the fit quality as observed on the difference plots.

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