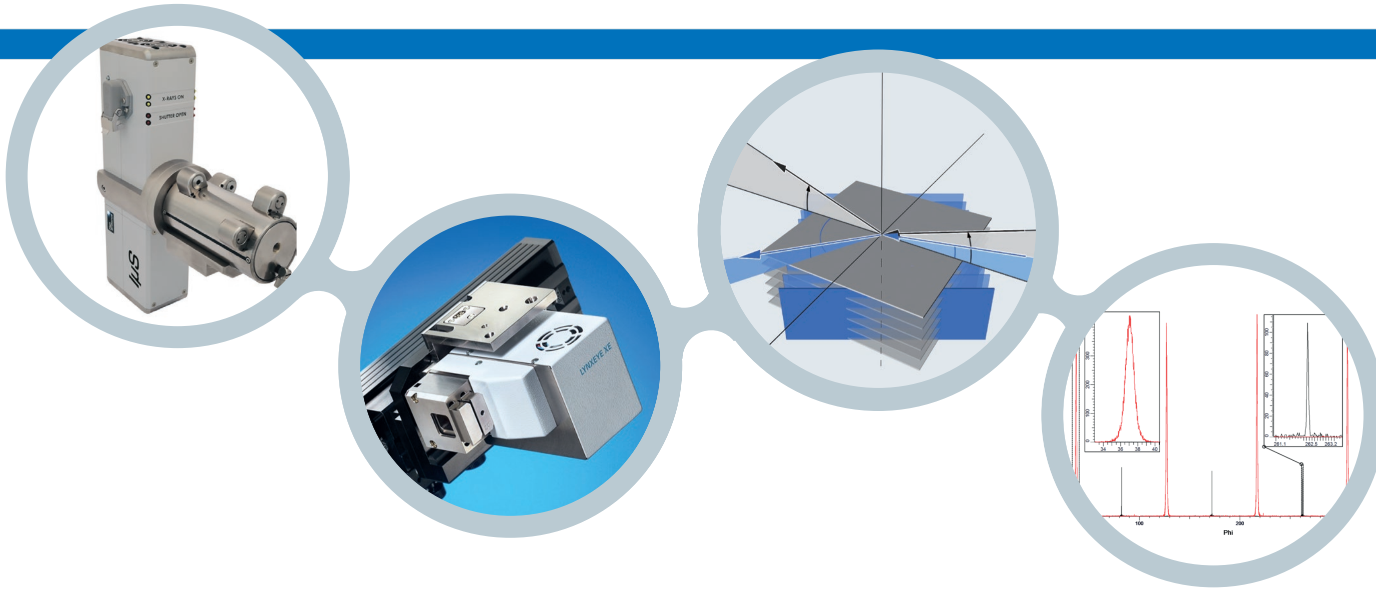


Application Note XRD 605

D8 DISCOVER with IμS Microfocus Source

Application: In-Plane Grazing Incidence Diffraction (IP-GID)

Introduction

The D8 DISCOVER combined with the INCOATEC IμS 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 In-Plane Grazing Incidence Diffraction (IP-GID) configuration. This particular setup enhances the signal from very thin epitaxial and polycrystalline layers, and allows probing properties in the plane of the sample surface.

In this report, the instrumental resolution in α_i, φ and 2θ will be characterized through the measurement of a (001) oriented Si wafer. Additionally, measurements of a polycrystalline copper thin film on a (001) Si wafer and measurements of a series of epitaxial $SrTiO_3$ on (001) Si thin films will be shown.

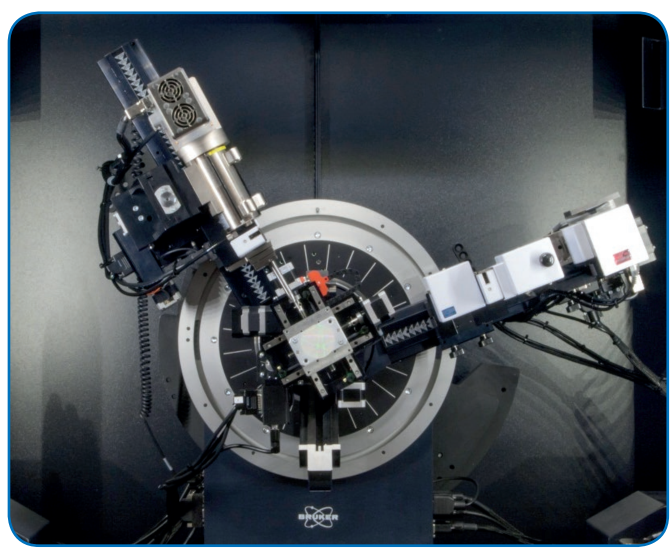


Figure 1. D8 DISCOVER with IμS and LYNXEYE XE detector configured for In-Plane Grazing Incidence Diffraction (IP-GID).

IµS In-Plane Grazing Incidence Diffraction (IP-GID)

Non-coplanar GID (Grazing Incidence Diffraction) also called In-Plane GID (IP-GID) is a technique for investigating the near-surface region of samples (ten or fewer nm beneath the air-sample interface). It exploits the high intensity of the total external reflection condition while simultaneously Bragg diffracting from planes that are nearly perpendicular to the sample surface. Figure 2 shows the experimental geometries used for coplanar and non-coplanar diffraction.

Traditional lab systems configured for IP-GID use a primary beam conditioned by a single multilayer mirror or polycapillary optic and may have low divergence perpendicular to the film, which gives good depth penetration control, but has relatively large divergence parallel to the film (in the scattering direction) which results in poor peak resolution. The configuration used for IP-GID is shown in Figure 1 and detailed in Table 1. The IµS and integrated MONTEL optic produces a very intense primary beam with 1 mm diameter spot size and equatorial and axial divergence of <0.1°. This results in both excellent depth control and equatorial resolution due to the symmetric divergence. A 0.2° equatorial soller is used in front of the LYNXEYE XE, which is used in 0D mode, giving a diffracted beam resolution of 0.2°. Figure 3 shows a schematic representation of the IP-GID geometry utilizing the IµS source.

Source	IµS Microfocus (Cu)
Optics	MONTEL
Collimator	1.0 mm
Stage	Centric Eulerian Cradle (CEC)
Secondary Optic	0.2° equatorial Soller collimator
Detector	LYNXEYE XE (0D)

Table 1. IP-GID instrument setup for the D8 DISCOVER with IµS.

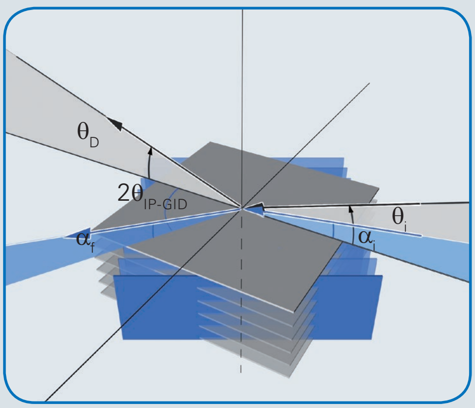


Figure 2. Coplanar diffraction geometry and with θ_i and θ_D (b) Non-coplanar diffraction geometry with incident angle (α_i) and exit angle (α_f) from the sample surface with $2\theta_{IP-GID}$ the Bragg scattering angle.

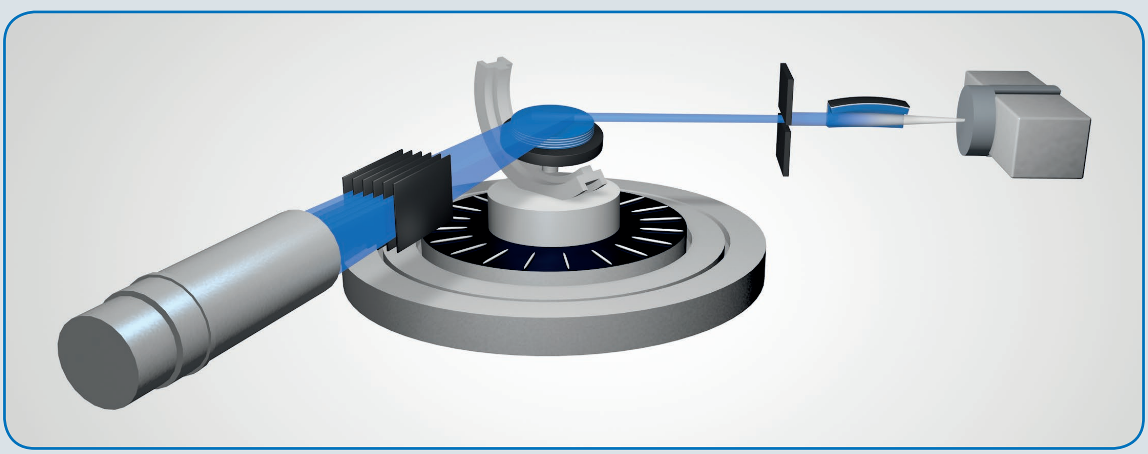
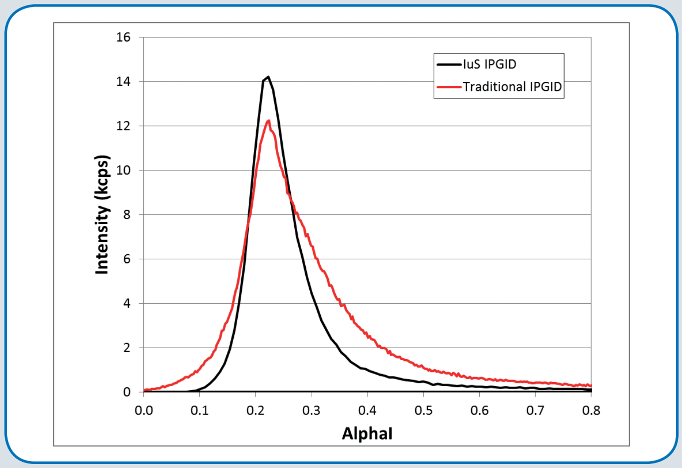
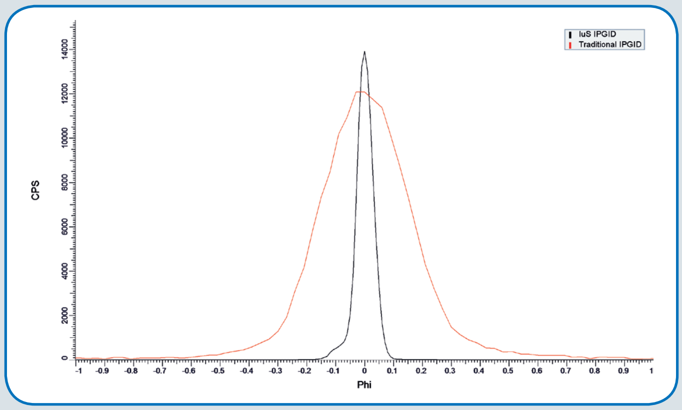


Figure 3. IP-GID geometry with the IµS, MONTEL optic, and secondary equatorial soller collimator.

Instrument Resolution Characterization using a (001) Si wafer

A 25 mm x 25 mm piece of a (001) Si wafer was used to characterize the instrumental resolution of both the traditional (single Göbel mirror) and IμS IP-GID instrument geometries. Figure 4a shows a scan of the inclination angle α_i on the (004) Si in-plane reflection. Both the resolution and intensity achieved with the IP-GID geometry based on the IμS is superior to the traditional method. Figure 4b shows a phi scan of the (004) Si in-plane reflection. The FWHM of the reflection is found to be 0.07°, consistent with the equatorial divergence of the MONTEL, where the traditional IP-GID geometry with FWHM of 0.3° is consistent with the 0.3° incident equatorial soller. Figure 4c shows a coupled 2θ-ω scan of the (004) Si in-plane reflection. $K\alpha_1$ and $K\alpha_2$ splitting is clearly evident in the enhanced IP-GID result (FWHM of $K\alpha_1$ = 0.14°), where the traditional IP-GID geometry instrumental function is too broad to observe splitting.





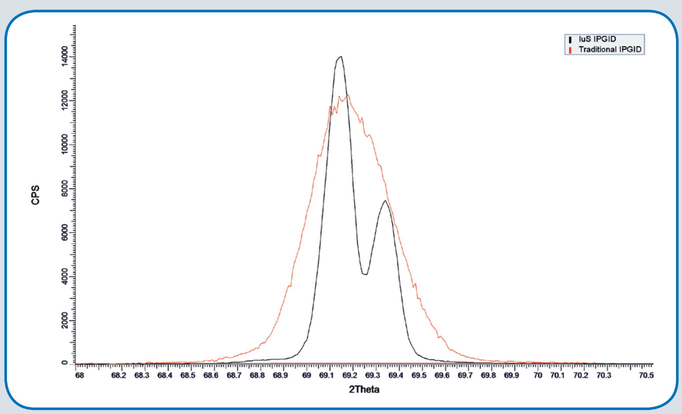


Figure 4. IP-GID measurements using the traditional IP-GID method with a line focus and single Göbel mirror in red and the IuS IP-GID method in black of the (400) in-plane reflection of a (001) Si wafer. (a) Scan of the inclination angle (α_i) (b) Phi Scan (c) Coupled 2θ-ω scan.

Measurement of a Polycrystalline Cu film on Silicon substrate

Many coatings are applied to materials to alter the properties of the surface while maintaining properties of the bulk. This could be to provide electrical conductivity along an insulating surface or to change the surface finish. These coatings can range in thickness from microns to nanometers. For micron thick layers, a geometry called Grazing Incidence Diffraction is commonly used, where the incident beam angle is kept very low, typically below 1°, while the detector is scanned over a wide range of angles. Since the incident beam angle is kept low, the beam does not penetrate to the substrate. Where as in a classic Bragg-Brentano coupled geometry the incident angle is scanned with the detector angle, resulting in an increase of penetration depth of the incident beam as the scan progresses. Additionally, it is important to note the direction being probed in the material. In a standard coupled scan, the direction being probed in the material is normal to the surface of the material. In the grazing incident method, the direction being probed is close to normal to the surface at the beginning of the scan, but tilts towards the plane of the sample surface as the scan progresses. In the IP-GID geometry, the angle of inclination to the surface (α_i) is fixed near the critical edge of the material while a detector scan is performed in a direction parallel to the surface of the sample. This results in extremely low penetration depth and an enhancement of the signal coming from the surface. In this case, surface sensitivity is achieved and the direction being probed is in the plane of the surface of the sample. Figure 4 shows a scan of a Cu thin film on (001) Si. The normal bisecting geometry scan shows only the (111) and (222) Cu peaks along with the (004) Si peak indicating the Cu [111] axis being oriented normal to the surface of the sample. The Grazing Incidence Diffraction scan was collected with a fixed incident angle of 1°. All Cu reflections are now present as the direction being measured is no longer normal to the surface. The Si (004) reflection is no longer present as the direction being probed is not normal to the surface of the sample. The In-Plane Grazing Incidence Diffraction scan shows not only the Cu peaks, but also peaks coming from a surface oxide phase of Cu_2O.

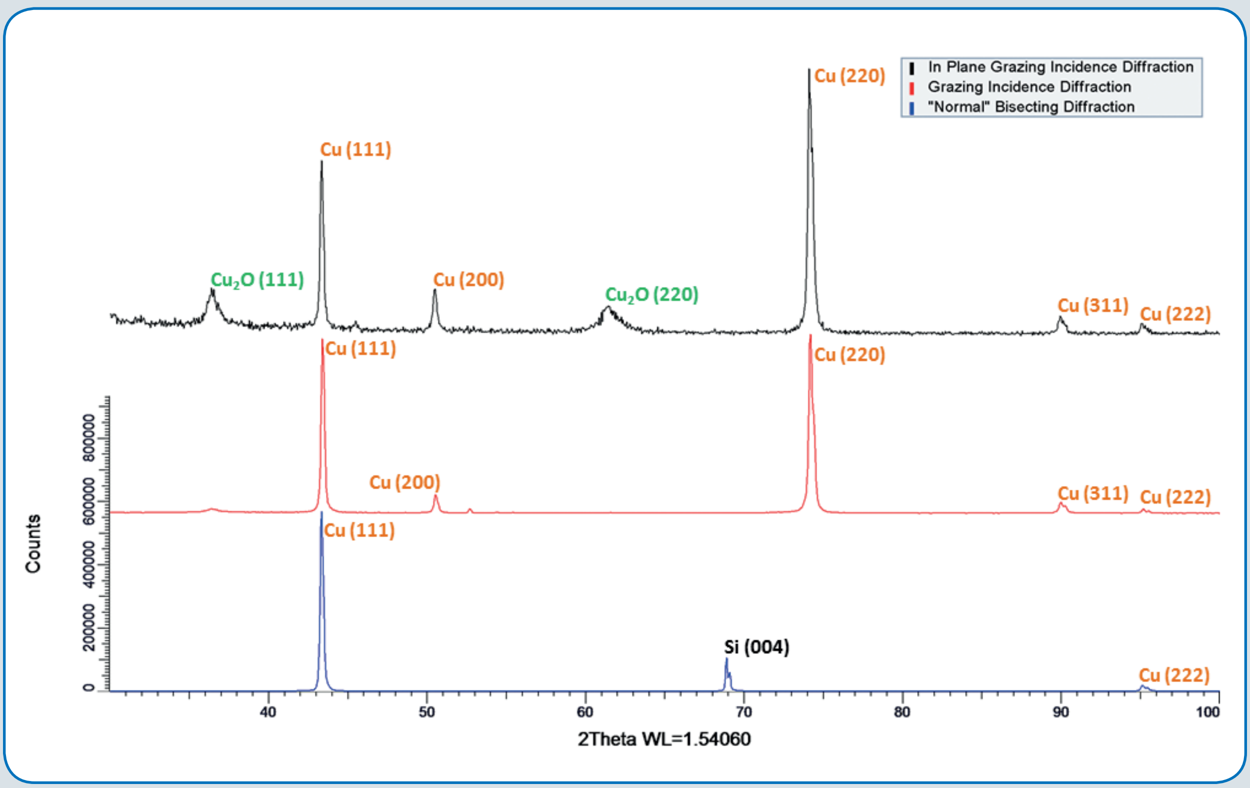


Figure 5. Normal "Bragg-Brentano" Diffraction (blue), Grazing Incidence Diffraction (red) and In-Plane Grazing Incidence Diffraction (black) measurements using the IμS IP-GID geometry of a copper film on (001) Si.

Measurement of Epitaxial $SrTiO_3$ on Si (001)

$SrTiO_3$ is commonly used as a buffer layer for the growth of perovskite based multiferroic materials, such as PMNPT or $LaAlO_3$, on Si. There is a fairly large lattice mismatch between $SrTiO_3$ (a = 3.905 Å) and Si (a= 5.437 Å) resulting in a 45° in-plane rotation of the $SrTiO_3$, aligning the $SrTiO_3$ (100) axis with the Si (110) axis (5.437/ √2 = 3.845 Å). This in-plane relationship can be seen in figure 6, where the (200) $SrTiO_3$ reflection shows a clear 45 degree rotations with respect to the (400) Si reflection.

A series of $SrTiO_3$-films of various thicknesses deposited via molecular beam epitaxy with thicknesses ranging from 8 nm to 100 nm were investigated for in-plane relaxations effects. Phi and α_i were optimized for the (220) Si reflection, then a 2θ-ω scan was collected and α_i optimized on the $SrTiO_3$ (200) reflection to optimize the signal coming from the film. The difference in optimization of α_i on the Si and $SrTiO_3$ reflection for the 40 nm film can be seen in figure 7. A 2θ-ω scan was then collected from 44° to 49° 2θ, with 0.025° step and 5 seconds per step, resulting in a total scan time of 17 minutes. The result is shown in Figure 8 with a similar analysis performed on the $SrTiO_3$ (220) and Si (400) reflections in Figure 9. In-plane reciprocal space maps were collected of the $SrTiO_3$ (200) and Si (220) reflections on the 8 nm and 100 nm films. The in-plane alignment of the $SrTiO_3$ remains consistent as a function of thickness, while the crystalline quality and in-plane mosaic spread, related to the height and width of the film spot, is reduced in the thicker film.

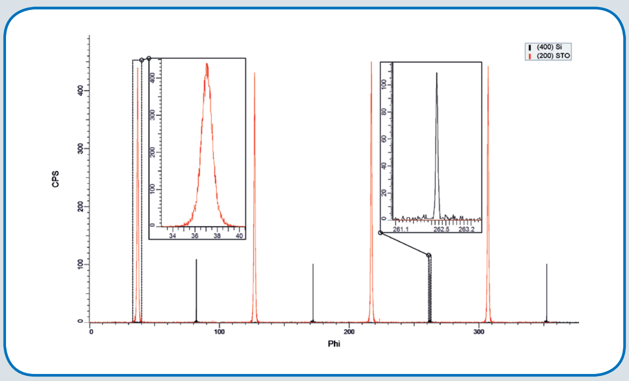


Figure 6. Phi scan of the in-plane (200) $SrTiO_3$ and (400) Si reflections measured using the IµS IP-GID geometry showing the in-plane 45° rotation of the $SrTiO_3$ relative to the Si.

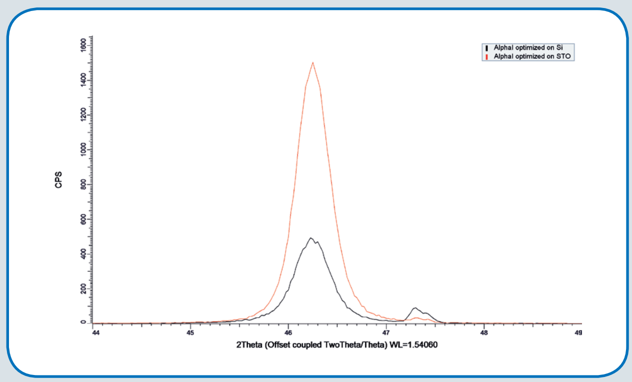


Figure 7. 2θ-ω scans of the in-plane (200) $SrTiO_3$ and (220) Si reflections with α_i optimized for $SrTiO_3$ (red) and Si (black) measured using the IµS IP-GID geometry.

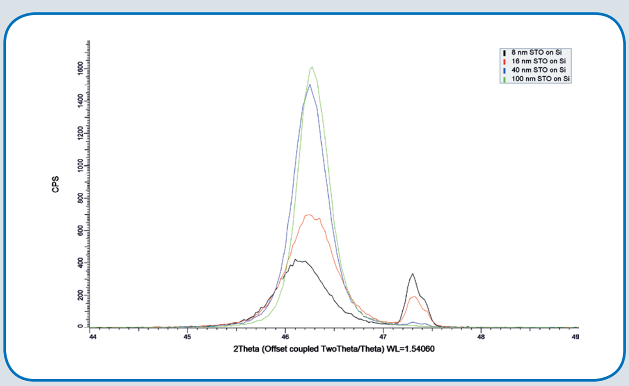


Figure 8. 2θ-ω scans of the in-plane (200) $SrTiO_3$ and (220) Si reflections for a series of $SrTiO_3$ film thicknesses ranging from 8 nm to 100 nm measured using the IµS IP-GID geometry.

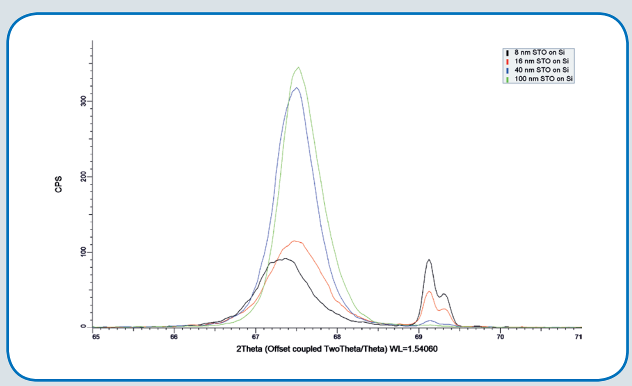


Figure 9. 2θ-ω scans of the in-plane (220) $SrTiO_3$ and (400) Si reflections for a series of $SrTiO_3$ film thicknesses ranging from 8 nm to 100 nm measured using the IµS IP-GID geometry.

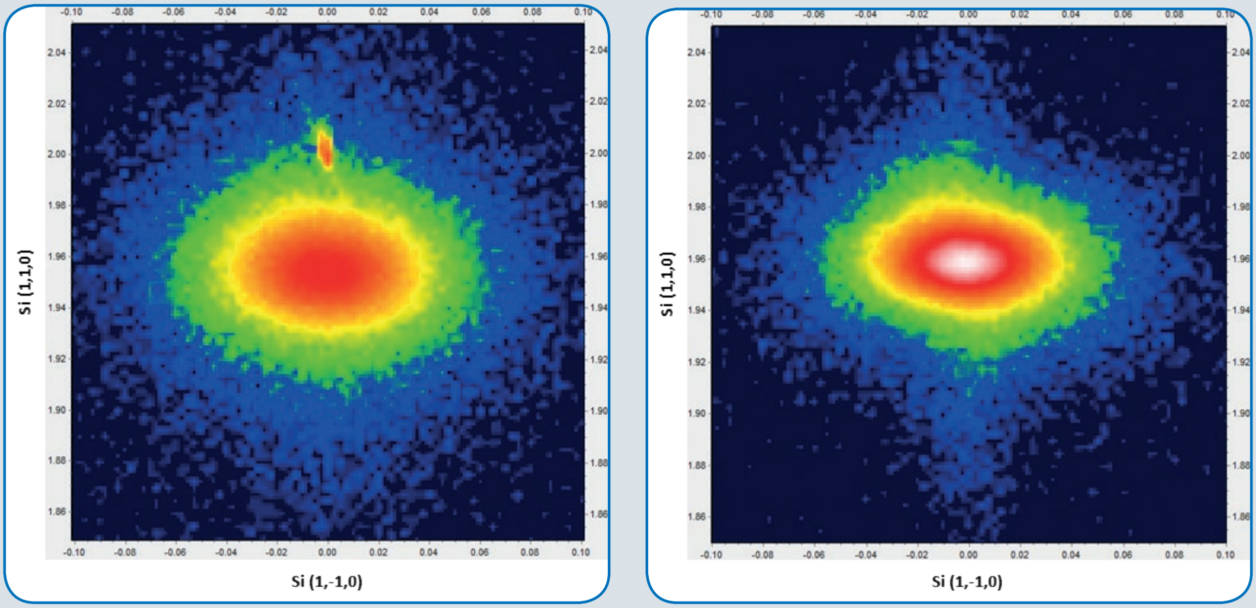


Figure 10. In-Plane Reciprocal Space Maps of the (200) $SrTiO_3$ and (220) Si reflections for the 8 nm and 100 nm thick $SrTiO_3$ on Si samples measured using the enhanced IµS IP-GID geometry.

Conclusion

The D8 DISCOVER with IµS was used to collect data in an In-Plane Grazing Incidence Diffraction geometry from a variety of samples including a bare Si wafer, fiber textured copper film on Si and epitaxial $SrTiO_3$ films on Si. The IµS combined with MONTEL optic creates a beam with extremely high flux-density and low divergence, resulting in an ideal configuration for In-Plane Grazing Incidence Diffraction as strong coupling with the surface can be achieved while high resolution in the equatorial scattering plane is maintained. In addition, flexibility of the D8 DISCOVER with IµS configured with a Centric Eulerian Cradle means that dedicated hardware for In-Plane Grazing Incidence Diffraction is not required, resulting in a system perfectly suited for the modern multipurpose materials research lab.

Author

Jon Giencke, Application Scientist, Bruker AXS Inc.

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Bruker AXS GmbH

Karlsruhe · Germany
Phone +49 721 50997-0
Fax +49 721 50997-5654
info@bruker-axs.de

www.bruker.com