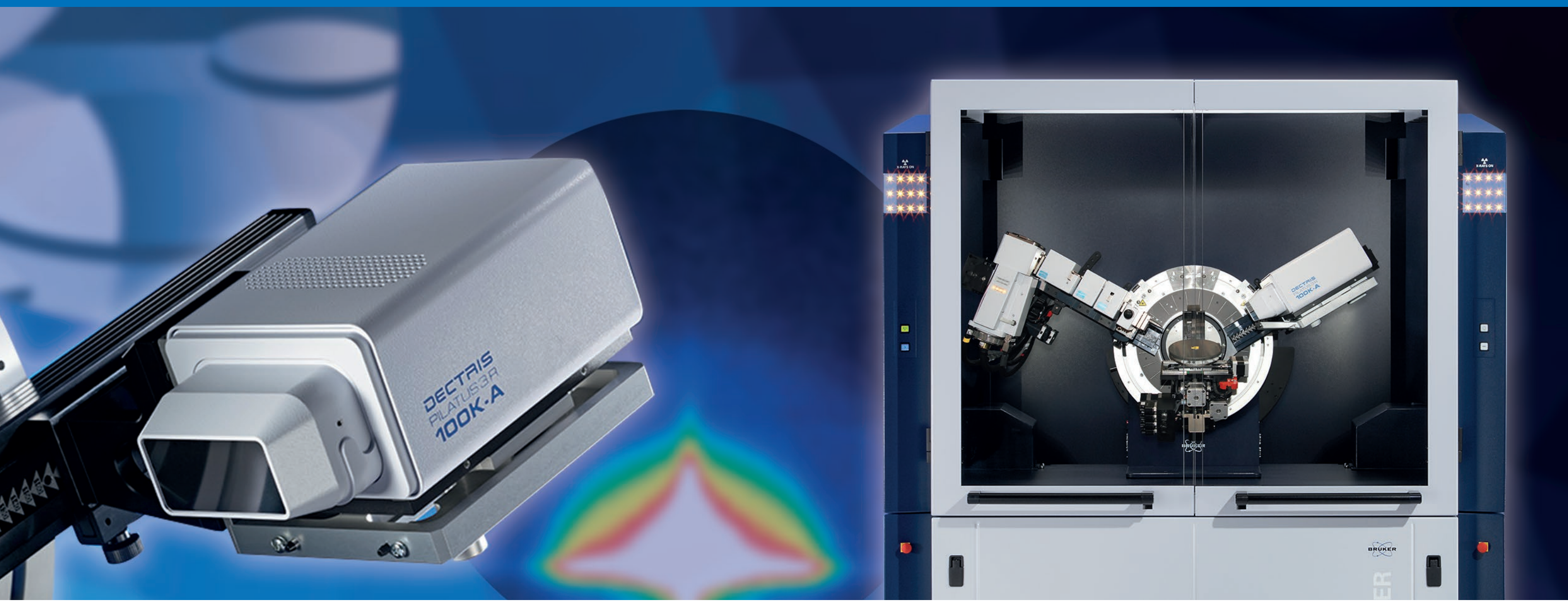

Application Report XRD 20

D8 DISCOVER with PILATUS3 R 100K-A 2D GI-SAXS

The D8 family of diffraction solutions combined with the DECTRIS PILATUS3 R 100K-A hybrid photon counting (HPC) pixel detector is an innovative 2D x-ray diffraction (XRD^2) solution that is uniquely suited for multipurpose modern materials research characterization. In this report, we present the capabilities of this system in a Grazing Incidence Small Angle X-ray Scattering (GI-SAXS) configuration for the analysis of nanostructured surfaces.

Introduction

Grazing Incidence Small Angle X-ray Scattering (GI-SAXS) is a technique that combines the surface sensitive nature of grazing incidence geometry with a scattering range that probes the small angle region which gives information on the nanostructures and nanoparticles within the surface layer. The scattering is ideally collected on a 2D detector as it gives access to lateral and vertical nanostructures.

Measurement

To create a nanostructured interface, 25 µl of 10 nm Au Nanoparticles in water were applied to a ~5 mm by ~15 mm area of the surface of a Si substrate. The solution was then allowed to dry, leaving a thin layer of nanoparticles. The samples were measured in reflection with a D8 DISCOVER equipped with the IµS micro-focus source and PILATUS3 2D detector, shown in Figure 1. The optimal angle of incidence was determined by using the PILATUS3 in 0D mode to collect a X-ray reflectometry (XRR) scan. In this case the angle was close the critical angle which is seen clearly in XRR data. Images were collected for 10 min each with a sample to detector distance of 33 cm and 500 µm primary beam collimator. The deposition process was then repeated to build the thickness in 25 µl increments until a total of 150 µl was applied (2 µl/mm^2).

Figure 2 shows PILATUS3 images collected with a nanoparticle concentration of 0, 1 and 2 µl/mm^2.

Results

The isotropic scattering pattern indicates that the nanoparticles arrange in a 3D manner. As expected, higher order reflections become visible as the nanoparticle concentration is increased. Traditionally, an elongated beam stop is required to block both the transmitted primary beam and also the specular reflection, but the PILATUS3, with instant retrigger technology resulting in high dynamic range, eliminates the need for a beam stop in this case.

The data was imported into DIFFRAC.LEPTOS and integrated along the horizon line to produce a plot of intensity versus Qy, shown in Figure 3. First, second and third order reflections from the 10 nm Au nanoparticles are clearly visible near the expected positions of $Q = 0.62\ nm^{-1}$, $Q = 1.26\ nm^{-1}$ and $Q = 1.89\ nm^{-1}$ ($Q=2n\pi/d$).

Author

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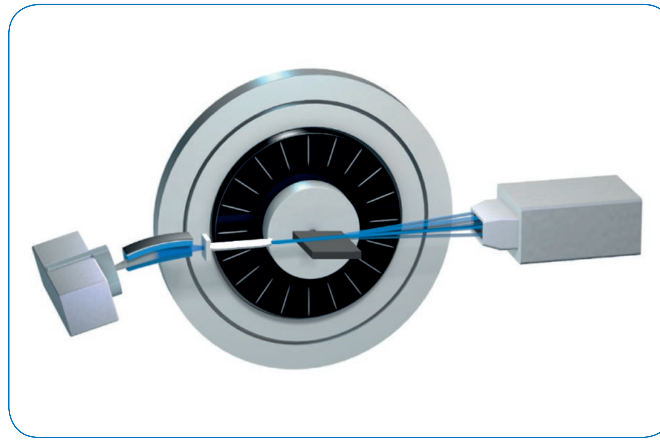

Figure 1: Instrumental setup used in this report.

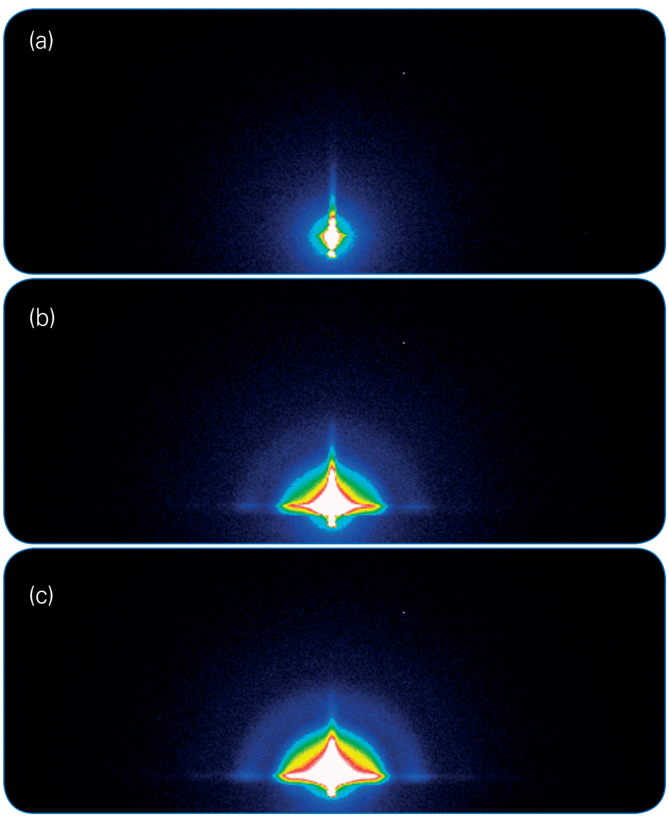

Figure 2: GISAXS result from PILATUS3 with various amounts of 10 nm Au nanoparticles applied to a Si wafer.
(a) 0 µl/mm² (b) 1 µl/mm² (c) 2 µl/mm².

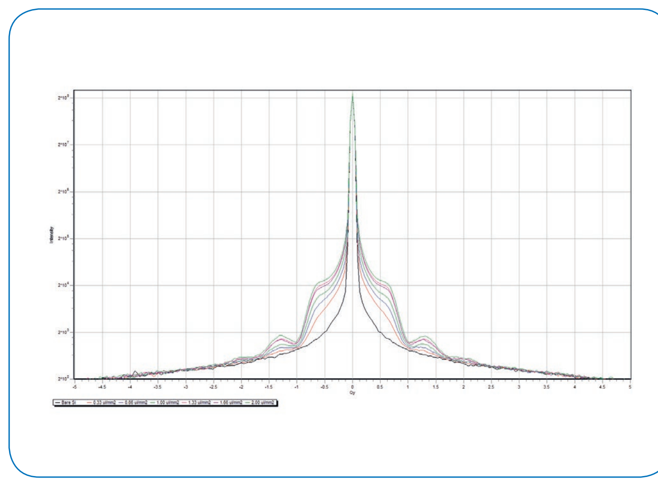

Figure 3: Qy integration of the data in Figure 2.

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