Analysis of ITO and *h*-BN thin films using GIXRD on ARL X'TRA Companion X-ray Diffractometer

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Introduction

Grazing Incidence X-ray Diffraction (GIXRD) is a specialized technique used to analyze thin films, surfaces, and nanomaterials. By utilizing a shallow angle of incidence, typically less than one degree, GIXRD enhances the sensitivity to the surface layers of a sample, making it ideal for studying thin films and coatings. This technique minimizes the penetration depth of X-rays, allowing for detailed analysis of surface properties and structures.

In a Bragg-Brentano X-ray Diffraction (XRD) instrument, GIXRD is performed by adjusting the geometry to maintain a low angle of incidence while the detector scans the diffracted X-rays. The Bragg-Brentano configuration, known for its high resolution and accuracy in phase identification, can be adapted for GIXRD by carefully controlling the incident angle and the alignment of the sample. Additionally, a parallel plate collimator (PPC) is helpful in this setup as it reduces the divergence of the X-ray beam, ensuring better resolution and accuracy in the diffraction data.

One of the key advantages of GIXRD is its capability for depth profiling. By varying the incidence angle, researchers can control the penetration depth of the X-rays, thus probing different layers within a multilayer system. This is particularly useful for studying the composition and structure of each layer in thin films and coatings. The absorption of X-rays in multilayer systems can also be analyzed, providing insights into the distribution of elements and phases throughout the depth of the sample. This comprehensive analysis enables detailed characterization of complex materials, essential for applications in materials science, nanotechnology, and surface engineering.

Figure 1. ARL X'TRA Companion X-ray diffraction system.

Instrument and software

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (Figure 1) is a simple, easy-to-use benchtop XRD instrument designed for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion XRD utilizes a decoupled θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry, coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a motorized beam knife. An integrated water chiller is available on demand. Thanks to the innovative solid-state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion XRD provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (laboratory information management system). To achieve grazing incidence geometry, a PPC with a selection angle of 0.2° is used. To ensure perfect sample positioning, pre-aligned containers are used which guarantee maximum precision and accuracy of the sample position. (cf. Figure 2)

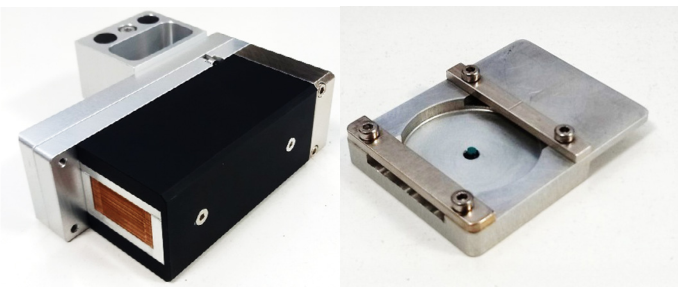

Figure 2. Pictures of PPC (left) and pre-aligned sample container (right).

Experimental

Measurements of an ITO on glass (Indium-Tin-Oxide; 100 nm) sample and two *h*-BN on silicon (130 nm and 50 nm) samples were carried out in GIXRD mode (1° incidence angle; 0.1 mm divergence slit, and anti-scatter slit; 0.2° PPC; detector in 0D mode) using Cu Kα (1.541874 Å) radiation (40 minutes per scan) (cf. Figure 3/4). The samples were put in pre-aligned sample containers in which they are pressed against a reference plane. Data processing was performed using Profex software [1].

Results and discussion

The two samples represent a simple (ITO) and a more complex (*h*-BN) example.

The GIXRD measurement (Figure 3) allows clear identification of ITO as the single phase. By adding or removing the Soller slit, it is possible to balance divergence and intensity. By removing the Soller slit, it is possible to increase the intensity by a factor of 4, but due to the increase in divergence, there is a loss of resolution (compare zoomed area in Figure 3 with normalized intensity).

In contrast to ITO, which is a good scatterer due to its high absorption and atomic weight, *h*-BN consists only of light elements and is therefore a poorer scatterer. This explains the lower intensity of the *h*-BN data in Figure 4. Nevertheless, both the 130 nm and 50 nm samples are measurable. Signals that don't belong to *h*-BN (brown indicators) are most likely due to the substrate (Si wafer)

Your Benefits

The ARL X'TRA Companion XRD excels in surface analysis through its optimized GIXRD configuration, featuring pre-aligned sample containers and a parallel plate collimator for precise measurements. The system's adaptable beam optics and solid-state pixel detector enable effective analysis of both strong and weak scattering materials. Combined with Profex software, this solution delivers efficient thin film characterization and depth profiling for advanced materials development.

[1] N. Döbelin, R. Kleeberg, *J. Appl. Crystallogr.* **2015**, 48, 1573-1580.

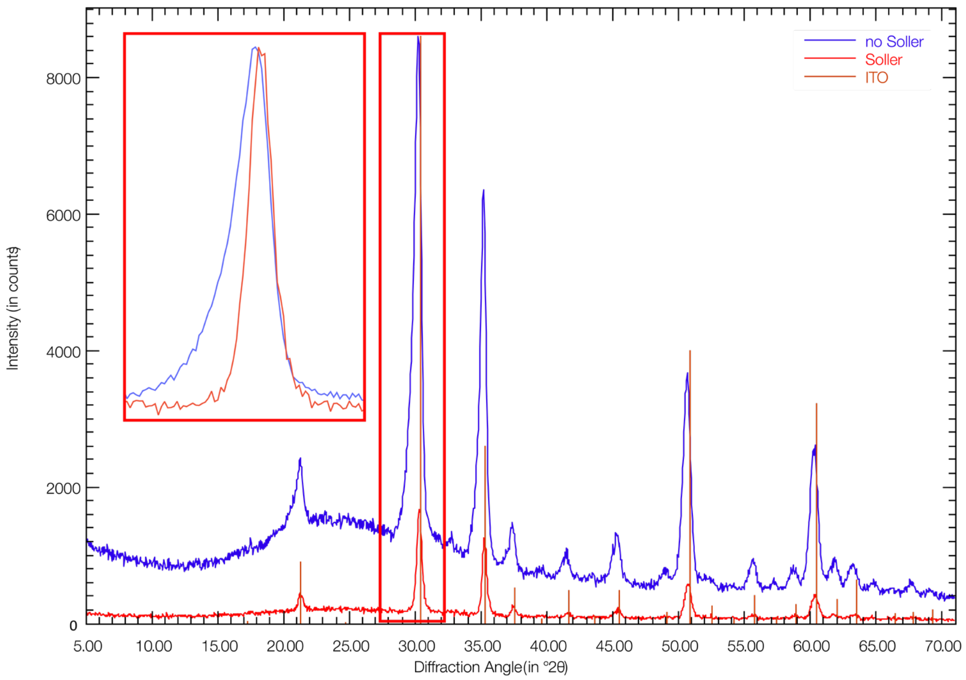

Figure 3. XRD pattern (1.0° incidence, 40 minutes measurement time) of ITO without (blue) and with Soller (red). Theoretical peak positions of ITO in brown. Normalized zoom from 28-32°2θ in red box.

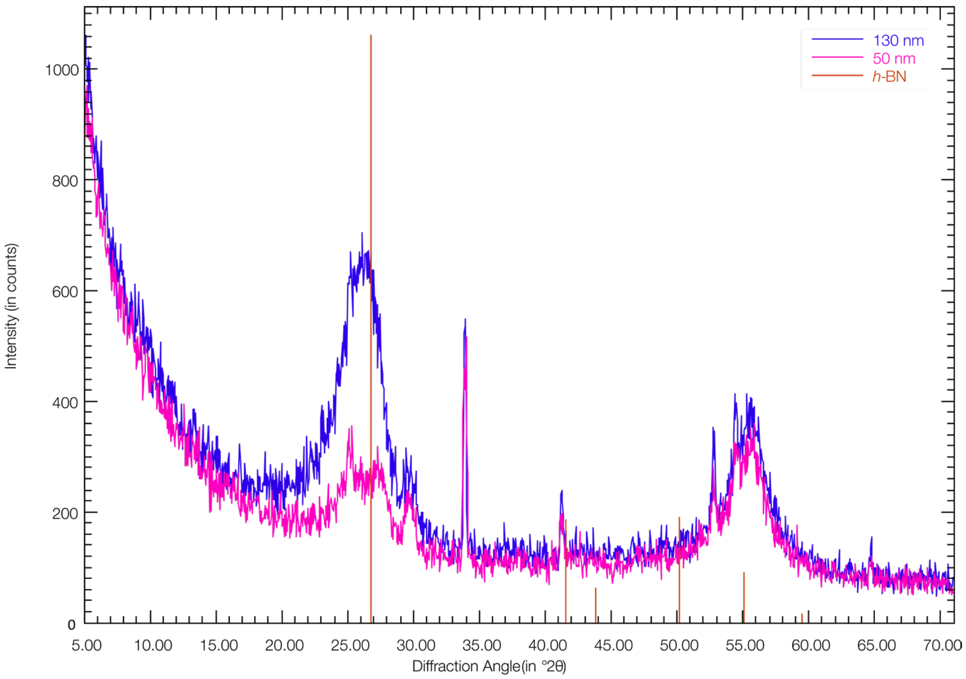

Figure 4. XRD patterns (1.0° incidence, 40 minutes measurement time, no Soller slit) of *h*-BN on a Si wafer with layer thickness of 50 nm (pink) and 130 nm (blue). Theoretical peak positions of *h*-BN in brown.

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