Contribution ID: **280** Type: **Contributed talk**

Grazing Incidence Diffraction and Scattering with High-Energy X-rays: Recent Innovations at Beamline P07 @ PETRA III

Friday 30 August 2024 15:45 (15 minutes)

Grazing incidence (GI) geometry, i.e. the illumination of a sample at very shallow angle on its surface and the creation of an evanescent wave parallel to the surface, is the foundation for x-ray diffraction and scattering studies of surfaces, interfaces and very thin films. At high x-ray energies (>50 keV), not only the Bragg angles are small, but also the critical angles of total external reflection assume low values <0.01° even for high-Z materials. In the second experimental hutch (EH2) of the high-energy beamline P07 at PETRA III [1], DESY offers the capabilities to routinely apply GI geometry by providing instrumentation of the necessary precision for sample alignment as well as tight focusing in the vertical to a few micrometers in order to keep the projected footprint on the surface short on the order of a few millimeters. The available large and fast area detectors for high-energy x-ray applications capture an extended range in reciprocal space in a single exposure within fractions of a second and thus record many different lattice planes simultaneously in real time that otherwise must be collected in time-consuming sequential measurements.
After the pioneering work on fast determination of surface reconstruction and phase formation during catalytic reactions at the gas-solid interface over single-crystal surfaces by Gustafson et al. [2], many more high-energy surface diffraction studies have followed using similar model catalysts and analyzing Bragg reflections and crystal truncation rods. This presentation highlights current advances of this technique at P07 e.g. operando (electro)catalysis with microsecond time resolution. [3]
Building onto the expertise in high-energy surface diffraction, we developed the beamline's capabilities for total scattering in grazing incidence to study the local atomic structure of thin films in real space by pair distribution function (PDF) analysis. The PDF is an intuitive tool to investigate the short-range order of amorphous and nanocrystalline materials as well as local disorder in periodic polycrystalline structures. We have achieved unprecedented sensitivity to layer thicknesses down to a few nanometers at time resolution in the range of seconds for the collection of individual 2D total scattering patterns up to high momentum transfer >20 Å-1 and even separated and resolved the signals from bilayer structures [4]. Recent in situ deposition and crystallization studies [5] showcase the method's strength as a unique tool to follow structural transitions in thin films between states of low and high degree of ordering. In this regard, especially the successful implementation of a laser-interferometer based sample stabilization system for variable temperature heat treatment is a major achievement to track the structural changes e.g. during post-deposition annealing typically applied in sputtering or atomic layer deposition.

[1] F. Bertram et al., AIP Conf. Proc. 1741 (2016) 040003, doi.org/10.1063/1.4952875
[2] J. Gustafson et al., Science 343 (2014) 758, doi.org/ 10.1126/science.1246834
[3] L. Jacobse et al., Rev. Sci. Instrum. 93 (2022) 065111, doi.org/10.1063/5.0087864
[4] A.-C. Dippel et al., Nanoscale 12 (2020) 13103, doi.org/10.1039/d0nr01847c
[5] M. Roelsgaard et al., IUCrJ 6 (2019) 299, doi.org/10.1107/S2052252519001192

I plan to submit also conference proceedings

No

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Session Classification: Mikrosymposium 5/2: Operando Investigations

Track Classification: 5. Diffraction and scattering for operando investigations