X-Ray Reflectivity and Reciprocal Space Mapping with a Micro-Focus Rotating Anode: From Micon-Sized Beams to Fractional-Order Bragg Peaks

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Historically, high beam flux and large detectors were accessible only at synchrotron light sources. Using the latest generation high-brilliance microfocus rotating anode generator and a large area hybrid photon counting detector in a custom-build diffractometer manufactured by Bruker AXS, we now have access to very weakly scattering features, such as non-integer Bragg reflections, that are notoriously hard to capture and were previously inaccessible to laboratory-based XRD tools.

In this talk, we will show some highlights of the research we have performed with this unique tool over the past six years at the MESA+ Institute for Nanotechnology at the University of Twente on epitaxial complex oxide thin films grown by the pulsed laser deposition technique. To showcase the unprecedented scanning and mapping capabilities of the state-of-the-art tool both in real space as well as in reciprocal space, we present three case studies, which all benefit from the availability of an intense, well-collimated, and monochromatic X-ray beam with a small footprint and a 2D detector with high dynamic range (EIGER2 R 500K by Dectris).

Firstly, we demonstrate how microfocus X-ray reflectivity (XRR) allows us to elucidate the lateral thickness gradient in a complex oxide heterostructure (LaAlO3 / SrTiO3) wedge sample of few unit cells thickness and correlate this data with reflection high-energy electron diffraction (RHEED) data – a widely used tool to study the growth dynamics of thin films, in situ and operando [1]. Going beyond reflectivity, we show how microfocus X-ray diffraction (XRD) allows us to systematically study compositional gradients in off-center deposited BaTiO3 thin film wedges [2].

Secondly, we show high-resolution X-ray diffraction (XRD) measurements on lithographically defined ferroelectric thin films (BiFeO3 / DyScO3 and PbZr0.55Ti0.45O3 (PZT) / SrTiO3), using an X-ray beam size of only 20 micrometer to probe individual microcapacitors and reconstruct 3D reciprocal space maps [2, 3]. Thirdly, we show how laboratory-source 3D RSMs can detect off-specular fractional-order Bragg peaks in a coplanar asymmetrical experimental geometry. Showcasing the example of PbZrO3/Pb0.9La0.1Zr0.52Ti0.48O3 multilayers, the analysis of weak superstructure reflections inaccessible to most thin-film diffractometers with line-focus beams and small detectors, revealed the formation of interfacial layers with rotated polarization that greatly enhanced the energy storage density and breakdown strength [5].

References

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