XRR with nanometre beams - Characterising micrometre surfaces

Steffen Tober[1], Andreas Stierle[2], Hans-Georg Steinrück[3], Vedran Vonk[4], Jiangtao Zhao[5], Steven Leake[5], Azat Khadiev[1], Wieland Corts[6], Toka Matar[6], Lisa Randolph[6], Breno Rabelo Couthino Saraiva[7]

[1] *DESY Photon Science, Deutsches Elektronen-Synchrotron (DESY), Germany*
[2] *Deutsches Elektronen-Synchrotron (DESY), University of Hamburg, Germany*
[3] *Forschungszentrum Jülich GmbH, Germany*
[4] *Deutsches Elektronen Synchrotron DESY, Germany*
[5] *ESRF, France*
[6] *Institute for a sustainable Hydrogen Economy, Forschungszentrum Jülich, Germany*
[7] *Deutsches Elektronen-Synchrotron (DESY), Centre for X-Ray and Nanoscience (CXNS), University of Hamburg, Physics Department, Hamburg/Germany*

Presentation type: Oral **Presentation time:** 09:30-09:45

The use of X-ray reflectivity (XRR), an established method for non-destructive probing of electron density profiles of semiconductor [1], polymer [2] or model catalyst [3] surfaces and interfaces with sub-nm-resolution, so far is limited to flat, mm-sized samples. The large effective footprint of the X-ray beam at relatively small incident angles results in a spillover that significantly reduces the reflected signal and increases the background for smaller samples. To study smaller samples such as dispersed catalyst particles or individual particles of multigrain catalysts, a significant reduction of the footprint is necessary.

As demonstrated for crystal truncation rods by Stubbs et al., the nm-sized X-ray beams of modern synchrotron sources with a correspondingly small footprint can be used for XRR on µm-sized samples [4]. Small sample and beam sizes, however, require a precise positioning procedure of the sample to ensure reliable measurements [5].

We present proof-of-principle studies comparing the nanobeam XRR of mm- and µm-sized thin-film samples (Au/Ti on Si) measured with 400 and 80 nm X-ray beams at ESRF ID01 focused using compound refractive lenses (CRL) or Fresnel zone plates (FZP), respectively [6]. Our results show a reasonable agreement between XRR on mm- and µm-sized parts of the sample and thereby the general feasibility of XRR of µm-sized samples using nm-sized beams. Adjustments to the scan procedure allowed a reliable, precise positioning of the beam on the sample and are expected to enable adapting our method for studies of catalysts under in situ and operando conditions.

References

[1] S. L. Moffitt et al., JPCC 127 23099 (2023)
[2] P. Lakner et al.,Langmuir 36 13448 (2020)
[3] R. Gleißner et al., ACS Appl. Nano Mater. 6 8004 (2023)
[4] J. Stubbs et al., Clays Clay. Miner. 69 688 (2021)
[5] V. Vonk et al. J. Appl. Cryst. 58 1978 (2025)
[6] S. J. Leake et al., J. Synchrotron Rad. 26 571 (2019)