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Novel fast-X-ray reflectivity setup for studying electrochemical interfaces

A fundamental understanding of atomic scale processes occurring at electrochemical interfaces is crucial for advancing the development of energy-related electrochemical systems such as ion batteries or fuel cells. For this purpose, operando experiments are imperative. X-ray reflectivity (XRR) is a non-destructive technique that enables operando analyses of buried structures with atomic resolution [1,2]. However, the time-resolution of typical XRR setups of minutes per XRR scan is limited by diffractometer motor motion and detector read-out times; this resolution is in many cases too slow to observe interfacial electrochemical phenomena [1,2].
To overcome these limitations, we developed a novel fast-XRR setup, which employs a fast quasi-continuous rotation stage in combination with a stationary, high frame rate area detector. With this setup, the sample can be rotated with a frequency of up to 10 Hz, yielding sub-second time resolution for an individual XRR scan. In contrast to complementary fast-XRR setups [3,4], the developed setup can easily be combined with complex sample environments, as typically required for the investigation of electrochemical interfaces.
We present first fast-XRR results obtained at PETRA III beamline P08 at DESY using 18 keV and a Dectris Eiger1M detector. We reached a time resolution below one second for an incident angular range between 0° – 6° (≙ 0 – 2 1/Å). We measured several multilayer samples and benchmarked the results to "standard" XRR measurements, where the measurements took several minutes. We find good agreement and conclude that our novel setup provides significant XRR data within less than one second, demonstrating its usability for operando XRR measurements.

Literature:

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Instrumentation and methods

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