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Grazing incidence X-ray ptychography for *in situ*

studies of thin sub-monolayer films of nanoparticles

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Introduction

X-ray ptychography [1] is a scanning coherent diffraction imaging (lensless) technique that provides unlimited fields-of-view for the sample reconstruction

and enables reconstruction of the generally unknown illumination function [2]. In ptychography a phase-retrieval algorithm plays the role of an imageforming lens by recovering the unknown phase numerically, using iterative algorithms [3]. Here, we present ptychographic imaging under grazing incidence as a technique suitable for investigation of surface properties of thin films. The grazing incidence configuration is of special interest for the study of sparse monolayers of nanoparticles that yield weak scattering signal in a conventional transmission configuration. The proposed method has a potential for *in situ* studies of particle-substrate interactions in a gaseous environment, under elevated temperatures and will allow describing timeevolution of an inhomogeneous sample structure.

Grazing Incidence Ptychography

In grazing incidence configuration, coherent X-ray scattering from substrate-supported nanostructures is measured below the critical angle of the sample substrate.



Figure 1: Schematic of grazing incidence ptychography.

Experimental results

The experiment was performed at the cSAXS beamline of the Swiss Light Source (SLS) facility in Switzerland. Figure 4 shows part of a Siemens star phantom that corresponded to the imaged area used for ptychographic reconstruction along with an amplitude of the reconstructed Siemens star and reconstructed illumination function [5]. (under a grazing incidence angle of 0.27 degrees, both corrected with respect to the aspect ratio of the reconstruction pixel size).



Figure 4: (a) Scanned part of the Siemens star phantom, the white region is showing entire size of the supporting wafer, black pattern is the Siemens star with highly anisotropic resolution. (b) Reconstruction of the amplitude signal reconstructed from diffraction data from the sample corresponding to the region simulated in (a). (c) Reconstruction of the illumination function

The shallow incident angle provides a high interaction cross-section with the sample because of the large footprint and the total external reflection of the incident beam from the substrate.





Figure 2: (a) Beam footprint on the sample at an incident angle of 0.27°. (b) Illuminated part of the Siemens star phantom with an aspect ratio of 1.

Preliminary simulations show that reconstruction of the sample can be achieved by the proposed method using modified propagation of the exit wave front from the sample plane to the detector [4].

(b)

Reactor chamber for *in situ* measurements



Figure 5: (a) Etched structure of an *in situ* grazing-incidence X-ray scattering micro-reactor flow cell. The gas inlets (1) and (2), the bypass (3), the capillary to the mass spectrometer (4), 10 µm-thick entrance and exit windows (5) and (6), beam path without supporting pillars (7) and supporting pillars to avoid reactor chamber collapse when working at lower than ambient pressure (8) are labelled on the image. (b) Close-up of the 10 µm-thick Si entrance window and pillar structure of an anodic-bonded closed reactor template before etching and dicing. (c) Schematic representation of the micro-reactor device [6].

Future work

Future improvements to the method will include grazing incidence ptychographic tomography for achieving isotropic resolution in object reconstruction. This requires better alignment of the measured projections, higher precision in the sample motion, and a new design of the reactor chamber for *in situ* studies.

Acknowledgments



(a)



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References

- 1. J. M. Rodenburg, et al. "Hard-X-ray lensless imaging of extended objects," Phys. Rev. Lett., vol. 98, no. 3, pp. 1–4, 2007.
- 2. P. Thibault, et al. "Probe retrieval in ptychographic coherent diffractive imaging." Ultramicroscopy 109.4 (2009): 338-343.
- 3. P. Gilbert, "Iterative methods for the three-dimensional reconstruction of an object from projections," J. Theor. Biol., vol. 36, no. 1, pp. 105–117, 1972.
- 4. T. Sun, et al. "Three-dimensional coherent X-ray surface scattering imaging near total external reflection." Nature Photonics 6.9 (2012): 586.
- 5. M. Odstrčil, A. Menzel, and M. Guizar-Sicairos, "Iterative least-squares solver for generalized maximum-likelihood ptychography," Opt. Express, 26(3), 3108, 2018
- 6. J. Kehres, et al. "Novel micro-reactor flow cell for investigation of model catalysts using in situ grazing-incidence X-ray scattering." J. Synchrotron Radiat. 23.2 (2016): 455-463.

Figure 3: Tilted plane correction of a diffraction pattern: (a) before and (b) after correction

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