Grazing-incidence X-ray ptychography for in situ studies of thin sub-monolayer films of nanoparticles

Slyamov, A. M.; Jørgensen, P. S.; Rein, C.; Odstril, M.; Silvestre, C. M.; Andreasen, J. W.

Publication date: 2019

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain.
- You may freely distribute the URL identifying the publication in the public portal.

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.
Grazing incidence X-ray ptychography for in situ studies of thin sub-monolayer films of nanoparticles

A. M. Slyamov1, P. S. Jørgensen1, C. Rein1, M. Odstrčil2, C. M. Silvestre3, J. W. Andreassen1†

1. Technical University of Denmark, Department of Energy Conversion and Storage, 4000 Roskilde, Denmark
2. Paul Scherrer Institute, 5232 Villigen PSI, Switzerland
3. Technical University of Denmark, DTU Nanolab, DK-2800 Kgs. Lyngby, Denmark

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 image-forming 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 time-evolution 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.

\[ \alpha_s = \arcsin \left( \frac{\lambda}{2n} \right) \]

\[ \alpha_s \] is the critical angle of the sample substrate.

\[ k_{\text{inc}} \] is the incident wavevector.

\[ k_{\text{sc}} \] is the scattered wavevector.

\[ \theta_i \] is the angle of incidence.

\[ \theta_r \] is the angle of reflection.

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 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).

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 [5].

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

This study was supported by the Marie Skłodowska-Curie Innovative Training Network MUMMERING (Multiscale, Multimodal, Multidimensional imaging for Engineers), funded through the EU research programme Horizon 2020 and by the Ministry of Higher Education and Science (DANSCATT grant, 7055-000078). The authors would like to acknowledge the support of the staff of the NanoMAX beamline at the MAX IV Laboratory, O. Carbone, A. Bijing, and A. R. Fernández and of the cSAXS beamline at the Swiss Light Source, A. Diaz. The authors thank D. Hansen, C. D. Damsgaard, and B. Chong from DTU Nanolab who provided basic structures for the experiment.

References