MATERIAL IDENTIFICATION AT NANOMETER SCALES USING SINGLE-SHOT HYPERSPECTRAL PTYCHOGRAPHY

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Summary: First proof of principle combination of hard x-ray ptychography with hyperspectral imaging at the I13-1 branch of DLS. Single shot discrimination of Ni versus Cu has been demonstrated at 96 nm pixel size using K-edge subtraction, as well as measurement of the Ni K-edge spectral profile from a single ptychographic acquisition.

1. INTRODUCTION

X-ray ptychography is a microscopic imaging technique that differs from conventional microscopy in a few key ways. Where conventional imaging achieves magnification using specific source geometries (cone beam, fan beam, …) or lensing optics, ptychography is a lensless technique based on a highly coherent light source. Instead of recording a single direct image, a very small coherent beam is scanned across the sample in an overlapping grid, and a far-field scatter pattern is recorded using a 2D detector for each grid point.
An iterative algorithm[1,2,3] is used to reconstruct the original 2D projection instead of an objective lens, allowing this technique to achieve a theoretical spatial resolution down to the diffraction limit, with practical applications reaching resolutions of 10nm and better.
Hyperspectral X-ray detectors are a class of cameras which provide full spectral information across an entire 2D image. These systems are usually photon counting and provide direct measurement of single-photon deposited charge through readout of raw ADC values. This provides a large number of energy bins, typically on the order of a few thousands of bins roughly a few eV wide.

We present the first proof of principle combination of both X-ray ptychography and hyperspectral imaging, allowing capture of both chemical information and very high resolution spatial details in a single ptychographic acquisition. Additionally, during this proof of principle test a few key points of future study were identified, and the implications of spectral ptychography using a pink beam on the reconstruction algorithms were explored.

2. EXPERIMENTAL METHOD

The experiments were performed at the I13-1 coherence branch[4] at Diamond Light Source under proposals MG22099 and MG23140.

Detector

The SLcam detector system used for this experiment is based on the pnCCD, a hyperspectral photon-counting sensor using a 450 µm thick Si active volume. The active area consists of 264 by 264 pixels of 48 µm pitch, read out as a CCD at a framerate of 400 Hz. Each sensor quadrant is read out by an 11 bit ADC, with the raw ADC values serving directly as input to a custom hyperspectral camera control and data processing framework[5] developed at Ghent University. Using this system, an energy resolution of better than 150 eV FWHM can be reached at the 5.9 keV Mn Kα line, approaching the performance of state-of-the-art single pixel energy dispersive detectors.

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Setup

A test phantom consisting of a Ni and a Cu mesh (10 µm width, 2 µm and 5 µm resp. thickness) overlayed on top of each other was imaged around the 8340 eV Ni K-edge. The beamline was configured using a Fresnel zone plate (FZP) setup including upstream central beam stop and an order selecting aperture (OSA). The beam spectrum was tuned to have a third harmonic centered at 8335 eV and 180 eV wide, and the beam size was around 9 µm at 8340 eV.
A 16x16 ptychography scan was performed over 1.5x1.5 μm^2 of the sample with 80 s exposures. The fluorescent spectrum was captured using an off-axis silicon drift detector.

3. RESULTS

As a first check a 2D ptychographic image of a Siemens star was acquired to explore detector performance and spatial resolution when used for ptychography. A reconstructed pixel size of 96 nm was achieved, and an integration time of 80 s per ptychography step was chosen based on flux and pixel statistics.

Pink beam ptychography

From a single ptychographic acquisition using a 180 eV wide pink beam a successful identification of the Ni mesh against the Cu mesh was possible. This discrimination of Ni versus Cu was confirmed via simultaneous measurement of the fluorescent spectrum at each ptychography sampling point, providing a coarse map of the materials in agreement with the K-edge subtraction image created from the hyperspectral data.

By further refinement of the acquired data a detailed K-edge spectral profile could be extracted from a single acquisition, convoluted with detector energy response and beam spectrum.
Using this data a transmission-energy plot was obtained at intervals of 26 eV between 8227 eV and 8461 eV. For a region inside a Ni mesh wire, this curve shows a transmission decreasing with higher energy, centered around the 8333 eV Ni K-edge. Applying this same method to the Cu mesh results in a flat transmission curve over the observed energy range.

In this presentation we show the measurement results of the phantom experiment, as well as a first practical application on zeolite catalyst particles around the Bromine K-edge. Furthermore, the implications of using a pink beam instead of a monochromatic beam on the ptychographic reconstruction is discussed, as well as the specific instrumentation challenges encountered.

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