850
doi:10.1017/S1431927622003786
Microsc. Microanal. 28 (Suppl 1), 2022


# Development of Operando X-ray Ptychography at the Advanced Light Source

David Shapiro[1*], Rich Celestre[1] and Young-Sang Yu[2]

[1.] Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA, USA
[2.] Department of Physics, Chungbuk National University, Chungbuk, Korea
* Corresponding author: dashapiro@lbl.gov

X-ray microscopy has developed over the last 50 years as a powerful complement to optical and electron microscopy [1]. It offers high sensitivity to electronic and magnetic states, penetration through bulk volumes of material and the feasibility of nanometer scale spatial resolution because of short wavelengths. Similar to electron microscopes, x-ray microscopes have suffered from imperfect lenses and the damaging effects of ionizing radiation. More recently, x-ray microscopy methods, like ptychography, based on phase retrieval from coherent scattering measurements, have achieved diffraction limited spatial resolution through a numerical separation of the illumination function [2]. This has allowed the achievable spatial resolution to approach the wavelength of the light, a few nanometers, but at the expense of long acquisitions times which reduce temporal resolution and lead to high radiation dose thereby potentially damaging the sample. These characteristics of a ptychographic microscope make operando measurements particularly challenging.

The Advanced Light Source has developed an x-ray ptychographic microscope which is dedicated to advanced sample manipulations like operando environments, tomography and cryogenic temperatures [3]. As a high resolution scanned probe method, ptychography is inherently slow so a focus of our development has been precise and fast positioning of the x-ray beam relative to the sample. Further, to facilitate correlative measurements with other types of probes and to speed the implementation of operando environments, our microscope is compatible with commercially available sample environments designed for transmission electron microscopes [4]. In fact, our facility makes available for general use sample environments capable of fluid flow with biasing or heating and also gas flow with heating. These have been implemented with no additional design effort.

Our microscope can operate as a conventional scanning probe x-ray microscope with 50 nm spatial resolution or in ptychographic imaging mode with as high as 8 nm spatial resolution. We present strategies for accelerating high resolution data acquisition for improved time resolution and reduction of radiation dose which can damage active materials. In particular, we show that high resolution chemical maps can be achieved with only a few energy points using x-ray energies away from the absorption resonance, thereby reducing x-ray dose [5]. We also show some of the signatures of radiation damage, like beam induced reduction, carbon deposition and shrinkage and suggest ways to isolate those affects from the material dynamics of interest [6].

[1] C Jacobsen, In *X-ray Microscopy* (Advances in Microscopy and Microanalysis, pp. I-Iv). Cambridge: Cambridge University Press.
[2] P Thibault et al., Science **321**(5887) (2008), p. 379.
[3] DA Shapiro et al., Science Advances **6**(51) (2020).
[4] HD Deng et al., Nature Materials (2022). https://doi.org/10.1038/s41563-021-01191-0
[5] M Farmand et al., Applied Physics Letters **110**(6) (2017), 063101
[6] M Yoo et al , . Energy Environmental Science **13** (2020) , p. 1231 .