**Mid-Term Plan for 2-BM Upgrade:**
**Integration of Microtomography and Nanotomography**
**Capabilities at Beamline 2-BM**

Yong S. Chu and Francesco De Carlo

Scientific Motivation

In recent years, hard x-ray imaging has found tremendous application in many scientific disciplines, especially in biology and materials sciences. The increasing scientific demand for 2D and 3D x-ray imaging is primarily aimed at higher spatial resolution, higher energies for deeper penetration, higher sensitivity using phase-contrast, and higher imaging speed for investigation of dynamics. At the APS, the growing interest in hard x-ray imaging is evidenced by the recent birth of a dedicated full-field x-ray imaging beamline at 32-ID and the existence of dedicated microtomography instruments (2-BM, 13-BM, and 5-BM) at the APS.

Despite the fast growing scientific user communities for x-ray imaging, there remains a huge discrepancy between the spatial resolution needed for solving scientific problems and the spatial resolution (~1 μm) delivered by the APS instruments. Moreover, the majority of microscopy investigation requires the capability to image a sample at multiple length scales from microns to nanometers. For example, understanding the complex electrochemical reactions occurring in a solid oxide fuel cell (SOFC) and modeling the multi-component gas transport at the triple-phase-boundary requires 3D structural arrangement of the pores and their connectivity before and during the operation of the cell (see the list of scientific cases below). The pore size can range from a few nanometers to a few microns. There exist a vast number of scientific problems across multiple scientific disciplines that have never been examined because of 1) the lack of spatial resolution in 3D x-ray imaging and 2) the limited probing depth of electron microscopy.

The goal of our plan is to develop a full-field nanotomography capability with the spatial resolution aimed at 30 nm, fully integrated with the existing microtomography capability at beamline 2-BM. The integration of nanotomography with the existing microtomography capability at beamline 2-BM will provide a unique x-ray microscopy facility to the general user community, in which three dimensional structure of variety of samples can be investigated at tens of microns to nanometers continuously using “zoom in” and “local” tomography techniques.

Targeted Scientific Problems

We identified several specific scientific problems that can be uniquely or efficiently investigated using the new capability outlined in this proposal:

- Investigation of Biological Structures (Yeukuang Hwu, Academia Sinica, Taiwan, Stuart Stock, Northwestern University): Most of the biological systems consists of multi-length scale structures from microns to nanometers. There exist a vast number of scientific problems that can be effectively investigated by the “zoom in” microscopy capability with the spatial resolution of tens of microns to tens of nanometers.
- *In situ* Structural Investigation of Solid Oxide Fuel Cells (W. Chiu, University of Connecticut): development of more efficient and robust solid oxide fuel cells requires 3D visualization, with microns to nanometers resolution, of the evolution of microstructures and chemical reactions occurring at the triple-phase-boundary. The 3D visualization of pores and their connectivity will lead to accurate modeling of the multiple gas transport and electrochemical reactions and help elucidate the fundamental mechanism of catalytic activities at the electrodes.
- 3D Structural Investigation of Integrated Circuit (IC) Devices for National Security (M. Bajura, University of Southern California): DARPA assigned a proof-of-concept grant for demonstrating the capability to detect possible design modification and/or tampering of ICs, supplied by the foreign manufactures, which are intended to be incorporated in the electronic instruments critical to the national security. This research project requires rapid inspection of

ICs over a large area with a spatial resolution better than 100 nm, which is only possible using synchrotron x-rays.

- *In Situ* Imaging of Nanoporous Metal Foam Formation (D. Dunand, Northwestern University): Nanoporous metals display novel physical, chemical and mechanical properties, accompanied with potential application in such diverse areas as catalysis, heat exchange, mechanical actuation, and sensors. X-ray nanotomography will reveal the formation, annealing and growth of nanopores and will help correlate the evolution of the nano and microstructure with the material properties.
- *In Situ* Investigation of Electrochemical Corrosion and Growth Processes (A. J. Davenport, University of Birmingham, United Kingdom, J. H. Je, Pohang University of Science and Technology, Korea): Multi-scale x-ray imaging with resolution from tens of nanometers to microns can visualize how the corrosion attack initiates at the grain boundaries and leads to structural failure. The same capability can be used to investigate how the electrochemical interface evolves during the nucleation and growth.
- Development of Full-field X-ray Diffraction Microscopy (Y. Chu, XOR and J. M. Yi, University of Science and Technology, Korea): Feasibility to image the diffraction contrast to visualize the crystalline defects at nanoscale has been demonstrated and quantitative analysis method is being developed.

Added Value of the Medium Term Upgrade

The goal of this proposal is to outline a cost-effective plan for developing a unique 3D x-ray imaging instrument with a spatial resolution from 10 microns to 30 nanometers at XOR beamline 2-BM with the imaging throughput necessary for real time or *in situ* application.

The XOR microtomography system at beamline 2-BM, developed over last several years, is fully operational with extreme measurement throughput up to 100 samples per day and a dedicated computing infrastructure, allowing online data analysis and 3D rendering. The critical limitation of this system is its intrinsic spatial resolution limit of about 1 $\mu$m, originating from the use of the visible-light optical lenses in the CCD system. Overcoming this resolution limit requires a completely different type of instrumentation known as transmission x-ray microscope.

In 2007-2 cycle, the X-ray Microscopy and Imaging Group of the APS launched a joint development of a state-of-art full-field transmission x-ray microscope (TXM) at beamline 32-ID under a partner-user proposal (PUP-64, active until 2011-1). Because of the magnification of the sample image by x-ray lenses, the intrinsic resolution of a TXM is not limited by the wavelength of the visible light but by the diffraction limit of the x-ray optics. The TXM at 32-ID has demonstrated a spatial resolution of 40 nm.

Presently, about 25% of the total available beamtime is allocated to this instrument, of which 20% is committed to the PUP experiments and only 5% is distributed to the general user experiments. In 2008-2 cycle, nine active general user proposals are competing for less than nine shifts available to the general users. Despite the overwhelming general user interests, this instrument is completely idle 75% of beamtime due to the experiments running on other instruments at 32-ID.

Our plan to integrate the nanotomography and microtomography capabilities into a single instrument consists of two phases. In the first phase, we propose to use the TXM at 2-BM during its idle time. We will develop an engineering solution to integrate the optical components of the TXM into the microtomography instrument at 2-BM, with an easy and reproducible method to position the optical components back to the TXM when it is used at 32-ID. In addition, we plan to install focusing optics at 2-BM, in order to ensure sufficiently high imaging throughput (< 2 seconds per frame) for nanotomography.

Before the end of PUP-64, granting our use of the TXM owned by the Academia of Sinica, we will seek external funding to acquire a new TXM at 2-BM and proceed into the second phase.

A new microscope owned by XOR will ensure the continuation of the scientific programs initiated by the integrated nano and microtomography capability developed in the first phase.

The added values of our plan are:

- To increase the utilization of the existing TXM from 25 to 75% (25% at 32-ID plus 50% at 2-BM) of the beamtime attracting a larger user community that cannot be supported with the current beamtime allocation at 32-ID. Furthermore, the increased beamtime will accelerate the development of more robust nanotomography instrumentation.
- To serve both micro and nano imaging communities, providing "zoom in" capability. This integrated approach will build user communities focused on science rather than centered on the instrument, will greatly expand the user communities in both biological and material sciences and will increase opportunities to attract external funding to support the second phase.
- To consolidate staff expertise and the computational infrastructure in one beamline.

Expected user communities

The scientific user communities for x-ray imaging are extremely diverse including biology, environmental science, material science, mechanical engineering, chemistry, and condensed matter physics. We can confidently project that about 50% of current microtomography users are interested in visualizing the 3D structures well below a micron.

Enabling technology and infrastructure

The following are the major components for this proposal.

- A full-field x-ray transmission microscope (TXM) capable of nanotomography: The TXM installed at 32-ID costs 1.5 million USD. In this proposal, we are not requesting XOR funds to purchase a microscope for 2-BM because we are planning to increase the utilization of this unique instrument.
- Smart Integration of the 32-ID TXM at 2-BM and Enhancement of Motion Control: We plan to implement kinematic mounting and positioning system for critical optical components of the TXM enabling us to transport them back and forth between 32-ID and 2-BM without need to perform time-consuming alignment procedures. In addition, we will enhance the performance of the sample rotation and x-y-z translation motion.
- Installation of x-ray focusing optic at 2-BM: Pre-focusing of the bending magnet x-rays is imperative to ensure a sufficiently high imaging throughput (< 2 seconds per frame). We are considering installation of either a single bendable toroidal x-ray mirror or a bendable saggital focusing 2$^{nd}$ crystal monochromator plus a vertically focusing x-ray mirror.

Partnerships and user interest

- Yeukuang Hwu, Academia of Sinica, Taiwan: He is the principal investigator of PUP-64, who purchased the TXM at 32-ID. He is in full support of this proposed upgrade plan and a key collaborator for enhancing the motion control of the TXM. Recently, prof. Hwu succeeded in the fabricating an x-ray Fresnel zone plate producing a 30 nm spatial resolution and will continually support in further enhancing the spatial resolution.
- Mike Bajura, University of Southern California, Information Sciences Institute: Under DARPA's "TRUST in Integrated Circuits" Program, he is interested in developing a high-throughput 3D imaging capability for ICs, in order to detect possible design modification and/or tampering of the ICs, supplied by the foreign manufactures, which are intended to be incorporated in the electronic instruments critical to the national security. DARPA has shown significant interest in x-ray nanotomogphy capability. Once the high-throughput 3D imaging capability is successfully demonstrated to DARPA, Dr. Bajura plans to coordinate with DARPA to develop a dedicated nanotomgraphy instrument at 2-BM.

- Wilson Chiu, University of Connecticut.
- Stuart Stock, Northwestern University.
- David Dunand, Northwestern University.
- Alison Davenport, University of Birmingham, United Kingdom.
- Jung Ho Je, Pohang University of Science and Technology.
- Ian K. Robinson, University London College.
- Philip Withers, University of Manchester, Aerospace Research Institute.
- Tilo Baumbach, ANKA.

Industry and technology transfer

- High throughput nanometer scale inspection facility for IC characterization.

Estimated Budget

| | Equipment Budget Table | | Phase 1 | Phase 2 | Phase 3 |
|---|---|---|---|---|---|
| | **2-BM Beamline Upgrade ($k)** | ***1340*** | 700 | 230 | 410 |
| **1.1** | **Beamline optics** | **400** | | | |
| 1.1.1 | x-ray focusing optics | | 400 | | |
| **1.2** | **Nano Tomography** | **580** | | | |
| 1.2.1 | Enhancement of Motion Control | | 200 | | |
| 1.2.2 | Ancillary equipment (Optical table, optimization-engineering for TXM rapid transfer-setup between 32-ID and 2-BM) | | 100 | | |
| 1.2.3 | Dedicated CCD Camera | | | 80 | |
| 1.2.4 | 50% cost for dedicated optics optimized for 2-BM | | | | 200 |
| **1.3** | **Micro Tomography** | **110** | | | |
| 1.3.1 | Enhancement of Motion Control (autocollimator feedback, air bearing rotary stage etc.) | | | | 70 |
| 1.3.2 | CCD camera update | | | | 40 |
| **1.4** | **Micro-Nano Shared Equipmenent** | **250** | | | |
| 1.4.1 | Pre-alignment station | | | 150 | |
| 1.4.2 | Computational Infrastructure update | | | | 100 |