2018 SAC Review of the NSLS-II Beamlines

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HXN – Hard X-ray Nanoprobe – 3-ID

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1. Executive Summary

The Hard X-ray Nanoprobe (HXN) provides world leading routine nanoscale microscopy with hard x-rays. HXN supports two microscopes, one currently a 50 nm Zone Plate (ZP) resolution optics with long working distance and the other a best-in-world ~10 nm resolution multilayer Laue lens (MLL) based microscope. HXN is used for three focus areas: (i) quantifying trace elemental composition in materials via X-ray Fluorescence Microscopy (XFM); (ii) imaging crystalline ordering or strain with scanning microscopy and ptychography on Bragg peaks; and (iii) structural evolution as a function of in-situ controls using XFM, Differential Phase Contrast (DPC) imaging and ptychography in a single multimodal capability with up to 8 nm spatial resolution.

Currently, materials science and condensed matter users appear to be the community best suited to HXN capabilities; the science program is well aligned and in turn is appropriately driving the technique development. The committee recommends that HXN continues with current improvement and upgrade plans. HXN is encouraged to further expand the user community and the beamline staff is advised to strongly favor potentially high impact 2D work in the near term, while selectively working with users and techniques on 3D capabilities of high scientific impact as they continue to develop.

The beamline staff at HXN is very highly regarded by the user community. We strongly support the addition of a new staff member, to backfill both for the effective reductions caused by reorganization and departure of beamline postdocs in the last year. This additional staff member is intended to focus on in-situ or spectroscopic capabilities at HXN. The support labs at HXN are well maintained by the staff and the plan to upgrade the current scanning electron microscope (SEM) is sensible. Currently there are no Partner Users at HXN. The committee believes this may be a missed opportunity to supplement staffing with on-site expert users and develop new capabilities.

HXN staff have a well thought out upgrade plan going into the future. The renewal of the Tomography stage is particularly important to reach routine 3D capabilities. The committee recognized that data analysis capabilities and the ability of users to connect the unique data coming out of HXN to their science is sometimes a barrier to publication. The addition of one group-level data analysis support staff is recommended to help develop and maintain codes as well as support users in the post experiment phase. This staff member would help bridge the gap from experiment to publication and improve the output of every beamline in the imaging group.

2. Beamline Capabilities

2.1. Committee observations of world-leading/unique capabilities:

HXN routinely provides ~10 nm focused hard x-ray beams using a MLL for microscopy into an instrument which has the stability and positioning to take advantage of them. In the same instrument, the beamline also provides a ZP lense with down to 50 nm beams and longer working distance. The 30 nm ZP optic was recently damaged, temporarily limiting the instrument to 50 nm. A replacement 30 nm ZP is currently being acquisitioned. If ZP testing is successful, HXN will be able to provide routine 30 nm beams with long working distance. This instrument complements the 10 nm MLL module for in-situ and Bragg diffraction microscopy. Multimodal imaging with simultaneous XFM, DPC, and ptychography is a unique facility at this high resolution. Ptychography provides sub-spotsize structural information of the sample and high resolution images of the x-ray spot itself that can be deconvolved from the XFM images with down to around 8 nm spatial resolution and improved contrast.

2.2. Committee comments regarding capabilities:

HXN is creating the community that will be served by its capabilities. Materials science and condensed matter applications are the strongest user base. A strong mix of BNL divisional science and university academics, with synchrotron characterization experience, are currently the most capable and productive users. The maturity of multimodal imaging at HXN is unique.

While hard x-rays naturally enable in-situ studies of materials, unfortunately such capabilities are particularly challenging at the nanoscale in an instrument like HXN. Reduction in effective staff levels has led to the de-emphasis of the in-situ experimental program.

2.3. Committee recommendations regarding HXN capabilities:

The committee agrees with and supports plans to add another staff member at HXN and the committee strongly supports the notion that this person should have expertise for in-situ or spectroscopy x-ray experiments.

The plan to replace the tomography stage for more accurate and efficient 3D imaging and at the same time upgrading the MLL instrument are strongly supported by the committee.

Problems with Bluesky and databroker have resulted in up to several days of lost beamtime for individual users. According to the beamline staff, this is due to simple configuration issues that may have been solved by a DAMA group member just coming to the beamline every once in a while and being readily accessible during experiments. For example, databroker still spends more than five minutes writing data after a measurement is finished. This is up to five times longer than online data analysis in place at HXN and effectively halts experimental operations for the duration of the function. In addition, after upgrades the system is prone to frequent crashes resulting in repeated loss of data and valuable run time. The committee strongly encourages NSLS-II management to aggressively pursue solutions to these crippling problems.

3. Science Program and Impact

3.1. Committee observations regarding science program and impact:

As noted above, materials science and condensed matter appear to be the most impactful applications of HXN at this time. The user community that exploits HXN is also very much driving the research program of the beamline. Given the unique capabilities of HXN and the complexity of the experiments, the beamline staff are required to be integral members of most experimental teams. Staff

research appears to be appropriately focused on technique development with an eye toward future capabilities.

There is a noticeable trend toward more impactful science with publications shifting from largely instrumentation and "opportunity reviews" to core science themed papers. Recent journal articles in *Nano Letters*, *Scientific Reports* and *Advanced Functional Materials* are an indication of good things to come. The committee looks forward to future papers in high impact journals.

3.2. Committee comments regarding science and impact.

A concern was expressed that part of the user community should demonstrate more progress with the data. One issue with this is the high complexity of data processing, especially for the 3D imaging. Currently, one-third of work is 3D imaging, with the other two-thirds going to what should be high-impact 2D microscopy requiring less sophistication to publish. The bottle-neck was presented as being the users taking the processed data and then connecting it to a scientific story. However, there are also additional bottlenecks upstream of this in terms of data processing that are being addressed by the staff. The staff believe about 80% of their effort goes to cementing current capabilities and making them more effective for users. This seems like an appropriate balance at this point.

3.3. Committee recommendations regarding science and impact.

With capabilities maturing and the types of science with greatest impact potential being identified, it is important to continue to advertise the microscope to new potential users to build a larger, more diverse, community. The committee strongly encourages the beamline to focus more on potentially high impact 2D experiments, while continuing to explore longer term but high impact 3D experiments, with the user community. The committee strongly supports the current plan to request a new staff person be hired to focus on in-situ or spectroscopy experiments.

4. User Support and Labs

The beamline staff are very highly regarded by the entire user community, a fact that is evident both from the talks given by users during the review and from the end of run reports provided by the facility.

Beamline staff provide and support most of the codes to acquire and process data, which is a heavy burden. The committee recommends that a data analysis scientist be added at the group-level to both support analysis codes for the HXN instrument and other imaging beamlines in the group, and to assist users in connecting images to science by creating specific analysis tools when needed by a less sophisticated user.

The SEM instrument at the beamline is a vital tool to experimental sample preparation. The current model is aging and the resolution specification is even less than that of the HXN instrument itself. Upgrading the SEM is a good idea, giving the importance to the experimental program. A SEM with an XRF spectrometer or EDS could give additional information for light elements.

Overall, the staff is encouraged to keep up the great work building the capabilities of the world's most advanced x-ray microscope!

5. Partner Users

There are no current partner users. The beamline staff may be missing an opportunity. The staff is strongly encouraged to consider partner users, especially when it could help build a program in either a

different branch of science or a new capability at HXN. In addition, partner users may become expert users that can support experiments with collaborators reducing stress on beamline staff.

6. Future Plans

6.1. Committee observations regarding future planning at HXN.

Staffing requests are well considered and based on identified needs, as described above.

Beamline upgrades enabling more efficient photon delivery to the instrument as planned are appropriate:

- *Replace CRL with a K-B mirror*
- *Enable spectro-microscopy*, especially to carry out oxidation state imaging, although extreme caution with respect to photodamage of chemical species under these conditions is warranted.
- *Improve flux on sample* by a factor 2 or more enabling more rapid tomography.

The indicated HXN instrument upgrades are well considered and important for the performance of the facility:

- *Upgrade of the tomography stage* to one with small runout and wobble, and including encoding for automated sample centering and alignment, will enable more efficient and accurate tomography.
- *The MLL carriage* will enable more rapid alignment of the instrument during MLL-ZP optics change or for spectro-microscopy experiments, thereby saving photons, and will yield improved stability and specto-microscopy for improved performance.
- *Wedge MLL development and implementation* will improve flux on the sample, which will enable more rapid scanning and throughput that is extremely important for tomography.

6.2. Committee recommendations regarding the future planning at HXN.

HXN is recognized world-wide for stability and has become a go-to beamline for optics characterization. However, the General User program involving optics characterization for other beamlines and light sources currently running on HXN is detracting from the science output. HXN should continue characterizing and testing optics in support of its own instrument, but should limit beamtime given to other optics developments that will not directly impact HXN performance.

The development of a new instrument optimized for tomography and a new generation of MLLs is of particular importance. To improve the overall through-put of the experiments, the envisaged flux increase should be balanced with: 1) the development of a faster sample change (load-lock system or in-vacuum robot) and 2) the implementation of fast and robust control software, both at low and high levels.

The committee recommends that beamline staff identify clear plans for implementation of newly developed capabilities. Staff should avoid ad-hoc implementation of current R&D such that new capabilities end up under-utilized. Of specific capabilities identified for future development, the committee recommends that Bragg ptychography with MLL should be pursued.

The committee strongly endorses adding a staff member at HXN with expertise for in-situ or spectroscopy x-ray experiments. In addition, adding a data analysis scientist at the group-level to both support analysis codes for the HXN instrument and other imaging beamlines in the group, and assist users in connecting images to science by creating and supporting specific analysis tools when needed by a less sophisticated user.