2018 SAC Review of the NSLS-II Beamlines

September 19-20, 2018

IOS – In situ and Operando Soft X-ray Spectroscopy – 23-ID-2

Committee:
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1. Executive Summary

IOS shares the cell 23 straight section with CSX in 50:50 equal beamtime sharing. Currently, IOS operates two experimental stations, APPES and IO-XAS, to support the general users' research needs which are aligning with NSLS-II strengths and strategic direction of "Operando Chemistry & Structural Science". The ambient pressure X-ray photoelectron spectroscopy (XPS) capability is world class and supports the research needs to study energy materials and processes in heterogeneous catalysis, fuel cells, photovoltaics, and batteries. IOS staff has been providing exceptional support, especially to the less experienced user groups. There is clearly a rapid increase in the product in the last two years before the challenging situation of the ambient pressure XPS instrument. IOS is currently running PUA between with the Interface Science and Catalysis group at the CFN, in which CFN receive 23% of the available user time for ambient pressure XPS. The PUA was activated upon the start of GU operation in January 2016. The PUA stipulates that the CFN receive 23% of the available user time for running their user program as well as their own research, in exchange for an upgraded endstation valued at $800K and some staffing contributions. However, besides the necessary replacements of failing components over the years (e.g., detector, pumps, valves), the endstation has not been upgraded since its first construction. The PUA with BNL Chemistry will be activated after the installation of in-situ XAS cells for GU use. Staff research has been demonstrated and is at an appropriate level and aligns with the mission of the beamline and involves some good collaboration with general users. Staff are appropriately planning a number of incremental upgrades of APPES and IO-XAS to improve the performance and productivity. There are plans to upgrade and replace APPES and IO-XAS to meet the current research trends.

The report is structured according to the specific questions posed in the charge to the Committee in five areas, including Beamline Capabilities, Science Program and Impact, User Support and Labs, Partner Users and Future Plans.

The key findings are listed as follows:

- In-situ chemistry capability at IOS using ambient pressure XPS is established and supported.
- The electron analyzer for ambient pressure XPS has been is out of operation since May 2018, which shows significant impact on NSLS-II users' program.
- Some current users require a too high a degree of user support due to insufficient experience with handling complexed experimental setups at IOS.
- A clear deficiency of the program is the apparent absence of *operando* capabilities.

- The synergy is missing in the PUA involving CFN, in which "partnership" is lost in the translation.
- Available shifts on IOS are highly oversubscribed under the current 50:50 beamtime sharing agreement between CSX and IOS

The recommendations are listed as follows:

- Identify what lessons have been learned from prolonged downtime of the ambient pressure XPS capability to avoid future impact.
- Encourage to develop a contingency plan for the ambient pressure XPS capability.
- Move forward with *operando* capabilities and research.
- Find a balance between supporting experience and non-experienced users to sustain the productivity of IOS.
- Encourage NSLS-II to establish a new relationship under PUA between CFN in regards to the instrumentation and supporting team.
- Compelling science case was missing for the INSPIRE proposal to replace APPES.
- Encourage the IOS team to have more discussions with CFN and Department of Chemistry on the uniqueness for the science cases of the new INSPIRE endstation.
- The Committee strongly supports the proposal to increase the canting angle between CSX and IOS to allow independent operation of the two
- Suggest to explore the option of moving the IOS beamline to a dedicated EPU or bending magnet source.

2. Beamline Capabilities

Are the beamline capabilities well established and supported to meet the research needs of the intended scientific communities?

IOS is a soft X-ray beamline on the cell 23 straight section, which has a pair of EPU49 sources shared with CSX. The focus of IOS is in-situ and operando chemistry using soft X-ray spectroscopic techniques, such as ambient pressure XPS and X-ray absorption spectroscopy. The in-situ chemistry capability of IOS, APPES and IO-XAS endstations, meets the research needs to study energy materials and processes in heterogeneous catalysis, fuel cells, photovoltaics and batteries.

Are these well aligned with NSLS-II strengths and strategic directions?

Yes, the capability of IOS aligns with NSLS-II strengths and strategic directions of "Operando Chemistry & Structural Science".

Are there opportunities to enhance the existing program or pursue new capabilities?

The electron analyzer for APPES has been out of operation since May 2018, and its absence has had a significant impact on the NSLS-II users' program at IOS. The Committee suggests that the team identify what lessons have been learned from these events and plan to avoid future impact. The IOS team may look into the possible connection between the aged endstation and the out-of-action ambient pressure XPS analyzer contributing current the lengthy downtime too as well. We encourage the development of a contingency plan for the APPES capability. For example, could a loaner of APPES equipment be found internally or externally in exchange for PU beamtime?

3. Science Program and Impact

Are there potential efficiencies to be gained in scheduling, programmatic balance, or other ways to enhance productivity and implications?

A clear deficiency of the program is the apparent absence of *operando* capabilities as indicated by the examples presented and the publications cited. Since it is part of the beamline's newly christened name and an identified priority area in the NSLS-II Strategic Plan, the Committee recommends that *operando* capabilities and research should be emphasized moving forward.

It is less clear what the programmatic balance should be between APPES and APXAS. Statistics were not presented that indicate how often these capabilities are requested. The allocation of staffing and capital resources should be driven by demand.

It is somewhat too early to judge the productivity and impact of the research at IOS. The beamline has served a considerable number of users resulting in a limited, although increasing, number of publications. Staff indicated that the typical "gestation" between experiment and publication is ca. two years. This seems long, and the committee suggests that perhaps proposal review should place increased weight on well-defined experiments with clear publication goals.

Staff also indicated that productivity was also hindered by poorly prepared users and samples that were not compatible with the capabilities. As noted below, this deficiency could possibly be addressed by utilizing parasitic beamtime to train users and screen samples.

Is the current science program mix appropriate for the beamline?

The majority of the IOS scientific program serves the heterogeneous catalysis community (~65%) (especially those with surface science capabilities), with additional programs in material science (25%) and batteries (10%). The beamline's capabilities consist of APPES and APXAS. The programmatic mix is likely driven by the proposals received and the beamtime allocated.

Is each science program productive and making sufficient impact in its scientific field?

The mix appears to be consistent with the beamline's capabilities, since the application of XPS in heterogeneous catalysis has historically been hindered by whether the existence of a so-called "pressure gap" limits the relevance of the measurements. The AP capabilities of this beamline do much to address this issue, and recent results showing reactivity (e.g., bonding of ligands to isolated metal sites) are strongly encouraging.

Is staff research at the appropriate level and aligned with the mission of the beamline?

Staff research expertise has been demonstrated and is at an appropriate level and aligns with the mission of the beamline.

4. User Support and Labs

Is the user support at the beamline of high quality and sufficient to enable and sustain high productivity and high impact?

IOS staff has been providing exceptional support for IOS users, which is indicated by an outstanding publication record. There have been 25 publications in the first few years of IOS

operation. The quality of publications from IOS is high and 2 of the publications appeared in DOE-designated high-impact journals, such as Proceedings of the National Academy of Sciences and Applied Physics Letters.

There is concern about the technical competence of users. Though accepted proposals have high scientific merit for NSLS-II, some current users require a too high a degree of user support. It is due to insufficient experience with operating IOS control, data acquisition, vacuum equipment and the requirement to handle complex experimental setups.

Are the support labs available and sufficiently equipped to enable the type of research at the beamline?

In principle, yes. The support labs are available near IOS. The type of on-going research at IOS underutilizes these resources.

Are there opportunities to improve user productivity?

To sustain the productivity, the Committee encourages IOS beamline staff to find a balance between supporting experienced and inexperienced users. For example, increase the number of experienced users through the Proposal Review Process (user groups' experience in ambient pressure XPS can be made as a criterion for the beamtime proposal scoring) or increase staffing level to support inexperienced users.

Turning incompetent new users into competent returning users will provide opportunities for improving user productivity.

5. Partner Users

Are the partner users contributing as planned, fulfilling their responsibilities, and satisfied with the partnership?

The APPES endstation is available through the PUA with the Interface Science and Catalysis group at the CFN. The endstation was constructed in the early 2010's and operated at NSLS beamline X1A1 in 2013-2014, then moving to NSLS□II after the permanent shut down of NSLS in September 2014. The PUA contributed the hardware, a whole endstation, having a value of $800K and three-year replacements of failing components over the years (e.g., detector, pumps, valves), and this contribution, in Committee's view, is significant. But the endstation hasn't been upgraded since its first construction which could be markedly longer than three years, probably more than six years.

The Committee didn't see a representative from CFN overseeing the IOS APXPS endstation during the review, and presumably there is no CFN staff helping to take care of the system. It would be essential to have CFN scientific staff who could interpret the science needs of the APXPS endstation for the CFN user groups when using their entitled 23% beamtime. The synergy is missing between CFN and NSLS-II—"partnership" is lost in the translation.

For beamlines with partner users, are the partner users productive and making sufficiently high impact on the beamline science programs?

Data are not available how well they are making an impact. The IOS program report may indicate direct statistics on the Partner User Productivity.

Are there opportunities to further improve the existing partnerships or to pursue new partnerships that would enhance beamline operations and productivity?

The three-year term of the PUA involving the CFN is ending by the next cycle. This is a good time for NSLS-II and CFN to establish a new relationship in regards to the instrumentation and supporting team. CFN in the past had a dedicated staff member who was well trained in APXPS when operating at the NSLS. It was the case possibly due to the planned upgrade of the endstation as included in the PUA at that time. The Committee considers the operation of such a complex and challenging experimental system to require a skilled APXPS staff person with strong surface science expertise. The IOS now does have the excellent scientific staff, but it would be important to have an interaction with CFN to assure that there is a good mutual understanding of the APXPS and its applications.

The IO-XAS capability is provided by the Catalysis: Reactivity and Structure group in the BNL Chemistry department. The PU contributes the Vortex detector, manipulator, and in situ cells (developed by the Advanced Light Source) for 8% of the available user time. The PUA is to be activated after the installation of in-situ cells available for GU. The timeline of this PUA is expected after January 2019. The PUA of the APPES endstation between NSLS-II and CFN can be a lesson in preparation for the planned PUA.

The new proposed INSPIRE endstation could be a new platform for the partner user agreements between NSLS-II and both CFN and the Department of Chemistry. The committee suggests that the IOS beamline staff have more discussions with CFN and the Department of Chemistry people regarding the uniqueness of the new endstation and the opportunities to support the science cases.

6. Future Plans

Is there an appropriate level of future planning at the beamline?

The staff presented plans for a number of near-term, incremental upgrades that should improve the performance and productivity of the beamline. These include: redesign of the load lock system to reduce time to exchange samples; upgrading of the APXPS manipulator controls, sputtering and gas dosing so that they are automated and more user-friendly, remove C-contamination on mirrors and gratings, upgrading sample heating capability; and improvement of beam stability by enhanced temperature control.

Upgrading the XAS endstation for ambient pressure and electrochemical cell capability, and *operando* analysis will be undertaken in partnership with the Catalysis: Reactivity and Structure group in the BNL Chemistry department.

The possibility of totally replacing the APPES endstation was also mentioned although it wasn't clear what timeframe this might occur over.

On a much longer time scale the staff would like to upgrade the endstation to include infrared vibrational spectroscopy using synchrotron radiation and resonant inelastic x-ray scattering (RIXS) as contained in their INSPIRE proposal. These capabilities would provide substantial upgrades.

Are these future plans appropriate given the current and evolving research trends in the community and funding climate?

Most of the future plans appeared to be appropriate considering the current research trends in the community.

The staff did not present a compelling science case for their INSPIRE proposal. Presumably this proposal evolved from the QIX proposal that was reviewed and approved during the beamline development process. The Committee is concerned with adding additional capabilities to an already oversubscribed beamline. We are also concerned with the investment that would be required in terms of staff time, funds, and instrument downtime that would be required to bring this proposal to fruition.

What other improvements and upgrades should be considered in the future?

An overarching concern is to develop plans to address the oversubscription / user access issues that are evident and are likely to increase in the future.

"Programmatic balance" in sharing beamtime between IOS and CSX is critical. It is clear from the proposal and allocations statistics that demand for IOS is increasing, and the beamline is becoming increasingly oversubscribed. With the current 50:50 beamtime sharing practice, the IOS usage is likely to plateau and the requests may in fact decline as potential users get discouraged and go elsewhere. Therefore, the Committee strongly supports the proposal to increase the canting angle between the two beamlines to allow independent operation. For the near term, the Committee recommends utilization of "parasitic" beamtime by IOS to perform activities such as training users, screening samples and allowing research that would not need to change the photon energy. In which the PU of CFN and Department of Chemistry can play an assisting role too.

Another option that could be explored is moving the IOS beamline to a dedicated EPU or bending magnet source.