National Synchrotron Light Source II

Photon Science Division Update

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Photon Science Division Director

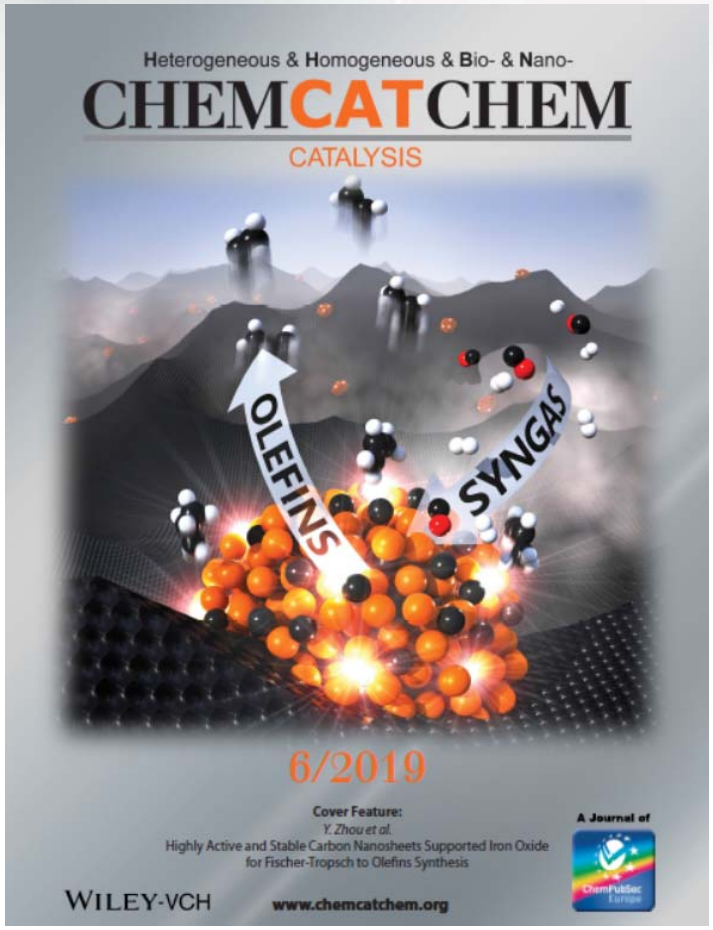
SAC Meeting – 28 March 2019

U.S. DEPARTMENT OF ENERGY | Office of Science

BROOKHAVEN NATIONAL LABORATORY

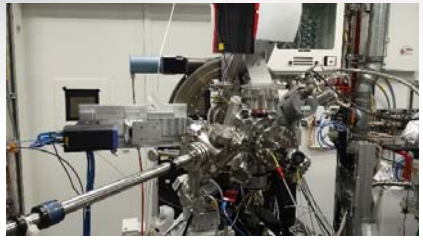
Outline

- Program operations updates
- Science highlights
- Beamline status updates
- Beamline Operation and Staffing Issues
- User Expectations
- Summary

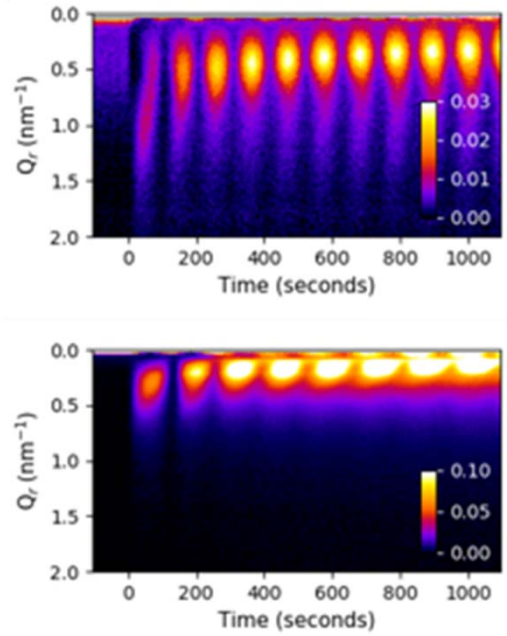


In-situ Scattering & Diffraction Program

- Established Partner User Program at ISR making time-resolved Pulsed Laser Deposition studies available to the user community.
- PDF beamline begins General User Program
- 100th publication at XPD due to appear early April.
- Large volume press open to GU (PUA with COMPRES and SBU)
- First light on diffraction-tomography endstation on 3/14 (PUA with NSUF Idaho and NE)
- HEX Final Design Review planned for 1-2 April 2019
- bidding for HEX SCW in April
- Start of construction work for the HEX satellite building in early April
- HEX hutch contract is awarded.

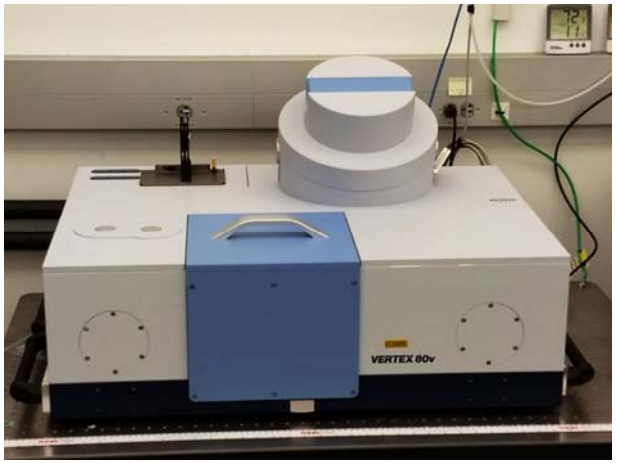


PLD system at 4-ID-D



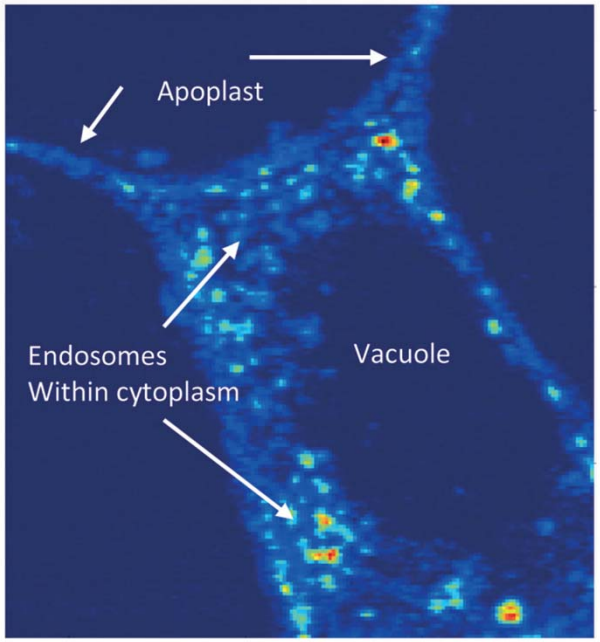
Soft X-ray Scattering & Spectroscopy

- The SIX beamline entered the general user program
- FIS and MET Passed their IRR and obtained first light. Commissioning is underway of the photon delivery system. Expecting FIS users in 2019-2 and MET users in 2019-3.
- Upgrade of FTIR spectrometer completed for MET completed thanks to Facility Improvement Project.
- MBE setup nearing final completion at ESM.
- IOS deployed EPICS/Bluesky software control of the SPECS analyzer, which integrates beamline systems for automated measurements like XAS with partial electron yield (PEY)
- AP-XPS capability back online



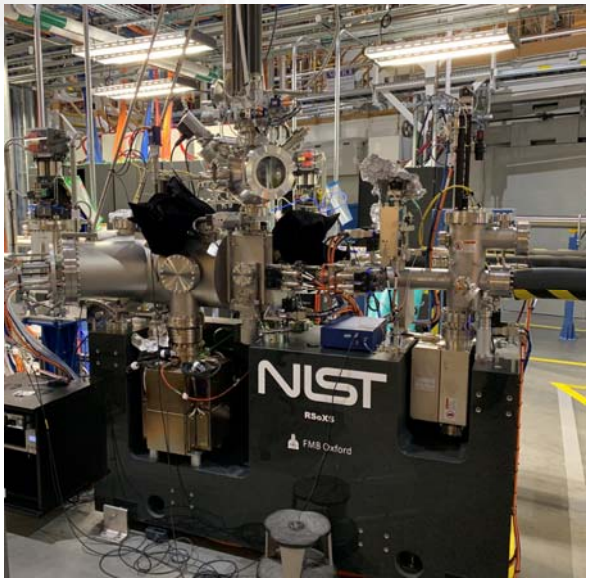
Imaging and Microscopy Program

- HXN is back to user operation after the ID failure
- TES will resume user operation, staring in May, 2019
- XFM and FXI enter General User Program
- CDI beamline CDR is completed.
- SRX KB mirror system on track; design review held in December
- Commissioning of MAIA detector on XFM



Hard X-Ray Spectroscopy Program

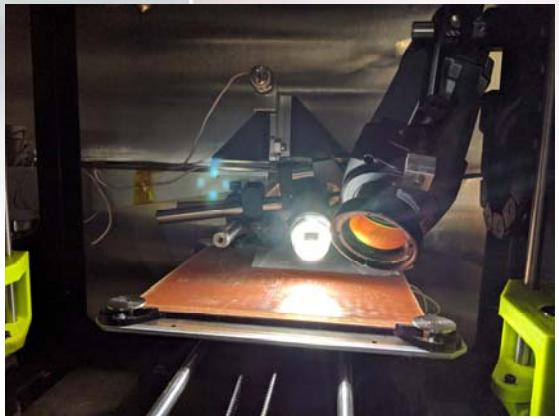
- The NIST RSoXS instrument was constructed, completed its IRR, and will begin technical & scientific commissioning – Delayed due to Gov't shutdown.
- The NIST full-field NEXAFS spectro-microscope (LARIAT-II) was commissioned and currently has its first science commissioning users
- Multi-reactor setups were developed on ISS for heterogeneous catalysis, electrochemical and battery experiments.
- LDRD on "Integrating of Experiments with Data Analytics for Quantum Materials", involving ISS staff, was granted in Oct 2018.
- Two pre-proposals (on QIS and Machine learning for solving complex problems) and a White paper (on Machine learning for XAS data interpretation) involving ISS staff were submitted to DOE
- The QAS portable gas handling system was commissioned and is now in regular use.
- Collaborative research by staff from ISS & QAS, BNL Chemistry Dept., and universities to study single-atom catalysts was among "*Top 10 BNL Discoveries of 2018*"



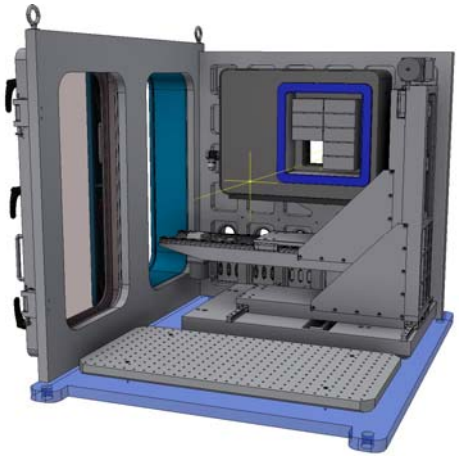
Location: NIST's NSLS-II Beamline SST-1
Source: 150 eV – 2000 eV EPU, C-N-O-F K-edges
RSoXS design led by Dr. Eliot Gann, NIST

Complex Scattering Program

- New CMS L-shaped WAXS detector received. Motion stage fabrication in progress. Installation planned for April shutdown
- Science commissioning of in-situ shear-SAXS and in-situ DAC-based high-pressure SAXS in progress at CMS
- First in-situ processing measurements done on SMI - 10:10:2 Hz measurements with SAXS:WAXS:MAXS detectors of printed semi-crystalline polymer filaments.
- IXS in technical commissioning for the 2019-1 & 2019-2 cycles to understand and improve efficiency of the optical components
- Collaborative research by CFN/NSLS-II staff using CMS among "*Top 10 BNL Discoveries of 2018*"
- An RPI graduate student, Liwen Chen, won the prestigious Padden award at the 2019 APS March meeting based on the research she conducted at CMS and SMI.



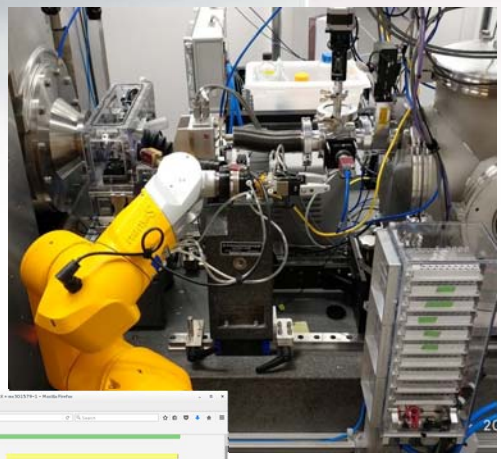
Lulzbot 3D printer on SMI with off-axis visible and IR imaging of printed filaments (Y. Shmueli et al., to be submitted)



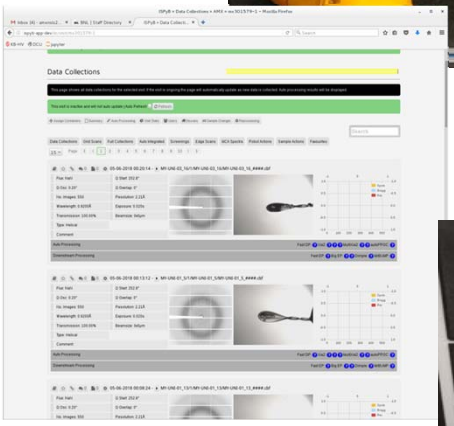
DECTRIS *Pilatus3 S 800K* L-shaped WAXS Detector to be installed on CMS

Structural Biology Program

- Automated Sample handling implemented at LiX.
- Implemented the ispyb database for MX.
- Cryo-EM construction Underway
  - First microscope installed and (almost) ready for use.
- Remote User Access grown to ~50% of use at AMX
- Serial Crystallography now routinely available at FMX and AMX
- NYX open for user operations – science commissioning
- XFP program has ceased bioXAS (funding)



LiX robot for SAXS

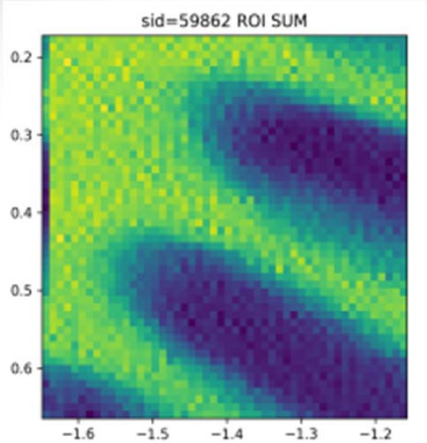


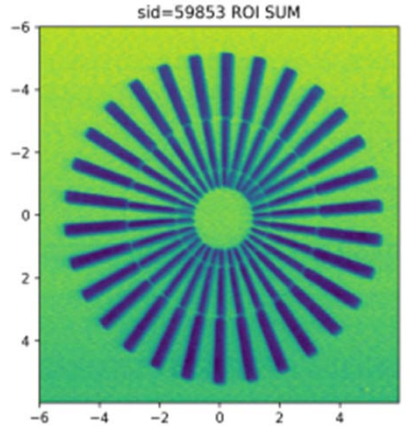
Ispyb database for MX

First microscope in the lab

Experiment Development Program

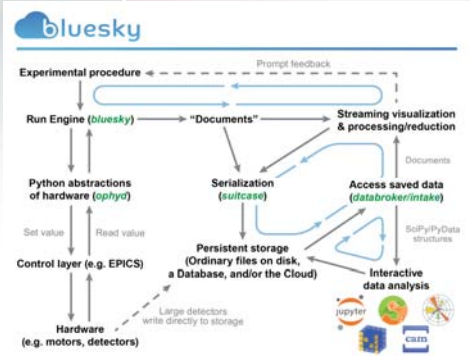
- Achieved 2D focus (18 keV) of 9 nm x 14 nm using 2 wedged MLLs (fig. axes units in microns)
- LOB 5 laboratory construction completed, in occupancy review stage.
- LOB 4 laboratory construction scheduled to complete by end of May 2019.
- Continued R&D on stitching metrology and slope metrology
- Optical simulations for Bragg CDI beamline completed and optical layout developed
- Development of sub-100 nm KB-based scanning microscope for SRX
- Fabrication of 10 diamond BPMs (collaboration with Instrumentation Division)
- Completion of Phase 2A SBIR on Development of Software Framework for X-ray Optics Simulations
- Completion of Phase 2 SBIR on Development of Fiber-optic based distributed atomic absorption spectroscopy for film growth monitoring

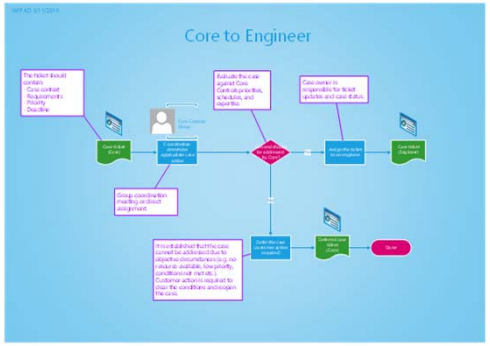




Controls Program

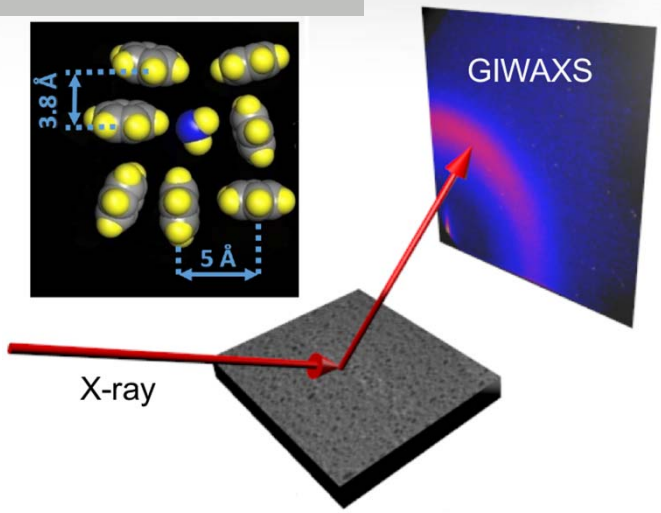
- Controls program success remain essential for NSLS-II success
- Controls action Plan guides overall work, steady progress. (Richard's presentation)
- Controls program organizational adjustments are leading to a more centralized structure, core group is established.
- The PoC role is expanded and better defined with structured priority setting and problem solving steps. Transitioning to a more operationally focused environment.
- Intermittent performance continues to frustrate staff, especially as data volumes increase. Steps, including outsourcing "what we can" continue.
- Networking is now being managed substantially externally by ITD.
- Continued improved communications are needed in both seminars and multi-disciplinary working groups
- Collaborations are showing benefits





Illuminating desalination membranes

Molecular motifs



The sketch shows x-rays scattering from the membrane surface into a detector to create a GIWAXS pattern. The top left image shows two different molecular scale packing motifs.. GIWAXS results suggest that the perpendicular motif (lower right) is correlated with superior filtration properties.

Q. Fu, N. Verma, H. Ma, F. J. Medellin-Rodriguez, R. Li, M. Fukuto, C. M. Stafford, B. S. Hsiao, B. M. Ocko. *ACS Macro Lett.* **8**:352-356 (2019).

Work was performed in part at Brookhaven National Laboratory

Scientific Achievement

Scientists revealed the molecular structure of polymer membranes materials used in reverse osmosis (RO) water purification

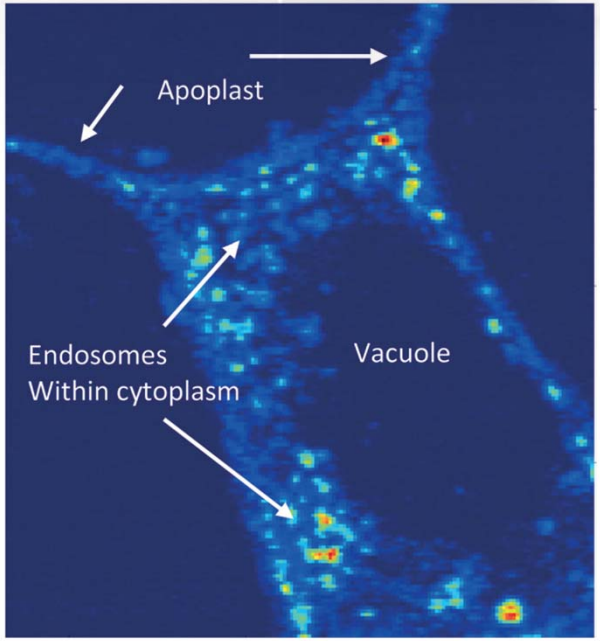
Significance and Impact

RO is the leading method of converting seawater into potable water; understanding the molecular structure and the correlation with filtration properties will aid in the development of more efficient membranes.

Research Details

- Thin, well-defined polymer RO barrier layers – similar to those used in commercially RO filtration– were prepared at the water/oil interface
- Grazing-incident wide-angle x-ray scattering (GIWAXS) at NSLS-II beamlines 11-BM and 12-ID was used to study the molecular packing.
- Two different molecular packing motifs were found, one of which appears to be better correlated with superior filtration properties.

A Nanomaterial's Journey through a Tomato Plant



This 60 nm resolution x-ray fluorescence image was taken from the stele of the root of a plant that was exposed to functionalized CeO_2. The brighter areas show the highest concentration of Ce within the apoplast and within endosomes.

J. Li, R. V. Tappero, A. S. Acerbo, H. Yan, Y. Chu, G. V. Lowry, J. M. Unrine. *Environ. Sci.: Nano* **6**, 273 (2019).

Work was performed in part at Brookhaven National Laboratory

Scientific Achievement

Scientists revealed how a manufactured nanomaterial (MNM) based on Ce travels through a tomato plant on a subcellular level.

Significance and Impact

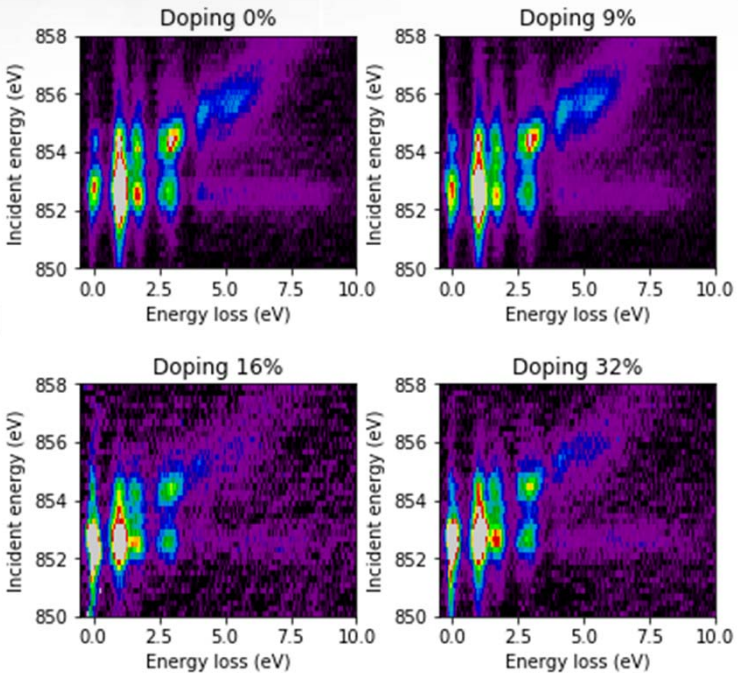
This study will enhance our ability to predict how properties of MNM such as CeO_2 – used in rechargeable batteries – influence the uptake, transformation, and transfer of nanomaterials in terrestrial food webs.

Research Details

- Mechanisms of functionalized CeO_2 entry into tomato roots and translocation within the plants were examined using X-ray nano- and microprobes at NSLS-II beamline 3-ID and APS beamline 13-ID-E
- Plant growth and Ce concentration in tissues were found to be functions of surface charge and translocation, which were greater for negatively charged CeO_2 than positive or neutral CeO_2.

SIX Performance & Plans

- SIX Has achieved world leading resolution of 26 meV at the Copper L-edge, corresponding to a resolving power of ~36000.
  - Congratulations to the SIX team!
- New replacement M4 mirror has been delivered to BNL and is currently in our Metrology lab to be measured. Mirror will be installed in next shutdown for commissioning in the next cycle.
- Sample transfer system has been installed and is now regularly used for user experiments.
- Over the last 6 months, we have been working with the vendor of the CCD detector to improve and develop the controls support for this detector.
  - 2-ID now offers users single photon counting in a centroiding mode to offer the highest resolution.
  - OPHYD/BlueSky has been implemented for control of the detector.
  - Work continues on data processing routines. Jupyter notebooks are currently offered to users to perform required data analysis.



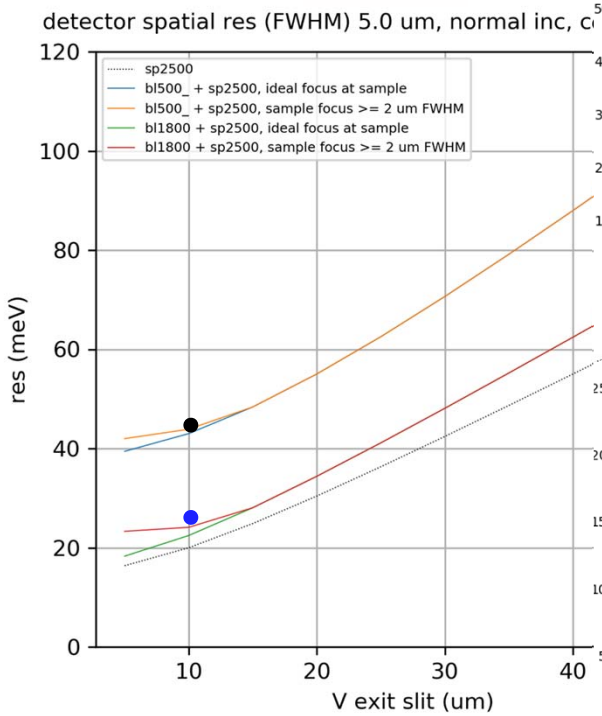
K-doped NiO Ni L edge 850 eV

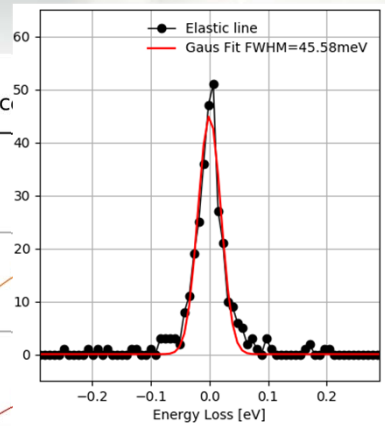
Optics Status at 2-ID

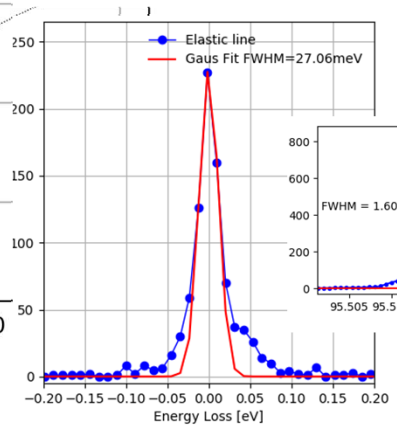
- 2-ID now has laminar holographic gratings for the spectrometer (2500 lines.mm^{-1}) and beamline (1800 lines.mm^{-1})
  - This combined with new M4 should allow 2-ID to reach its ultimate design resolution of 15 meV at the Copper L-edge.
  - This will be commissioned in 2019-2.
  - Upon successful commissioning, this would be offered to users in the 2019-3 cycle.
- Continue to work with Inprentus to obtain blazed gratings which would allow for improved throughput.
  - Inprentus have made significant improvements in their process.
  - Our plan is to have Inprentus rule and deliver at 1200 lines.mm^{-1} (beamline) grating by the end of CY19 followed by the 1250 lines.mm^{-1} (spectrometer)
  - Strategy here is to see Inprentus deliver less challenging gratings first to verify their process for long gratings.
  - Addition of these gratings would offer a routine, very high throughput mode.
  - If these gratings are acceptable, then we will ask Inprentus to rule the ultra-high resolution gratings to improve throughput from current laminar gratings.

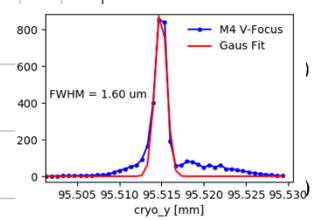
SIX Energy Resolution: achieved results with ~2um M4 V focus

1000 eV

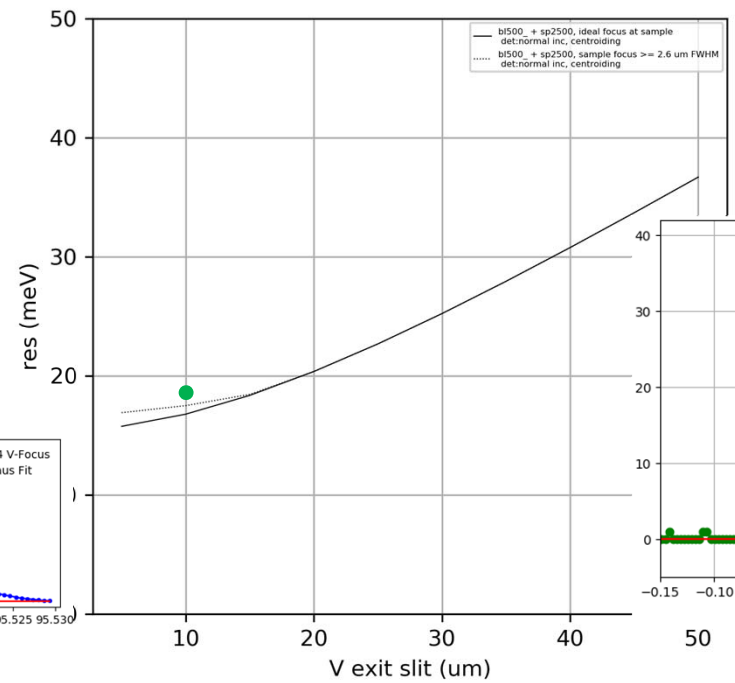


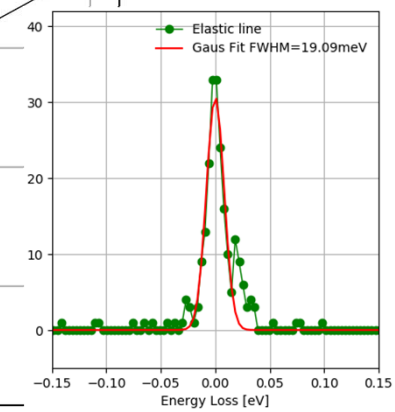






500 eV





Currently used grating configuration for ~1000 eV:

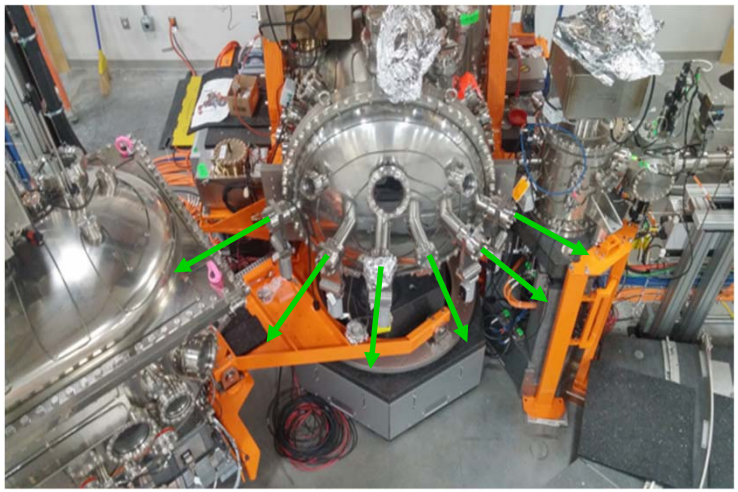
- **BL 500 l/mm** (medium res) or **1800l/mm (high res)**
- Spectrometer 2500 l/mm (medium res)
- M4 vertical focus is ~ 2 um

Currently used grating configuration for ~500 eV:

- **BL 500 l/mm** (medium res)
- Spectrometer 2500 l/mm (medium res)
- M4 vertical focus is ~ 2 um

Triple Rotating Flange – A Path Forward

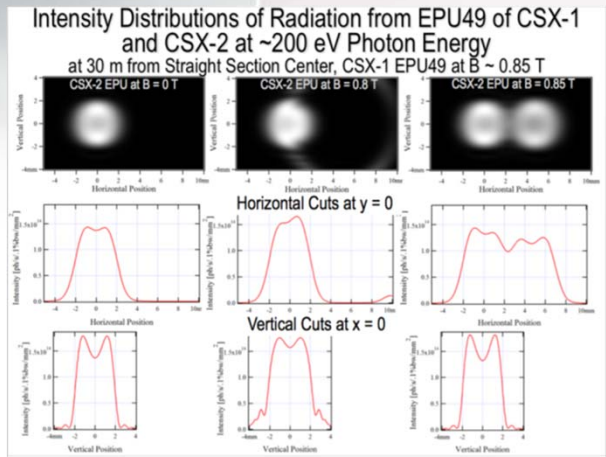
- NSLS-II is developing the following 3 strategies in seeking a fix to the TRF at 2-ID to allow continuous movement of the spectrometer arm under vacuum.
  - Continue to manage the contract with BESTEC and pressure the vendor to deliver the TRF. Within a few months we will know if the vendor will deliver the TRF or we have canceled the contract.
  - Market research shows that a suitable vendor could be found to develop a replacement TRF. Plan to develop a SOW/SPEC and contract which would split design and fabrication. Design would take approximately 6 months at which point NSLS-II would evaluate the design and decide whether to fabricate. Fabrication is likely an additional 12 months.
  - Over the next 6 months evaluate what engineering would be needed to incorporate the sliding seal (as used at Diamond and ESRF) into the 2-ID beamline. At the end of 6 months a plan to execute this solution would be available.



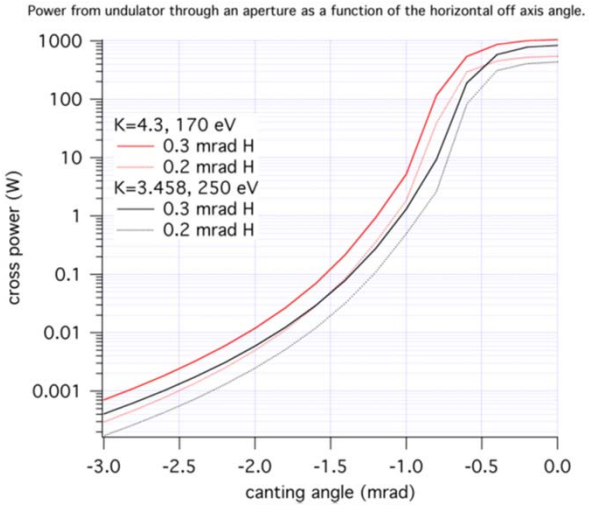
- In ~6 months, evaluate which option we would pursue. In the absence of a TRF from BESTEC decide on whether to build a replacement TRF or go with the sliding seal.

IOS/CSX

- In the canted mode 2 beams are produced – one going to each branch beamline.
- Interference effects between the two undulators prevent simultaneous operations
- As a result, each beamline currently operates only 50% of the time
- Ideally, each branch would exist on a separate straight section, but resources are not available to do this
- Increasing the canting will eliminate the interferences to allow each beamline to run full-time
- IOS is one of the most oversubscribed beamlines and would greatly benefit from running full-time
- CSX would lose the opportunity for phasing the undulators, but gains a factor of 2 in beamtime. Improvement in detector performance (S/N) would be larger impact for CSX.

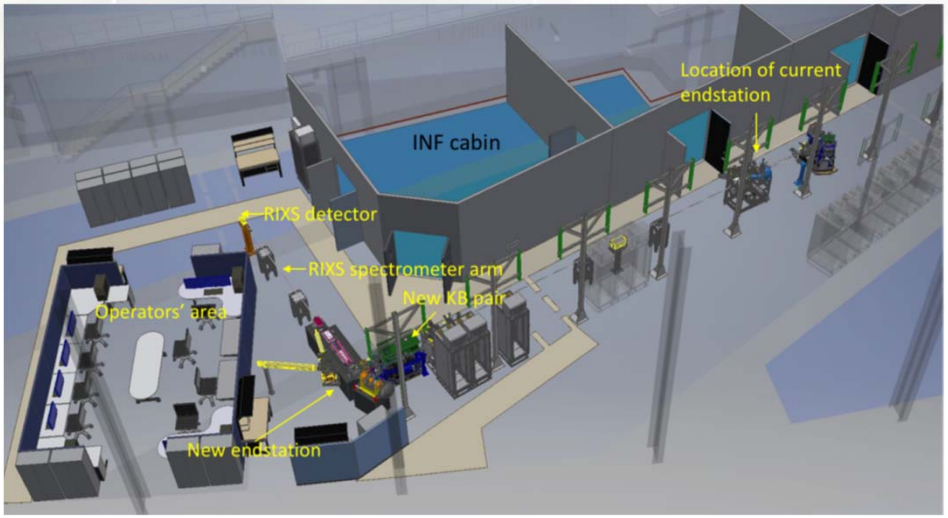


CSX cross contamination vs canting angle
(calculations done with Spectra)



INSPIRE

- Upgrade at IOS consisting of an endstation that combines AP-XPS, RIXS, IRRAS in ambient pressures and a refocusing KB mirror pair
- Fixes current problems at IOS – poor beam focus and degrading endstation
- Combination of techniques provides deeper insight into an energy system in real conditions
- Atmospheric pressure XPS in ms time scale, high-res RIXS map in ~sec time scale, IRRAS in ms time scale (3 orders of magnitude brighter and extended range with IR from INF)
- Workshop planned to refine the science case



ESM "Hands-on" Assessment (29-30 April)

- Performance goals not yet realized at ESM. Prior hands-on assessment at SRX was very valuable
- Assess current technical capabilities of ESM compared with design goals
- Assess the current strengths and weaknesses of the ESM Beamline, compared with other similar beamlines around the world.
- Identify the areas of technical development where resources might be directed with the highest priority over the next two years
- Evaluate the general user program of the ESM Beamline

Assessment Team

- Jessica McChesney (chair)
- Riccardo Comin
- Martin Fuchs
- Andrei Fluerasu
- Eliot Gann (NIST)

Technical Commissioning Update of 10-ID

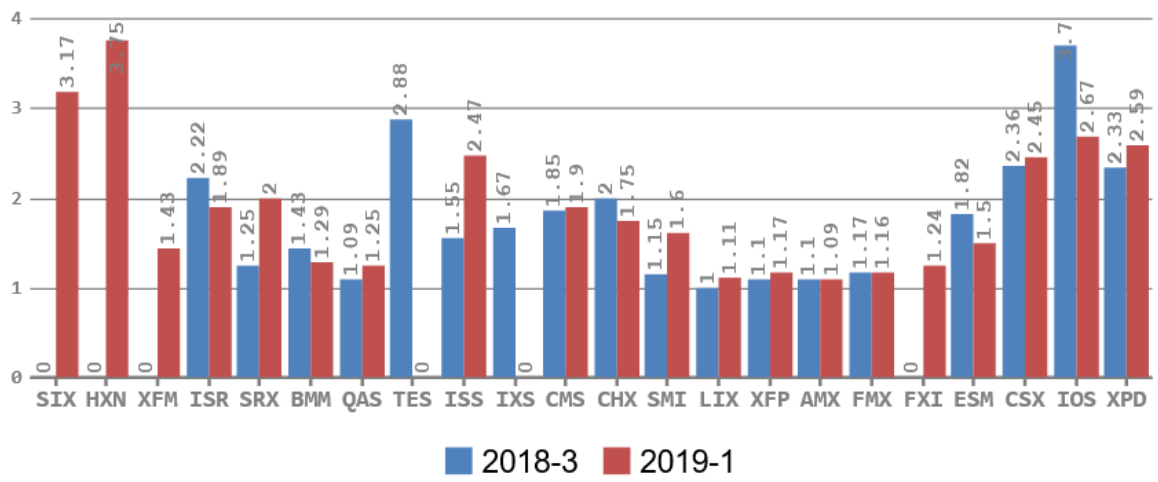
- IXS in technical commissioning for 2019-1 and 2019-2 cycles to understand and improve the efficiency of the optical components.
- Hutch temperature control system nearly complete (final sensors hook-up scheduled)
- Key losses and deficiencies identified thus far include
  - ~40% loss between the KB focusing mirrors and the sample, mostly due to the mask of the horizontal mirror partially blocking the beam.
  - Mounting stress and angular vibration of the HRM crystals suspected to be the leading causes of energy broadening of the HRM due to its high angular sensitivity (~1 meV/0.3 urad).
  - Overall spectral efficiency of the CDW analyzer was ~5.2%, about a factor 4 less than the ~20% measured during the R&D phase using the central area of the optical components. Evaluation of the likely cause pointed to the poor transmissivity of the C crystal, ***a major challenge of the current analyzer scheme.***
- Near-term plans:
  - Focus effort on recovering major losses and improving the overall counting efficiency using new 2-meV high resolution optics
  - Complete the optics station in C hutch to support high-resolution optics R&D
  - Explore new ways to improve the quality of the C crystal.
  - Research new analyzer optical schemes that offer substantial gain in counting efficiency

IXS Management Review (17 April)

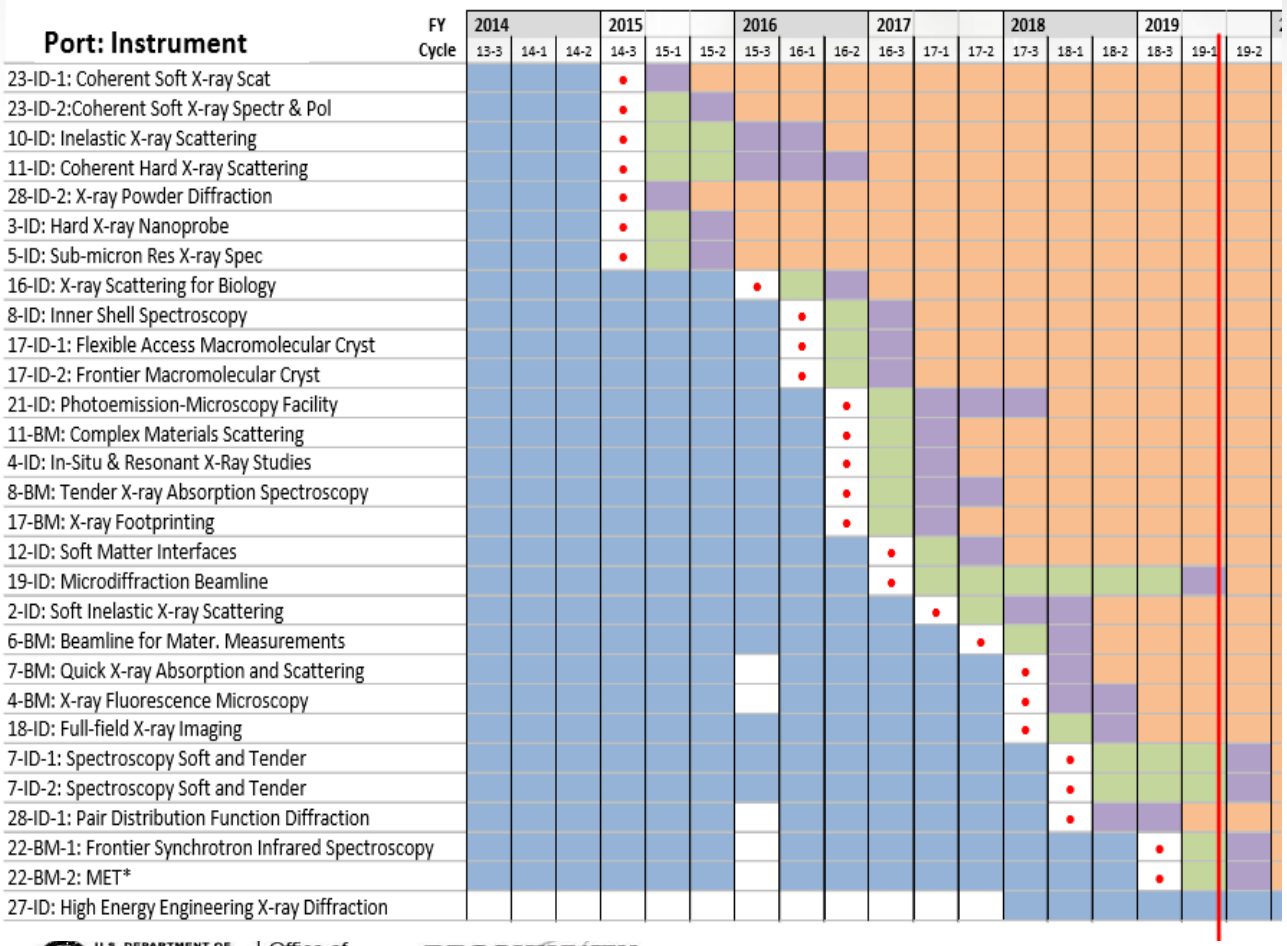
- Review current optical systems of the IXS beamline and the spectrometer in terms of resolution, lineshape and spectral efficiency.
- Identify potential sources of any major deficiencies compared to theoretical expectations.
- Review a plan to rectify the deficiencies while maintaining the total energy resolution profile to within 2 meV width maintaining very sharp elastic peak (very low intensity tails).
- Assuming the corrective action plan can be successfully applied at 10-ID, where would the subsequent performance of the instrument place it on the world stage?
- External members: Tom Toellner & Michael Krisch

Beamline Operations

- HXN insertion device repair completed – user operations resumed
- ISS Crystal replacement completed and user operations resumed
- PSD operations efforts reduced to support Beamline Developments
- Government shutdown impacted user operations at BMM
- Klaus Attenkofer departed NSLS-II. Eli Stavitski is Interim Lead Beamline Scientist; Ron Pindak is Interim Program Manager
- Beamline over-subscription rates (based on proposals) are sustainable



Publications by Beamline (CY)



Primary Beamline	Total 2018	Total 2017	Total 2016
23-ID-1	2	4	2
23-ID-2	19	6	1
10-ID	3	3	2
11-ID	4	4	3
28-ID-2	34	28	19
3-ID	13	13	8
5-ID	6	7	4
16-ID	3	4	1
8-ID	23	10	0
17-ID-1	14	1	1
17-ID-2	7	1	1
21-ID	2	1	0
11-BM	33	5	0
4-ID	0	0	0
8-BM	1	1	1
17-BM	3	2	1
12-ID	4	0	0
19-ID	0	0	0
2-ID	1	0	0
6-BM	0	0	0
7-BM	2	0	0
4-BM	2	0	0
18-ID	1	0	0
7-ID-1	0	0	0
7-ID-2	0	0	0
28-ID-1	1	0	0
Total	178	90	44
Premier	44	24	11

Photon Science Division staffing update

There are a number of open positions within the Photon Science Division currently:

- NSLS-II Core Controls technology analyst position filled (25 March 2019): Nathanael Maytan
- HEX: Lead beamline scientist position filled (11 March 2019): Michael Drakopolous
- CDI beamline scientist position filled (21 Feb 2019): Yuan Gao
- SMI beamline scientist position filled (22 Jan 2019): Guillaume Freychet
- ESM operations post-doc position filled (22 Jan 2019): Turgut Yilmaz
- FMX scientist position filled (31 Dec 2018): Wuxian Shi
- XFM: Beamline scientist position filled (10 Dec 2018): Sarah Nicholas
- SRX beamline scientist position filled (pending visa): Yang Yang
- SIX beamline scientist position filled (starting 29 April 2019): Jonathan Pelliciari
- TES lead beamline scientist position filled (starting 8 April 2019): Yonghua Du
- ISS lead beamline scientist: Interviews to begin.
- QAS: Position has been reposted to obtain a larger candidate pool. Interviews underway.
- AMX beamline scientist position posted.
- DAMA: 2-3 open positions; several offers declined
- Cryo-EM Group Leader position posted.
- Cryo-EM scientist position posted.

Recent award successes

- Our staff have been very successful in competing for external funding – a sign of the strength of our staff:

  - **Ryan Tappero**: DOE-BER: full field fluorescence microscopy (collaboration between XFM, Detector group, Metrology group and Instrumentation Division) ($3.75 M)
  - **Sean McSweeney**: DOE-BER: CryoEM operations ($2.3 M)
  - **Valentina Bisogni**: DOE Early Career Award ($2.5 M)
  - **Abdul Rumaiz**: DOE BES Ge detector technology ($800k)
  - **Oleg Tchoubar**: DOE-BES simulation software ($800k)
  - **Chu, Wang, Lee**: IAPRA ($200k)
  - **Tchoubar, Nazaretski**: SBIRs ($265k)
  - **Attenkofer, McSweeney, Mazzoli**: 3 LDRDs
  - **Ocko, Attenkofer, Dooryhee**: 2 x EFRC participation
  - **Steve Hulbert** (on behalf of numerous staff): SLAC, ALS, APS upgrade projects

Communications and Staff Activities

- Continue Monthly Photon Science Division Seminar and Staff Meeting
- Continue Division Director Weekly Office Hour – routinely used
- Suggestion box continues to be rarely used
- Weekly Q&A session with NSLS-II management added to facilitate improved communications/access
- Increased presence of management team on the experiment floor
- Adjustments to Controls Town Hall meetings to increase participation
- Staff are incredibly dedicated and often invest heroic efforts to support our mission
- Performance Appraisal modifications by BNL should improve uniform scale across the Lab
- Scientific staff progression at BNL – Bob Tribble has charged a Committee to Review Classification Titles for Science and Technology
- Group lunches to recognize special efforts of specific groups or teams

Setting Expectations – User Beamline Guide

1. Accelerator 24 hour coverage
2. Beamline operations not able to provide 24 hour coverage
3. Users expected to contact staff prior to proposal and visit
4. User staffing expectations
5. Staff availability
6. Collaborations
7. Commissioning
8. SAF
9. Post-experiment clean-up
10. Data transfer
11. Publications
12. End-of-run survey

Summary

- Operating beamlines continue to mature and increase productivity
- Outstanding NSLS-II capabilities together with the expertise and dedication of the staff are producing high impact science
- Most beamlines in the general user program are still working hard to improve performance and commission additional capabilities for the user community.
- Hiring staff for beamline operations continues
- Flat or declining budgets will impact Photon Science Division operations