Quick X-ray Absorption and Scattering (QAS) Beamline IRR Functional Description

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1 INTRODUCTION

1.1 Primary Research Capabilities

QAS (Quick x-ray Absorption and Scattering) will enable *in situ* studies of chemical and physical transformations by both quick and conventional x-ray absorption and scattering measurements. It will provide for the investigation of fast kinetics of samples from the fields of catalysis, energy storage/conversion and other scientific disciplines. It will allow measurements using separate and combined XAS and XRD techniques, as well as combining these techniques with those provided by end stations with IR and Raman spectroscopy capabilities, in different sample environments.

The QAS beamline was proposed to serve the chemical and energy sciences community. Its scientific program moved from that of NSLS X18A, X18B and the high energy part of X19A. It will enable the development of new chemical process and catalysts, and be able to measure reactions on sub-second and longer times scales.

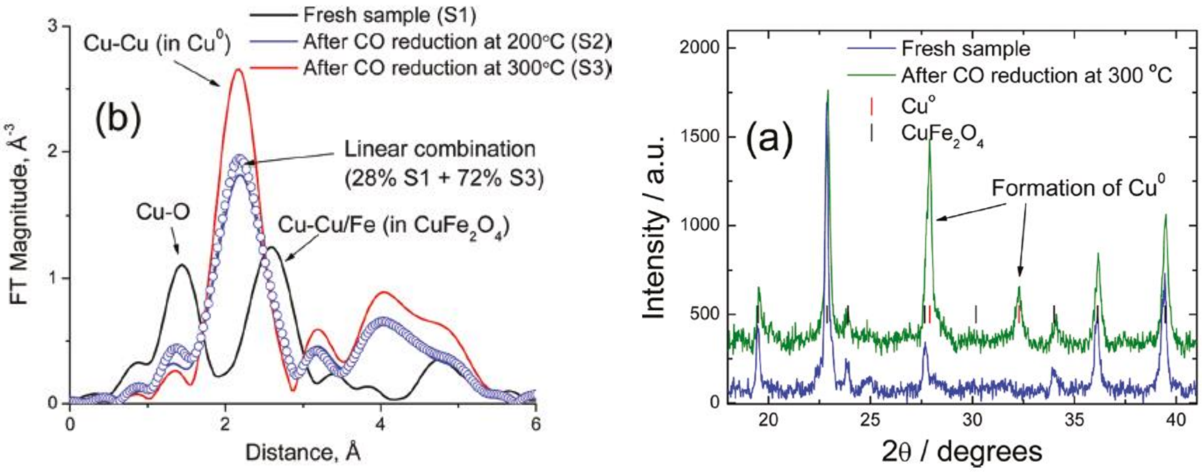

Figure 1: Combined XAS and XRD on reduction of $CuFe_2O_4$ metal oxide catalyst

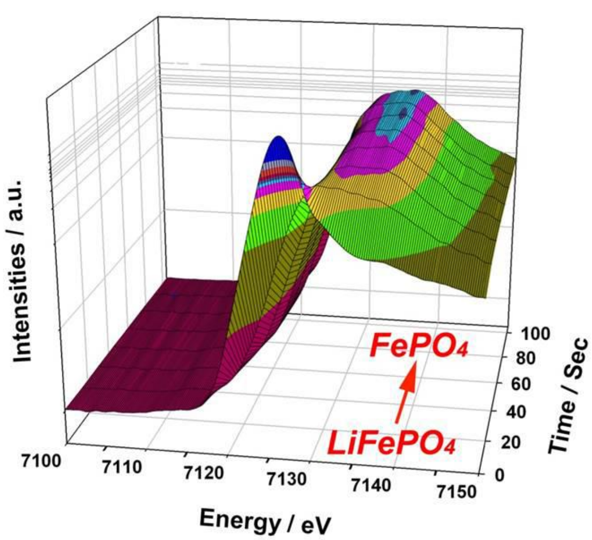

Figure 2: High rate delithiaton of LiFePO4 in Li ion batteries studied by quick XAS

1.2 Beamline Staff

Lead Beamline Scientist	Steven Ehrlich	
Authorized Beamline Staff	Klaus Attenkofer	ISS Lead Beamline Scientist
	Paul Northrup	TES Lead Beamline Scientist
	Eli Stavitski	ISS Beamline Scientist
	Syed Khalid	Research Engineer
Beamline Support Staff	Gary Nintzel	Mechanical Technician
	Lukas Lienhard	Mechanical Engineer
	Chanaka De Silva	Controls Engineer

2 BEAMLINE DESIGN AND COMPONENTS

2.1 Beamline Performance Goals

QAS is designed for quick and conventional XAS and XRD experiments on samples in a variety of sample environments at different energies. The energy range was chosen to allow access to absorption edges from titanium (4.9664 keV) to tin (29.2001 keV), to study elements of interest to catalysis, battery research and more. Table 1 summarizes the designed performance of the QAS beamline. The flux performance was estimated using ray-tracing simulations of the source and beamline optical components

Table 1: Designed Performance of the QAS Beamline

Parameter	**Specification/Description**
Source	3-pole wiggler
Operating energy range	4.7 – 31 keV
Monochromator	Quick scan capable double crystal channel-cut Si(111) ($\Delta E/E \approx 10^{-4}$)
Spot Size	1.8mm (V) x 10mm (H) (collimated, unfocused); <0.5mm (V) x 1.0mm (H) (toroidal focusing mirror)
Energy scanning	1000+ eV scans, in continuous mode up to 10 scans/sec (5 Hz)
Flux at 500 mA storage ring current	7.9×10^{11} ph/s at 10 keV; 2.9×10^{11} ph/s at 20 keV

2.2 Beamline Layout

The estimated performance, above, is based on the conceptual layout of the QAS beamline, shown in Figure 3. The source for the QAS beamline is a three-pole wiggler (3PW) installed at 7-BM. A flat, bendable mirror in the front end both collimates the beam for better energy resolution and is used for harmonic rejection. A double crystal channel-cut Si(111) monochromator (DCM) selects the photon energy and provides the capability for both conventional and quick energy scans. The toroidal mirror provides horizontal and vertical focusing and is also used for harmonic suppression. The endstation is equipped with beam defining slits, diagnostics and sample stages. Detectors include ion chambers for transmission XAS, Si dift detectors for fluorescence XAS and an area detector for x-ray diffraction.

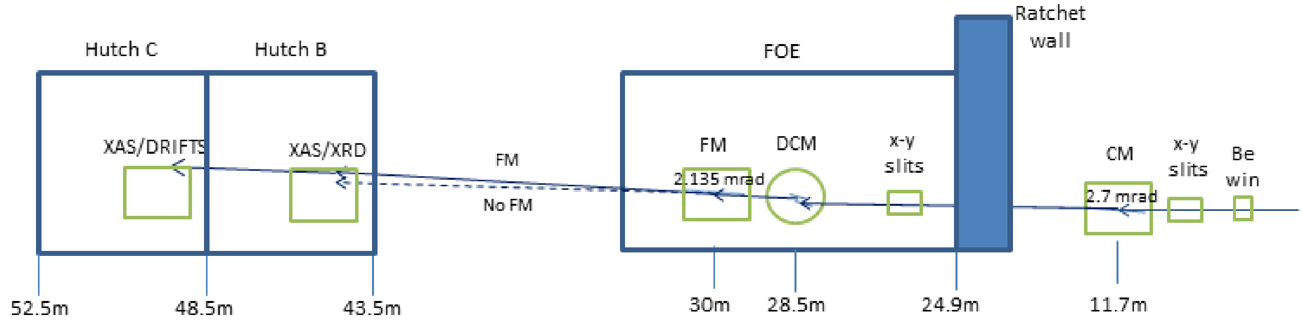

Figure 3: Optical Layout of the QAS Beamline (beam direction is right to left)

2.2.1 Source

The 3PW source characteristics are summarized in Table 2. The aperture for the first fixed mask in the QAS front end has an acceptance of 2.0 mrad H x 0.3 mrad V; the corresponding pink-beam power out of the Front End is estimated to be about 70 W.

Table 2: Source Parameters for the NSLS-II 3PW

Parameter	Specification/Description
Peak Field	1.14 T
Length	0.25 m
Number of Periods	0.5
Magnetic Gap	28 mm
Critical Energy	6.8 keV
On-axis Power Density	260 W/mrad^2
Maximum Total Power	320 W

2.2.2 Optics and Diagnostics

The overall layout of the QAS beamline is shown below in Figure 4. The collimating mirror is located inside the front end. The first optical enclosure (FOE) houses the DCM and the toroidal focusing mirror. The tables list the properties of the collimating mirror, monochromator and toroidal focusing mirror.

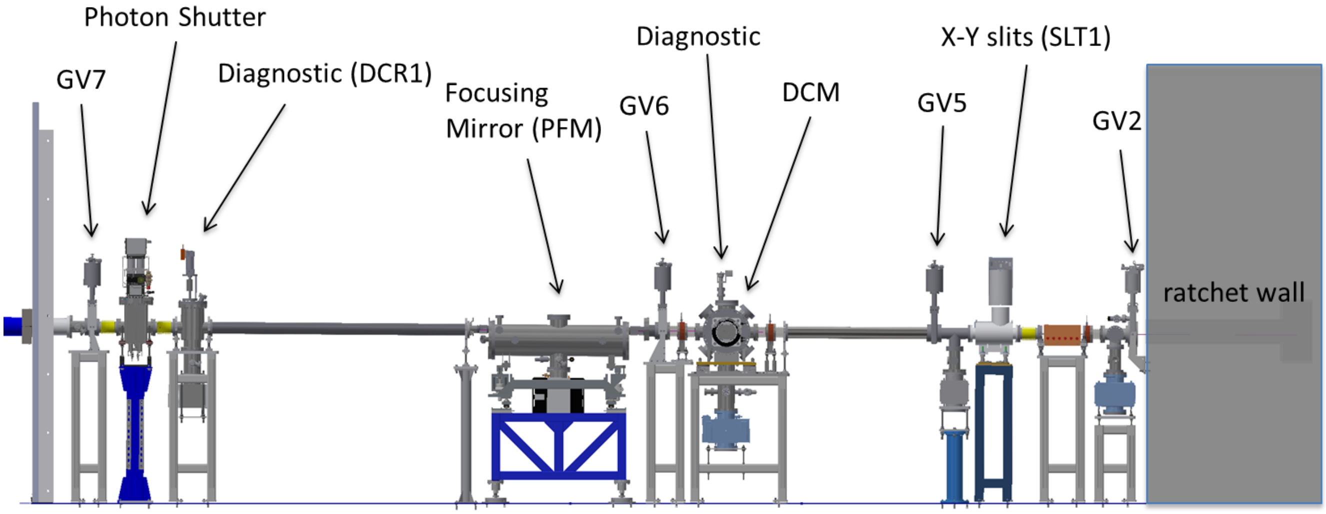

Figure 4: Optical Layout of the QAS Beamline (beam direction is right to left)

Collimating Mirror Parameters

Dimensions	700 mm x 100 mm x 50 mm
Substrate	Single crystal Si flat
Coating	Rh and Pt stripes
Pitch	2.7 mrad
Cutoff energy	Rh: ~25 keV; Pt: ~31 keV
Positioning	roll, pitch, yaw
Bender	SESO

Monochromator

Type	Double crystal, channel-cut Si(111); quick scan capable
Energy range	4.7 – 31 keV (~3.6 - 26 degrees in Bragg angle)
Crystal gap	3.75 mm
Scan modes	Step and continuous
Drive	Direct drive servo-motor
Cooling	Water-cooled Ni-coated Cu block with Ga-In eutectic

Toroidal Focusing Mirror

Dimensions	1000 mm x 100 mm x 60 mm
Substrate	Glass cylindrical (42.7 mm sagittal radius)
Coating	Rh
Pitch	2.135 mrad
Cutoff energy	31.5 keV
Positioning	roll, pitch, yaw
Bender	NSLS design

Diagnostics

Location	Type
Front end	White beam slits with drain current measurements upstream of collimating mirror
Front end	Fluorescent screens on fixed mask 2 downstream of collimating mirror
Front end	Insertable fluorescent screen downstream of collimating mirror
FOE	Pink beam slits with drain current measurements
FOE	Insertable fluorescent screen downstream of monochromator crystal
FOE	Insertable fluorescent screen downstream of focusing mirror
End station	Fluorescent screen and ion chamber

3 BEAMLINE SAFETY

3.1 Radiation Shielding

The design of all radiation shielding (hutches and radiation safety components) for the QAS beamline follow set guidelines to reduce radiation levels external to the beamline enclosures during normal operation to as low as reasonably achievable. This is confirmed by detailed calculations (see separate Tech Notes) that show that for all areas outside the FOE, the maximum dose is < 0.05 mrem/hr. Shielding of hutches is as follows:

Hutch 7-BM-A (lead and steel hutch):

- Lateral wall: 18 mm lead
- Downstream wall: 50 mm lead
- Roof: 4 mm lead

Beam Transport Pipe:

- Pipes: 5 mm lead
- Ion pump enclosure: 5 mm lead

Hutch 7-BM-B & 7-BM-C (steel hutches):

- Lateral wall: 3 mm steel
- Downstream wall: 6 mm steel
- Roof: 2 mm steel
- Beam stops: 0.5 m x 0.5 m x 12 mm thick lead

A photon shutter of NSLS-II design (30 x 60 mm aperture) is installed at the downstream end of the 7-BM-A hutch.

3.2 Radiation Safety Components and Configuration Control

These are the components required to contain the synchrotron radiation from the 3PW source and the gas Bremsstrahlung radiation from the storage ring. The major components are shown in Figure 5 and described in detail below.

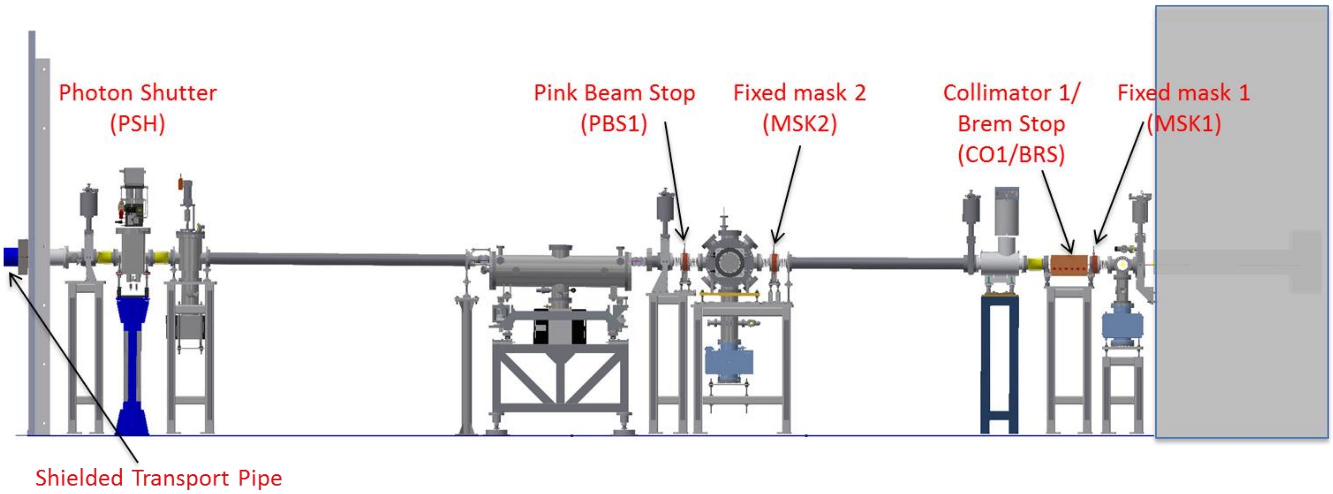

Figure 5: QAS Layout with Major Radiation Safety Components (beam direction is right to left)

The radiation safety components in the front end (not shown) include fixed masks and standard lead Bremsstrahlung collimators and shadow shield. In the FOE, there is one 12" thick lead collimator/stop (CO1/BRS), which serves as the primary Bremsstrahlung stop, and a standard 50 mm thick lead guillotine at the downstream end of the FOE. There are 2 fixed masks, MSK1 and MSK2, and a pink beam stop, PBS1, which stops the pink beam from exiting the FOE. FOE labyrinths include the PPS labyrinth on the outboard wall and 5 roof labyrinths. Hutches B and C include 4 roof and one user labyrinth each, and a 0.5 m x 0.5 m x 12 mm lead beam stop on the downstream wall of each hutch, with the one in hutch B including a PPS interlocked door for letting beam into hutch C. Refer to the raytracing drawings and the 7-BM radiation safety component checklist for more information.

3.2.1 Primary Bremsstrahlung Management

Primary Bremsstrahlung radiation is fully blocked in the FOE at Collimator 1/Brem Stop (CO1/BRS).

3.2.2 Secondary Bremsstrahlung Management

Secondary Bremsstrahlung radiation is contained in the FOE so no additional secondary Bremsstrahlung shielding is required.

3.2.3 Configuration Control

All radiation safety components are under configuration control, in accordance with the NSLS-II Radiation Safety Component Configuration Management procedure (PS-C-ASD-PRC-055).

3.3 Area Radiation Monitors (ARMs)

The results of top-off radiation analysis show that the total dose rates on the walls and roof of the QAS FOE are less than 100 mrem/h, therefore there is no need for an ARM.

3.4 Personnel Protection system (PPS)

The PPS controls access to the hutches through the interlock system and beamline-specific search and secure procedure, to ensure personnel safety during normal operation of the beamline.

3.5 Hazard Identification and Mitigation

The QAS beamline is similar to other beamlines already in operation at NSLS-II with respect to any hazards that might be encountered. A USI evaluation has been conducted and it was determined that the anticipated activities at the beamline do not violate the existing SAD and ASE. All relevant NSLS-II procedures and safety practices were followed during the design and construction of the beamline to mitigate the hazards identified in these documents.

4 INSTRUMENT READINESS

4.1 Survey and Alignment

The beamline components are installed according to the specifications and respective final designs. Installation of the components is verified and documented by travelers with input from the NSLS-II Survey Group working closely with the beamline staff.

4.2 Utilities

The following services/capabilities are deployed at the beamline:

- Electrical power distribution to all electrical power outlets, light fixtures, fans, etc., in the hutches and along the beamline
- Distribution of process chilled water
- Compressed air
- Dry nitrogen gas
- Liquid nitrogen in hutch B
- Network connectivity
- Cabling and piping support for all utilities, including EPS and PPS

4.3 Vacuum System and Pressure Safety

The vacuum pressure downstream of GV2 is expected to be 10^{-8} - 10^{-9} torr range. There is a water-cooled Be window at 9.1 m from the source, in the front end, separating the beamline vacuum from the ring vacuum.

The end-of-beamline Be window has been pressure-tested to 2.0 atm differential pressure (higher pressure downstream). This window terminates beamline vacuum at the upstream end of Hutch B. A vacuum failure in a given beamline vacuum section results in the closing of the front end photon shutter and the closing of the adjacent gate valves. When venting any of these beamline vacuum sections to dry nitrogen gas, a pressure relief valve with a very low relief pressure (1/3 psi) will be used to prevent any internal overpressure condition.

The front end collimating mirror, FOE slits and monochromator are the only components to have any source of potential pressurization (internal water cooling), and are equipped with a certified burst disc to prevent overpressure.

4.4 Controls

All motorized components have been tested by the Controls Group and documented in the travelers. Controls System Studio (CSS) screens have been prepared to access the motors on the components. The individual motors are also accessible using standard EPICS Extensible Display Manager (EDM) screens.

4.5 Equipment Protection System (EPS)

The EPS at the QAS beamline performs the following functions:

- Vacuum pressure monitoring and interlock for required vacuum sections of the beamline
- Temperature monitoring and interlock for non-safety-related components, including the front end collimating mirror and the monochromator
- Water flow monitoring and interlock for cooling of the front end collimating mirror, front end diagnostic screens, FOE slits and the monochromator
- Experimental Physics and Industrial Control System (EPICS) interface for components that require I/Os installed on the EPS Programmable Logic Controller (PLC), including the readout of thermocouples, control of venting, evacuation valves, and pumps