X-ray Fluorescence Microprobe (XFM) Beamline

IRR Functional Description

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1 INTRODUCTION

1.1 Primary Research Capabilities

XFM is designed for X-ray Fluorescence (XRF) imaging and high-quality X-ray Absorption Fine-structure Spectroscopy (XAFS) at spatial resolutions from the mm to μm scale. The design of XFM is optimized for the 2.05 - 23 keV energy range (Phosphorus to Technicium K edges). XFM will serve key scientific needs of a broad user community and support frontier research along the entire spectrum of agricultural, biological, Earth, energy, environmental, and materials sciences. Advanced capabilities as an "XAFS Microprobe" enable a scientific program that specifically addresses multi-scale, multi-component and dynamic systems. Such systems include those in soil and environmental sciences, such as plant-root-microbe systems; geologic and cosmologic materials; energy-related functional materials and devices (e.g., energy storage, photovoltaics, fuel cells, catalysts); systems critical to or indicative of climate-change, including terrestrial, atmospheric and marine components; and both biological and geological carbon sequestration.

1.2 Beamline Staff

Lead Beamline Scientist	Ryan Tappero	
Authorized Beamline Staff	Alvin Acerbo	Beamline Scientist
	Paul Northrup	TES Lead Beamline Scientist
Beamline Support Staff	Mike Maklary	Mechanical Technician
	Ed Haas	Mechanical Engineer
	Jun Ma	Controls Engineer

2 BEAMLINE DESIGN AND COMPONENTS

2.1 Beamline Performance Goals

XFM is designed for microbeam XRF imaging and high-quality XAFS spectroscopy. Spatially-resolved XAFS spectroscopy requires optimization of beamline design that is distinct from either bulk-XAS beamlines or imaging beamlines (even those with some XAS capabilities). Some of the unique capabilities and requirements that define an "XAFS Microprobe," such as XFM and its tender-X-ray sister beamline TES, are listed in Table 1. The ultimate challenge is full EXAFS spectroscopy of a particle or domain the same size as the beam. To achieve this, beam on the sample must not change position, size, shape, flux distribution, energy distribution, or harmonics distribution, over an energy scan of up to 1000 eV. A long working distance is needed to accommodate *in-situ* measurements and large or odd-shaped samples. Combined XAFS/XRF imaging can include both image-stack and energy-scan-per-pixel modes, to perform oxidation-state mapping or quantitative speciation imaging by XANES PCA or XAFS Fourier-transform imaging. Execution of XAS imaging in realistic time necessitates fast on-the-fly energy scanning for sub-minute EXAFS, with adequate flux for dilute concentrations. Tunable flux density is important for samples susceptible to radiation damage.

Table 1. XFM Requirements

Parameter	**Design target**
Spot Size	User-tunable from ~1 μm to ~1 mm, with independent control of horizontal and vertical size
Positional Stability	Within ~1% of spot size, over 1000+ eV energy scan, over 12+ hours
Sample Stage Scanning	Up to 300x200 mm area, in both step and on-the-fly modes
Energy scanning	Up to 1000 eV, in both step and on-the-fly (at up to ~1000 eV/min) modes
Energy Stability/Repeatability	0.1 eV or better, scan-to-scan and over 24+ hours
Flux	Up to 10^{11} ph/sec at 500 mA ring current
Sample Environment	Ambient atmosphere with accommodations for Helium atmosphere, in-situ cells or large samples

2.2 Beamline Layout

The optical layout of the XFM beamline is shown in Figure 1. A toroidal mirror is used to focus the beam (H & V) onto a secondary source aperture (SSA), and beam diverging from the SSA is directed to a vertical collimating mirror and monochromator before final focusing by Kirkpatrick-Baez (KB) micro-focusing mirrors.

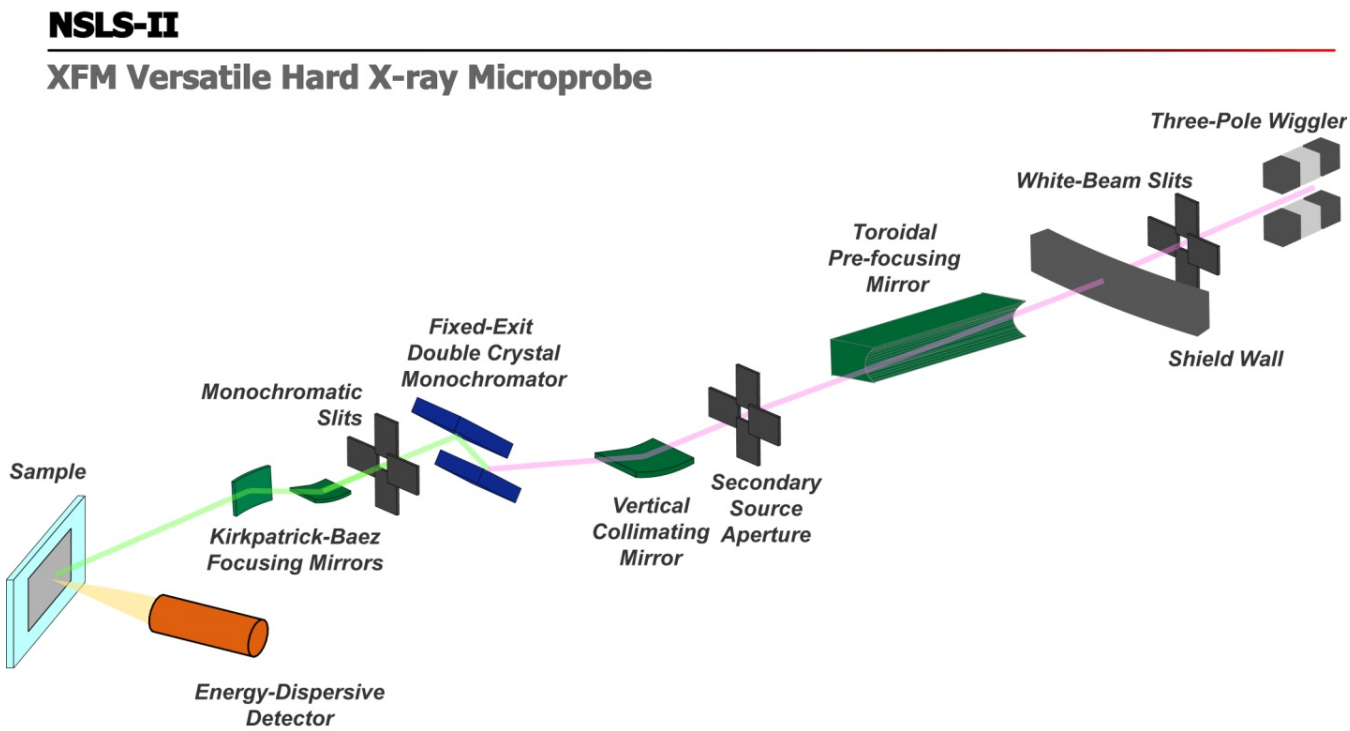

Figure 1: Optical Layout of the XFM Beamline (beam direction is right to left)

2.2.1 Source

XFM source requirements are well matched by the NSLS-II 3-pole wiggler (3PW) source. XFM requires a small source size, a broadband source without spatial or energy-dependent structure within the fan accepted, and having a critical energy around 6 keV to provide good flux with minimal high-energy power. Acceptance required to provide an adequate beam fan to fully illuminate the XFM optics is 0.4 mrad (V) x 0.117 mrad (H).

Table 2. Designed Performance of the XFM Beamline

Parameter	Specification/Description
Source:	3-pole wiggler (3PW)
Acceptance:	0.4 mrad V x 0.117 mrad H
Operating Energy Range:	2.05 – 23 KeV
Monochromator:	Fixed-exit double crystal monochromator(25 mm offset)
Beam size at sample (FWHM):	1 to 10 μm (tunable) or 1 mm
Flux at sample at 500 mA storage ring current:	Up to 2 x 10^{11} ph/sec

2.2.2 Optics and Diagnostics

The overall layout of the XFM beamline is shown in Figure 1 and described above. Specifications of the mirrors and monochromator are tabulated below.

Mirror 1:

Optical length	1000 mm
Substrate	Single-crystal Si
Coating	Rh on Cr base
Optical quality	0.8 urad slope error, 0.25 nm roughness
Positioning	Roll, pitch, yaw
Pitch	3.0 mrad (inboard deflection)
Bender	cylindrical

The SSA is a JJ X-ray IB-C30 UHV precision slits unit, with 30 x 30 mm range and 0.3 um precision. It is mounted on a granite block for vibrational stability.

Mirror 2:

Optical length	600 mm
Substrate	Single-crystal Si
Coating	Rh on Cr base or bare Si
Optical quality	0.5 urad slope error, 0.25 nm roughness
Positioning	motorized, independent positioning of each end for height, pitch and yaw control
Pitch	3 mrad (upward deflection)
Bender	cross-lever type with independent actuators for each end

Monochromator:

Type	Double crystal, fixed exit
Cooling	Indirect water cooling of 1st crystal
Offset	25 mm upward
Energy range	2.05 - 23 keV
Angular range	0° to 75° theta
Crystals	Remote exchangeable sets including Si(111), Si(220), and Si(311)
Scan modes	step- and on-the-fly-scanning
Coarse adjustments	Tilt and roll adjustments on 2nd crystal
Fine adjustments	Tilt and roll adjustments on 2nd crystal

KB Mirrors:

Optical length	200 mm
Substrate	Single-crystal Si
Coating	Rh on Cr base or bare Si
Optical quality	<1.0 urad slope error, 0.25 nm roughness
Positioning	motorized, independent positioning of each end
Pitch	up to 7 mrad
Bender	cross-lever type with independent actuators for forces on each end
Working distance	100 mm from end of VFM

Diagnostics:

For beam diagnostics, XFM has drain current measurements on FE white beam slits. Fluorescence screens for beam visualization are located on Mask 1 & Mask 2 and at the midpoint of the Mirror 1 focus. In addition, high-resolution beam imaging units are located just downstream of the SSA and the monochromator. The Endstation has several ion chambers and pin diode to measure beam intensity.

3 BEAMLINE SAFETY

3.1 Radiation Shielding

The design of all radiation shielding (hutches and radiation safety components) for the XFM 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 at 1 foot distance (< 0.5 mrem/hr at contact for downstream wall). Shielding of hutches is as follows:

Hutch 4-BM-A (lead and steel hutch):

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

Beam Transport System:

- Pipes: >5 mm lead
- Enclosures: >5 mm lead

Hutch 4-BM-B & 4-BM-C (steel hutches):

- Lateral wall: >3 mm steel
- Downstream wall: >6 mm steel
- Roof: >2 mm steel
- Beam stop: >12 mm lead

A photon shutter of NSLS-II design (30 x 60 mm aperture) is installed at the downstream end of the 4-BM-B 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 2 and described in detail below.

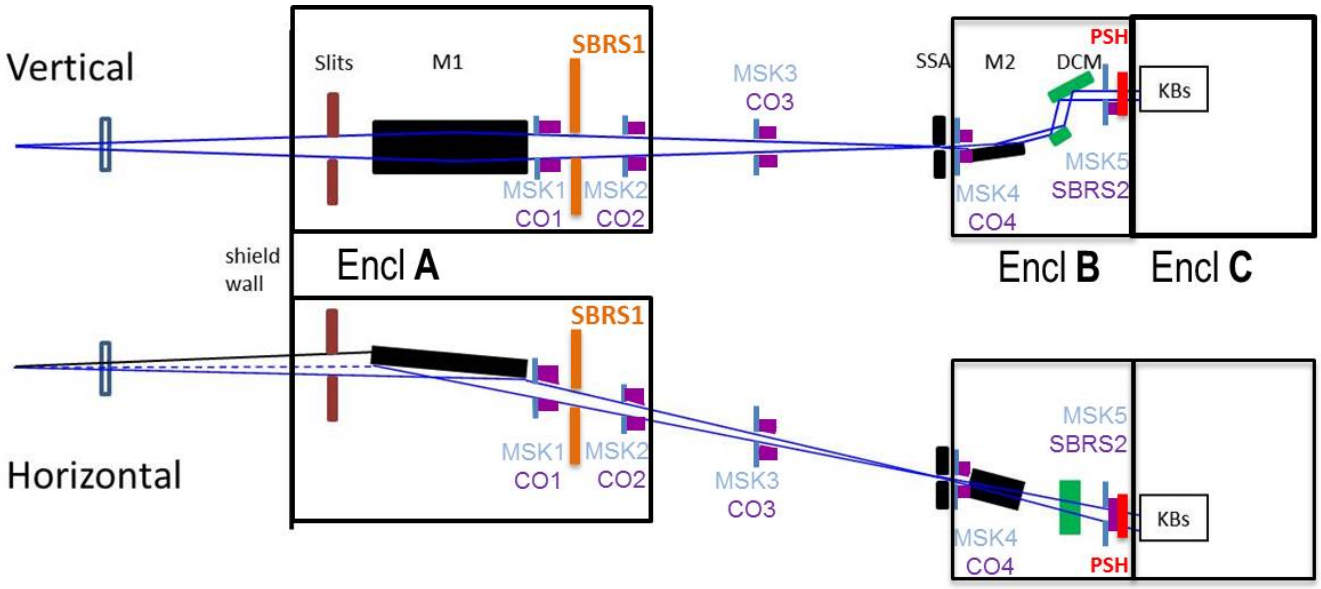

Figure 2: XFM Layout with Major Radiation Safety Components (beam direction is right to left)

The radiation safety components in the Front End (not shown) include fixed masks and standard lead Bremsstrahlung collimators. In the FOE, we employ two standard lead collimators as primary Bremsstrahlung radiation shields/stops (CO1 & CO2, respectively), and a standard lead guillotine at the downstream end of the FOE. Collimator 2 serves as the primary bremsstrahlung stop and Mask 2 serves as the white beam stop. A secondary bremsstrahlung shield (SBRS1) is also located in the FOE between Collimator 1 and Collimator 2. FOE labyrinths include the PPS labyrinth on the outboard wall and the 4 roof labyrinths. The radiation safety components located between the FOE and Endstation hutch include additional Masks (3, 4 & 5) and standard lead collimators (3 & 4) and a tungsten secondary bremsstrahlung stop (SBRS2). Refer to the raytracing drawings and the 4-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 2.

3.2.2 Secondary Bremsstrahlung Management

Secondary bremsstrahlung radiation arises from scattering of primary Bremsstrahlung radiation off Mirror 1 and downstream fixed mask (MSK1). This is collimated by collimators 1 and 2 in the FOE, collimator 3 in the beam transport and collimator 4 in the SOE, and then stopped by a secondary Bremsstrahlung Stop in the SOE. Secondary bremsstrahlung scatter outside of collimators 1 and 2 in the FOE is blocked by the secondary bremsstrahlung shield and FOE walls. FLUKA simulations were carried out to verify effectiveness of shielding at a ring current of 500 mA.

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)

Radiation levels in the area will be actively monitored through an area radiation monitor (ARM) installed on the outboard wall at the upstream end of the FOE. The monitor will be certified in accordance with the procedure PS-C-ASD-PRC-008, *NSLS-II Area Radiation Monitor PPS Test.*

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.

The PPS also monitors beamline gate values (GV3-GV6). In the event of a vacuum trip, the PPS system closes the Front End photon shutter to shut off the beam.

3.5 Hazard Identification and Mitigation

The XFM 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
- 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^{-9} - 10^{-10} mbar range. There is no physical barrier between the beamline vacuum and Front End vacuum. The beamline vacuum for the white-beam (M1) mirror downstream, is differentially separated from the accelerator vacuum by a 2 um graphite window located just upstream of the M1 mirror. The accelerator vacuum is protected by a fast valve located in the Front End, with a fast value sensor located on M1 mirror vessel.

The end beryllium window, which has been pressure-tested to 2.0 atm differential from downstream and 1.0 atm differential from upstream, does not experience vacuum downstream. This window terminates beamline vacuum at the upstream end of Hutch C. 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 relieving pressure (1/3 psi) will be used to prevent any internal overpressure condition.

The M1 mirror 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 XFM 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 white-beam (M1) mirror and monochromator
- Water flow monitoring and interlock for cooling of the white-beam (M1) mirror, retractable diagnostic screens and 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