Tender Energy Spectroscopy and Imaging (TES) Beamline

IRR Functional Description

Contents

1 INTRODUCTION

1.1 Primary Research Capabilities

TES is designed for high-performance extended X-ray absorption fine-structure spectroscopy (EXAFS) in heterogeneous and structured materials, at spatial resolutions from the mm to µm scale, and under various in-situ or in-operando sample conditions. The scientific mission of TES is the application of tender-energy spatially resolved EXAFS to studies of multi-scale heterogeneous and dynamic systems. It focuses on core DOE:BES research in energy and climate, with strong Partner User programs extending from this core into studies of geological, chemical and extraterrestrial materials, plus a broad multidisciplinary user program. TES will 1) serve a critical need to obtain chemical specificity complementary to, and unobtainable at, other current and anticipated NSLS-II beamlines, and provide new capabilities unique among all DOE synchrotron facilities; 2) continue and enhance current and future research programs utilizing <8 keV XAS and microprobe techniques developed at the NSLS and elsewhere, and exploit untapped science in this area; and 3) take advantage of the superlative characteristics of the NSLS-II BM source, to create a best-in-class beamline. The design of TES is optimized for the 1.2-5 keV energy range; up to 8 keV will be accessible to meet important scientific needs. TES will provide advanced capabilities as an "EXAFS Microprobe" to enable a scientific program that specifically addresses multi-scale, multi-component and dynamic systems using 1) spatially resolved XAS at mm to µm scales for heterogeneous, structured and single-particle samples, and combined XAS and XRF imaging, and 2) high-performance, *in-situ*, *operando* and rapid-scanning XAS for chemistry and materials characterization and kinetics/catalysis research.

TES will serve key scientific needs of a broad user community and support frontier research along the entire spectrum of agricultural, biological, catalysis, Earth, energy, environmental, and materials sciences. The design of TES is optimized for the tender 1.2-5 keV energy range, an operational range between those of standard soft <2 keV and hard >5 keV XAS and microprobe beamlines. The absorption edges of greatest interest in the <5 keV energy range are 1) K-edges of Mg, Al, Si, P, S, Cl, K, Ca, and Ti, which are major elements in geomaterials, key elements in catalysts and energy-storage materials, and vital bionutrients, 2) L-edges of As through I, including important metals for novel materials and superconductors, environmental contaminants and biological toxins, and 3) M-edges of heavy REEs to Pu, including Hg and radionuclides of concern to DOE, catalysts, and metals important to functional nanomaterials. Advanced capabilities as an "EXAFS 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	Pau Northrup	
Authorized Beamline Staff	Ryan Tappero	XFM Lead Beamline Scientist
Beamline Support Staff	Gary Nintzel	Mechanical Technician
	Ed Haas	Mechanical Engineer
	Chanaka DeSilva	Controls Engineer

2 BEAMLINE DESIGN AND COMPONENTS

2.1 Beamline Performance Goals

XAS in the tender energy range requires specific optimizations of beamline design that are different from the approaches taken by either hard or soft X-ray facilities. The source must have a low critical energy; optics operate at higher incidence angles, requiring geometric considerations; specialized monochromator crystals are needed; harmonic-rejection requires more attention; mirror coatings must be carefully considered to avoid absorption edges such as Rh L and Pt M; windows and beam position monitors must be transparent to lower energy than typically used; sample environment, in particular when samples are not vacuum-compatible, must be helium; sample mountings and in-situ cells must be designed for low-energy transparency; and sample thickness and particle size must be considered on a smaller scale than for hard X-rays.

Spatially resolved EXAFS likewise requires optimization of beamline design that is distinct from either bulk-XAS beamlines or imaging beamlines (even those with some spectroscopy capabilities). The unique capabilities and requirements that define an EXAFS Microprobe -- such as TES and its hard-X-ray sister beamline XFM -- include the following. A versatile spot size from 1 μm to 1 mm, with independent horizontal and vertical size under user control, is necessary to match beam footprint to the area to be measured. The beamline must deliver high-performance fluorescence and transmission EXAFS at all available spot sizes. The ultimate challenge is full EXAFS 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 1500 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 EXAFS 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. In order to meet the needs of its driving science programs, TES is designed to deliver the following specifications.

Table 1. General Specifications

Parameter	Design target
Energy Range	1-8 keV, optimized for 1.2-5 keV.
Spot Size	user-tunable from ~1 μm to ~1 mm, with independent control of horizontal and vertical size.
Flux	up to 1012 ph/sec at 500 mA ring current.
sample stage scanning	up to 100x100 mm area, in both step and on-the-fly modes.

Energy scanning	up to 1500 eV, in both step and on-the-fly (at up to ~1000 eV/min) modes.
Positional stability	within ~1% of spot size, over 1000+ eV energy scan, over 12+ hours.
Energy stability/repeatability	0.1 eV or better, scan-to-scan and over 24+ hours.
Harmonic rejection	tunable up to 105.
Sample environment	Helium atmosphere, accommodations for in-situ cells or large samples.

2.2 Beamline Layout

The basic layout of TES includes three optics components located in the Front End: white beam slits, a downward-deflecting bent flat collimating Mirror 1, and an upward-deflecting flat redirecting Mirror 2 at matching pitch for parallel beam at 25 mm vertical offset. This Ni-coated mirror pair will operate at a range of pitch from 6.8 to 20 mrad to provide appropriate energy cut-off for harmonic rejection, while maintaining a fixed offset by virtue of a longer Mirror 2. In the First Optics Enclosure (FOE) is be a thin graphite filter to absorb visible and very-low-energy photons, a fixed-exit double-crystal monochromator, and a 1:1 toroidal (bent cylinder) focusing Mirror 3. A beam position monitor near the point of focus will be used for feedback for beam position and energy calibration. Mirror 3 will focus to a secondary source aperture (SSA) which will provide a user-tunable virtual source for microfocusing optics for the endstation. This necessitates a long beam transport tube from the FOE to the endstation which contains a pair of KB mirrors for achromatic focusing to the micron scale.

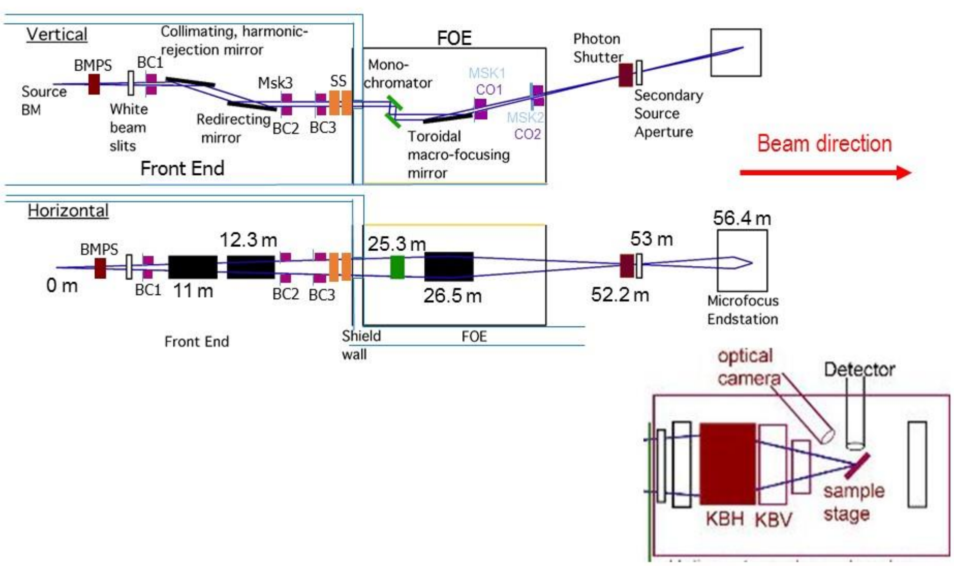

Figure 1: Conceptual Layout of the TES Beamline

2.2.1 Source

TES source requirements are well matched by the NSLS-II dipole Bend Magnet source, without the presence of a 3PW. TES requires a small source size, a broadband source without spatial or energydependent structure within the fan accepted, and having a critical energy around 2.5 keV to provide good flux with minimal high-energy power. Acceptance required to provide an adequate beam fan to fully illuminate the TES optics is 0.4 mrad (vertical) x 2.5 mrad (horizontal).

Tabel 2: TES Requirements and Source Parameters for the NSLS-II BM

Parameter	TES Requirement	NSLS-II specifications
Source type	BM	BM
Location	even sector downstream of fixed-gap ID, close to ISS	8-BM
Source size	as small as possible	125 H x 13.4 um V (sigma)
Critical energy	ideally ~2.5 keV	2.4 keV
Energy spectrum	smooth continuous broadband	typical BM flux curve
Energy range	1-8.3 keV	<1 to ~10 keV
Acceptance	0.4 mrad V x 2.5 mrad H below 4 keV; 0.23 x 2.5 above 4 keV	up to 0.6 x 3.0 mrad
Uniformity	uniform flux and energy distribution within acceptance	nearly uniform
Flux	maximized within acceptance	excellent
Total power	minimized relative to flux	~40 W

2.2.2 Optics and Diagnostics

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

Mirror 1:

Size	813 mm long, 100 mm wide, 60 mm thick.
Optical surface	80 mm wide, full length.
Substrate	Si.
Coating	Ni on Cr base.
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	variable, over the range of 6.8 to 20 mrad.
Bender	cross-lever type with independent motorized actuators for each end.

Mirror 2:

Size	1200 mm long, 70 mm wide, 40 mm thick.
Optical surface	50 mm wide, full length.
Substrate	Si.
Coating	Ni on Cr base.
Optical quality	0.5 urad slope error, 0.25 nm roughness.
Positioning	motorized, independent positioning of each end for height and pitch control.
Pitch	6.8 to 20 mrad, matched to Mirror 1 pitch while maintaining constant vertical offset.

Monochromator:

Type	Double crystal, *fixed exit*.
Cooling	Indirect water cooling of 1st crystal.
Offset	35 mm downward.
Energy range	1 to 8.3 keV.
Angular range	10° to 80° theta.
Crystals	Exchangeable sets including Si(111), InSb, Quartz, and Beryl.
Acceptance	At least 5 mm vertical at above 12° theta, 65 mm horizontal.
Scan modes	step- and on-the-fly-scanning. Scan speeds of 150 to 1000 eV/min.
Fine adjustments	Tilt and roll adjustments on first crystal.
Feedback capabilities	Compatible with TES optical design feedback requirements.
Positional stability	With feedback operational, within 20 um at SSA during scan and over 12+ hours.
Energy stability	Repeatability within 0.1 eV scan-to-scan and over 12+ hours.
Vacuum	High 10^{-9} range.

Mirror 3:

Size	960 mm long, 100 mm wide, 80 mm thick.
Optical surface	80 mm wide, full length.
Sagittal radius	186.0 mm.
Substrate	Si.
Coating	Ni on Cr base.
Optical quality	0.8 urad slope error, 0.25 nm roughness.
Positioning	motorized, independent positioning of each end for height, pitch and yaw control.
Pitch	7 mrad for Endstation 1, 5 mrad to deliver beam to Endstation 2.
Bender	cross-lever type with independent actuators for each end.

The Secondary Source Aperture is a JJ X-ray 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, and can translate along beam over more than 500 mm to align with toroid focus.

KB Mirrors:

Size	HFM 320 mm, VFM 180 mm.
Substrate	Si.
Coating	Ni on Cr base.
Optical quality	0.5 urad slope error, 0.25 nm roughness.
Positioning	motorized, independent positioning of each end.
Pitch	up to 15 mrad for HFM, 10 mrad for VFM.
Bender	cross-lever type with independent actuators for forces on each end.
Working distance	80 mm from end of VFM.
Manufacturer	IDT

For beam diagnostics, TES has drain current measurements on white beam slits and SSA slits. Fluorescence screens for beam visualization are located just downstream of Mirror 1, at the downstream end of the Front End, at both the entrance and exit of the monochromator, just downstream of Mirror 3, at the downstream end of the FOE, at the mid-point of the toroidal mirror focal length, at the BPM 2 m upstream of the SSA, at the SSA, at the entrance to the endstation, and at the beam stop in the endstation. Beam intensity monitors are located at the monochromator entrance, the BPM, and at Diagnostic 3 just upstream of the endstation. The Endstation has two ion chambers.

3 BEAMLINE SAFETY

3.1 Radiation Shielding

The design of all radiation shielding (hutches and radiation safety components) for the TES 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 8-BM-A (FOE, white beam hutch):

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

Hutch 8-BM-B (monochromatic beam hutch):

- All walls, ends, top and bottom 1.06 mm lead
- Beam stop 4.2 mm lead equivalent

Beam transport system:

- Pipes, vacuum components: >1mm iron
- Bellows shields: >1 mm iron
- Viewports: >1 mm lead equivalent glass

A monochromatic beam shutter of the standard NSLS-II design is installed between the BPM and SSA near the endstation.

3.2 Radiation Safety Components and Configuration Control

These are the components required to contain the synchrotron radiation from the BM source and the gas Bremsstrahlung radiation from the storage ring. The major components are shown in Figure 1. The radiation safety components in the Front End include fixed masks and standard lead Bremsstrahlung collimators; Mask 3 serves as the white beam stop, and Collimator 2 serves as primary Bremsstrahlung stop. In the FOE, we employ two standard lead collimators as secondary Bremsstrahlung radiation shields, and a standard lead guillotine at the downstream end of the FOE. 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 hutches include several bellows inserts. Refer to the raytracing drawings, and the 8-BM radiation safety component checklist.

3.2.1 Primary Bremsstrahlung Management

Primary Bremsstrahlung radiation is fully blocked in the Front End at Collimator 2.

3.2.2 Secondary Bremsstrahlung Management

Secondary Bremsstrahlung radiation arises from scattering of primary Bremsstrahlung radiation off Mirror 1 and upstream masks. This is collimated by collimators 2 and 3 in the Front End, and stopped by the collimators in the FOE. 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, as is the entire beam transport between the FOE and the endstation hutch, in accordance with the NSLS-II Radiation Safety Component Configuration Management procedure (PS-C-ASD-PRC-055).

3.3 Area Radiation Monitors (ARMs)

Following detailed FLUKA calculations of all possible top-off loss electron beam trajectories (which are stopped at or upstream of Front nd Mask 3), it was determined that 8-BM does not require 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. 8-BM-B (Endstation) is secured using a Kirk Lock mechanism, as it is too small for person access.

3.5 Hazard Identification and Mitigation

The TES 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 are 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 the 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 inside the FOE for the monochromator heat exchanger and rack cooling, and to the endstation rea near 8-BM-B for water-cooled racks.
- Compressed air
- 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} mbar or better. There is no physical barrier between the beamline vacuum and Front End vacuum. The beamline vacuum fom the monochromator downstream is differentially separated from the accelerator vacuum by a 2 um graphite window located just upstream of the monochromator. The accelerator vacuum is protected by a fast valve located in the Front End as far upstream as possible; sensors are located at Mirror 1 and just inside the FOE. Vacuum components of TES are as follows:

- Front End GV4.
- Mirror 1 with ion pump and NEG.
- Mirror 2 with ion pump and NEG.
- Front End GV3
- Front End ion pumps
- GV2
- Slits box with ion pump and NEG.
- Graphite differential-pumping window 1.
- GV5
- Monochromator with integral protected turbo pump.
- GV6
- Graphite differential-pumping window 2.
- Mirror 3 system with ion pump and NEG.
- GV7
- Masks 1 (Pink Beam Stop) and 2, and lead collimators 1 and 2.
- Diagnostic 1 with ion pump.
- GV8
- Beam transport pipe outside FOE includes ion pumps 4, 5, and 6 and two NEGs. GV9 and GV10 along beam pipe.
- Beam Position Monitor with ion pump and NEG.
- GV11
- Photon Shutter with pump cross and ion pump.
- Secondary Source Aperture.
- Diagnostic section 3 with ion pumps and NEG.
- GV12
- Beam tube and Be window at entrance to endstation Hutch Box (8-BM-B).

The final beryllium window, which has been pressure-tested to 2.0 atm differential from downstream and 1.0 atm differential from upstream, cannot experience vacuum downstream. This 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 relieving pressure (1/3 psi) will be used to prevent any internal overpressure condition.

The Monochromator is the sole component to have any source of potential pressurization (internal water cooling), and is equipped with a certified burst disc to prevent overpressure.

4.4 Controls

All motorized components necessary for the photon delivery system have been tested by the controls group and documented in the appropriate travelers. Controls screens have been prepared to access the motors on the components.

4.5 Equipment Protection System (EPS)

The EPS at the TES beamline performs the following functions:

- Vacuum pressure monitoring and interlock for required vacuum sections of the beamline
- Temperature monitoring and interlock for all non-safety-related components, including the monochromator.
- Water flow monitoring and interlock for cooling of 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, actuation of the diagnostic screen in the FE, and monitoring of experimental shutter state.