Beamline for Material Measurements (BMM)

IRR Functional Description

Contents

1 INTRODUCTION

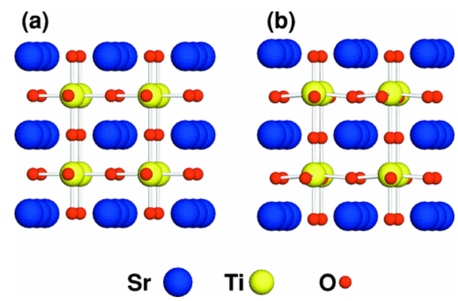
Primary Research Capabilities

The National Institute of Standards and Technology (NIST) and the Department of Energy (DOE) have a long-term partnership spanning over three decades, beginning at the National Synchrotron Light Source and continuing into operations at NSLS-II. In that time, NIST has developed advanced synchrotron measurement methods and delivered excellence in material science, impacting important societal challenges in energy, health, environment, national security, and improving our quality of life. This partnership promotes innovation and enhances US industrial competiveness in areas such as inorganic and organic semiconductors, photovoltaics, self-assembled monolayers (SAMs), biological and environmental materials, batteries, catalysts, fuel cells, polymers, superconductors, ferroelectrics, and ferromagnets. At the NSLS, the NIST Synchrotron Methods Group operated a suite of three state-of-the-art spectroscopy beamlines (U7A, X24A, and X23A2) that spanned the absorption-edge energy range of the entire periodic table to establish structure function relationships in advanced materials. More than 200 industry and academic researchers each year used the NIST NSLS Beamline Suite to accelerate the development of new materials into devices and systems with advanced functionality for a broad spectrum of industries. Building upon this success, NIST is currently developing at NSLS-II a suite of three state-of-the-art, high-throughput spectroscopy beamlines, along with imaging and X-ray Diffraction capabilities. Taken together, the NIST Spectroscopy Beamline Suite at NSLS-II will be capable of measuring electronic, chemical, and structural properties of almost any material, often at the nanoscale, and over a very broad energy range. NIST has committed to the full funding of construction and operation of its Spectroscopy Beamline Suite and to continuous, world-leading improvements in synchrotron measurement science and technology.

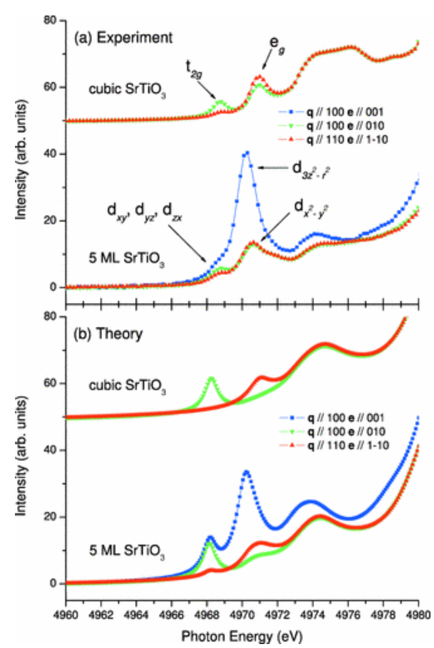
The Beamline for Materials Measurement (BMM) beamline is one of this suite of state-of-the-art, high-throughput, spectroscopy beam-lines at the NSLS-II developed by the National Institute of Standards and Technology (NIST). It is located at port 6BM and was constructed as a partnership between NIST and NSLS-II. With the figure to the right, we offer one example of the many kinds of work that will be enabled by BMM.

Strain engineering via epitaxial thin-film growth is an effective method by which the electronic and mechanical properties of a material may be custom tailored. Industrial applications range from enhanced electron mobility devices to the recently demonstrated realization of ferroelectric memory conjoined with silicon. Strain also alters both the nature

Figure 1



(a) Structure of cubic SrTiO3. (b) Structure of strained $SrTiO_3$ on Si(001) as calculated by DFT. The structure in (b) reveals both antiferrodisplacive and ferroelectric distortions.



(a) Ti K near-edge spectra for cubic $SrTiO_3$ and a 5 monolayer (2 nm) $SrTiO_3$ thin film grown coherently on Si(001).
(b) Theoretical spectra calculated from the structures above.

and temperature of ferromagnetic, ferroelectric, and superconducting phase transitions.

With the BMM beamline, users will combine high-resolution, polarization-dependent, near and extended XAFS measurements with XRD to determine the strain and local structural distortions in strained, epitaxial thin films grown on substrates with dissimilar lattice constants. These measurements will have the goal of improving both first principles and phenomenological theoretical modeling of the unique structures and rich phase diagrams that these materials reveal.

As an example of work performed by NIST scientists in this area, Figure 1 shows the polarization-dependent near edge XAFS spectra from a thin, 5 monolayer (2 nm) $SrTiO_3$ film grown coherently on Si(001) by Motorola. The enhancement of the pre-edge peak is direct evidence for the presence of a room temperature ferroelectric polarization in the film normal to the film/substrate interface, and it is reproduced by ab initio density functional theory calculations of both the atomic and electronic structure of strained $SrTiO_3$ (Figure 1.3). $SrTiO_3$ is normally not ferroelectric at any temperature, so the strain imposed on the film by the substrate has created a directly observable phase of $SrTiO_3$ that does not exist in the bulk material. As a direct result of this research, initial steps towards the industrially relevant goal of high-density, nanoscale, non-volatile ferroelectric memory written and read on a 2 nm $SrTiO_3$ thin film on Si were made.

The spectra shown in Figure 1 were collected at the NIST beamline X23A2 in a glancing incidence, thin-film geometry to limit the X-ray penetration into the Si substrate. As X23A2 had no focusing optics, each spectrum required 2 to 3 days (6 to 9 eight hour shifts) of beam time to obtain high quality EXAFS data. Thin-film research will therefore greatly benefit from the capabilities of BMM.

Beamline Staff

NIST Project Leader	Daniel Fischer	
Lead Beamline Scientist	Bruce Ravel (NIST)	
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	Jean Jordan-Sweet (IBM)	Beamline Scientist
	Johnny Kirkland	Controls Engineer

2 BEAMLINE DESIGN AND COMPONENTS

2.1 Beamline Performance Goals

Table 1 summarizes the design performance of the BMM beamline. A paraboloid collimating mirror (M1), located in the front end at a distance of 13 meters from the source allows the beamline to realize the full energy resolution of the monochromator without excessively restricting the vertical extent of the 20 mm (V) x 5 mm (H) beam aperture. Si(111) crystals are used in the monochromator to provide high flux operations, with a horizontal translation to Si(311) when XANES studies require higher energy resolution. A cylindrical mirror (M2) is mounted on a bender to provide focusing to a spot below 300 μm at any point in the end station. Finally, a flat mirror (M3) is used to provide harmonic rejection and to direct the unfocused beam to the end station. In this way, XAS and XRD experiments may be performed using the focused beam or the large, collimated, unfocused beam. Different aspects of the experimental program will use these different beam delivery modes.

Table 1. Designed Performance of the BMM Beamline

Parameter	Specification/Description
Insertion Device	Three-pole wiggler
Operating Energy Range	4500 eV to 23000 eV
Monochromator	Double crystal monochromator, Si(111) and Si(311), lateral translation between crystal sets
Beam size at sample (FWHM)	5 mm (V) x 20 mm (H) (collimated, unfocused) 300 μm (toroidal focusing mirror)
Flux at sample at 500 mA storage ring current	Si(111): $2x10^{12}$ ph./sec at 10 keV; $6x10^{10}$ ph./sec at 20 keV Si(311): $4x10^{11}$ ph./sec at 10 keV; $1x10^{10}$ ph./sec at 20 keV
Energy resolution	Si(111): $1.3x10^{-4}$ ΔE/E; Si(311): $3x10^{-5}$ ΔE/E
Detector system	Ionization chambers, silicon drift detectors

2.2 Beamline Layout

The estimated performance above is based on the conceptual layout of the BMM beamline shown in Figure 2. The source for the BMM beamline is a three-pole wiggler installed in the 06-BM bend section, 13 meters upstream of the first mirror (the right-most component in Figure 2).

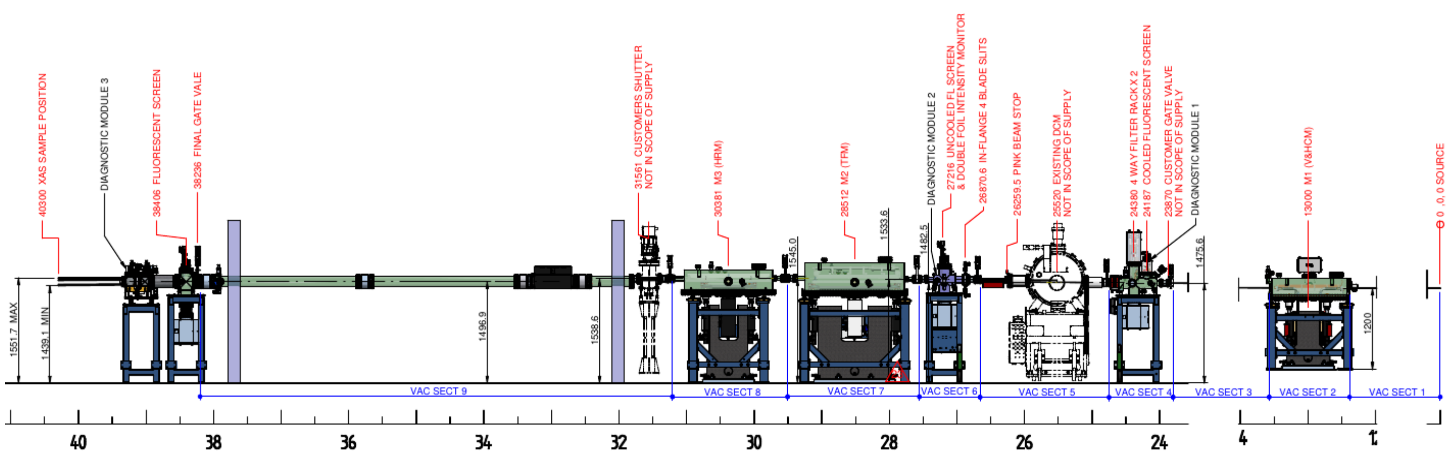

Figure 2. The optical layout of BMM.

Table 2. Main characteristics of the TPW for BMM.

*Note that the maximum power density includes power from the surrounding bend magnets. The power incident upon the beamline optics is only portion that passes through the aperture defined by the front end slits.

	Resolving Power
Photon energy range	<0.01 keV – 25 keV
Device length	0.25 m
Number of periods	0.5
Magnetic gap	28 mm
Peak field	1.14 T
Critical energy	6.8 keV
Maximum total power	320 W
On-axis power density*	260 kW/mrad^2
Beam aperture	2.0 mrad x 0.24 mrad 26 mm x 3 mm at M1

The light from the source is collimated by M1 in the front end. The collimated light is intercepted by the double crystal monochromator. The monochromating elements, Si(111) and Si(311), are accessible by horizontal translation of the monochromator vacuum vessel. Monochromatic light is optionally focused by the toroidal (bent cylinder) M2. Harmonic content is removed by the flat M3, which is also used to direct the beam through the beam pipe and into the end station.

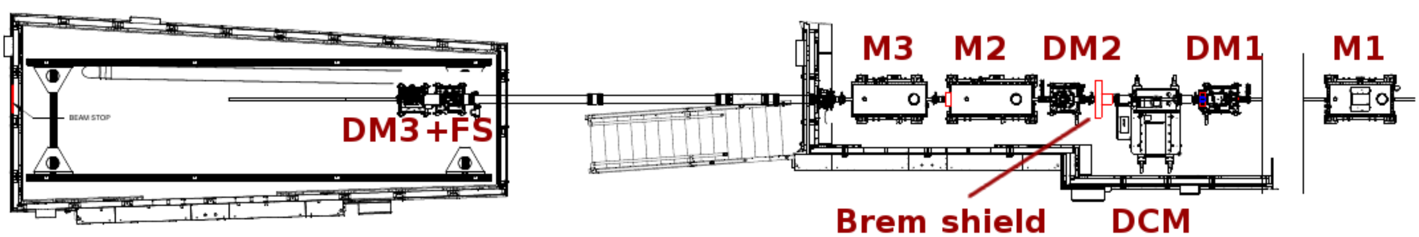

Figure 3. An overview of the components at the BMM beamline.

Key to component acronyms:

- M1 = Mirror 1 (Paraboloid collimating mirror)
- M2 = Mirror 2 (Toroidal focusing mirror)
- M3 = Mirror 3 (Flat harmonic rejection mirror)
- DCM = Double crystal monochromator
- DM1 = Diagnostic module 1 (Fluorescent screen + filters)
- DM2 = Diagnostic module 2 (Fluorescent screen + instrumented slits + intensity monitor)
- DM3 = Diagnostic module 3 (Instrumented slits + intensity monitor + beam position monitor)
- FS = Fluorescent screen

3 BEAMLINE SAFETY

3.1 Radiation Shielding

The design of all radiation shielding (hutches and radiation safety components) will be reviewed to ensure that it follows guidelines to reduce radiation levels external to the beamline enclosures during normal operation to < 0.05 mrem/hr and as low as reasonably achievable. The shielding wall thicknesses follow released shielding guidelines:[1]

Hutch A (FOE, pink beam hutch):

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

Transport section:

- Transport pipe: 19 mm steel + 8 mm lead
- Ion pump coffin: 18 mm steel + 8 mm lead

Hutch B (FOE, monochromatic beam hutch):

- Side walls: 3 mm steel
- Upstream wall: 3 mm steel
- Downstream wall: 6 mm steel
- Roof: 2 mm steel
- Beam stop: 19 mm lead

A beam shutter of the large-aperture, NSLS-II design is installed at the downstream end of the 6-BM-A hutch.

3.2 Radiation Safety Components

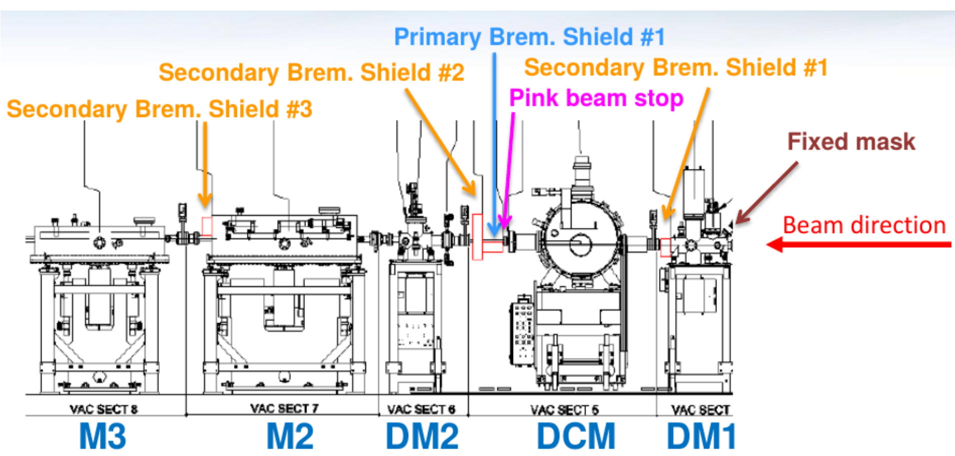

Figure 4. Radiation safety components located in the FOE. Details are given in the text.

These are the components that help contain the synchrotron radiation from the three-pole wiggler source and the gas Bremsstrahlung radiation from the storage ring. The major components are shown in Figure 4 and described in detail below. The location and dimensions of these components are determined based on the ray-tracing analysis and documented in the ray-tracing drawing. Radiation

[1] W.-K Lee et.al., *Guidelines for the NSLS-II Beamline Shielding Design*, (LT-C-ESH-STD-001).

safety components also include devices such as labyrinths and guillotines. A full list of components are identified in the 6-BM radiation safety components checklist (PS-R-XFD-CHK-019).

3.2.1 Heat Load Management (white, pink, and monochromatic beams)

The incident total power at 500 mA ring current from the three-pole wiggler onto the first mirror, located at 13 m from the source and within the front-end, is expected to be 88 W. The first mirror is water-cooled and delivers 70 W of power to the filter assembly in the FOE. The filter assembly contains a sequence of filter thicknesses, appropriate for different energy ranges within the operational energy range of the beamline. The power delivered to the monochromator is between 24 W and 57 W, depending on the filter settings. The power delivered by the monochromatic beam is less than 20 mW.

Mirror 1 is a fixed angle mirror. Once commissioned, it's angle will not change. It is protected by a water-cooled, disaster mask that keeps white beam from striking the side of the mirror optic.

The monochromator is water-cooled. The Si(111) and Si(311) crystals are clamped between two cooled, copper blocks. The filter assembly, also water-cooled, is positioned to provide filtration appropriate to the different operating energy ranges. In all cases, the power delivered to the monochromator is suitable for optimal performance.

In the case where the monochromator first crystal is removed from the path of the pink beam, the pink beam is intercepted by a water-cooled, copper pink beam stop.

It is possible to have the mirrors following the monochromator in a position such that the monochromatic beam can strike the wall of a vessel or a bellows within the FOE. If both mirrors are removed from the path of the monochromatic beam, the beam will strike the housing of the photon shutter. It is also possible to use one of the mirrors to steer the monochromatic beam such that it strikes the wall of the transport pipe. None of these cases poses a heat load management problem due to the very small power (<20mW) contained within the monochromatic beam.

All water cooling circuits are included in the beamline EPS.

3.2.2 Primary Bremsstrahlung Radiation Management

Along with components in the front end for managing primary Bremsstrahlung, there is one primary Bremsstrahlung component in the FOE. The primary Bremsstrahlung stop sits at about 26.5 m, just downstream of the monochromator. See Figure 4.

3.2.3 Secondary (Scattered) Bremsstrahlung Radiation Management

Secondary Bremsstrahlung radiation arises from scattering of primary Bremsstrahlung radiation off the white beam mask, the M1 mirror, and other front end components. Based on NSLS-II guidelines, a series of secondary gas Bremsstrahlung shields have been installed downstream in the FOE. The first is mounted on the downstream flange of the first diagnostic module. The second is on the same stand as the primary Bremsstrahlung stop. The third is mounted on the downstream flange of mirror 2. Diagnostic module 1, the Bremsstrahlung shielding stand, and mirror 2 are all under configuration control. See Figure 4.

3.2.4 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). Examples are shown in Figure 5.

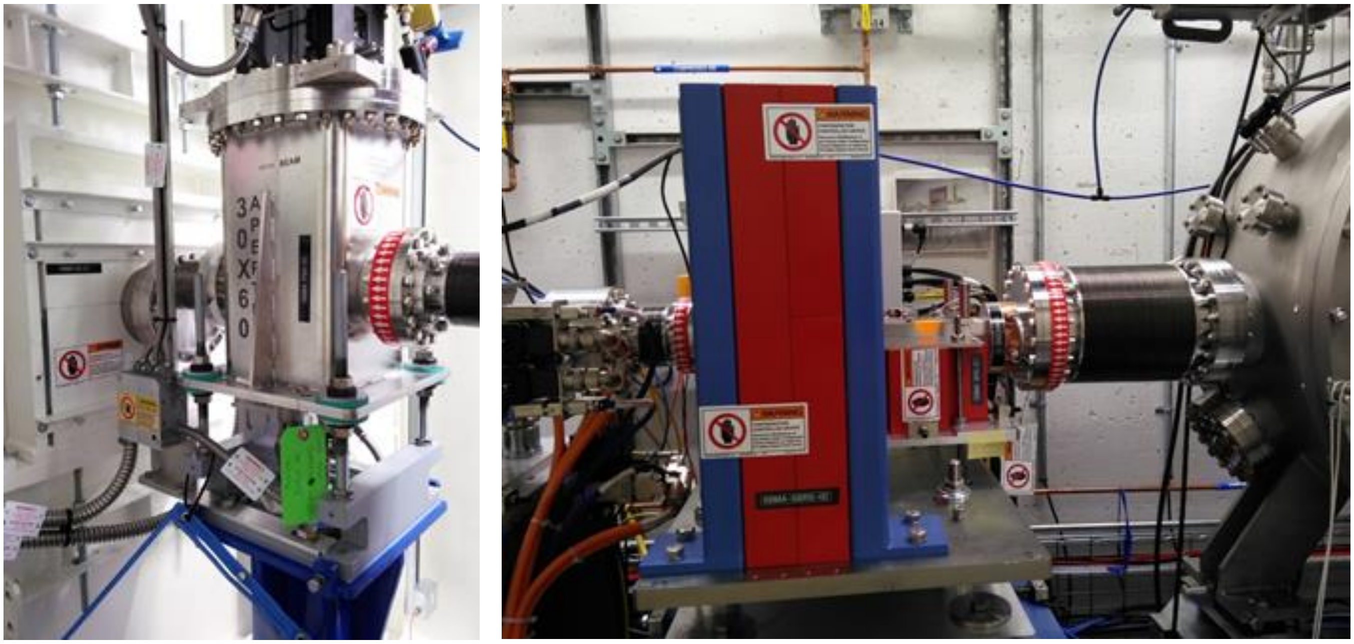

Figure 5. Examples of configuration control on radiation safety components. Left: the photon shutter in the FOE; right: the pink beam stop, the primary Bremsstrahlung shield, and second secondary Bremsstrahlung shield, all located in the FOE.

3.3 Area Radiation Monitor (ARM)

The use of an area radiation monitor (ARM) was considered as part of the top-off safety analysis. It was determined that an ARM is not required at 6-BM. This is documented in NSLSII-TOS-RPT-012, 06-BM (BMM) Top-Off Radiation Safety Analysis and Tech Note #249, 06-BM BMM Beamline Radiation Shielding Analysis – Addendum.

3.5 Personnel Protection System (PPS)

The PPS controls access to the hutches through an interlock system and search and secure procedure, to ensure personnel safety during normal operation of the beamline. The FOE hutch is equipped with a PPS-interlocked user labyrinth to facilitate temporary equipment access during user experiments.

The PPS also monitors critical DI water flow to safety critical components to ensure the safe operation of the beamline. In the event that water flow is lost, the PPS system closes the front end photon shutter to shut off the beam.

3.6 Hazard Identification and Mitigation

Overall, the BMM beamline is similar to other beamlines that are already in operation at NSLS-II. 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 document.

4 INSTRUMENT READINESS

4.1 Survey and alignment

The beamline components are installed according to the specifications and the respective final designs. Installations of the components are verified and documented by 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 deionized water for high heat-load components
- Distribution of process chilled water, water-cooled racks
- Compressed air: for pneumatic valves
- Dry nitrogen gas
- Liquid nitrogen in the B hutch (note that the supply to the B hutch is a part of the SST LN2 distribution system, which is fully approved for operation)
- Ventilation in the hutches
- Network connectivity
- Cabling and piping support structures, for all utilities including EPS and PPS.

4.3 Vacuum System and Pressure Safety

The vacuum pressure for all beamline components is expected to be in the 10^{-9}-10^{-8} mbar range. There is a Be window as a barrier between the beamline vacuum and front end vacuum. This protects the storage ring in case of vacuum failure in the beamline vacuum system.

All vacuum vessels are installed with a pressure relief valve to avoid over pressure when the vessel is vented using dry nitrogen.

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 and the XDAC system.

4.5 Equipment Protection System (EPS)

The EPS at the BMM beamline performs the following functions:

1. Vacuum pressure monitoring and interlock for all the vacuum sections of the beamline.
2. Temperature monitoring and interlock for all non-safety related components, including components exposed to heat load in the white beam mirror system and monochromator.
3. Water flow monitoring and interlock for the cooling of the monochromator, and the fluorescent screen and filter assembly in the first diagnostic module.
4. EPICS interface for components that require I/Os installed on the EPS PLC.