Complex Materials Scattering (CMS) Beamline

IRR Functional Description

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1 INTRODUCTION

1.1 Primary Research Capabilities

The Complex Materials Scattering (CMS) beamline at NSLS II will be dedicated to studies of the bulk and interfacial structures of complex materials. The beamline will provide instrumentation for small- and wide-angle x-ray scattering (SAXS, WAXS) as well as scattering in grazing incidence geometry (GISAXS, GIWAXS).

The over-arching challenge in materials science is the rational design of new materials, where given the required characteristics, the material structure is predicted; and for that particular structure, we can design appropriate constituents and assembly processes. Achieving this goal requires detailed understanding of material assembly at multiple length-scales. The CMS will enhance the pace of exploration by screening materials more rapidly and intelligently. High-throughput techniques have been shown to allow discovery of previously hidden structures and patterns. As such, we anticipate entire new classes of materials may arise from such explorations. Perhaps more importantly, however, by mapping out complex parameter spaces, the CMS will provide fundamental scientific insights in material behavior and assembly processes, thereby paving the way for truly rational material design.

CMS will provide robust x-ray scattering methods to enable structural probing in a wide variety of systems, with emphasis on:

- A highly versatile beamline design, allowing for arbitrary sample environments, in-situ studies, and stimuli-responsive experiments
- A broad *q*-range for measurements, including simultaneous SAXS and WAXS, allowing for the study of hierarchical materials and next-generation nano- or meso- structures
- Microfocusing for performing grazing-incidence (GISAXS/GIWAXS) experiments on surfaces and thin films
- Microfocusing for performing mapping of heterogeneous materials

The ultimate goal of CMS is to provide users with high-throughput and automation capabilities, enabling the efficient exploration of non-trivial phase diagrams and kinetics of materials assembly. These high-throughput capabilities are being envisioned as a future upgrade; day-one designs will provide a fully-functioning x-ray scattering beamline with the capability to smoothly integrate new automation capabilities.

In order to achieve these scientific goals, CMS aims to offer the following capabilities:

1. The ability to measure simultaneous small-angle and wide-angle scattering using area detectors, with variable detector positions to accommodate experimental needs.
2. Routine beam size at the sample position of 200 μm (H) by 100 μm (V), with the ability to modify the beam size to suit user needs. In particular, the ability to access higher-flux configurations with larger horizontal beam sizes (1 mm), and the ability to focus down to 50 μm (H) by 25 μm (V).
3. The ability to operate the instrument with a resolution of $\sigma = 0.0005$ Å^{-1} (equivalent to a divergence of FWHM = 0.2 mrad), while noting that some users may tolerate a relaxed resolution of $\sigma = 0.003$ Å^{-1} (equivalent to a divergence of FWHM = 1.4 mrad).
4. Flux on the order of 10^{10} ph/s to 10^{12} ph/s at the sample position depending on acceptable beam size and divergence.
5. Moderate tunability of x-ray energy (10-17 keV).

1.2 Beamline Staff

Lead Beamline Scientist	Masa Fukuto	
Authorized Beamline Staff	Kevin Yager	CFN Partner User PI Beamline Scientist
	Rick Greene	Mechanical Technician
Beamline Support Staff	Lukas Lienhard	Mechanical Engineer
	Oksana Ivashkevych	Controls Engineer

2 BEAMLINE DESIGN AND COMPONENTS

2.1 Beamline Performance Goals

Table 1 summarizes the designed performance of the CMS beamline. The beam size will be dictated by the needs of each individual experiment. The flux performance is estimated using ray-tracing simulations of the source and beamline optical components; actual flux for each experiment will depend on the configuration of the optical components specific to the experiment.

Table 1: Designed Performance of the CMS Beamline

Parameter	**Specification/Description**
Insertion device:	Three-pole wiggler
Operating energy range:	10 – 17 keV
Monochromator:	Fixed-exit Ru/B_4C multilayers (200 layers, 2.0 nm d-spacing, $\Delta E/E = 1\%$)
Beam size at sample (FWHM):	Horizontal variable from 0.8 to 0.05 mm; vertical variable from 0.3 to 0.025 mm
Flux at sample at 500 mA storage ring current:	10^{10} ~10^{13} photons/s depending on beam size

2.2 Beamline Layout

The estimated performance, above, is based on the conceptual layout of the CMS beamline, shown in Figure 1. The source for the CMS beamline is a three-pole wiggler (3PW) installed at 11-BM. A double multilayer monochromator (DMM) selects the photon energy. The toroidal mirror privides primary horizontal and vertical focusing and is also used for harmonic suppression. The KB mirror, located in the Endstation hutch, provides the capability for secondary focusing; when the KB mirror is used, the primary focus is set to Slit S2, which serves as the secondary source aperture (SSA). When the KB mirror is not used, the primary focus is set to slit set S4, immediately upstream of the sample position. The endstation is equipped with a series of additional beam-defining slits (S1, S3, S5), beam diagnostics, sample stages, and dedicated area detectors to enable SAXS/WAXS and GISAXS/GIWAXS experiments.

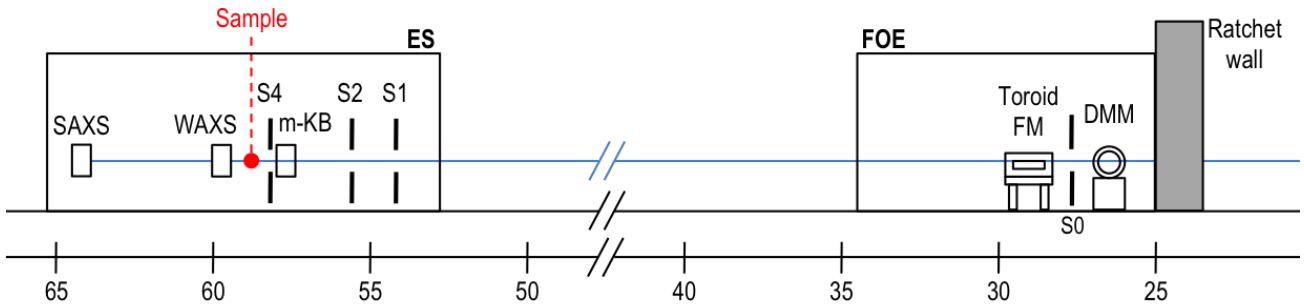

Figure 1: Conceptual Layout of the CMS Beamline

2.2.1 Source

The 3PW source characteristics are summarized in Table 2. The expected flux for the 3PW source at the bandwidth of ΔE/E = 1%, consistent with the DMM installed at CMS, is shown in Figure 2 for the relevant 10-17 keV range. The aperture for the first fixed mask in the CMS Front End has acceptance of 1.5 mrad H x 0.15 mrad V; the corresponding white-beam power and the monochromatic beam flux out of the Front End are estimated to be 60 W and 10^{14} ph/s/1%bw, respectively.

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

[Source: "NSLS-II Source Properties and Floor Layout," April 2010]

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

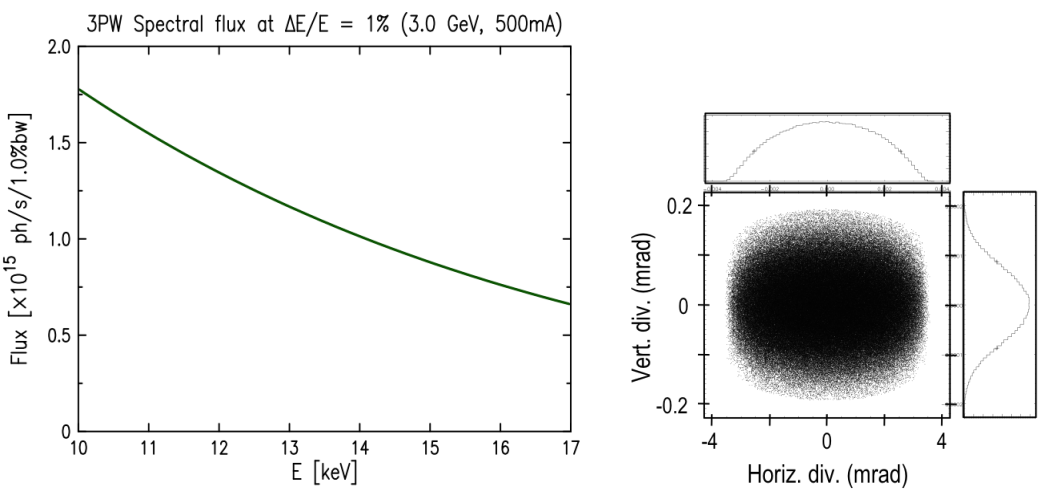

Figure 2: Calculated spectral flux versus photon energy (left) and beam divergence distribution at 13.5 keV (right) for the NSLS-II 3PW source at 3 GeV, 500 mA, and 1% energy bandwidth

2.2.2 Optics and Diagnostics

The overall layout of the CMS beamline is shown below in Figure 3. The first optical enclosure (FOE) houses the DMM and the toroidal focusing mirror. The Endstation hutch houses a series of beam-defining and beam-conditioning slits, the KB mirrors for secondary focusing, the sample/WAXS detector chamber, in-vacuum WAXS area detector, SAXS flightpath, and SAXS area detector.

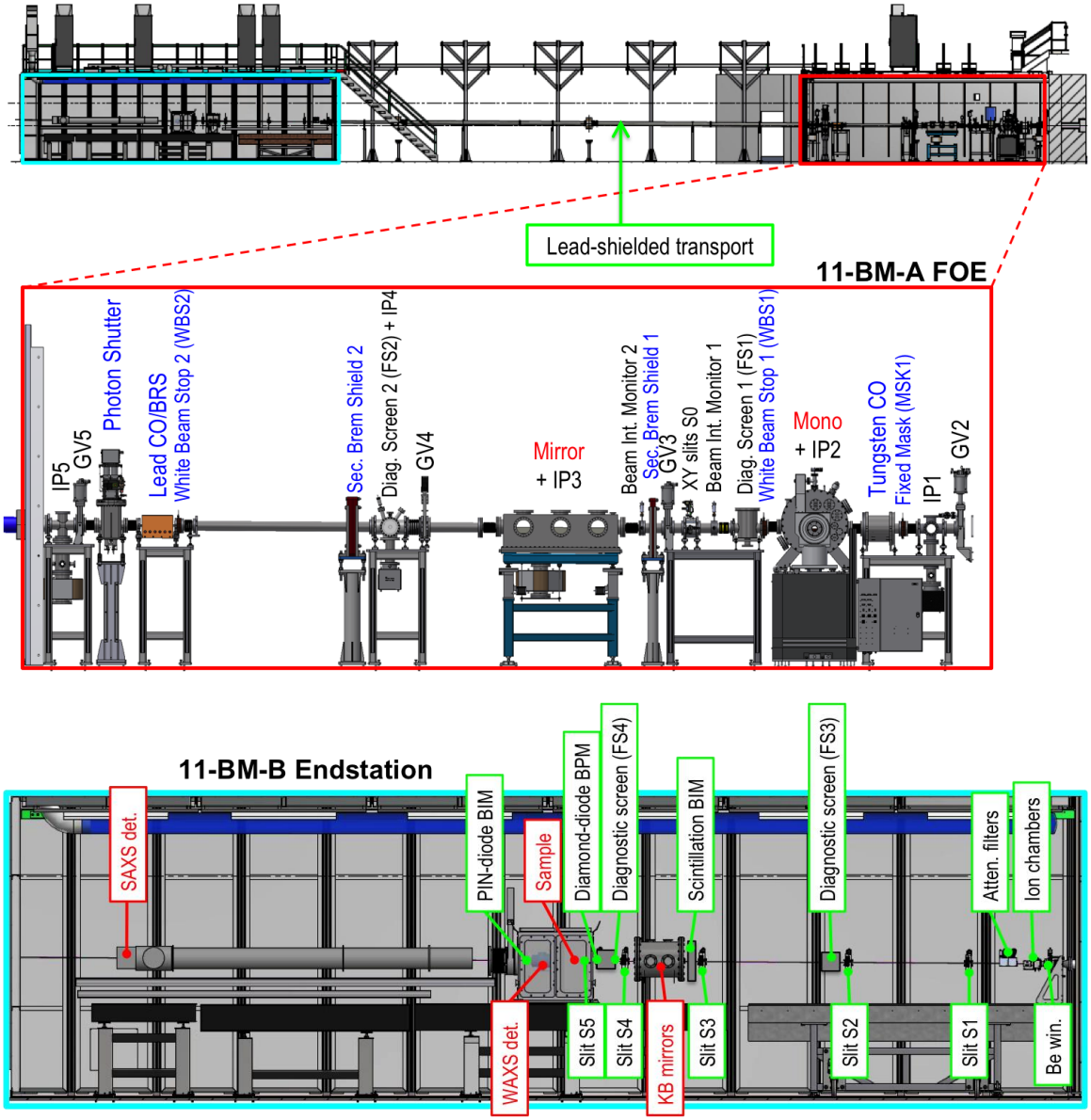

Figure 3: CMS Beamline Component Layout

The CMS beamline has a flexible focusing scheme. Primary focusing is provided by the toroidal mirror in the FOE, while secondary focusing is provided by the KB mirrors in Endstation. When the KB mirror(s) is used to provide secondary focusing, the primary focus is set to Slit S2 in the Endstation, which serves as the SSA. The two main optical configurations to be used for regular operation are:

- <u>SAXS/WAXS mode</u>: Vertical and horizontal primary focusing at Slit S4 in Endstation; KB mirrors are not used.

- GISAXS/GIWAXS mode: Horizontal primary focusing at Slit S4, and vertical primary focusing at Slit S2 as SSA. The vertical KB mirror is used for secondary focusing, while the horizontal KB mirror is not used.

In addition, a third configuration that is anticipated to be used is:

- Microbeam mode: Vertical and horizontal primary focusing at Slit S2 as SSA. Both vertical and horizontal KB mirrors are used for secondary focusing.

In all of the above configurations, Slit S1 is used to control the beam divergence, S3 for diagnostic purposes, and S5 as guard slits to eliminate parasitic upstream-slits scattering at small angles.

For beam diagnostics, the CMS beamline has a set of XY slits in the Front End that also serve as beam position monitors by means of drain current measurements. The FOE has two fluorescence screens for beam visualization and two beam intensity monitors that are based on metal-mesh screens that allows for drain-current measurements. The Endstation has two diagnostic fluorescence screens for beam visualization; a set of ion chambers, a scintillation beam-intensity monitor, and a diamond-diode beam position monitor, located upstream of the sample; and a PIN diode, located downstream of the sample, for sample alignment. The SAXS area detector also serves as an additional tool for beam diagnostics.

3 BEAMLINE SAFETY

3.1 Radiation Shielding

The design of all radiation shieldings (hutches and radiation safety components) for the CMS beamline follows strict guidelines to reduce radiation levels external to the beamline enclosures during normal operation to as low as reasonably achievable. For the area downstream of the downstream wall of the FOE, the maximum dose is < 0.5 mrem/hr, while all other areas are shielded to < 0.05 mrem/hr. The shielding wall thicknesses follow released shielding guidelines:[1]

Hutch A (FOE, white beam hutch):

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

Hutch B (monochromatic beam hutch):

- Lateral and upstream wall: 3 mm steel
- Downstream wall: 6 mm steel
- Roof: 2 mm steel

Shielded beam transport system:

- Pipes, pump enclosures, flange covers: 5 mm lead
- Hutch interfaces: 10 mm lead

A monochromatic beam shutter of the standard NSLS-II design is installed at the downstream end of the FOE. A monochromatic beam stop, welded on the downstream wall of Hutch B, consists of a lead plate of dimensions 0.5 m wide, 0.5 m high, and 12 mm thick.

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

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 3. The radiation safety components in FOE include: a fixed mask (MSK1), a tungsten Bremsstrahlung collimator, two white beam stops (WBS1, WBS2), a lead Bremsstrahlung radiation stop, two secondary Bremsstrahlung radiation shields, the photon shutter, the guillotine at the downstream end of the FOE hutch, the PPS labyrinth on the outboard wall, and the 5 roof labyrinths. The radiation safety components located between the FOE and Endstation hutches include: the hutch interfaces, the lead-shielded beam transport pipes, two lead-shielded flange enclosures, and two lead-shielded ion-pump enclosures. The radiation safety components in Endstation include: the guillotine at the upstream wall, the monochromatic beam stop on the downstream wall, a user labyrinth on the outboard wall, and the 5 roof labyrinths. The locations and dimensions of the tungsten Bremsstrahlung collimator, the two white beam stops, and the lead Bremsstrahlung radiation stop are determined based on the raytracing analysis and are documented in the ray tracing drawing (PD-CMS-RAYT-0001). The 11-BM radiation safety component checklist (PS-R-XFD-CHK-014) is the complete list of all components.

3.2.1 Primary Bremsstrahlung Management

There are two components for primary bremsstrahlung shielding. The tungsten Bremsstrahlung collimator is located close to the upstream end of FOE, immediately downstream of the fixed mask and immediately upstream of the monochromator; the tungsten collimator reduces the maximum fan of the primary Bremsstrahlung radiation. The lead Bremsstrahlung stop is located close to the downstream end of FOE, immediately upstream of the photon shutter. An aperture in the Bremsstrahlung stop, which is sufficiently distant from the extremal rays of the primary bremsstrahlung, permits the monochromatic beam to pass through.

3.2.2 Secondary Bremsstrahlung Management

Secondary Bremsstrahlung radiation arises from scattering of primary bremsstrahlung radiation off MSK1, monochromator, WBS1, toroidal focusing mirror, and WBS2. Based on NSLS-II guidelines, two secondary Bremsstrahlung shields are installed; one between WBS1 and the toroidal mirror, and the other between the toroidal mirror and WBS2. FLUKA simulations were carried out to verify that the shielding satisfies the requirements described in Section 3.1 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 level in the area is actively monitored through an ARM installed on the narrow downstream wall of the FOE bump-out section (Figure 4). The ARM has been certified in accordance with the procedure PS-C-ASD-PRC-008, *NSLS-II Area Radiation Monitor PPS Test.*

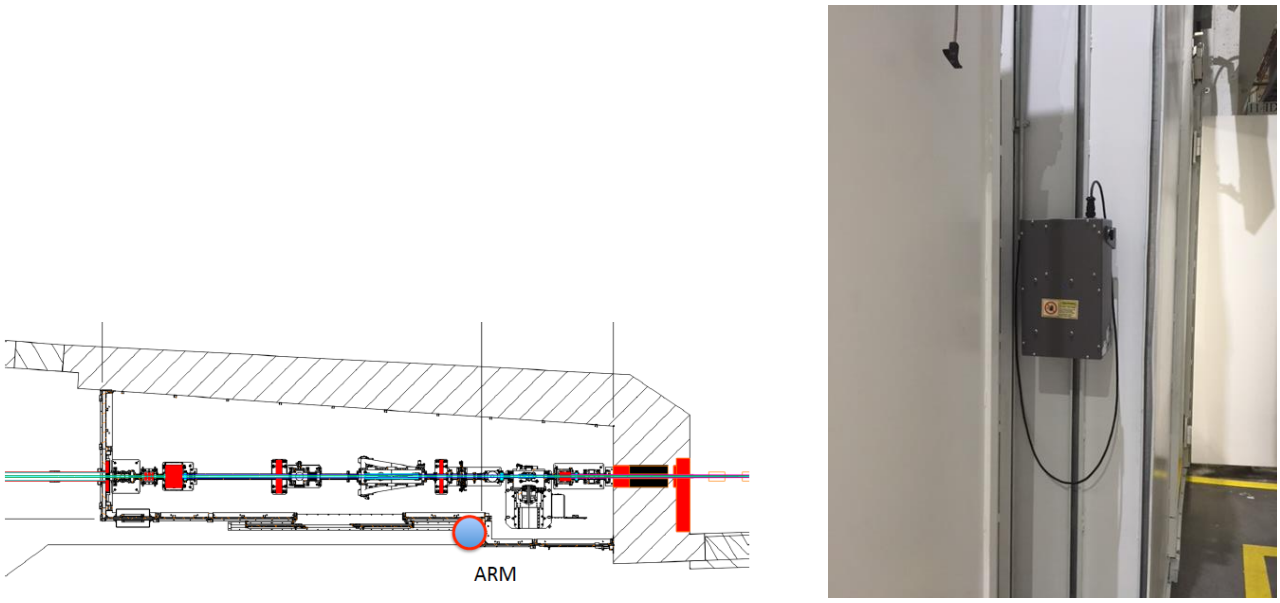

Figure 4: ARM Installed on the Beamline

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. Hutch B (Endstation) is equipped with one PPS-interlocked user labyrinth to facilitate temporary equipment access during user experiments.

3.5 Hazard Identification and Mitigation

The CMS 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 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 to the roof of the FOE for the monochromator heat exchanger, to the roof of Hutch B for detector chillers, to inside Hutches B for water-cooled roots pumps, and to all water-cooled racks on the FOE and Hutch-B roofs

- 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 for FOE components (downstream of GV2) is expected to be 10^{-7} mbar or better. There is no physical barrier between the beamline vacuum and Front End vacuum. The beamline vacuum is separated from the accelerator vacuum by a beryllium window located in the Front End. The beamline vacuum, downstream of GV2, consists of four sections (Figure 3). Section I, containing MSK1, tungsten Bremsstrahlung collimator, and the monochromator, has two ion pumps, one upstream of MSK1 to achieve ultrahigh vacuum immediately downstream of GV2 and the other on the monochromator tank. Section II, containing the toroidal mirror, has one ion pump on the mirror tank. Section III, containing the diagnostic screen #2, WBS2, the lead Bremsstrahlung stop, and the photon shutter, has one ion pump on the diagnostic cross. Section IV, corresponding to the beam transport section between the FOE and Hutch B, has three ion pumps. The final beryllium window, which has been pressure-tested to 1.0 atm, terminates Section IV inside, and 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.

Two water-cooled dry pumps maintain the vacuum for all Endstation components in Hutch B, to a pressure of $< 10^{-1}$ mbar. The FOE vacuum is separated from the Endstation vacuum by the beryllium window at the upstream end of Hutch B, a GN2/air gap, and a Kapton window (circular, 16 mm diameter, 25 um thick) at the upstream end of the Endstation vacuum section. The Endstation vacuum is terminated with the final Kapton window (circular, 200 mm diameter, 127 um thick) at the downstream end of the SAXS flight path. The design of the terminal Kapton windows used for the Endstation vacuum have been reviewed. Pressure tests shall be performed on the final Kapton window that terminates the SAXS flight path. The Endstation vacuum will be vented to air and presents no overpressure condition.

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 System Studio (CSS) screens have been prepared to access the motors on the components.

4.5 Equipment Protection System (EPS)

The EPS at the CMS 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 system, the toroidal mirror system, and water-cooled electronic racks
- Water flow monitoring and interlock for cooling of the monochromator, the FOE slits, the first diagnostic fluorescence screen, and the WAXS detector.
- 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 dry pumps in the experimental station, actuation of attenuation filters and diagnostic screens in the experimental station, and monitoring of experimental shutter state.