

# DESIGN, CONSTRUCTION AND COMMISSIONING OF TWO HIGHLY INTEGRATED EXPERIMENTAL STATIONS FOR MICRO-FOCUSING MACROMOLECULAR CRYSTALLOGRAPHY (MX) BEAMLINES AT NSLS-II

Dileep K. Bhogadi <sup>a)</sup>, Martin R. Fuchs, Jean Jakoncic, Babak Andi, Stuart Myers, Bruno Martins, Lonny E. Berman, Dieter Schneider, Robert M. Sweet, Dieter K. Schneider and Sean McSweeney

*National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973, USA*

<sup>a)</sup> dbhogadi@bnl.gov

## Abstract

We present the final engineering design and first commissioning results of two highly integrated experimental stations for the micro-focusing (FMX) and the highly automated (AMX) MX beamlines at the NSLS-II. These beamlines will support a broad range of biomedical structure determination methods from serial crystallography on micron-sized crystals, to structure determination of complexes in large unit cells. These experimental stations are completely designed and fabricated in-house to meet challenging requirements resulting from the small beam size of 1  $\mu\text{m}$  and the extremely short working distance of only 190 mm from the beam exit window to the FMX focal spot.

The beam conditioning unit contains, within 140 mm, a beam position monitor, an attenuator, primary slits, an intensity monitor, a sub-millisecond shutter, and secondary slits. The diffractometers consist of an interchangeable high precision air bearing based main goniometer and a secondary goniometer for crystallization plates, both with a SOC of 100 nm on horizontal axes, an on-axis microscope with a customized reflective optics, x-ray fluorescence detector and dynamic beam shaping slits. Both these robotic end stations are integrated in a compact space on a granite machine bed with high modularity for future upgrades and extensions. Novel automation concepts are being implemented to increase the throughput of the cryogenic samples.

## INTRODUCTION

The Frontier Microfocusing MX beamline (FMX) and the Highly Automated MX beamline (AMX), together with their companion beamline for Life science X-ray scattering (LIX), form a suite of Advanced Beamlines for Biological Investigations with X-rays (ABBIX) (1,2). Both the layouts and the beam properties of these new beamlines pose very specific instrumentation challenges.

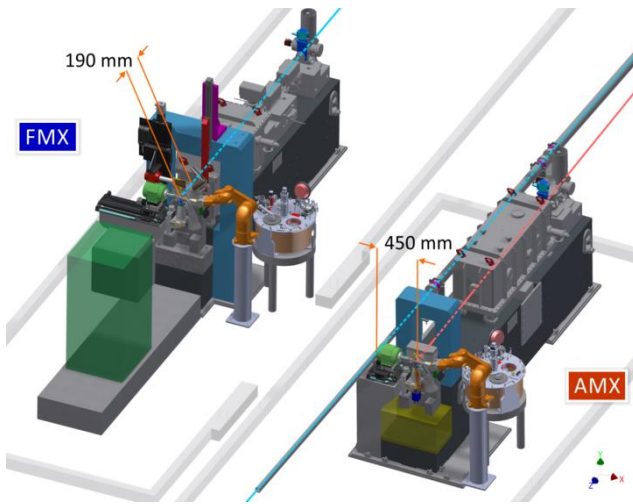
These beamlines are designed to have high vibrational stability of the x-ray optics, while the canted undulator arrangement greatly limits the space for the optical components and the experimental stations (**Error! Reference source not found.**).



Figure 1: Layout of the FMX and AMX photon delivery systems in sector 17-ID

In the design of the experimental stations, two central restrictions had to be overcome which result from the beamlines' layout (Figure ). At AMX, the lateral distance between adjacent FMX beam pipe and AMX focal spot is less than 450mm which greatly restricts the space for the main goniometer. At FMX, the short working distance from the mirror tank's downstream face to the sample leaves a total distance of 190 mm along the beam. Within this space, the central components of the end station like Beam Conditioning Unit (BCU), the on axis microscope, and a retractable scatter guard tube have to be accommodated.

The BCU contains – from upstream to downstream – an x-ray beam position monitor (Sydor Instruments SI-DBPM-M405) (4), an attenuator unit, an HV slit pair, an intensity monitor, a photon shutter and a pair of so-called dynamic slits (Figure 1). This order of devices is deliberate – the DBPM requires the full monochromatic flux for highest position resolution. The slit pair and the intensity monitor upstream of the shutter enable an intensity-based choice of slit openings without exposing the sample. The final dynamic slit pair is placed as close as possible to the sample to allow for beam size changes during crystallographic data collection. By dynamically matching the beam size to non-isotropic crystal shapes, scattering from the buffer solution can be minimized.



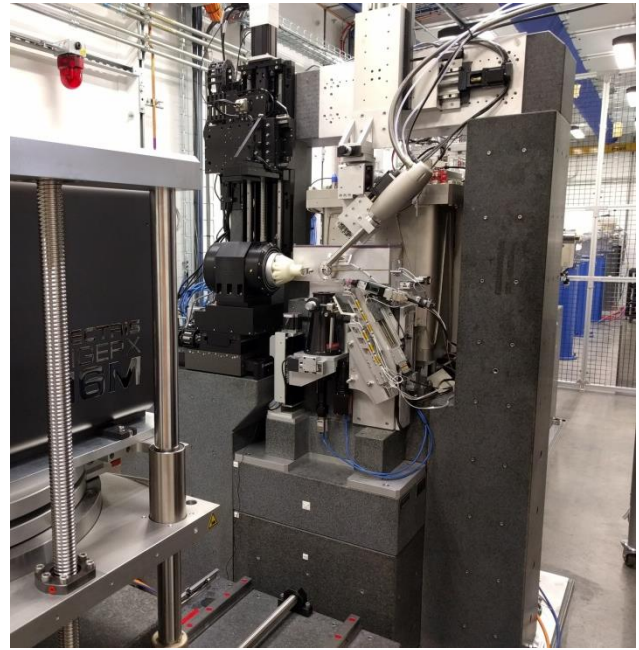
**Figure 2. Layout of the FMX (left) and AMX (right) experimental stations.**



**Figure 3a: Construction update of the AMX End Station**

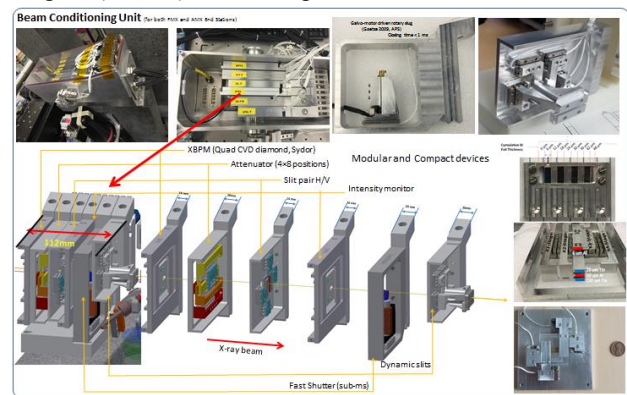
Several constructional details are key to fitting all devices including their enclosure within 140 mm along the beam. The attenuator is constructed as a slim stack of 4 sliders with 8 positions. In order to provide wide dynamic FMX's energy of 5-30 keV, a layered thin Al and Sn foils, an octal system of increasing thickness is used. Compact stick-slip piezo positioners with nm resolution (SmarAct SLC-17XX) and integrated encoders were used for their compactness and precision throughout the BCU devices. The fast shutter is derived from a highly compact design developed at the APS (5) which uses a galvanometer-motor (Cambridge Technologies 6220H) to rotate two tungsten blades bracketing a slit to pass the beam. This design provides opening times of a few 100  $\mu$ s, which is required to limit the sample exposure before the actual

data collection in high-flux experiments. To minimize the dimensions of the BCU enclosure, it will be filled with He to avoid the additional bulk of a vacuum tank.



**Figure 3b: Construction update of the FMX End Station.**

Both beamlines will feature a horizontal high-precision goniometer axis (Nelson Air SP150) with newly developed light weight extension arm and a stick-slip piezo XY centering stage (SmarAct SLC-1720). The total spindle runout 50 mm above the rotation table has been determined to  $\pm 20$  nm. Based on experiences with previous centering tables utilizing the SLC-1720 SmarAct centering tables (6), we expect to achieve a sphere of confusion of 100 nm peak-peak at the sample position through implementation of active runout-corrections using a lookup table of the repeatable errors. To locate the presence of a sample mounted on the goniometer, a smart magnet (Arinax) will be implemented.



**Figure 1. Beam Conditioning Unit (BCU). Left: Its six subcomponents are mounted on a rail using a single screw. Right: Exploded view of BCU components. (1) X-ray beam position monitor, (2) attenuator (3) slits (4) intensity monitor (5) fast shutter (6) dynamic slits**

In addition to classical single-axis crystallography, we expect to support a multitude of new sample delivery methods. A secondary goniometer has been designed for FMX and made provisions at AMX to flexibly support data collection from crystallization plates and to enable the development of new instrumentation such as customized plates or jets, without compromising the precision of the main goniometer.

The on-axis microscope is based on a design using a catadioptric telescope in a Maksutov Cassegrain configuration (Questar QM100) (7). The light is deflected downward by a drilled in-house designed flexural mirror deflection stage into the objective with a custom designed housing for a maximum numerical aperture of 0.185 at a 100 mm working distance. Due to the high UV transmission of the reflective optics, sample location using UV-excited intrinsic protein fluorescence can be employed to support crystal location (8).

The Beam diagnostics are being implemented and tested to dynamically shape the beam using wire EDM based pin collimator and scintillator/diode mounting on a retractable piezo stages. Overall integration of these endstation with compact devices mounted around the sample environment; pose a new challenge for sample delivery and handling methods. For this purpose, a six axis robotic arm and 24 puck sample dewar are used in both FMX and AMX end stations using novel methods. Novel automation methods are being developed to increase the successful throughput of the cryogenic samples. Figure 3a, 3b shows the current development and construction status of AMX and FMX experimental stations at sector 17ID at NSLS-II facility.

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