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ForMAX: A BEAMLINE FOR MULTI-SCALE AND MULTI-MODAL STRUCTURAL CHARACTERISATION OF HIERARCHICAL MATERIALS

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Abstract

ForMAX is an advanced beamline at MAX IV Laboratory, enabling multi-scale structural characterisation of hierarchical materials from nm to mm length scales with high temporal resolution. It combines full-field microtomography with small- and wide-angle x-ray scattering (SWAXS) techniques, operating at 8-25 keV and providing a variable beam size. The beamline supports SWAXS, scanning SWAXS imaging, absorption contrast tomography, propagation-based phase contrast tomography, and fast tomography. The experimental station is a versatile in-house design, tailored for various sample environments, allowing seamless integration of multiple techniques in the same experiment. The end station features a nine-meter-long evacuated flight tube with a motorized small-angle x-ray scattering (SAXS) detector trolley. Additionally, a granite gantry enables independent movement of the tomography microscope and custom-designed wide-angle x-ray (WAXS) detector. These features facilitate efficient switching and sequential combination of techniques. With commissioning completed in 2022, ForMAX End Station has demonstrated excellent performance and reliability in numerous high-quality experiments.

INTRODUCTION

Both natural and man-made materials often possess a hierarchical nature, with distinct structures evident across various length scales. Understanding the relationship between structure and function in these materials necessitates characterizing the structure across these scales, coupled with sufficient temporal resolution to observe in-situ processes. The ForMAX instrument efficiently addresses this challenge by combining two complementary techniques: full-field tomographic imaging covering µm to mm scales and SWAXS targeting nm scales.

The primary technical obstacle in integrating full-field tomography with SAXS arises from spatial limitations behind the sample. In full-field tomography, one observes the x-ray beam transmitted through the sample in a forward direction. In contrast, SAXS captures the x-ray beam scattered at small angles, $\leq 3^\circ$, essentially in a near-forward direction. At ForMAX, the innovative strategy is to conduct sequential tomography and SWAXS experiments. This is facilitated by a motorized detector gantry, enabling swift translation of the tomography microscope (and the WAXS detector) into and out of the x-ray beam. This design promotes a rapid and efficient transition between experimental modes.

In the following conference paper, we provide an in-depth overview of the ForMAX beamline's design. Table 1 list the main components of the beamline and their distance from source.

Table 1: Main ForMAX Components and Their Distance From Source

ForMAX Components	Distance from source (m)
Undulator	0
Front end movable mask	19.5
White-beam slits	23.9
Double multilayer monochromator	25.0
Double crystal monochromator	27.0
Vertically focusing mirror	30.2
Horizontally focusing mirror	31.0
Monochromatic slits	28.1, 32.3, 36.3, 41.5 - 41.8
Diamond window	35.8
Attenuator system	35.9
Fast shutter	36.1
X-ray prism lens	36.6
Compound refractive lenses	40.5
Experimental table	42.0
Full-field microscope	42.0 – 42.3
WAXS detector	42.1
SAXS detector	42.9-49.5

Throughout this article, we adhere to MAX IV's coordinate system: the lateral x-axis (outboard direction from the ring), the vertical y-axis (upward direction), and the longitudinal z-axis (downstream direction from the source). The direction of each rotation around the Cartesian axes (Rx, Ry, and Rz) adheres to the right-hand rule.

OPTICS

The primary optics of ForMAX comprises a double crystal monochromator (FMB Oxford), a double multilayer monochromator (Axilon), dynamically bendable vertical and horizontal focusing mirrors in Kirkpatrick-Baez (KB) geometry (IRELEC), a photon shutter (Axilon), and four diagnostic modules (FMB Oxford). These modules contain a fixed mask, a high-band-pass diamond filter for heat-load management, a white-beam stop, bremsstrahlung collimators, slits, beam viewers, and beam intensity monitors. This section explores the monochromators and mirrors. For an overview of the main components, see Fig. 1.

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ForMAX can operate using either a double crystal monochromator (DCM) or a double multilayer monochromator (MLM) based on experimental requirements.

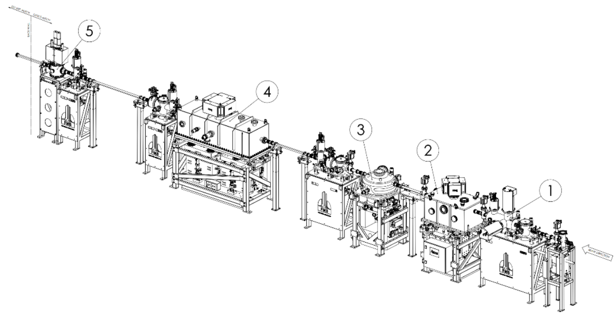

Figure 1: Main components of the optics: (1) white-beam slits, (2) MLM, (3) DCM, (4) mirrors, and (5) photon shutter.

The Si (111) DCM, which deflects horizontally, is situated 27 m from the source. Its compact and rigid design, assisted by a minor horizontal offset between the crystals, ensures superior stability [1]. The upstream crystal is anchored directly onto the Bragg goniometer (Ry) without additional motorized axes. However, the second crystal possesses motorized adjustments for pitch (Ry), roll (Rz), and perpendicular motion. The monochromator also features motorized lateral (x) and vertical (y) translations. Both crystals employ side cooling, achieved by clamping them to liquid-nitrogen-cooled Cu blocks.

The horizontally deflecting MLM is positioned 25 m from the source. Primarily intended for full-field imaging experiments demanding high temporal resolution, it may also be employed in select, photon-intensive scattering experiments. The multilayer mirrors on both units use flat Si (100) substrates, overlaid with 200 layers of W/B4C and 250 layers of Ru/B4C stripes. The monochromator's Bragg rotation (Ry), the upstream mirror's fine roll (Rz), and the downstream mirror's fine pitch (Ry) are all facilitated by linear actuators and unique flexure setups. Given the monochromator's extensive angular range, a longitudinal (z) translation of the downstream multilayer assembly is essential. Additional motorized motions incorporate the monochromator's lateral (x) and vertical (y) translations, as well as the perpendicular movement of the downstream multilayer assembly. Multilayer mirrors are adequately cooled using braids from water-cooled Galinstan baths.

The mirror system houses vertically (VFM) and horizontally (HFM) focusing mirrors in KB geometry within a singular vacuum chamber. Each operates at a consistent incidence angle of 3 mrad. These mirrors serve dual functions: providing harmonic rejection and focusing capabilities. To accommodate the beamline's broad energy range, each mirror possesses distinct stripes of Si, Rh, and Pt. Furthermore, each mirror can be adjusted to bend between approximately 5 and 100 km, enabling versatile focusing, collimation, or non-focusing operations. Each mirror is equipped with a few stiff, motorized axes, including lateral (x) and vertical (y) translation stages. Additionally, pitch rotation (Rx for VFM, Ry for HFM) employs a precision actuator and flexure components. The HFM further incorporates a motorized roll rotation (Rx) using a similar high-resolution actuator and flexure components.

EXPERIMENTAL STATION

As illustrated in Fig. 2, the primary components of the experimental station include two beam conditioning units (BCUs), an experimental table, a detector gantry, and a SAXS flight tube, all of which have been custom-designed at MAX IV [2]. Given the distinct—and at times conflicting—technical prerequisites of SWAXS and full-field tomography, we placed significant emphasis on seamlessly integrating these components into a singular instrument. The modular design of the experimental station, further detailed below, necessitated the integration of a dedicated PLC system to guarantee safe operation.

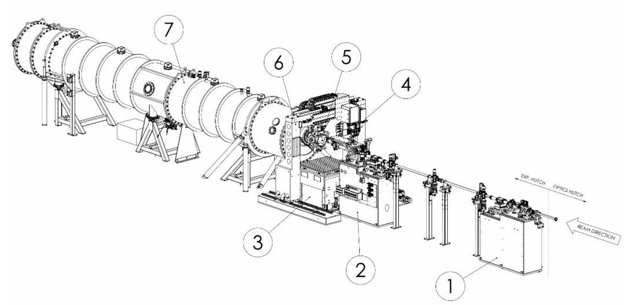

Figure 2: Main components of the experimental station: (1) BCU I, (2) BCU II, (3) experimental table, (4) tomography microscope, (5) WAXS detector, (6) detector gantry, and (7) flight tube.

Beam Conditioning Units

The experimental station features two beam conditioning units, BCU I and BCU II, positioned upstream of the experimental table. They adjust beam characteristics like size, microfocus, and attenuation based on the experiment.

BCU I, located 5 meters upstream of the sample table, has a diamond vacuum window, attenuator system, fast shutter, and slits. An upcoming addition is a motorized overfocusing x-ray prism lens for beam expansion during tomography.

BCU II, directly before the sample table, houses polymeric compound refractive lenses (CRLs) for SWAXS microfocusing, a beam diagnostic module with three intensity monitors, a YAG crystal screen, and slits. A telescopic vacuum tube minimizes the x-ray beam's air path.

Experimental Table

Located 42 meters from the source, the experimental table serves as a platform for equipment essential to SWAXS and tomography studies. With its 800 x 800 mm surface, it allows for adjustments in vertical (±105 mm), lateral (±100 mm), and pitch (±10 mrad) orientations of the sample environment. Its design, mirroring the "skin" concept from ALBA Synchrotron, ensures both stability and high-resolution performance [3].

Constructed with a granite base secured to the floor, the table features two movable lateral plates attached to the

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base. These plates, driven by ball screws and stepper motors, support a top plate connected by flexure hinges. An added lateral motion stage is positioned atop this structure. The table's carrying capacity is 200 kg.

Detector Gantry

Positioned 42 meters from the source, the granite detector gantry surrounds the sample location. Both the tomography microscope (Optique Peter) and the WAXS detector (X-Spectrum Lambda 3M), tailored specifically for the ForMAX beamline though commercially available, will be secured to the gantry. Two distinct linear stages, set on the gantry lintel, facilitate the lateral movement (≈670 mm) of both the microscope and detector, enabling them to easily enter or exit the beam's path. Additionally, two separate vertical stages allow for the vertical adjustment of both the microscope and the detector (±22.5 mm and ±7 mm, respectively). Each stage is constructed with ball screws and linear guides, driven by stepper motors. The entire assembly is mounted on motorized rails on the floor.

The motorized floor rails, extending from 41 to 42.5 meters from the source, enable phase-contrast imaging and accommodate large sample setups on the experimental table. The two lateral stages allow users to quickly switch between multiple techniques in a single experiment. Additionally, the design grants clear access to the sample table from the end-station's side.

Vibrations at the microscope's tip were measured using an LDV vibrometer, confirming rms amplitudes of less than 20 nm in both lateral and longitudinal directions [4].

Flight Tube

The flight tube links the sample to the SAXS detector (Dectris EIGER2 X 4M) and minimizes scattering and beam absorption. It's an eight-meter-long, one-meter-diameter stationary vacuum chamber divided into five sections. The SAXS detector is housed inside [5], on motorized rails, allowing for sample-to-detector distance adjustments between approximately 800 and 7600 mm.

The rail system, powered by an external servo motor, uses a belt drive. A girder, mechanically isolated from the vacuum chamber by bellows, supports the setup, ensuring stability from potential chamber vibrations during pumping. The SAXS detector has vertical and lateral adjustments, and a tungsten central beamstop with a GaAs diode is attached to its trolley, monitoring the x-ray beam's flux.

Safety measures include the MAX IV Personal Safety Systems, a rigorous search procedure, and emergency pull-wire switches to ensure no unintended personnel remain inside during operations.

Sample Manipulation

Given the diverse experimental techniques offered by ForMAX, each possessing unique sample manipulation requirements, ForMAX incorporates three distinct stage stacks for sample manipulation.

For standard SWAXS experiments, a high-load five-axis assembly (Huber). It comprises motorized pitch Rx (±13°), roll Rz (±12°), lateral x (±25 mm), longitudinal z (±25 mm), and vertical y (±20 mm) axes.

For scanning SWAXS, another assembly (Huber) is used. Its base has motorized lateral x (±25 mm), vertical y (variable ±10 or ±45 mm), and longitudinal z (±25 mm) axes. A yaw Ry axis and a wide-range pitch axis Rx (±45°) enable SWAXS tomographic imaging. A manual goniometer head assists in the precise alignment of the sample.

For tomography experiments, a five-axis assembly (Lab Motion) is employed. From the bottom up, this assembly consists of a motorized longitudinal z axis (with approximately 380 mm range) suitable for propagation-based phase-contrast imaging, a vertical y axis (±20 mm) designed for helical imaging, a tomographic yaw axis Ry paired with a rotary union that accommodates a fluid and an electrical slip ring, and horizontal xz axes (±5 mm each) for precise sample alignment. This electrical slip ring also features nine spare wires, allowing the integration of sample environments. The assembly's modular nature often leads to its operation without the vertical axis and the rotary union.

EXPERIMENTAL MODES

The versatile motions of the detector gantry facilitate a diverse range of experimental configurations. The design allows for the independent movement of the WAXS detector and the full-field microscope, providing a variety of experimental modes.

SAXS

In the SAXS setup (see Fig. 3), both the WAXS detector and the full-field microscope are moved out of the x-ray beam's path. An evacuated nose cone is also attached to the flight tube, reducing the air path downstream of the sample.

Figure 3: SAXS setup of the endstation.

SWAXS

For the SWAXS setup (as shown in Fig. 4), the x-ray beam is directed at the WAXS detector. Meanwhile, the SAXS signal (along with the unscattered beam) travels through the WAXS detector's central hole, eventually reaching the SAXS detector (and the central beam stop). During this mode, the full-field microscope is translated out of the x-ray beam's pathway.

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Figure 4: SWAXS setup of the end station.

Full-Field Tomography

In this mode, the WAXS detector is moved out of the x-ray beam's trajectory (see Fig. 5). As a safety precaution, a gate valve at the entrance of the flight tube is closed, ensuring that the SAXS detector isn't exposed to x-rays.

Figure 5: Full-field tomography setup of the endstation.

Sequential SWAXS and Full-Field Tomography

For the combined SWAXS and full-field tomography setup, both the SAXS and WAXS detectors are aligned with the x-ray beam. The full-field microscope is then vertically moved in and out of the x-ray beam path for either full-field imaging or scattering modes, respectively (as depicted in Fig. 6). This vertical repositioning of the microscope takes no more than 15 seconds.

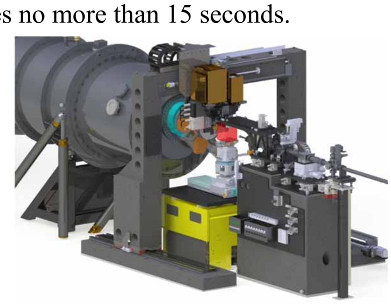

Figure 6: Sequential SWAXS and full-field tomography setup of the endstation.

Others

The spatial design of the ForMAX endstation, particularly in the areas surrounding the sample table and between the BCUs, can accommodate temporary configurations for unique experimental techniques. This includes niche methods like Ultra-fast Full-field Tomography and Multi-projection Imaging [6] among others.

Additionally, the beamline will provide dedicated sample environments, facilitating multiscale structural characterisation during complex rheological or mechanical tests, all under precisely controlled temperature and humidity conditions.

CONCLUSIONS

We have introduced the ForMAX beamline, highlighting its innovative design for multi-scale structural characterisation by combining full-field tomography and SWAXS. The adaptability of the experimental table and flight tube designs enables state-of-the-art SWAXS experiments across a variety of sample environments. The detector gantry's design supports multiple experimental modes, ranging from conventional SAXS to SWAXS and full-field tomography. The integration of full-field tomography and SWAXS experiments within the same experiment is facilitated by swift and fast transitions between setups. After completing its commissioning in 2022, the ForMAX End Station commenced regular user operations in 2023, consistently demonstrating outstanding performance and reliability in numerous high-quality experiments.

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