

MINIMIZING EXPERIMENTAL SETUP TIME AND EFFORT AT APS BEAMLINE 1-ID THROUGH INSTRUMENTATION DESIGN

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Abstract

Sector 1-ID at the APS accommodates a number of different experimental techniques in the same spatial envelope of the E-hutch end station. These include high-energy small and wide angle X-ray scattering (SAXS and WAXS), high-energy diffraction microscopy (HEDM, both near and far field modes) and high-energy X-ray tomography. These techniques are frequently combined to allow the users to obtain multimodal data, often attaining 1 μm spatial resolution and $<0.05^\circ$ angular resolution. Furthermore, these techniques are utilized while the sample is thermo-mechanically loaded to mimic real operating conditions. The instrumentation required for each of these techniques and environments has been designed and configured in a modular way with a focus on stability and repeatability between changeovers. This approach allows the end station to be more versatile, capable of collecting multi-modal data *in-situ* while reducing time and effort typically required for set up and alignment, resulting in more efficient beam time use. Key instrumentation design features and layout of the end station are presented.

BEAMLINE 1-ID

Focus

The 1-ID beamline at the APS delivers high brilliance, monochromatic ($\Delta E/E \approx 10^{-3}$) high-energy X-rays (>40 keV) resulting in exceptional penetrating power and spatial-angular resolution. The Materials Physics and Engineering (MPE) group operates the beamline and takes advantage of this combination to investigate a wide variety of polycrystalline material systems *in situ* / *in operando*, such as structural alloys, chemical compounds used in batteries, and bones to name a few.

Techniques

User experiments are typically performed at the 1-ID-E end station. Three primary techniques are supported at the 1-ID-E end station:

Small and wide angle X-ray scattering (SAXS/WAXS) While these two techniques are typically supported individually, the unique WAXS detector

array at the 1-ID-E end station allows the combination of the two techniques. WAXS provides information such as phase volume fraction, preferred crystallographic orientation, and aggregate-average strain. SAXS provides information such as precipitate or void size distribution. The combined technique is capable of interrogating a wide range of length scales and is particularly useful in investigating polycrystalline materials with small grains (1 μm or less) and/or large prior deformations.

High-energy diffraction microscopy (HEDM) This novel technique is capable of non-destructively characterizing an individual constituent grain in a polycrystalline aggregate. Several variants of this technique exist; depending on the location of the detector, the morphologies and intragranular crystallographic orientation information of the grains (near-field) or the average crystallographic orientations, positions, and stress tensors associated with the individual grains (far-field) can be obtained.

Tomography While tomography is a widely available technique, the ability to combine SAXS/WAXS or HEDM with high resolution (1 μm) phase or absorption contrast tomography is quite unique and allows the users to gain a more complete picture of material behavior at these length scales.

Users

The user groups supported by the 1-ID beamline are diverse, ranging from university research groups investigating the micromechanics of materials at a more fundamental level to large corporate users interested in process optimization. Large polycrystalline aggregates with a variety of applications are investigated.

Examples of research performed at beamline 1-ID:

- Understanding the micromechanics associated with microcrack nucleation in nickel based superalloys used for turbine blades. [1]
- Strain development in bovine bone under uniaxial compression. [2]
- Investigating the effects of irradiation on materials used in nuclear engineering. [3]

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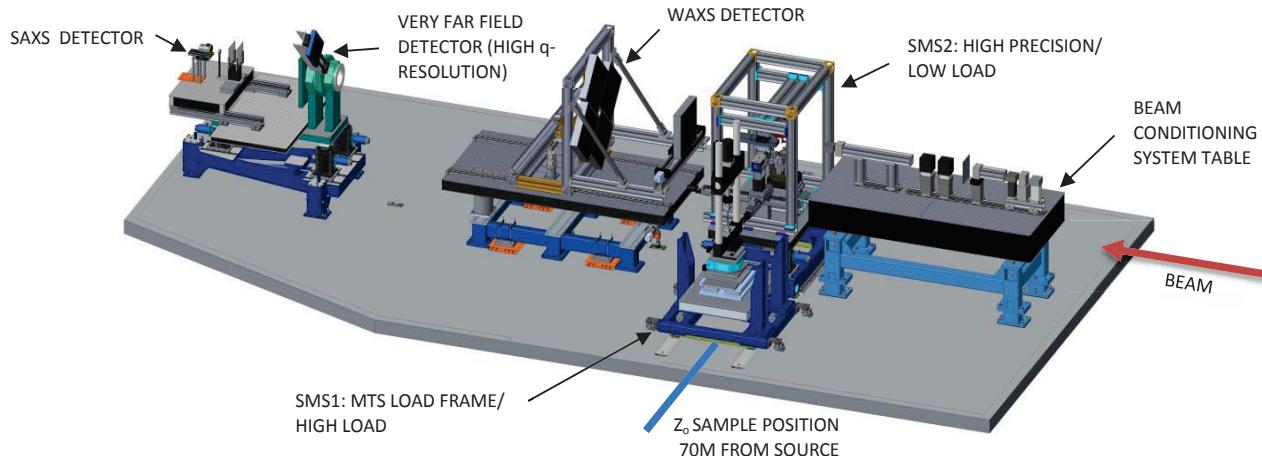


Figure 1: Standard configuration of instrumentation at beamline 1-ID E-hutch.

E-HUTCH INSTRUMENTATION

Layout

The configuration of the hutch has been largely driven by user requirements. In addition to utilizing the existing instrumentation, many users want to bring their own experimental components such as custom load frames, environmental chambers or heaters. The modular configuration and organization of the hutch are critical to accommodating these custom components and handling frequent setup changeovers with the least amount of time and effort (see Figure 1).

Beam Conditioning System (BCS) Table

The BCS table is located at the upstream end of the endstation. It is equipped with ion chambers capable of monitoring the flux and beam position, slits, horizontal and vertical focussing sawtooth refractive lenses and a fast shutter. The lenses are equipped with full degrees of motion necessary to provide 1 μm (vertically) and 15 μm focussed beam (horizontally) on demand. The table is also equipped such that new high energy optics can be deployed and tested without disturbing the existing optics. The individual optical elements can be utilized flexibly according to the actual experimental needs for the given technique.

Sample Location and Manipulation Systems

Immediately downstream from the BCS table is the sample support which is located at a fixed APS-Z position of approximately 70 m from the source. Samples are typically mounted in one of two independent sample manipulation systems (SMS) which can be brought into and out of the beam position. An SMS is essentially a predefined experimental setup for different demands which can be used as a baseline configuration for a sample environment combined with a combination of techniques.

The available SMS at Sector 1 are listed below:

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1. **Large vertical translation and high load capacity table with an MTS load frame (permanent)** – An optical table capable of large Y motion range is equipped with an appropriate stage stack to rotate and translate a 15 kN uniaxial tension/compression MTS load frame. An infrared furnace can be added to the setup. The MTS load frame can be removed to utilize the table and stage stack for other large size sample environments (e.g. furnace, welder, or microwave chamber). While predominantly used for SAXS/WAXS types of experiments, a series of successful HEDM measurements has been performed using this setup.
2. **High precision and low load capacity sample manipulation table (permanent)** – A standard APS optical table is equipped with a set of high-end translation and arc stages and an air bearing rotation stage (see Figure 2). Optionally a compact size load frame or a furnace can be deployed on the top of the stage stack. This table is to routinely deliver the stability and motion necessary for HEDM.

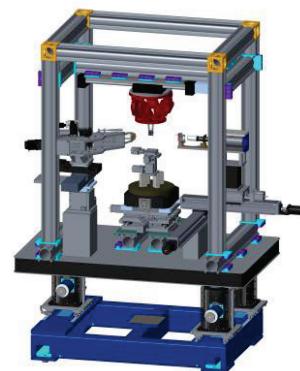


Figure 2: High precision SMS with near field and tomography detectors and overhead rail system supporting the conical slit cell.

Beam Lines

End Stations and Sample Environments

3. Open spot for custom user instrumentation

- The clearance required for the two permanent SMS to be positioned into the beam also serves as space for custom instrumentation to be installed.

Floor Rails

A set of two parallel linear profile rails have been installed in recessed pockets in the concrete floor. Each SMS is permanently mounted to the rail system and can translate transverse to beam direction into or out of position. The motion is manual and can be easily be done by one person. Manually moving the SMSs, reduces the risk for collision between auxiliary equipment which changes continually. The rails allow multiple SMS to occupy the same spatial area maintain their alignment in APS-Z and Y.

In the case that custom equipment which does not use the rails must be brought in, a set of low profile trough covers are put into position to cover the rail system. Equipment can then be wheeled over the rails and people can safely walk over the troughs as well.

The floor rail system, however, is flexible and a new SMS can be adapted through the use of locating components on the support base bottom (see Figure 3). The locating scheme utilizes custom made case hardened steel conical locators which also serve as feet for the SMS which can be removed from the rails if needed. The receivers, similar to a kinematic mount, include two flats, one vee and one cone (see Figure 4).

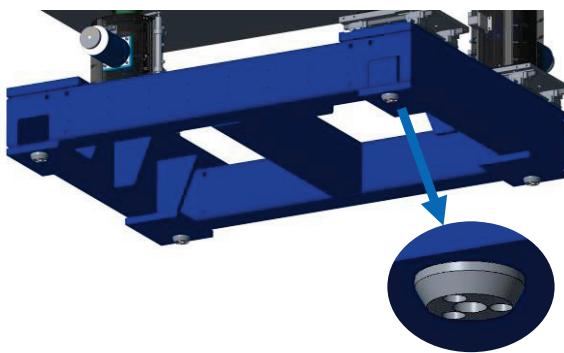


Figure 3: Locators on the bottom of a SMS support table.

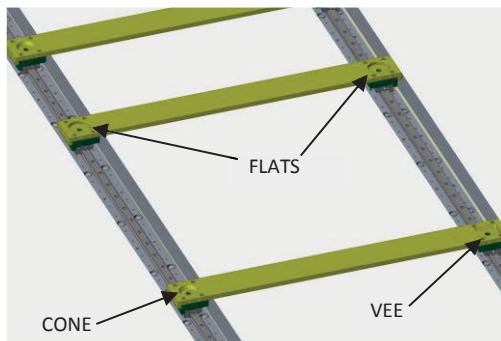


Figure 4: A set of receivers on “straps” mounted to the floor linear rails.

Beam Lines

End Stations and Sample Environments

Overhead Rail

The high-precision SMS is equipped with an extruded aluminium structural rail frame mounted to the support base for the SMS. The cube shaped structure is used for mounting auxiliary components around the sample including:

- Digital imaging correlation camera (DIC)
- Conical Slit cell and manipulator
- Heater
- Keyence laser system
- Shielding

This framework was constructed relatively quickly and was intended to be temporary. A larger more permanent version of this system is currently under design.

WAXS Detector Array

A four panel GE detector array used for WAXS fills the central portion of the hutch area. It was initially designed for a static installation with a panel adjustment of 2m in Z obtained through the support table’s rack and pinion system. Since the initial installation, the detector’s support table has been retrofitted with air pads and upstream and downstream locators to increase its range and coverage. The alignment scheme consists of one vee on the downstream end of the table and one cone on the upstream end of the table. Two sets of receivers installed into the concrete floor are in line with the beam and allow the table to gain an additional 1m in panel throw (see Figure 5).

The air pads and locating system are also used to position the detector panel array asymmetrically from the beam to increase or change coverage or to be moved out of the way completely for use of the VFF detector setup.

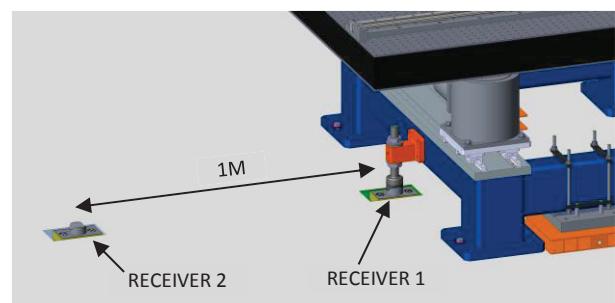


Figure 5: A locator on the support frame and receivers in the concrete floor allow for quick alignment of the WAXS detector.

FUTURE PLANS

Permanent Overhead Rail System

Although the temporary overhead rail system has achieved expectations for the high-precision SMS over the short term, a more permanent structure made out of structural steel tubing is currently in design. The new structure will span the width of the hutch and provide a universal framework with additional motions incorporated (both manual and motorized) which can allow these

components to be moved into position as needed without repeated mounting and alignment. The near field and tomography detectors may join the group of components which will be suspended from the structure. Currently these two detectors, mounted from below, must be relocated and aligned based on the experiment.

SUMMARY

Beamline 1-ID accommodates a number of complimentary techniques and sample environments in the same spatial envelope. The configuration of the hutch has evolved to minimize the time and effort required for changeovers between techniques. Instrumentation design features have been incorporated to achieve maximum stability and repeatability of both the instruments and sample.

In addition, flexibility exists for users to install their own custom sample environments. New instrumentation can be designed or modified to adapt for installation to

the floor rail system. The configuration of the end station will continue to evolve with the needs of users.

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