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Variable q-range x-ray scattering chamber *for chemical and materials science at the Advanced Photon Source*

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Variable q-range x-ray scattering chamber *for chemical and materials science at the Advanced Photon Source*

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Abstract. We present here the design of a novel variable q-range x-ray scattering instrument recently installed at Sector 12 of the Advanced Photon Source. This device provides automated, computer-controlled q-range changes for x-ray scattering experiments by varying the sample-to-detector distance within a large vacuum chamber. Eliminating the need to vent the system when changing camera lengths allows for quick and efficient change-overs between experimental setups. The detector cannot operate in a vacuum environment; therefore it is housed within an air chamber open to atmospheric pressure. A large carbon window isolates the detector from vacuum while allowing high x-ray transmission. An array of motorized beam stops mounted directly upstream of the window protects the detector from the direct x-ray beam for various types of scattering experiments. A smaller detector protrudes into the lower front section for simultaneous wide-angle x-ray scattering data collection. A fully automated support structure aligns the vacuum chamber to the x-ray trajectory.

1. Introduction

X-ray scattering has proven to be a premier measurement technique for quickly obtaining information about the structure and composition of materials. In an x-ray scattering experiment, a sample is illuminated by a beam of x-ray photons, and the scattering pattern that is produced is measured as a function of the angle between the incident and scattered beams. The length scale that is examined within a sample is a function of the photon energy and the scattering angle; thus, studying complex materials in a comprehensive way often requires a combination of small and wide-angle measurements.

Figure 1. X-ray Scattering Chamber

Small-angle x-ray scattering (SAXS) is useful for obtaining nanoscale structural information for a variety of samples, while wide-angle x-ray scattering (WAXS) resolves features in the sub-nanometer regime. An area detector collects data that spans a particular angular range; this range depends on the size of the detector and its distance from the sample. The angles that are measured by the detector are proportional to the scattering vector, q. With fixed energy, the q-range can be manipulated by changing the distance between the sample and the detector; this allows for different sizes of interest within a sample to be probed. An evacuated flight path is required downstream of the sample to reduce background scattering from air and to allow for detection of a clear signal from the sample.

Sector 12 at the Advanced Photon Source at Argonne National Laboratory was recently upgraded to include a new, fully dedicated x-ray scattering station. Completed in the fall of 2010, beamline 12-ID-B was formed from canting beamline 12-ID. The beamline utilizes a large offset side-bounce monochromator and a set of focusing mirrors [1] to deliver desired beam to the endstation. The available energy range at this beamline is 7.8 to 14 keV, but the beamline is optimized for and typically run at a fixed energy of 12 keV. At this energy range, a vacuum chamber is necessary to prevent scattering and absorption by air.

The diversity of themes being studied at 12-ID-B together with the high demand for beam time requires a setup that incorporates quick and simple sample-to-detector distance changes and efficient changeovers between experimental setups.

2. Construction

Similar in design to the much larger neutron scattering chambers at the National Institute of Standards and Technology [2], the variable q-range x-ray scattering chamber, shown in Figure 2, was built to facilitate rapid sample-to-detector changes by housing the detector inside the vacuum chamber, so that the q-range can be varied without the need to vent the system. This feature is unique to the Sector 12 instrument and enhances the efficiency and user experience.

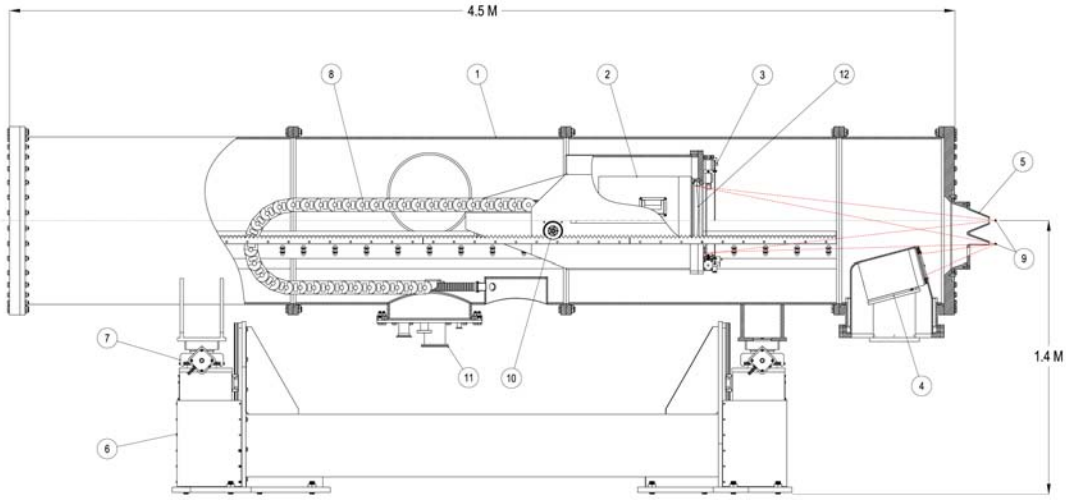

Figure 2. Variable q-range x-ray scattering chamber cutaway: 1) vacuum chamber, 2) SAXS detector housing, 3) beamstops, 4) WAXS detector housing, 5) interface flange, 6) support structure, 7) kinematic mount, 8) flex hose/cable carrier, 9) sample position, 10) gear drive and rack, 11) feedthrough ports, 12) carbon window.

2.1. Vacuum chamber

The vacuum chamber provides a scatter-free flight path between the experimental sample and the detector. The camera length of the main SAXS detector can be continuously varied from 400 mm to 3.5 meters along the length of the chamber. To minimize overall weight, ease fabrication, and provide significant cost savings, the chamber was fabricated in four main sections out of 6061 aluminum. O-rings provide all of the vacuum seals, and various ports allow for electrical feedthroughs, vacuum connections, and maintenance access. A rectangular flange on the bottom of the upstream section allows for a smaller removable detector housing for simultaneous WAXS data collection. Average pump down to 10^{-4} Torr is commonly achieved within 24 hours.

2.2. SAXS detector housing

Not designed for in-vacuum applications, the Pilatus 2M detector is nestled within a welded aluminum housing maintained at atmospheric pressure. A large 13-in-diameter carbon window, comprised of 3 layers of 125-µm-thick unidirectional carbon-fiber sheet, isolates the detector from vacuum and minimizes x-ray absorption. Data cables, power, and water cooling lines enter the main vacuum chamber from underneath and are fed to the detector housing through three flexible stainless steel vacuum hoses. The hoses are mounted within a flexible cable guide to prevent kinking and to allow for the full range of detector travel along the length of the vacuum chamber. The detector housing is supported and guided by a single linear rail on one side, eliminating the need to precisely align two rails over the large distance. The other side of the box rests on a NEXEN® precision roller pinion system that moves the chamber via direct drive from an in-vacuum stepper motor. An inline 50:1 gearbox provides a linear resolution of 12 µm/step, and the position can be precisely monitored by means of a rotary encoder fixed to the rear output shaft of the motor. The rail and rack are aligned to within 200 µm in both lateral and elevation, allowing q-range adjustment of the detector along its entire travel range without the need to realign the beam stops.

2.3. Beamstops
Remotely operated beamstops mounted directly upstream of the carbon window protect the detector from the direct x-ray beam. The beamstops are mounted to motorized mechanical slides by thin stainless steel rods. Each beamstop has a minimum travel of ± 20 mm and can be driven completely out of the detector area. A retractable aluminum plate shields the detector during beamstop alignment. For grazing incident measurements, a long 3-mm-wide tungsten strip, coupled to the horizontal and vertical motion of the lower beamstop, can be rotated upwards to block the reflected beam.

2.4. WAXS detector housing
To simultaneously collect wide-angle scattering data, the smaller Pilatus 300K detector protrudes into the lower front section of the main vacuum chamber. The detector is positioned to minimize dead pixel space between SAXS and WAXS measurements. A smaller carbon window isolates the detector from vacuum. The housing is removable to allow the main detector to achieve shorter distances to the sample if required.

2.5. Interface flange
Interchangeable inlet cones mounted to the upstream flange of the vacuum chamber accommodate various scattering angles and sample distances while minimizing the air gap between the sample and the detector. Kapton, mica, and/or Mylar windows glued to the apertures provide an adequate vacuum seal and minimize x-ray absorption. A thin tungsten septum plate installed within the cone blocks the possible scattering produced from the direct beam on the window to the WAXS detector.

2.6. Support structure
The support structure is designed to provide vertical as well as pitch, roll, and yaw manipulation of the chamber in order to align the detector travel axis to the x-ray beam trajectory. Three independently operated lifting columns are mounted to a rigid, welded steel frame. Each lifting column consists of a carrier, guided by a pair of vertically mounted linear rails, and driven by a 24:1 machine screw jack directly coupled to a high torque Nema 34 size stepper motor. Each column has an overall travel range of 12 inches, a load capacity of 1500 lbs, and a vertical resolution of 1.3 µm per motor half step. Both the aluminum vacuum chamber and the steel support base are externally powder coated to match for a consistent finish across the device.

2.7. Kinematic mounts
Arranged in the typical cone, vee, and flat configuration and affixed to the top of each lifting column, special kinematic mounts were designed in order to fully constrain the system, yet allow for the full range of motion required. Crossed roller bearing slides, mounted to efficiently designed aluminum frames, allow for a maximum travel of ± 1.5 inches in the x and z directions, and a maximum load capacity of 1000 lbs. each. A large spherical bearing allows for a tip-tilt misalignment of up to 8° and

an in-plane rotational misalignment of 15°. An integrated spherical rod end, aligned at the center of the spherical bearing, captures the assembly in the unlikely chance that the chamber would lift off. Over-travel is prevented by limit switches mounted to each axis, backed up by internal hard-stops. The cone and vee supports, driven via direct-drive hybrid linear stepper actuators, provide additional yaw alignment capability, with a resolution of 12.7 µm per step. As the vertical columns are independently actuated, the kinematic mounts fully constrain the chamber while allowing for the necessary degrees of freedom.

3. Operation

The beamstops, nozzles, and support structure motions allow the SAXS chamber to operate in three experimental modes, providing measurement capabilities for centered SAXS, off-centered SAXS/WAXS, and GISAXS. When the setup is at its centered configuration, the x-ray beam enters the chamber through the top nozzle, and the detector is protected by a small 3-mm-diameter beamstop located just upstream of the center of the detector face. Using this configuration, data is collected over the entire range of the detector; as such, the images contain information for the full radius of scattering, which is often required for anisotropic samples.

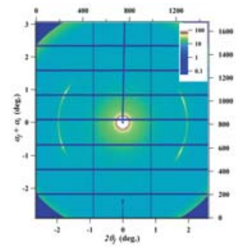

Figure 3. SAXS

To run off-centered SAXS/WAXS, the chamber is raised so that the beam enters the lower nozzle and window. A corresponding 3-mm-diameter beamstop is positioned to block the direct beam. This offset configuration provides a wide dynamic q-range, as the lower beam position allows for a greater scattering angle to be collected at the SAXS detector. Furthermore, this setup makes use of the WAXS detector, which is installed in the lower front section of the chamber. Merging data from both detectors provides a q_{max}/q_{min} ratio of about 150 and an overall q-range from 0.003 to 2.4 Å^{-1}.

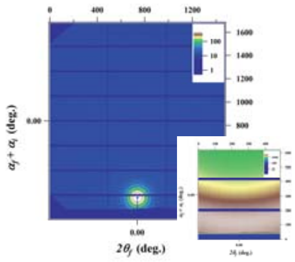

Figure 4. SAXS/WAXS

In addition to transmission SAXS/WAXS, the chamber is also equipped for collecting data in grazing incidence geometry. When the beamline runs GISAXS, an additional rotary motor is used at the sample area for careful alignment of films and flat surfaces. The scattered beam enters the chamber through the lower nozzle, and a long beamstop strip is used concomitantly with the off-centered SAXS beamstop to block the reflected beam. GISAXS measurements are useful for characterizing density correlations and analyzing nanoscale features at the surface of a sample or at an interface within a sample.

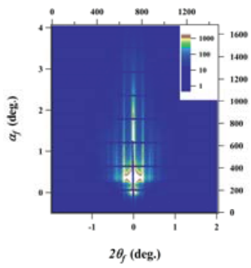

Figure 5. GISAXS

4. Conclusions

The installation of the SAXS chamber has greatly improved the versatility and efficiency of the 12-ID-B beamline. Because the setup can be changed quickly and accurately, a wider array and larger number of user groups can be easily accommodated. Additionally, many users take advantage of the chamber's capability for simultaneous SAXS/WAXS measurements. The quality of the data has also improved, due to the accuracy by which alignment and scanning procedures can be done.

5. References

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