Small-angle Scattering at the Complex Materials Consortium Collaborative Access Team (CMC-CAT) Beamline 9-ID

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Introduction

A new small-angle x-ray scattering/wide-angle x-ray scattering (SAXS/WAXS) instrument has been commissioned at CMC-CAT beamline 9-ID. It significantly expands experimental capabilities in the areas of soft matter and nanotechnology research. The instrument offers a modular, easy to use, highly adaptable approach. The sample-detector distance can be varied from a few centimeters to more than 5 m. In preliminary work, q ranges of ~3 $Å^{-1}$ for WAXS to 5×10^{-4} $Å^{-1}$ for SAXS have been achieved. The instrument is in its early form but is already operational. A number of experiments have been carried out successfully. However, significant upgrades are planned to enhance the instrument's capabilities beyond the demonstrated results.

Methods and Materials

The instrument is located in the 9-ID-C station at CMC-CAT. In accordance with the multipurpose nature of this beamline, the SAXS instrument shares the same x-ray optics with other upstream instruments, thus minimizing turnaround time. The beamline components are shown schematically in Fig. 1. Briefly, the optics consist of a Si(111) cryogenically cooled double-crystal monochromator, immediately followed by a Kirkpatrick-Baez (K-B) mirror pair [1], which allows 1:1 focusing in both directions at any position along the beam path.

Two 4-blade slits at 59 and 64 m from the source provide beam shaping and collimation. Each is followed by a removable ion chamber, allowing precise aligning. Beam sizes of 100 μm × 100 μm have been routinely used.

The main K-B mirrors also provide harmonic rejection. However, for low energies (<8 keV), an additional flat rejection mirror (Rh/ultralow expansion [ULE]) at 61 m from the source can be moved into the beam if needed.

The SAXS apparatus itself is on 7-m-long optical rails starting at 71 m from the source. Each component can easily slide along the rails or be removed, adding flexibility. For SAXS, the components include a four-blade guard slit, a reconfigurable sample stage, and a modular (1- to 5-m) evacuated flight path. A lead beam stop is placed on the last Kapton® window. The 2-D detector is a Bruker charge-coupled device (CCD). The flight path is motorized and can move in both directions around the sample position. For WAXS, the flight path is removed, and a wire-mounted beam stop is added.

Results

A number of experiments have been carried out successfully, some of which are discussed in this compilation of user activity reports. These include position-resolved, element-sensitive WAXS/SAXS studies of worm jaws biomineralization [2], silica microsphere colloidal crystals [3], biaxially stretched polymer films [4], metallic nanowire arrays [5], and nanofabricated structures on Si wafers[6].

Discussion

The SAXS/WAXS instrument at CMC-CAT beamline 9-ID has been commissioned and is already operational and productive in its early stage. Among the planned upgrades are pinhole collimation, optical sample aligning, active beam stops, and beam position monitors.

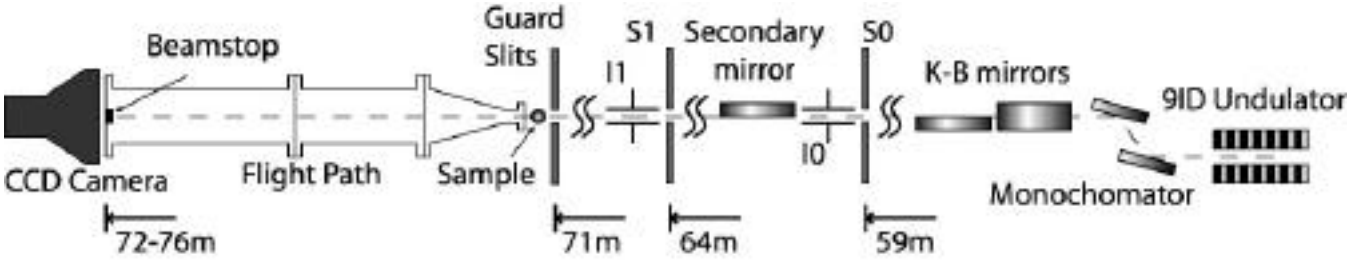

FIG. 1. Schematic beamline optics and components for SAXS (refer to text for detailed description). The distances shown for some components are distances from the source. S0 and S1 are beam collimating slits. I0 and I1 are their associated ion chambers. The double wavy lines indicate sections of evacuated beam pipe.

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References

[1] C. T. Venkataraman et al., in *Advanced Photon Source Activity Report 2001*, ANL-02/06 (Argonne National Laboratory, Argonne, IL, Dec. 2002).

[2] H. Lichtenneger et al., in *Advanced Photon Source Activity Report 2001*, ANL-02/06 (Argonne National Laboratory, Argonne, IL, Dec. 2002).

[3] P. A. Heiney et al., in *Advanced Photon Source Activity Report 2001*, ANL-02/06 (Argonne National Laboratory, Argonne, IL, Dec. 2002).

[4] R. Kolb (unpublished).

[5] C. T. Venkataraman (unpublished).

[6] W.-L. Wu (unpublished).