Ultra-small-angle x-ray scattering on the UNI-CAT instrument at the APS

Gabrielle G. Long[+], Andrew J. Allen[+], Jan Ilavsky[+], Pete R. Jemian*[+], and Paul Zschack*

[+]*National Institute of Standards and Technology, Gaithersburg, MD 20899 USA*

**University of Illinois at Urbana-Champaign, Urbana, IL 61801 USA*

Introduction

Bonse-Hart ultra-small-angle scattering (USAXS) instruments [1] make use of two pairs of crystals, where the first pair acts as a collimator and the second pair acts as an analyzer. These instruments offer excellent sensitivity and resolution for small-angle scattering measurements below scattering vector q = 0.01 $Å^{-1}$, where the scattering range of pinhole instruments usually begins. The data from such instruments are slit-smeared because of the angular collimation of the crystals. However, the addition of an orthogonal crystal pair, one crystal before and one after the sample, yields effective pinhole-collimated, unsmeared USAXS data [2].

A new USAXS instrument [3] has been commissioned as part of the UNI-CAT facility on the 33-ID beamline at the Advanced Photon Source (APS). The instrument offers continuously tunable optics for anomalous USAXS, 1000 times the throughput of earlier USAXS instruments [4, 5], high sensitivity and high resolution at low scattering vector, and a scattering range from below q = 0.00015 $Å^{-1}$ to above 0.5 $Å^{-1}$. The addition of side-reflection optics, in an optional configuration of this instrument, enables USAXS measurements of anisotropic as well as isotropic materials.

Methods and Materials

The new instrument is installed on UNI-CAT sector 33, on the insertion device beamline, after the monochromator (for photon energy selection) and the mirrors (for harmonic energy rejection). It has high sensitivity to scattering at low q and improved signal-to-noise at high q. The q range using Si(111) optics is 0.00015 $Å^{-1}$ to 0.5 $Å^{-1}$, which can be scanned in 15 minutes. The collimator pair is channel-cut Si(111), in which the first crystal is triangular. The innovative shape of this crystal allows the selection of two, four, six, or eight reflections in the pair simply by putting the x-ray beam in the appropriate horizontal position. The analyzer pair consists of two separated-function Si(111) crystals, where an adjustable gap allows the selection of matching two, four, six, or eight reflections. The x-ray beam size is 0.4 mm vertical by 1.5 mm horizontal, through which 2×10^{13} ph/s are incident at 7 keV. The photodiode detector and the associated electronics are linear over a 10-decade range. This capability means that the detector can measure a blank spectrum, with no sample in place, without the use of filters, and enables a primary calibration of the absolute intensity [4]. The resolution is $\Delta q \approx 1.5 \times 10^{-4}$. The sample-to-detector distance, which determines the high end of the q-range, can be as low as 250 mm, but can be made longer to accommodate a sample environment. The energy range currently available is 7 keV to 14 keV, but there are plans for the commissioning of higher order optics that will allow USAXS measurements over the full energy range of the monochromator in the future. Of particular interest for obtaining scattering data from samples that fluoresce is the excellent fluorescence and inelastic scattering rejection. The USAXS geometry offers an opportunity to measure of microstructure via scattering measurements (which are typically 10^{-6} below the primary beam) in the presence of copious fluorescence (which are typically only 10^{-3} below the primary beam). Finally, side-reflection Si(111) optics have been added [2, 6, 7] to enable the effective pinhole measurement of anisotropic ultra-small-angle x-ray scattering. This optional configuration delivers unsmeared, rather than slit-smeared, USAXS data.

Results

A measure of the performance of the instrument is given in Figure 1, which shows a blank scan taken with the instrument at 10 keV with no sample in the beam. This scan, measured at 10 keV, made use of six reflections in the collimator and six reflections in the analyzer. Within the rocking curve, the instrument profile falls off as q^{-12}, as expected from theory. Outside the rocking curve, the profile falls off as q^{-4}. Depending on the strength of the scattering contrast, microstructures in the range from 20 Å to 8 μm can be measured.

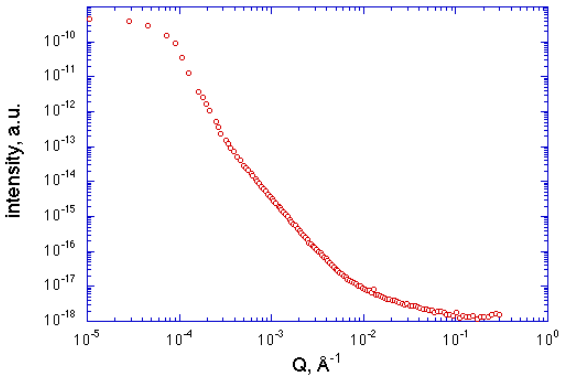

Figure 1: Blank scan taken with the instrument using 10 keV photons and no sample in the beam.

Discussion

The new USAXS instrument on UNI-CAT sector 33 is a very practical addition to the portfolio of facilities at third-generation synchrotron sources. Data can be taken over a 10-decade range in intensity, and scattering can be measured over nearly four decades in scattering vector. The number of reflections can be selected in the collimator and the analyzer pairs for high flux (two or four reflections) or high resolution and high sensitivity (six or eight reflections). Varying the position of the x-ray beam on the first crystal pair and varying the distance between crystals on the second crystal pair allows easy access to a wide range of x-ray energies and resolutions. The flux on the ID beamlines at the APS is sufficiently great that USAXS imaging can be realized.

Acknowledgements

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