USAXS/SAXS/WAXS instrument for materials research

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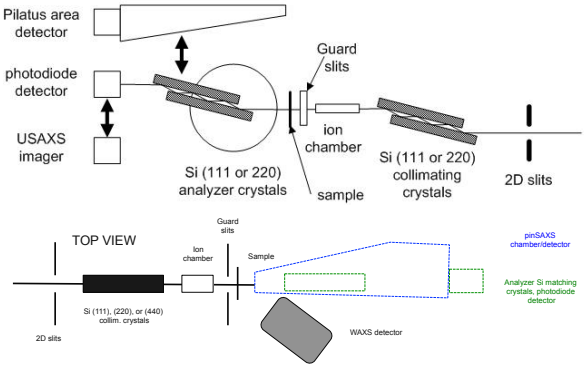
The ultra-small-angle x-ray scattering (USAXS) instrument at the Advanced Photon Source has opened up new areas of microstructure characterization in physics, chemistry, polymers, biology, and materials science by combining the high brilliance and variable beam size with a highly flexible instrument design. Various geometries, including 1-D and 2-D collimated USAXS, USAXS combined with high-q pinhole SAXS ("pinSAXS") and WAXS, USAXS imaging, and USAXS XPCS provide set unique scattering and imaging tools for materials characterization. The poster present status of the most used USAXS/pinSAXS/WAXS geometries available for users at the APS.

1-D collimated ("slit smeared") USAXS/pinSAXS/WAXS :

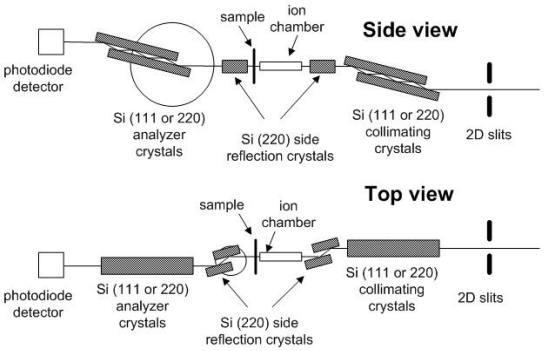
Slit smeared USAXS is the most used geometry of the APS USAXS instrument. It combines World-class unmatched scattering vector *q* range with matched dynamic intensity range. It can be used for strong to relatively weakly scattering from isotropic microstructures. Due to slit-smearing the data require either modeling with software capable of smearing the models (e.g., *Irena* package) or use of desmearing routine prior to analysis.
By combining slit smeared Bonse-Hart instrument with an 500mm long pinhole SAXS camera and 200mm long WAXS camera, fully automated and integrated into the operation, we improve the instrument to include quality data at high-*q* values.



2-D collimated USAXS :

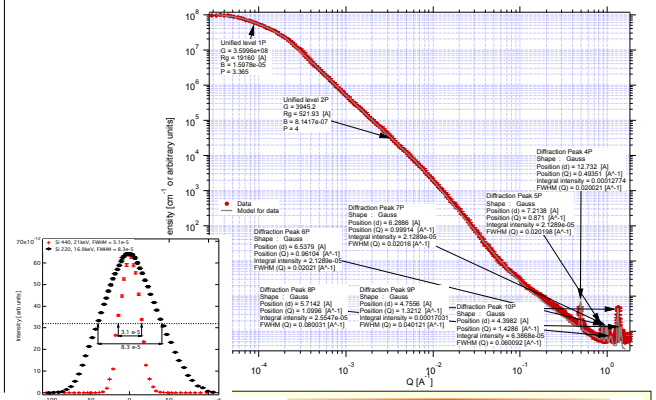
2-D collimated USAXS removes slit smearing by adding horizontally scattering pair of single piece channel cut crystals. By using this method the instrument can be used for measurements of anisotropically scattering samples. Multiple measurements per sample are needed to analyze the anisotropy.

By combining vertically and horizontally scattering crystals we gain high-q resolution in both directions, but the efficiency of counting is significantly reduced. Therefore this geometry is suitable only for strongly scattering samples. Further, the geometry limits the maximum scattering vector *q* to about 0.1 A^{-1}.



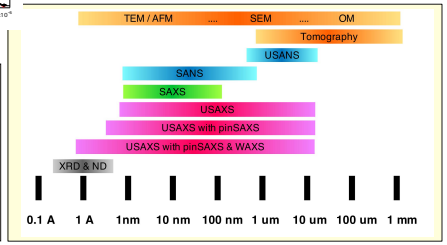
High-Energy USAXS/pinSAXS/WAXS

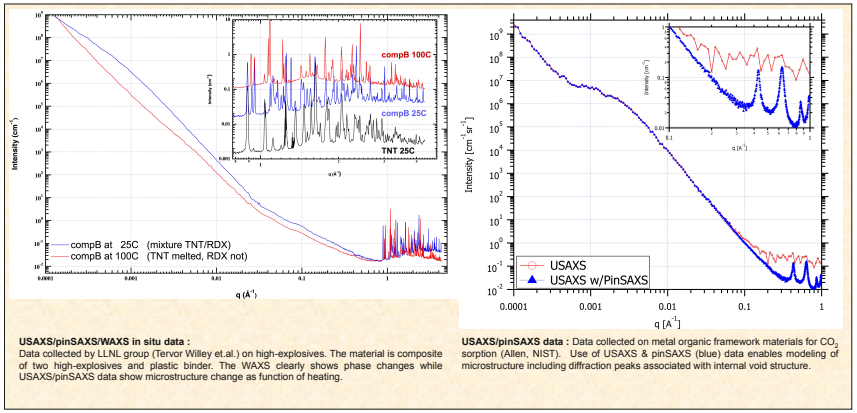
The operations with 20-30keV X-rays, employing Si 440 crystals was proven to be possible and is currently under development for user operations. This results in Q resolution ~ 3x10^{-5} A, increase in the maximum scatterer sizes up to ~20 micron, and reduction of absorption in the samples and sample environments.



1-D collimated USAXS/pinSAXS/WAXS capabilities:
USAXS/pinSAXS 0.8 x 10^{-4} < Q < 1.5 Å^{-1}, Q resolution 0.8 x 10^{-4} Å^{-1}
WAXS (user selectable) ~ 4 Å > d > 0.8 Å
Size range measured: *0.8 Å to 2 micrometers* (one scan) (up 30 micron in the future)
Absolutely calibrated intensity data (quantitative modeling results)
9 – 18 keV X-ray energy range (up to 28keV in the future)
Intensity range up to *11 decades*
Data collection time : *average 10 min/sample*, fastest ~ 3 minutes/sample
Beam size up to : 1 mm (vert.) - 2 mm (hor.)

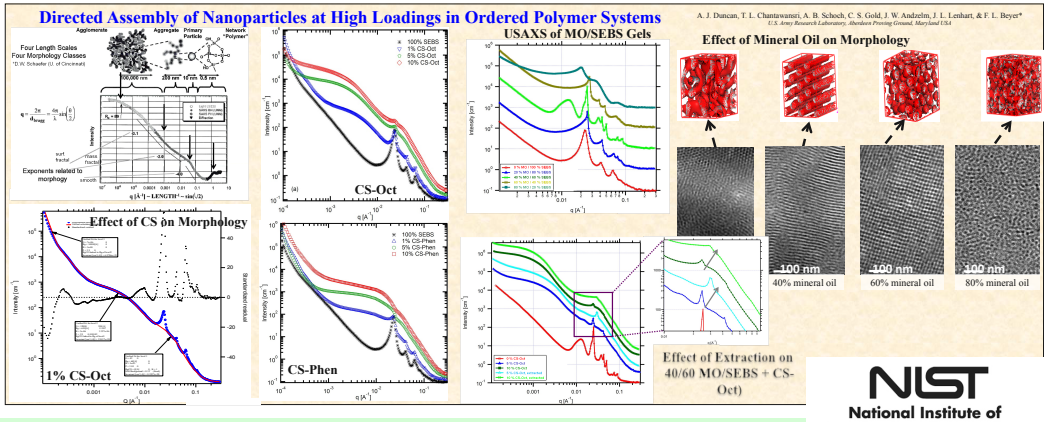
2-D collimated USAXS capabilities:
0.8 x 10^{-4} < Q < 0.1 Å^{-1}, Q resolution 0.8 x 10^{-4} A^{-1}
9 – 18 keV X-ray energy range
Intensity range up to 8 decades
Beam size up to
0.4 mm - 3 mm USAXS horizontal
0.04 mm – 0.4 mm USAXS vertical
0.2 x 0.2 mm pinSAXS
Pinhole collimated data – studies of anisotropic scatterers possible





USAXS/pinSAXS/WAXS in situ data :
Data collected by LLNL group (Tervor Willey et.al.) on high-explosives. The material is composite of two high-explosives and plastic binder. The WAXS clearly shows phase changes while USAXS/pinSAXS data show microstructure change as function of heating.

USAXS/pinSAXS data : Data collected on metal organic framework materials for CO_2 sorption (Allen, NIST). Use of USAXS & pinSAXS (blue) data enables modeling of microstructure including diffraction peaks associated with internal void structure.



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APS USAXS/pinSAXS/WAXS is a unique, powerful facility for characterization of the microstructures in many areas of science, including materials science physics, chemistry, polymers, and biology. It provides unique and unparalleled microstructure data covering size range from nanometer to over 1 micron.

NIST National Institute of Standards and Technology
U.S. Department of Commerce

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