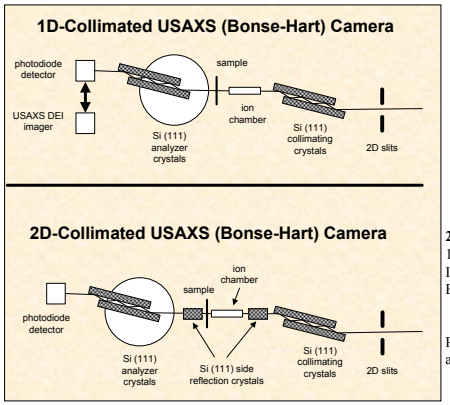
APS USAXS facility - capabilities and examples of science

J. Ilavsky and **P. R. Jemian**
Advanced Photon Source, Argonne national Laboratory, Argonne IL
Submitting author: J. Ilavsky, e-mail: ilavsky@aps.anl.gov, FAX: (630) 252-0862

Abstract

Ultra-small-angle x-ray scattering (USAXS) instrument at a third generation synchrotron source has been proven to open up new areas of microstructure characterization in materials science by combining the high brilliance and small beam size with a highly flexible instrument design. We will present overview of current instrument capabilities of the APS USAXS instrument in both slit-smeared and 2D collimated geometries. Further we will present examples of science documenting the unique data which this instrument provides for broad range of applications.

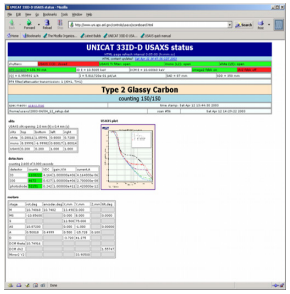


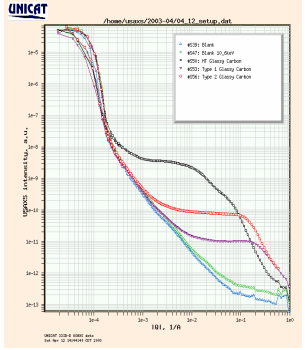
1-D collimated USAXS:
$1.2 \times 10^{-4} < Q < 1$ Å^{-1}
Intensity range up to 9 decades
Beam size up to
0.4 mm - 3 mm horizontal
0.04 mm – 0.4 mm vertical
Slit smeared data (need for numerical desmearing) – limited to isotropic scatterers

2-D collimated USAXS:
$1.2 \times 10^{-4} < Q < 0.1$ Å^{-1}
Intensity range up to 8 decades
Beam size up to
0.4 mm - 1 mm horizontal
0.04 mm – 0.4 mm vertical
Pinhole collimated data – studies of anisotropic scatterers possible

Common features:

Standard less automatic absolute intensity calibration.
Standard data set (150 points, $Q_{max} \sim 1$ Å^{-1}) collection time ~20 min
CCD imager available to image samples in radiography mode.
Heating cell for temperatures up to 130 deg. C
Flexible sample area accommodates complicated experimental setups, Multi sample holders.
Data reduction using set of macros ("Indra") for Igor Pro (Wavemetrics Inc., www.wavemetrics.com)
Use of spec (Certified Scientific Software) for instrument control.
Simple user interface with only few simple commands needed.
View live USAXS on the Web:





USAXS imaging:

Ultra-small angle scattering used as contrast mechanism
Can provide contrast under conditions when radiography and phase contrast imaging cannot
Images produced at different scattering vectors highlight different microstructural features within the same sample volume - separately characterize specific population using their size and/or orientation

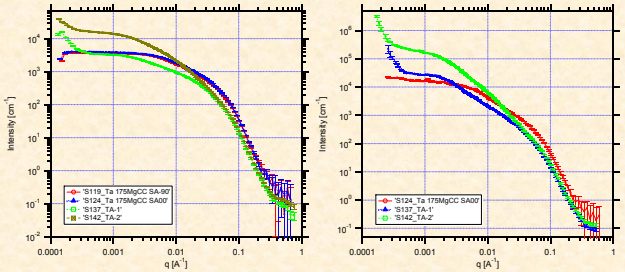
Data evaluation ("Irena" package):

Freely available package for SAS data evaluation supported by staff.
Windows and Mac platform (one package), set of macros for Igor Pro
Within one package provides various ways to look on the SAS data
Seamlessly works with data from various sources.
Consistent GUI amongst analytical methods to reduce user learning curve.
Following methods are incorporated:

- Size distribution using regularization method, maximum entropy and total-non negative least squares & number of built in particle shapes/form factors
- Direct SAS modeling from up to 5 populations of scatterers of various sizes, shapes and scattering contrasts. Optional least square refinement available.
- Unified fit model for up to 5 levels (*http://www.eng.uc.edu/~gbeaucag/PDFPapers/Beaucage2.pdf*)
- Fractal model.
- Gels model (Debye-Bueche).
- X-ray/neutron reflectivity (Parratt's code)
- Graphing tool.
- Scattering contrast calculator (including anomalous effects)
- Import/export/data manipulation tools.

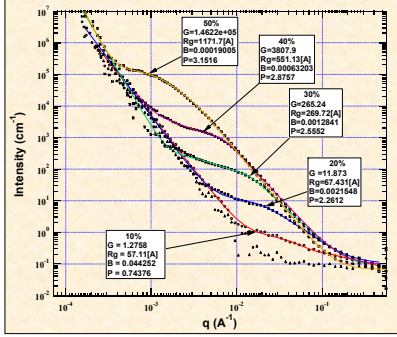
For details see:
http://www.uni.aps.anl.gov/usaxs USAXS instrument web page
http://www.uni.aps.anl.gov/~ilavsky/irena.html Irena 1 web page

Example : Aerogels



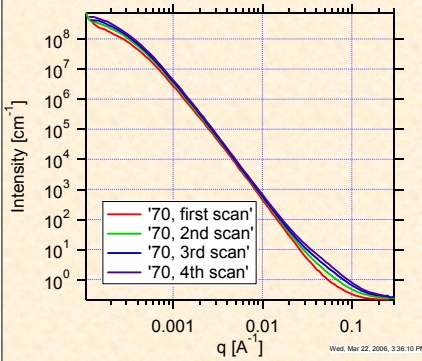
Example of small angle scattering from Ta aerogels. Slit smeared data left graph, same data desmeared right graph. Aerogels are unique materials with very low density which are considered for many applications in aerospace industry. Graphs from work by Ted Baumann, Joe Satcher, Trevor Willey, and Tony Van Buuren, LLNL.

Example : Liquid crystals in polymers

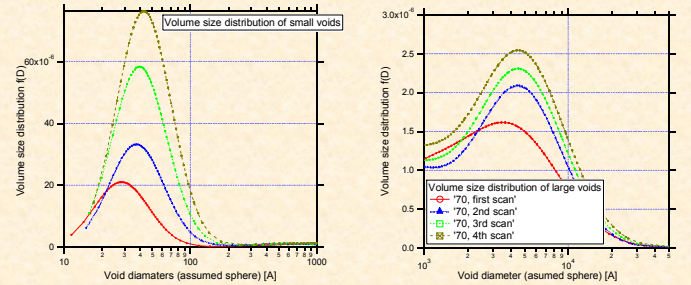


USAXS data from Polymer-dispersed liquid crystals. The loading of liquid crystals in polymer changes the structure over wide size range accessible only by USAXS. Polymer-dispersed liquid crystals (PDLCs) are of technological importance for electro-optic applications such as privacy windows, electro-optic shutters, and large area flat-panel displays. Graph from current work by Ryan S. Justice, Dale Schaefer, Richard Vaia, David Tomlin, and Timothy Bunning, Interface morphology and phase separationin polymer-dispersed liquid crystal composites, accepted to *Polymer*. Authors are from University of Cincinnati, Air Force research Lab, and UES Incorporated.

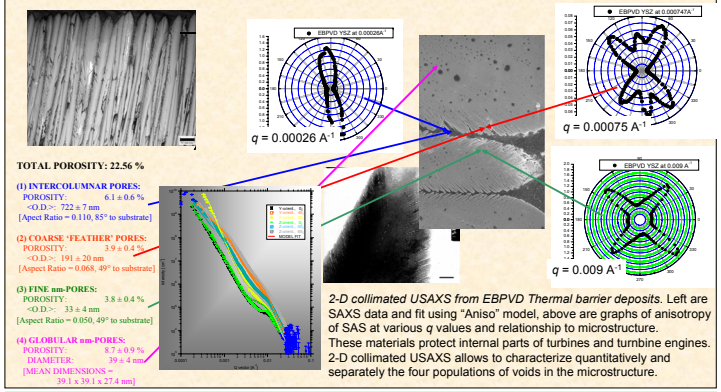
Example : High explosives



USAXS data from in situ measurements of highly insensitive energetic materials based on 1,3,5-triamino-2,4,6-trinitrobenzene (TATB). Various TATB formulations experience an irreversible volume growth event that is a function of both temperature and time, generally referred to as ratchet growth. This affects significantly the detonation velocity of these highly insensitive explosives. Of particular concern are the voids in the nanometer to micron size scale intrinsically associated with the detonation process. Such small porosity in bulk material is not easily investigated using various techniques, however, ultra small angle scattering (USAXS) technique is ideally suited for characterization of structure on this scale in energetic materials.
Presented data are from in situ experiment, each scan represents one thermal cycle between -30C and 80C. Graph is from work preformed by Trevor M. Willey, Tony van Buuren, and Jonathan R. I. Lee, LLNL.



Example : EBPVD Thermal barrier Deposits



2-D collimated USAXS from EBPVD Thermal barrier deposits. Left are SAXS data and fit using "Aniso" model, above are graphs of anisotropy of SAS at various *q* values and relationship to microstructure. These materials protect internal parts of turbines and turnbine engines. 2-D collimated USAXS allows to characterize quantitatively and separately the four populations of voids in the microstructure.

Conclusions:

USAXS 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.

Argonne National Laboratory is a U.S. Department of Energy laboratory managed by The University of Chicago