

Versatile USAXS (Bonse-Hart) facility for advanced materials research

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Abstract

A Bonse-Hart USAXS facility developed by NIST and UNICAT is a world-class resource for advanced materials research. The facility consists of a Bonse-Hart-type camera, fully-automated instrument control, data reduction, and evaluation software packages, and dedicated staff to support users. Instrument performance parameters include: undulator X-ray source with $\sim 10^{15}$ photons/sec at 10keV, 7 – 19 keV energy range, 0.0001 Å⁻¹ - 1 Å⁻¹ Q-range, 9 decade intensity range, primary (standless) absolute intensity calibration, fluorescence rejection in the scattered beam, beam sizes from 1.2 mm² down to 0.04 mm² with a 1-D spatial resolution down to 40 μm. Instrument capabilities encompass: semi-automated data reduction, analysis using state-of-the-art structure factors and models, anomalous SAXS, and USAXS-imaging capabilities. Both 1-D collimated (slit smeared) and 2-D collimated configurations are routinely available.

Both internal and general user applications presented elsewhere at this meeting include characterization of the formation of nanoparticles within a flame, sintering-resistance in advanced thermal coatings, gradient microstructures in fuel cell layers, nano-particulate agglomeration and assembly, and dislocation structures in aluminum. Polymer studies include hierarchical structures, in-situ and ex-situ deformation, nano-composites, and carbon nanotube fillers. Environmental science studies include diesel soot formation and soil structure studies.

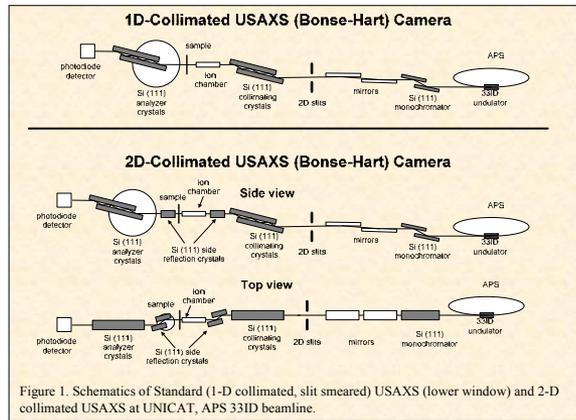


Figure 1. Schematics of Standard (1-D collimated, slit smeared) USAXS (lower window) and 2-D collimated USAXS at UNICAT, APS 331D beamline.

Common features:

Standard data set (150 points, $Q_{max} \sim 1 \text{ \AA}^{-1}$) collection time ~ 22 minutes
CCD imager available to image samples in radiography mode.

Heating cell for temperatures up to 130 deg. C
Multi sample holders.



Acknowledgements

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1-D collimated USAXS:

- $1.2 \times 10^{-4} < Q < 1 \text{ \AA}^{-1}$
- 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:

- $1.2 \times 10^{-4} < Q < 0.1 \text{ \AA}^{-1}$
- Intensity range up to 8 decades
- Beam size up to
 - 0.4 mm - 1 mm horizontal
 - 0.04 mm - 0.4 mm vertical
- Unsmear data – studies of anisotropic scatterers possible

Examples of results

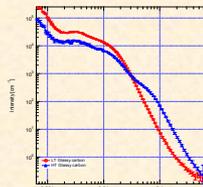


Figure 2. Example of 1-D collimated USAXS (numerically desmeared) data - 2 different types of glassy carbon

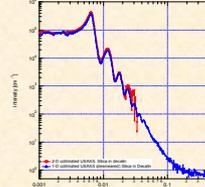


Figure 3. Comparison of 1-D collimated USAXS (numerically desmeared) data and 2-D collimated (no numerical desmearing necessary) data of Silica spheres with narrow size distribution in Decalin. Note difference in Q range and intensity range. Error bars removed for clarity.

Use of spec (Certified Scientific Software)™ custom macros for instrument control and data collection:

- Simple user interface – 4 commands needed:
 - Change geometry (*useModeRadiography & useModeUSAXS*)
 - Collect data (*qdo usaxs.mac*)
 - Reset instrument when necessary (*ResetUSAXS*).
- Use of command file to automate data collection, simple command to run one sample (*preUSAXStune: USAXS_SAMPLE_THICKNESS=1.0 : USAXSscan 12.5 0.0 "Sample Name"*)
- View live USAXS on the Web:

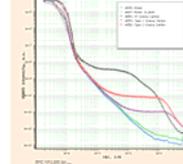


Figure 4. Examples of live instrument data on web. Left instrument parameters overview, right graph with live data.

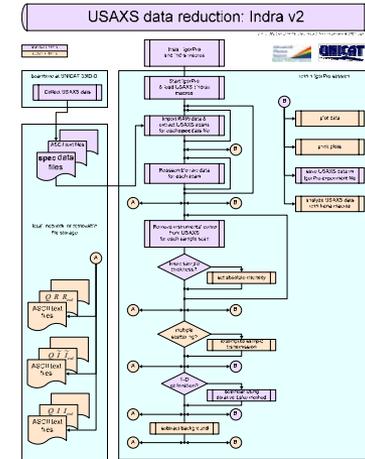
Conclusions:

USAXS facility provides a practical means to characterize the microstructures in materials science.

* Information on commercial products is given for completeness and does not necessarily constitute or imply their endorsement by the National Institute of Standards and Technology

Data reduction:

Custom set of macros ("Indra" v. 2) for Igor Pro v. 4 (Wavemetrics Inc.)^{*}



SAS data evaluation package ("Irena" ver. 1):

Provided free to anyone.

Windows and Mac platform (one package)

Custom made set of macros for Igor Pro (Wavemetrics Inc.)

Within one package provides various ways to look on the SAS data

Seamlessly works with data from:

- USAXS reduction routines
- "qrs" named data
- any other SAS data structure with Q – Intensity – Error

Consistent GUI amongst analytical methods to reduce user learning curve.

Following methods are incorporated:

- Size distribution using regularization method (using spheroidal particle shapes)
- Size distribution using maximum entropy method (Windows only) (using spheroidal particle shapes)
- 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>)
- Configurable graphing tool.
- under development: standard SAS plots and fits (Porod, Guinier, Kratky,...)

For details see:

- <http://www.uni.aps.anl.gov/~ilavsky/sas.htm> USAXS instrument web page
- <http://www.uni.aps.anl.gov/controls/usaxs/scoreboard.html> USAXS live data
- http://www.uni.aps.anl.gov/~ilavsky/indra_2.htm Indra v2 web page
- http://www.uni.aps.anl.gov/~ilavsky/irena_1.htm Irena 1 web page
- <http://www.uni.aps.anl.gov/~ilavsky/APSUM2003.pdf> this poster

