## APS Workshop 7: Ultra-small, Ultra-powerful: APS-U-USAXS' Role in Advancing Materials and Manufacturing

**Wednesday, May 8, Morning**

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| 8:00 – 8:05 | Workshop Organizers<br>*Opening Remarks* |
| 8:05 – 8:30 | Jan Ilavsky (Argonne National Laboratory)<br>*A New Era of USAXS with APS-U* |
| 8:30 – 8:55 | Andrew Allen (National Institute of Standards and Technology)<br>*Addressing the Nation's Standards and Measurement Needs to Quantify Phenomena in Complex Materials by Exploring* Operando *Structural Science across All Length Scales* |
| 8:55 – 9:20 | Trevor Willey (Lawrence Livermore National Laboratory)<br>*Advanced USAXS Capabilities to Characterize Engineered Materials for Novel Applications* |
| 9:20 – 9:45 | Lilo Pozzo (University of Washington)<br>*AI-driven High-throughput Small Angle X-ray Scattering for Accelerated Materials Discovery* |
| 9:45 – 9:55 | Break |
| 9:55 – 10:20 | Lawrence Anovitz (Oak Ridge National Laboratory)<br>*Thoughts on Geological Applications of the Future (U)SAXS Instrument at the APS-U* |
| 10:20 – 10:45 | Greg Beaucage (University of Cincinnati)<br>*Polymer Nanocomposites: Structural Emergence and Quantification of Dispersion and Distribution at Different Size Scales* |
| 10:45 – 11:30 | Panel Discussion<br>*Shaping the Future of USAXS: Priorities and Innovations Post-APS-U Restart* |
| 11:30 | Adjourn |

A New Era of USAXS with APS-U

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The combination of the new APS-U source and the newly acquired USAXS hardware, part of the APS-U project, presents a generational opportunity for the USAXS-SAXS-WAXS instrument. Initial operations and commissioning at 20-ID prior to the APS-U shutdown have validated our design decisions, demonstrating substantial enhancements in both performance and stability of the instrument. Although it may take a year or more to fully harness the capabilities of APS-U and the new USAXS hardware, it is imperative to begin contemplating the prospective opportunities now available to our staff and user community. In the presentation, the USAXS team will outline the observed and anticipated improvements and discuss potential new capabilities for consideration. This will initiate a dialogue with speakers and attendees, ultimately leading to a comprehensive document detailing the user community's future priorities for the USAXS instrument.

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Addressing the Nation's Standards and Measurement Needs to Quantify Phenomena in Complex Materials by Exploring *Operando* Structural Science across All Length Scales

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Working originally with Northwestern University, NIST developed a practical USAXS capability at NSLS in the late 1980s and transferred it to the APS as part of UNICAT in the late 1990s. Since then, NIST has partnered with APS staff to develop the USAXS facility and apply it to address increasingly complex materials measurement challenges of technological importance. Areas of application have included problems in thermal barrier coatings, heat treated alloys, densification of ceramics, solid oxide fuel cell degradation issues, solution-mediated nanoparticle formation and ordering, cement hydration, carbon capture, crystallization problems in glass, and additive manufacturing. The original restricted q range of the facility has been continually extended to encompass the conventional SAXS regime, x-ray diffraction, and imaging. Meanwhile, the intrinsic first-principle absolute intensity calibration of USAXS measurements has enabled NIST to work with APS in developing a traceable NIST Standard Reference Material for SAXS intensity calibration, now used around the world. Nowadays, complete structural and microstructural characterization can be achieved in a few minutes using x-ray energies of 21 keV and above, allowing *in-situ* studies of phenomena occurring across many length scales under a range of realistic sample environments [1]. With the APS upgrade, a radical upgrade of the USAXS instrument, itself, and a planned upgrade of the wide-angle x-ray scattering (WAXS) detector for XRD, the APS USAXS facility will provide truly world-leading capabilities among small-angle scattering facilities [2] for rapid real-time quantitative microstructure and structure characterization addressing the nation's standards and measurement needs in increasingly complex applications such as additive manufacturing of composite systems, and carbon dioxide reduction via direct air capture, carbonation and mineralization.

[1] J. Ilavsky, F. Zhang, R.N. Andrews, I. Kuzmenko, P.R. Jemian, L.E. Levine & A.J. Allen; "Development of combined microstructure and structure characterization facility for *in-situ* and *operando* studies at the Advanced Photon Source," *J. Appl. Cryst.*, **51**, 867-882 (2018).
[2] A.J. Allen; "Selected advances in small-angle scattering and applications they serve in manufacturing, energy and climate change," *J. Appl. Cryst.*, **56**, 787-800 (2023).

# Advanced USAXS Capabilities to Characterize Engineered Materials for Novel Applications

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For many years, the USAXS beamline has been invaluable for characterizing various engineered materials for novel applications at Livermore. A few examples range from studies of mesoscale voids in high explosives essential to detonation via hot-spot formation, to ultra-low-density materials (for example, aerogels) and their various applications, to target material developments for inertial confinement fusion, to metal damage mechanisms and helium inclusions due to alpha decay. The presentation will summarize a few results in some of these areas, and discuss persistent open questions, as well as new research areas that could greatly benefit from possible USAXS enhancements. APS-U will certainly provide new opportunities with much higher brightness and flux, enabling *in-situ*, *operando*, and imaging capabilities unrivaled worldwide.

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AI-driven High-throughput Small Angle X-ray Scattering for Accelerated Materials Discovery

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Artificial intelligence (AI), when paired with laboratory automation and advanced metrology (e.g., SANS, SAXS, USAXS), can greatly accelerate materials optimization and scientific discovery. For example, it can be used to efficiently map a phase-diagram via intelligent sampling along phase boundaries [1], or in 'retrosynthesis' problems where a material with a target structure is desired but a synthetic route is not yet known [2]. These approaches are especially promising in soft matter, and polymer physics, where design parameters (e.g., formulation, chemical composition, MW, topology, processing conditions) are vast and where properties and function are intimately tied to these design features. In this context, USAXS is especially useful because of the broad range of length-scales that are characterized, the non-destructive nature of the technique, and its generalizability to a large range of material systems. However, for AI algorithms to operate efficiently using scattering data in multidimensional design spaces, they must also be 'encoded' with relevant domain expertise specific to the problems being tackled. This talk will cover recent advances in the use of synergistic SAXS/USAXS, automation hardware, and software tools for accelerated materials optimization for colloids, polymers, and soft matter systems. Finally, it will also outline new opportunities that will be opened by the availability of improved USAXS capabilities at the APS after its upgrade.

[1] K. Vaddi, K. Li, L. Pozzo, (2023) "Metric geometry tools for automatic structure phase map generation," Digital Discovery, 2, 1471-1483.
[2] K. Vaddi, H. Thart Chiang, L.D. Pozzo, (2022), "Autonomous retrosynthesis of gold nanoparticles via spectral shape matching," RSC Digital Discovery, 1, 502-510.

Thoughts on Geological Applications of the Future (U)SAXS Instrument at the APS-U

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While, to my knowledge, the first published study of small angle scattering from a geological material was a neutron study of Hall, Milder, and Borst (1983) on a “shaly rock,” and may subsequent studies focused on the fractal properties of these rocks, over the last 20 years both x-ray and neutron scattering studies have focused on how data acquired in this manner can be used to address problems of geological interest. In particular, (U)SAXS studies have permitted both acquisition of data with spatial and temporal resolution not available by other means. Geologic materials also tend to show pore or other scattering structures over a very wide range of scales and may contain critical data near the SAXS/WAXS interface. Thus, the future (U)SAXS instrument provides a number of new opportunities in the geological sciences such as careful spatial mapping of changes in porosity near and across reaction interfaces, or perhaps near other features such as roots in soils, analysis of fluid structures, and determination of the kinetics of crystal nucleation and crystal growth and the relation of these features and processes to *in-situ* stress. The effects of the beam on sensitive and fluid samples will, however, have to be considered, especially when the beam is focused to give high spatial resolution. Given the breadth of the geological sciences, it will be an interesting task to show members who might never have contemplated such work how it can provide critical data to help in their most pressing problems.

Polymer Nanocomposites: Structural Emergence and Quantification of Dispersion and Distribution at Different Size Scales

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Dispersion of fillers, pigments and other additives to polymers is of industrial importance. Many fillers and pigments are hierarchical nano to macro scale materials. Often, the desired properties such as reinforcement, optical scattering, electrical conductivity, permeation depend on the emergence of macro scale features from these nano-scale materials, for instance tear resistance is the main feature enhanced by the addition of silica or carbon black to rubber and is a decidedly macroscopic phenomena though the silica and carbon blacks used for this purpose are ramified nanoparticle aggregates. Compounding must be seen as a hierarchical feature. Using USAXS, we can understand the relationship between various mixing technologies and dispersion (breaking apart) and distribution (moving the pieces towards a more uniform distribution) at nano, colloidal, micro, and macro scales and how these emergent structures impact properties such as the dynamic mechanical response of a rubber compound or the electrical conductivity of a battery anode. Our most current interests are how the complex branched structure of immiscible compounds can be tuned to accommodate cyclic mechanical deformation.