2024 APS/CNM Users Meeting Poster Session

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Posters are indexed by category then author last name.

Categories

Lettering in front of the number indicates facility/program:

- A# – Advanced Photon Source
- C# – Center for Nanoscale Materials
- U# – Upgrade
- ESRP# – Exemplary Student Research Program

BIOLOGY

The GM/CA@APS Structural Biology Facility Upgrade Plan

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The National Institute of General Medical Sciences and National Cancer Institute Structural Biology Facility at the Advanced Photon Source (GM/CA@APS) operates a national user facility for structural biology with synchrotron beamlines specializing in intense, tunable micro-beams for crystallography. The facility includes canted-undulator beamlines, 23-ID-B, and 23-ID-D, that provide stable, intense x-ray beams of user-selectable size down to 5-micron, an intuitive user interface for experiment control, and an automated data processing pipeline. The beamlines have high-capacity automounters and PAD detectors (Dectris), allowing rapid data collection. GM/CA users have been very productive, resulting in almost 2000 publications and over 3350 protein data bank deposits. Our micro-crystallography developments supported the research of Brian Kobilka, who was awarded the 2012 Nobel Prize in Chemistry for studies of G-protein-coupled receptors (GPCRs).

We plan to upgrade the beamlines during the APS dark period to exploit the high brightness of the APS-U. New state-of-the-art focusing optics and endstation instrumentations will be installed. The focusing optics will be replaced with EEM-polished mirrors (JTEC) in mechanical benders (AXILON) and compound refractive lenses (CRLs) (RXOPTICS, AXILON). The mirrors could focus the full beam down to 5 microns with an intensity of over 5×10^{13} photons/sec, and with the CRL transfocator, the beam could be focused to sub-micron dimensions with an intensity greater than 1×10^{13} photons/sec at 12 keV. The new optics will provide extremely intense, clean, stable, and rapidly adjustable beam sizes between 1-30 microns. The monochromator on 23-ID-D will be modified to increase thermal and mechanical stability and raise the maximum energy to 35 keV to exploit the high intensity of the APS-U at high energy. Each endstation will be replaced, and one high-stability table will support the CRL translocator and sample environment. The new goniometer will allow data collection on crystals as small as one micron and provide rapid scanning of random or periodic fixed target samples. A Dectris Eiger2 16M CdTe detector will allow high-speed, high-efficiency x-ray detection on 23-ID-D. The new pyBluIce GUI and beamline control software will enable sophisticated data collection routines such as 3D-rastering and helical data collection, fully automated (unattended) data collection, and routine serial crystallography data collection from fixed target and injector-based sample delivery systems. Here we will present the new designs and game-changing opportunities for structural biology research enabled by these small, ultra-intense, high-energy beams.

Time-resolved Pump-probe X-ray Solution Scattering Capabilities at BioCARS 14-ID Beamline, Advanced Photon Source

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Fundamental properties of proteins such as folding, binding, signal transduction and other basic protein functions are determined by a concerted motion of many atoms. An essential problem in biological sciences is to connect biological function of a molecule with its structure. This is typically achieved by initiating a reaction in a biomolecule, and then following structural changes by methods of spectroscopy, NMR, x-ray scattering, etc. Here we present time-resolved capabilities in x-ray solution scattering at BioCARS, General User Facility and a synchrotron resource for structural dynamic in biology, located at 14-ID beamline at the Advanced Photon Source. After APS-U, solution scattering experiments will be typically conducted with the polychromatic x-ray beam of 12 keV, 2.5% bandpass and beam size at the sample position of 10x10 μm^2, typical exposure times of 3.6 μs and $1.6*10^{11}$ photons per exposure. Exposures with a single x-ray pulse of FWHM of 250ps and $3.3*10^9$ photons per pulse are also possible. BioCARS solution scattering setup is suitable for experiments with Q-range of ~0.01-5 1/Å and allows studies of molecules up to 300 Å in size. At BioCARS, we use powerful pulsed UV, VIS and IR lasers to initiate a reaction. The reaction may be initiated either by absorbing a laser pulse by a chromophore in a photosensitive solute molecule, or more generally by using heating of the solvent by a laser pulse, or by de-caging by a laser pulse small functional molecules which then initiate the reaction. By using laser light for reaction initiation, we can obtain structural information of biomolecule undergoing the reaction with time-resolution 250 ps and longer. Multiple sample environments are available for these time-resolved pump-probe experiments.

ESRP: Effects of Pesticides on Soil Microbiomes

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Alterations in the chemical composition of soil have a direct impact on its biodiversity. Recognizing the impact of chemicals, such as those in pesticides, on soil microbes is important to understanding chemical effects in the environment. In our project, we identify relationships between soil biodiversity and the application of various pesticides. Since pesticides are used frequently in modern agriculture, knowing their effects on bacteria will help develop a deeper understanding of the human impacts on the environment. We exposed soil samples to five commonly used pesticides (Phorate, Malathion, Atrazine, Fipronil, and Urea) in varying concentrations and exposure times. After the samples were aged appropriately, we used 16S sequencing to quantify their bacterial diversities. Using R, we modeled exposure time, pesticide type, and pesticide concentration against bacterial concentrations resolved from the 16S sequencing. These models enable us to draw conclusions on the overall impact of pesticide use on bacterial diversity.

ESRP-5

ESRP: Identification and Characterization of a Metal Binding Site in a Protein Crystal

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Metal ions are essential components of biological systems, contributing to a wide range of physiological processes essential for life. A wide range of metals, from alkali and alkaline earth metals to transition metals, plays an important role in biological process. Metals such as Na^{+}, K^{+}, Mg^{2+}, and Ca^{2+}, play a structural role, while transition metals are often involved in both structure stabilization and catalysis. Some of the examples where metals' ions play important roles are as follows: enzymatic catalysis, electron transfer, oxygen binding and transport, DNA and RNA binding, signal transduction, oxidative stress defense, and structure stability, etc. The concentration of metals required for physiological functions of living organisms varies from millimolar concentrations to trace amounts. Metals also play important roles in the medicinal field in the form of anti-cancer drugs like cisplatin and carboplatin. In all the above cases, the interaction of metals with proteins or DNA impacts their biological function.

Single crystal x-ray crystallography played an important role for the past several decades in identifying and characterizing metal ion binding sites in proteins. Hen egg white lysozyme (HEWL) is used as a tool to study the metal binding sites at crystalline state. The crystals of HEWL were soaked with 10mM concentration of K_2PtCl_4 for one hour. The native and Pt-soaked crystals were frozen in the LN2 and shipped to NSLS-II at Brookhaven National Laboratory for the general (or metal), extended x-ray absorption fine structure (EXAFS) scans and data collection. Several important results based on the spectroscopic and crystallographic experiments will be presented.

ESRP: The Uptake and Effects of Cadmium Selenide Quantum Dots on Broccoli and Mustard Seedlings

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Quantum dots (QDs) are semiconductor nanoparticles with unique quantum-mechanical properties that are frequently used in the medical field and in the manufacturing of microLEDS. Cadmium selenide (CdSe) QD manufacturing produces waste fragments that contaminate the environment when released into the air, water, or land. These QDs may then be absorbed by plants. Cadmium salts are toxic inorganic compounds capable of negatively impacting human, plant, and environmental health. Concentrations of CdSe QDs as low as 25 nanomolar are considered cytotoxic and genotoxic to plants. When a 50-nanomolar CdSe concentration is reached in their tissues, *Allium cepa* (onion) plants have exhibited extensive DNA damage [1].

To evaluate the potential impact on food crops, *Brassica oleracea* (broccoli) and *Brassica juncea* (mustard) seeds were germinated and allowed to grow in a medium infused with CdSe QDs. After two weeks, scanning electron microscopy (SEM) and Raman microscopy (RM) were used to image root and leaf surfaces, enabling analysis of morphological characteristics and the elemental compositions of the superficial leaf and root tissues of these plants. Compared to control, no significant presence of cadmium in the plant tissues was detected, and no significant toxic effects were observed. Biomass measurements and elemental analysis indicate that CdSe QDs presence in the growth medium promoted plant growth and may have enhanced the efficiency of photosynthesis compared to control, whereas exposure to $CdCl_2$ affected plant growth and induced stress. These results suggest that broccoli and mustard seed plants, which are known to be hyperaccumulators, do not readily uptake CdSe QDs from their growth media, and that the presence of quantum dots in their environment might enhance photosynthetic processes.

[1] Ritesh Banerjee, *et al*. “Cadmium Selenide (CdSe) Quantum Dots Cause Genotoxicity and Oxidative Stress in Allium Cepa Plants.” *Mutation Research/Genetic Toxicology and Environmental Mutagenesis*, Elsevier, 19 Feb. 2021, www.sciencedirect.com/science/article/abs/pii/S1383571821000292.

Time-resolved Macromolecular Crystallography Frontiers at BioCARS: Serial Laue Micro-crystallography and Electric Field Jump

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BioCARS is a University of Chicago National User Facility with more than 20 years of scientific focus on synchrotron-based, dynamic studies in structural biology, initially using time-resolved protein crystallography and, more recently, time-resolved solution scattering (see poster by Kosheleva et al.). We are located at Sector 14, at the Advanced Photon Source, Argonne National Laboratory. After the APS-U, our 14-ID beamline will use two U21 in-line undulators to continue to provide high-flux polychromatic capability with <3% to 5.5% bandpass at 12 keV. With a series of x-ray shutters, single x-ray pulses of 250 ps can be isolated with up to ~5.8×10^9 photons per pulse in 48-bunch mode, focused to 5 x 10 μm^2 (H x V) beam size at the sample. As before, longer exposures, in the μs time domain, are also possible. We are equipped with ps and ns lasers (tunable from UV to IR) used for reaction initiation in photosensitive proteins, by using caged compounds or by temperature jump. With the ability to isolate single x-ray pulses, we can follow reactions in protein crystals in pump-probe experiments from 250 ps to seconds. This poster focuses on addressing main challenges for advancing structural dynamics studies and expanding such studies to a wider range of important biomolecules. These challenges involve effective studies of irreversible reactions where a rapid sample exchange is necessary and going beyond photo-initiation by using more general means of reaction initiation. First challenge is addressed at BioCARS by implementing serial Laue micro-crystallography for minimized sample consumption and second by implementing electric field jump and mix-and-inject approaches.

Improved Sample Preparation and Mounting for Fixed Target Serial Synchrotron Crystallography

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In the last decade, a wide array of sample preparation and delivery technologies have been demonstrated for XFEL- and synchrotron-based serial crystallography. Drawing upon this work, we have developed an integrated system that addresses key issues in serial crystallography in a robust way while maintaining flexibility required to address diverse real-world crystal handling challenges [1]. The key elements of this system are: (1) sample supports incorporating microfabricated thin films that are fully compatible with existing infrastructure for high-throughput cryo-crystallography including SSRL-developed *in-situ* crystallization plates [2], allow efficient removal of excess surrounding solvent and positioning of microcrystals at particular locations, generate ultra-low-background scatter while allowing easy optical imaging, and allow both room-temperature data collection and rapid cooling for cryogenic data collection; (2) a sample loading station that allows easy dispensing and subsequent removal of liquid (e.g., ligand- and/or cryoprotectant-containing solutions, buffer to facilitate dispersing or positioning crystals) from the sample supports via precisely controllable time-varying suction; and (3) a humid "gloveless" glovebox for crystallization tray manipulations, crystal soaking, and sample support loading and sealing that, unlike commercial humidity chambers, can generate and maintain the near saturating humidities (>95% r.h.) required to maintain microcrystals at their as-grown hydration and maximize crystal isomorphism while maximizing allowable working times. This system's ease of use, flexibility, and optimized performance make it attractive not just for serial microcrystal crystallography but also for routine single- and few-crystal data collection.

[1] G. Illava et al. Integrated sample-handling and mounting system for fixed-target serial synchrotron crystallography. Acta Cryst. D77, 628-644 (2021).
[2] E. L. Baxter et al. High density grids for efficient data collection from multiple crystals. Acta Cryst. D72, 2-11 (2016).

A-6

X-Ray Fluorescence Microscopy Showing the Distribution of Cisplatin in the Cochlear

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Cisplatin is a chemotherapeutic drug widely used in cancer treatment. However, cisplatin ototoxicity, which is featured by outer hair cell (OHC) loss, may account for 100-300 thousand hearing loss cases in cancer patients annually in the US. Cisplatin treatment leads to damages in various structures and cells, including the organ of Corti (where the OHCs reside), nerve tissue, and stria vascularis (SV). However, the primary target of cisplatin in the cochlea is still debating. Moreover, cisplatin is delivered intravenously (i.v.) in chemotherapy but intraperitoneal (i.p.) in most animal studies. This raises the question of whether ototoxic effects and target structures are different for the two different delivery methods. We would like to address these questions using a state-of-the-art technique, x-ray fluorescence microscopy (XFM).

Cochlear tissue were harvested after different days (1, 7, 14) after cisplatin treatment (i.v. and i.p.). After decalcification, they were cryo-sectioned into 20 μm slices and placed on SiN windows. XFM scans were performed at the beamline 2-ID-E at APS and 5-ID at BNL. The results showed that platinum signal was higher in the cochlea after cisplatin i.v. injection than i.p. injection. A high platinum deposit was shown in the SV and nerve tissue. Weaker but obvious platinum signal was also observed in the organ of Corti, in which OHCs had weaker signal than the inner hair cells. These results indicate that SV might be the primary target in cisplatin-induced hearing loss, and the mechanism might due to the failure of maintaining the endocochlear potential due to the accumulation of toxic platinum in the SV.

The study is supported by NIH R01 (1R01DC019434-01A1) and the American Hearing Research Foundation awarded to XT. The research uses resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357. The work also used National Synchrotron Light Source-II which is supported in part by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences Program under contract number DESC0012704 (KC0401040).

CHEMISTRY

A-11

Influence of Lattice Stoichiometry on Electron vs. Hole-trapping Character of Sulfur in Anatase Titania

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The activity of a photocatalyst is dependent on the lifetime of the excited electron-hole pair, which can be extended by the separation of the electron from the hole [1]. There has been recent interest in improving photocatalytic activity in TiO_2 by using sulfur as a dopant to induce localized mid-gap states which can "trap" an electron or hole and thus achieve charge separation, but whether these states behave as electron traps or hole traps depends upon their occupancy and has been a point of confusion in the literature [2,3]. Understanding the speciation of sulfur which induces these states is critical to understanding how to reproducibly prepare and accurately model these states. Our group has recently provided the first direct evidence of mid-gap electronic states associated with sulfur in anatase titania [4]. UV-vis spectra for excitation of electrons from the trap state to the conduction band bear a remarkable similarity to predicted UV-vis spectra of sulfur substituted on Ti lattice sites [5], and the speciation was assigned accordingly. We also propose that the occupancy of these states is depleted with increasing titanium vacancy concentration. Thus, the states behave as electron traps if the lattice becomes electron-deficient due to a stoichiometric excess of oxygen.

To independently confirm the local environment and the effect of TiO_2 non-stoichiometry on the occupancy of the sulfur induced states, we performed sulfur K-edge x-ray absorption spectroscopy on several samples of sulfur-containing commercial anatase samples, with and without the trap states prepared via calcination. We find that the preparation of the trap states is associated with a sharpening of XANES features. We will discuss the local environment around the sulfur obtained from EXAFS fitting, as well as the assignment of two pre-edge features in the x-ray absorption spectra located at the surprisingly low energies of 2467 and 2471 eV, demonstrating an extremely electron-deficient environment around the sulfur.

[1] Rockafellow, E. M. et al. *Catal., B* **2009**, *91*, 554−562.
[2] Bakar, S. A. et al. *RSC Adv.* **2016**, *6*, 36516−36527.
[3] Vequizo, J. J. M. et al. *ACS Catal.* **2017**, *7*, 2644−2651.
[4] Rahmani-Chokanlu, A. et al. *J. Phys. Chem. C* **2023**, *127*, 6754–6767.
[5] Harb, M. et al. *J. Phys. Chem. C* **2013**, *117*, 8892−8902.

Investigation of Key Electronic States in d^0 Layered Mixed Transition Metal Chalcogenides

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The exploration of high-energy-density, long-lasting, and cost-effective battery technologies has driven extensive research into novel cathode materials, particularly those involving anion redox, which enhances the energy density of batteries by beyond contributions only from cationic redox. The anion redox in Li-rich 3d transition metal oxides materials has shown to result in an unconventionally high capacity. However, these materials suffer from irreversible capacity loss and voltage fade due to severe structural transformations resulting from unstable electronic configurations. In this context, sulfides and selenides have been identified as attractive cathode materials, but little is known about the underlying electronic phenomena responsible for their electrochemical activation.

In this work, we investigated the underlying electronic and structural evolution in $Li_2TiS_{3-y}Se_y$ (y = 0, 0.6, 1.5, 0.5 and 3) cathode materials for lithium-ion batteries using ligand and metal x-ray absorption spectroscopy (XAS) and extended x-ray absorption fine structure (EXAFS). This work presents spectroscopic evidence of the formation of S_2^{n-} and Se_2^{2-} from oxidation during the charging process and sheds light on the specific states contributing to the electrochemical capacity of $Li_2TiS_{2.4}Se_{0.6}$. These findings provide insight into the trends in electrochemical activity with the amount of Se^{2-} substitution and can aid in the continued development of high-capacity cathode materials based on environmentally friendly elements.

A-10

Development of Time-resolved X-ray Absorption Spectroscopy at APS 25-ID-E

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The time-resolved x-ray absorption spectroscopy (TR-XAS), developed from static XAS within a laser pump-x-ray probe configuration, allows us to concurrently measure the photoinduced electronic and structural dynamics in materials with the picosecond time-resolution and element and site sensitivities at the atomic level. This information is crucial for the design, fabrication, and application of materials for photovoltaics, optoelectronics and photocatalysis. With the accomplishment of APS-Upgrade, it opens doors to develop some new, previously inaccessible TR-XAS capabilities. In this talk, the TR-XAS capabilities that have been developed at APS 25-ID-E based on current integration and photon counting methods will be presented through a few scientific examples, which studied solution, nanocrystal, and thin film samples. Additionally, the future development of TR-XAS with novel asynchronous x-ray multiple-probe data acquisition technique will be included. A setup derived from TR-XAS sample platform for TR-XRD measurements will be also introduced briefly, which can be used to characterize the photoinduced phase change of nanocrystal samples.

ESRP: Not So ‘Forever’ Chemicals: A Study on Removing PFAs from Contaminated Water through Various Filtration Materials

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Per- and polyfluorinated substances, also known as ‘Forever Chemicals,’ such as PFAs and PFOAs, have become increasingly present in our daily environment. These chemicals are widely used in everyday products and are toxic even at low levels, increasing the risk of cancer by damaging the liver and immune system. Chemically, they are synthetic organofluorine compounds that consist of multiple fluorine atoms attached to an alkyl chain. They are not biodegradable, making it difficult to dispose of them safely. This research aimed to find effective methods to filter PFOAs out of water. Several materials were tested: coffee grounds, Biochar (fertilizer), ground coconut, sawdust, rayon, and cut-up water bottles. Each filter sample was 5g. The perfluorooctanoic acid solution included 0.5L distilled water and 0.5g of PFOA, representing concentration found in highly contaminated areas. The solution was heated and stirred until the PFOA had completely dissolved into the water. Three trials were conducted per filter sample, including a base trial where no filtrate was present, and the solution went through filter paper, simulating the filter-sample-included trials. Each sample was assessed by the infra-red spectroscopy in attenuated total reflectance mode (ATR-IR) and high-performance liquid chromatography (HPLC). The FTIR results show bands that could be assigned to vibration modes encountered in the PFOA compound for all filtrates. The HPLC results showed the presence of PFOA in the filtrate, and quantification analysis is being carried out to determine the exact amount of PFOA removed. Both results indicated the filtrates were capable of removing some amount of PFOAs from the sample. The best filtration materials were coffee grounds and Biochar, while plastic bottles and rayon fabric were the worst at filtering out PFAs, filtering out as much as regular filter paper. Sawdust and coconut were also not effective options. No options fully filtered out PFAs in the first filtration attempt; however, both coffee grounds and Biochar were somewhat successful.

U-55

Molecular Understanding of CO_2 Capture and Conversion at The Air/Aqueous Interface

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Negative emissions technologies (NETs), such as direct air capture (DAC), are considered essential for combating climate change, caused by carbon dioxide and other greenhouse gases. Addressing this challenge require a wide range of tools, in addition to the commonly used DAC sorbents such as amines and alkali and alkaline earth hydroxides. For instance, d^0-transition metal ions such as peroxovanadate and orthomolybdate anions have been reported as potential candidates for CO_2 DAC [1,2]. Given the crucial role of the interface as the first gate to the capture of CO_2 and transfer into the bulk of the DAC sorbent solution, molecular understanding of the air/liquid sorbent interface and related interfacial processes during capture events is necessary for facilitating and enhancing CO_2 capture and conversion at the interface and in the bulk.

In this work, we use surface-specific vibrational sum frequency generation spectroscopy (SFG), to probe CO_2 capture and conversion at the air/aqueous orthovanadate interface in a CO_2-saturated atmosphere. *In-situ* SFG measurements reveal CO_2 enhancement at the air/aqueous interface over a wide range of orthovanadate concentration, seen in the appearance of the asymmetric stretching signature of CO_2. Interestingly, the CO_2 signature is lost at 2.0 M orthovanadate, possibly due to the binding of CO_2 to the orthovanadate and conversion to carbonate. Upon longer exposure of highly concentrated orthovanadate solution to CO_2, we see evidence of carbonate formation at the interface. The results provide important physical insights into reactivity and transport of CO_2 with oxo-anions at aqueous solution interfaces.

This work was supported by the Department of Energy under the grant DE-SC0022278.

[1] Ribó, E.G., et al., *Implementing vanadium peroxides as direct air carbon capture materials.* Chemical Science, 2024. 15(5): p. 1700-1713.
[2] Knopf, I., et al., *Uptake of one and two molecules of CO2 by the molybdate dianion: a soluble, molecular oxide model system for carbon dioxide fixation.* Chemical Science, 2014. 5(5): p. 1772-1776.

CONDENSED MATTER PHYSICS

A-12.....Chu, Zhaodong
A-13.....Dayeen, Fazle
C-43.....Durham, Daniel
C-44.....Koll, William

A-12

Revealing Surface Soft Phonons in Quantum Paraelectrics

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Understanding surface dynamics in quantum materials is crucial for advancing quantum technology. However, the detection of surface vibrational modes at meV energies remains challenging due to the ultralow sampling volume and the needed spectroscopic resolution. Here, a surface-sensitive terahertz spectroscopy based on spin-polarized ultrafast electron transport is developed to uncover the softening of surface transverse optical phonons in two incipient ferroelectrics: $KTaO_3$ and $SrTiO_3$, providing unprecedented spectroscopic and time-domain details of surface phonons. For example, the softening of the transverse optical phonon in $KTaO_3$ deviates from the Curie-Weiss law and levels at finite energy, in agreement with quantum paraelectricity. In contrast, the transverse optical phonon in $SrTiO_3$ continues to soften and its frequency extrapolates to zero. However, the phonon peak broadens significantly below 35 K, indicating the fluctuation of soft phonon modes in the quantum paraelectric phase regime. Our findings underscore the ultralow-energy and surface sensitivity of this technique for revealing unique quantum states at surfaces and interfaces.

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A-13

Interfacial Structure of a Pulmonary Surfactant

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We study the structure-function relationship in pulmonary surfactants. Calf Lung Surfactant Extract (CLSE) was chosen for this purpose because it is presumed to closely resemble human surfactants. CLSE comprises a blend of dipalmitoyl phosphatidylcholine (DPPC), lipids, and surfactant proteins that reduce surface tension within the lungs and are an important element for preparing therapeutic surfactants for treating infants who suffer from pulmonary edema. Previously, it was thought that the condensed surfactant phase, which resisted collapse, consisted solely of DPPC, with cholesterol playing no role. Thus, nearly every manufacturer of commercial surfactant replacements eliminates cholesterol from their formulations, a practice we believe to be incorrect. However, our findings (at both 22 and 37 degrees Celsius) indicate that the structure of the condensed phase formed by DPPC/chol is notably distinct from that of DPPC alone, particularly when $X_{chol} > 0.05$. Grazing incidence x-ray diffraction (GIXD), specular x-ray reflectivity (XR), etc., are used to understand different kinds of phase transition and molecular structure within crystalline domains. Compression on a Langmuir trough altered both the interfacial density and surface tension, while GIXD offered insights into the structural characteristics and x-ray reflection shows that the film remained in a single molecular layer. We observe a striking similarity between the structures of CLSE and DPPC/chol mixtures (with X_{chol} approximately 0.25 or 0.3). Additionally, we examine how proteins and temperature influence the structure of CLSE. Our findings suggest a correlation: the structure of the condensed phase in pulmonary surfactant closely resembles that of the DPPC/cholesterol mixture, while most other surfactant components are predominantly in the disordered phase. We have demonstrated that cholesterol indeed integrates into the crystalline phase and plays a significant role therein.

Electrical Switching Dynamics of Charge Density Waves Visualized by Ultrafast Electron Microscopy

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Electrical switching of charge density waves is of great interest because the underlying electron-lattice interactions potentially open new, efficient pathways for electronic control. However, the switching mechanisms are often debated as electric field, current, carrier injection, heat, and strain can all contribute across varying length and time scales. I will present our recent experiments which visualize and disentangle these factors during voltage pulse switching of the room temperature, nearly commensurate charge density wave in 1T-TaS_2. We employed a unique voltage-triggered ultrafast electron microscopy mode recently developed at the Center for Nanoscale Materials [1]. This allows to record diffraction patterns and real-space images during electrical pulse stimulation with both nanometer spatial resolution and nanosecond time resolution. The charge density wave order dynamics following voltage pulses down to 20 ns duration are consistent with a Joule heating mechanism, showing robustness to field and carrier-injection effects. On the other hand, imaging reveals MHz-scale acoustic resonance modes which appear to modulate the order through dynamic strain. These measurements clarify the switching process at room temperature in TaS_2 and motivate further exploration of the dynamics during electrical switching across the myriad of charge density wave and other strongly correlated systems.

This material is based upon work supported by the U.S. Department of Energy, Office of Science, for support of microelectronics research, under contract number DE-AC0206CH11357. Work performed at the Center for Nanoscale Materials, a U.S. Department of Energy Office of Science User Facility, was supported by the U.S. DOE, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

[1] TE Gage & DB Durham, H Liu, S Guha, I Arslan, and C Phatak. “Nanosecond electron imaging of transient electric fields and material response.” **2023**. arXiv:2306.01171.

C-44

Towards RF-coupled Scanning Tunneling Microscopy of Single Molecule Spins

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Electron and nuclear spins are promising degrees of freedom for quantum computation and quantum sensing applications. Spins in metal-ion coordination complexes have the added advantage that their local environment is tunable via the chemical design of their molecular ligands. We have demonstrated that single molecule magnets such as vanadyl phthalocyanine (VOPc) can be studied on both metal (Ag) and insulator-on-conductor (hBN) surfaces using scanning tunneling microscopy (STM). At sub-monolayer coverage, we observe two distinct adsorption configurations of isolated molecules, corresponding to the central O atom pointing toward (O-down) or away (O-up) from the substrate. At higher coverage we find a strong preference for bilayer formation with O-up and O-down molecules in alternating layers, suggesting stabilization by dipolar interactions. We are now extending this work by implementing radiofrequency (RF) transmission to the tunneling junction, which in principle should enable the detection and coherent control of electron spin transitions with atomic precision.

This work was supported by funding from the National Science Foundation (NSF) QII-TAQS under award No. MPS-1936219, and by the Asian Office of Aerospace Research and Development (AOARD) (FA2386-20-1-4052).

ENVIRONMENTAL SCIENCE & GEOLOGY

ESRP-14

ESRP: Iron Nanoparticles in Common Plants, Fruits, and Vegetables

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Iron is an essential micronutrient in plants because of its use in various physiological and biochemical pathways. As the fourth most abundant element in the Earth's crust, iron is naturally found in soil, but the iron that is up-taken by plants comes from secondary oxides that were either absorbed or precipitated. However, iron is highly toxic in plants when seen in excessive amounts. Excess iron intake leads to stunted root systems and leaf discoloration. Iron toxicity may also affect uptake and distribution of other elements in the plant tissues, most notably potassium, disrupting the growth of the plant which is vital to plant health. Recent concerns cover iron-rich pollution nanoparticles, which are being increasingly emitted from industry and traffic-related sources. This research would show how these nanoparticles would interfere with the structure and inner workings of plants. Iron in the human body is a necessity, but excessive amounts of iron can be detrimental. If iron is consumed in high doses, it can lead to health issues such as organ failure, diabetes, and heart failure.

Through Lemont High School's Exemplary Student Research Program (2018-2023), students have the ability to work closely with Dr. Olga Antipova of Argonne National Laboratory to examine the relationship between the contamination of common plants, fruits, and vegetables and toxic metals such as chromium, cobalt, and arsenic. For the past research at Lemont High School, ESRP students used the APS technology at the 2-ID-E beamline. Students were able to examine the growth of arabidopsis, lettuce, tomato, and carrot in agarose gels and the effects of different toxic metals on root uptake. This year, we have explored other tools to track the effects of the iron nanoparticles in these plants. Using the technology at the CNM, students have evaluated structure and composition of nanoparticles and in leaves and roots using TEM, SEM, SEM EDX, and Raman spectroscopy, with focus on porousness of the surface of the root and to find the location of the uptake.

Elastic Property Changes in NaAlSi2O6 Glass: Insights from High-pressure Brillouin and Raman Spectroscopy

Young-Jay Ryu[1], Vitali Prakapenka[1], Yanbin Wang[1], Tony Yu[1], Stella Chariton[1], Peter Eng[1], Joanne Stubbs[1], and Mark L. Rivers[1]

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Silicate melts are the ubiquitous components of igneous processes in the Earth's crust and mantle, and they serve as a crucial key transport agent for physical and chemical differentiation and potentially produce dynamic scenarios of the Earth's interior. In order to understand the origin and significance of deep melts in Earth's interiors, it is imperative to thoroughly explore the fundamental roles of phase relationships of silicate melts and/or glass play in shaping the Earth's crust and mantle. Unfortunately, the experimental difficulties posed by the high-pressure and high-temperature environment have led to limited knowledge regarding the structures and properties of silicate melts and glasses throughout the Earth's pressure regime. Understanding the properties of amorphous silicates under pressure, encompassing densification, viscosity, chemical composition, and thermal differentiation, is intimately tied to changes in the structures and coordination numbers of silicate melts and glasses. Jadeite is the second major component of the Earth's upper mantle in volumetric distribution [1]. In fact, it is known that most of the subducted crusts are containing aluminous with incompatible alkaline metals, containing sodium has an important role in the fundamental studies of understanding the subducted crust and how they recycle within the deep Earth [2]. In this study, we have investigated the pressure-dependent elastic properties of $NaAlSi_2O_6$ glass up to 80 GPa. The experiments were performed at GSECARS 13-BM-D using the Brillion spectroscopy technique. Poisson's ratio and bulk, shear and Young's moduli are calculated as a function of pressure. Notably, the longitudinal and shear velocities reveal signatures of structural modifications around ~2, ~6, ~9, ~22, ~40, ~60, and 80 GPa. Additionally, Raman spectra of the glass were collected up to 70 GPa, providing further insights into its structural modification under pressure.

[1] Gasparik, T. (1992) *Contr. Mineral. And Petrol*. 111, 283-298.
[2] Green, D. H and Falloon, T. J (2015) *Sci. Bull*., 60, 1873-1900.

ESRP-17

ESRP: Bioremediation of Polyurethane in Water Using *Pestalotiopsis Microspora*

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As plastics are ubiquitous in consumer products, microplastics are a very common and prevalent problem, and will be long into the future. Plastics can end up in water and soil, where they leach chemicals and contaminants into these natural resources. A specific plastic, polyurethane, is used for furniture, thermal insulation, bedding, and in some medical equipment. Products like these eventually make their way into landfills and take decades to fully decompose and break down into polyurethane microplastics.

Researching how polyurethane interacts with water can lead to ways to treat this contamination. While the fungus *Pestalotiopsis microspora* has displayed potential in decomposing polyurethane, there is currently no research confirming its effectiveness in water; however, exploring its efficacy in aquatic environments is our intended focus for testing. In our project, we tested *Pestalotiopsis microspora* in simulated salt water, real river water, and deionized water samples, aiming to see some form of degradation of polyurethane in water. We analyze using Raman spectroscopy analysis and mass comparisons.

We want to thank Ben Diroll, The ESRP program, Kelly Sturner, Sandy Scott, and the West Aurora Science Department.

ESRP-87

ESRP: Creating Rubies in a Microwave Using Aluminum Oxide, Chromium Oxide, and Brillo Pads

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Chromium (III) oxide and aluminum oxide, undergo a chemical reaction resulting in the formation of rubies. This reaction is a typical example of metal-nonmetal interaction yielding ionic compounds characterized by positively and negatively charged ions derived from neutral atoms and molecules through electron transfer. Specifically, when chromium (III) oxide is subjected to high temperature in the presence of aluminum oxide, chromium atoms displace aluminum atoms periodically within the crystalline lattice. This introduction of chromium impurities serves as the fundamental structural units in the formation of rubies.

Our experimentation involved varying ratios of aluminum oxide, chromium (III) oxide, and a catalyst, such as a Brillo pad, enclosed in crucibles of different sizes. We opted for microwave heating due to its efficiency and widespread accessibility, capable of generating plasma. Among the nine samples produced, two exhibited promising characteristics resembling rubies. To assess their resemblance to rubies, we evaluated their fluorescence properties, identifying samples two and five as the most akin to rubies. Further analysis through techniques such as Raman spectroscopy, fluorescence spectroscopy, scanning electron microscopy, and x-ray diffraction was conducted on samples two and five.

We want to thank Ben Diroll, Kelly Sturner, Julie Zaborac, Sandy Scott, and the West Aurora Science Department.

[1] “Oxidation states of Chromium | MEL Chemistry” https://melscience.com/US-en/articles/oxidation-states-chromium/ Accessed Mar 21, 2024.
[2] “Science of Stones: Ruby- International Gemstone Association” https://www.gemstone.org/science-of-stones-ruby Accessed Mar 21, 2024.

A-16

Feature-specific Segmentation Approaches for Highly Variable Micro-CT Datasets in Earth Science

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Synchrotron micro-computed tomography (micro-CT) enables the rapid acquisition of large, highly detailed 3-dimensional (3D) datasets. However, as 3D images increase in size and number, they become less feasible and more prohibitive to manually segment and analyze while thoroughly probing all relevant elements. The need for robust automated analysis pipelines capable of interrogating the complexities of 3D image data beyond qualitative assessment of representative slices has given rise to multiple automated and semi-automated segmentation approaches. Image data processing tools such as intensity thresholding, watershed expansion, filtering, and edge detection have been assisting automated segmentation efforts for decades. Such methods have been explored to the point of creation of commercial and open-source software for easy use such as ImageJ, SimpleITK, Avizo, and Dragonfly. However, the need to segment complex or noisy datasets, such as those composed of multiple similar material types or acquired with difficult, artifact-prone techniques, prompted the development of new automated segmentation tools to deal with the varying differences in contrast levels, textures, and intensities. Combinations of traditional approaches with newer programs using machine learning algorithms such as Ilastik allow for solutions to specific image segmentation and classification problems containing highly varied volumes of interest. Here, we present our approach to such semi-automated segmentation methods as applied to image datasets acquired at GSECARS containing shear-induced faults, melt inclusions, and granular substrates. Such methods can save resources and standardize the process across multiple images, allowing downstream quantitative characterization based on attributes such as shape, texture, abundance, or location of the volumes of interest.

HIGH PRESSURE

A-20

Exploring Diamond Inclusions via Combined Microtomography and Single-crystal X-ray Diffraction at GSECARS

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Most of our knowledge on the Earth's deep interior is obtained through surface observations and geophysical surveys in combination with experiments and computational models. Diamond inclusions represent a direct window into the deepest layers of our planet and although rare specimens, the information they carry is invaluable in determining the mantle chemistry and understanding processes such as mantle convection and volatile cycling. A suite of techniques may be used to study diamond inclusions and to identify the phases entrapped. However, many are considered destructive, often requiring exposure of the inclusion to the surface by diamond polishing. Such approaches pose a significant risk to the analysis and results interpretation due to possible alteration of the inclusions by exposure to the atmospheric air and pressure as well as loss of fluid phases. Synchrotron single-crystal x-ray diffraction (SCXRD) is a powerful and unique tool in unravelling complex mineral assemblages by allowing accurate structure determination and phase identification. However, studying sub-surface inclusions (i.e., not exposed to the diamond surface) presents a challenge during alignment procedures, especially in the cases where the entrapped phases are weak absorbers. At the 13-BM-D beamline at GSECARS we combined microtomography and SCXRD to approach this problem. Diamonds are first scanned with pink or monochromatic beam producing a series of high contrast absorption images, followed by a filtered back-projection analysis that produces their 3D reconstructions revealing sample features such as density contrast, cracks, zoning, fluid rims, etc. This allows the precise visualization, location, and placement of selected inclusions on the rotational axis aligned with the x-ray beam to collect high resolution SCXRD patterns. This well optimized user-friendly procedure is ideal for pilot and advanced measurements on rough unpolished diamonds or other samples without invading the microenvironment of the inclusion, thus providing critical information essential for understanding the structure of the deep Earth's interior. We will present the detailed methodology, discuss the merits and limitations as well as the developments that the APS-U synchrotron upgrade will offer on this dynamic duo of techniques.

Cutting-edge Upgrade of Diamond Anvil Cell Program at GSECARS

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The Advanced Photon Source is undergoing a complex upgrade replacing its original electron storage ring with a new multi-bend achromat lattice to provide extremely enhanced coherent flux and increased hard x-ray brightness by a hundred times. To take full advantage of these improved capabilities we have started the process of comprehensive technical improvements and developments of multiprobe techniques across a suite of beamline stations at GSECARS.

The laser-heated diamond anvil cell technique combined with high energy tightly focused x-ray beam and fast detectors for spectroscopic measurements is the workhorse method for exploration of deep Earth mineral physics and chemistry. To provide new constraints on models for planetary evolution and origin, essential properties (melting, structure, phase relation, chemical reactions and kinetics, transport, elastic, electronic and optical properties) of a wide range of minerals must be studied *in situ* at extreme conditions of pressure and temperature. However existing data sets are often inconsistent or too poorly constrained to provide unique answers. This underscores the need for unique beamline capabilities: high-energy high-flux sub-micron x-ray beam to probe ultra-small samples (micron-sized) in megabar pressure range, high resolution large area fast x-ray and optical detectors for time-domain experiments, sample emissivity and absorption measurements to improve radiative temperature metrology, etc. To accomplish that we will construct a vibration-free granite-table system at 13-ID-D station, install a pre-shaped sub-micron x-ray focusing system (300 nm) coupled with high precision sample positioners including an air-bearing rotary stage, replace our optical spectrometer and acquire a new Eiger2 CdTe 9M X-ray detector. We plan to upgrade the x-ray and laser optics (Raman, Brillouin, fluorescence, absorption) at 13-ID-D, 13-BM-C, and 13-BM-D stations to accommodate higher x-ray energies in a tighter focused beam, which grants a significant boost in the reciprocal space explored at high temperatures with increased spatial resolution.

Recent results and details of future developments of the cutting-edge synchrotron and optical techniques for comprehensive characterization of materials in-situ at extreme conditions will be discussed.

Pressure-tuned Structures and Optoelectronic Properties of a Dion-Jacobson Type 2D Hybrid Organic-inorganic Lead Iodide Perovskite

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Two-dimensional (2D) Hybrid organic-inorganic perovskites (HOIPs) exhibited enhanced chemical and thermal stability against humidity and light compared to their 3D counterparts. The use of external high pressure can induce modifications to the structures allowing for tuning of the optoelectronic properties. Here, we report the first high-pressure study on $DPDAPbI_4$ (DPDA = N,N-dimethylphenylene-*p*-diammonium), a Dion-Jacobson type 2D HOIP with a highly rigid DPDA dication as the organic spacer. We used photoluminescence (PL), UV-visible spectroscopy, vibrational spectroscopy, and *in-situ* synchrotron powder x-ray diffraction (XRD) to study the structures and optoelectronic properties at high pressure in a diamond anvil cell (DAC) assisted by density functional theory (DFT) calculations. An isostructural phase transition around 1.5 GPa is observed, evidenced by a prominent change in both the intensity of the major PL peak originated from emission of free excitons, along with further vibrational spectroscopy measurements. Furthermore, synchrotron XRD measurements indicate considerable anisotropic compression along the *c* axis. Surprisingly, structural analysis shows a decrease in lead-iodine octahedral distortion in the low-pressure region, in significant contrast to previously studied other 2D HOIPs. The DFT calculations help to further the understanding of the structural mechanisms behind the proposed phase transition and pressure-tuned optoelectronic properties. Our findings underline the significance of the interplay between a highly rigid organic spacer and the inorganic octahedra in tuning the optoelectronic properties at high pressure, which provides insights into the design of future 2D HOIPs with practical applications.

From Structure to Dynamics: High-pressure Crystallographic Research at the GSECARS 13-BM-C

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The GSECARS 13-BM-C beamline at the Advanced Photon Source (APS) has been providing crystallographic research capabilities to the high-pressure community since 2015. This beamline utilizes focused x-rays at two fixed energies: 15 and 29 keV, and a unique 6-circle heavy duty diffractometer. The instrument is optimized for a variety of advanced crystallography experiments including interface studies, powder and single crystal structure determination, equation of state studies and atomic dynamics research. Currently we support high-pressure and variable-temperature experiments using diamond anvil cells, resistive-/laser-heating and cryostats. We have achieved P-T conditions of 100 GPa and 150-3000 K. Results of multiple recent experiments, including powder and single crystal diffraction over a range of P-T conditions, equations of state, and atomic dynamics will be presented to demonstrate the experimental capabilities. These new capabilities are available to all researchers interested in studying deep earth materials through the APS General User Proposal system.

INSTRUMENTATION

U-56

Advancing Techniques in Structure-based Drug Discovery and Development at the IMCA-CAT Beamline

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The IMCA-CAT beamline at the Advanced Photon Source (APS) is undergoing significant upgrades aimed at improving data quality, precision, and throughput. The optical upgrades include new mirrors, compound refractive lenses (CRLs) and a new monochromator (Axilon AG). Other upgrades include new robotics (Rigaku) and a new goniometer (AIM Concepts). The integration of advanced technologies and robust automation into the beamline infrastructure promises to accelerate drug discovery and development processes for pharmaceutical companies, including IMCA members and IMCA-CAT subscribers. The implementation of state-of the-art mirrors with improved micro-focusing capabilities and the CRLs will permit rapid refocusing of the beam with little loss of flux in order to best meet the needs of each experiment. A new monochromator will result in improved energy resolution, beam stability and reduced background noise. Faster robotics and improved sample handling systems will allow streamlined data collection processes, significantly reducing experimental time and enabling higher throughput. Enhancements to the goniometer system improve sample rotation and translation precision, leading to more accurate data acquisition and analysis. These upgrades lead the way for future advances in protein crystallography, with automation playing a central role in facilitating higher throughput and accelerating drug discovery and development efforts. The upgrades to the IMCA-CAT beamline represent a significant step forward in the capabilities of the facility, which is poised to play a pivotal role in shaping the future of structure-based drug discovery and development.

U-57

The PILATUS4 Detector: Advancing High-energy Synchrotron Research

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The advancement of hybrid photon counting (HPC) x-ray detectors is pivotal for pushing the boundaries of synchrotron research, offering unparalleled detection capabilities without noise and with advanced acquisition modes [1,2].

The PILATUS4 detector emerges as a groundbreaking successor in this innovative lineage, setting new benchmarks for performance and versatility in x-ray science. Particularly compared to its predecessor, PILATUS4 distinguishes itself by combining frame rates of up to 4 kHz over the entire active area with the continuous readout technology. This combination ensures the highest possible duty cycle at high frame rates, which effectively increases the amount of collected photons by 2-fold at 8-fold higher frame rates, compared to its predecessor PILATUS3. Particularly for time-resolved experiments, these advancements will enable pivotal experiments at unprecedented temporal domains.

Here, we show examples of the PILATUS4 paired with the High-Z CdTe sensor used at 3rd and 4th generation synchrotron sources, including Spring8 and MaxIV.

[1] Förster, A., et al. (2019) *Philos. Trans. R. Soc. Math. Phys. Eng. Sci.* **377**, 20180241.
[2] Donath, T., et al. (2023) *J. Syn. Rad.* **30**, 723-738.

A-38

The Next-generation of Beamline Detectors: In-beam Pixelated Imaging

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[1]Advent Diamond, Inc., Scottsdale, AZ 85257

Real-time, in-beam measurements of x-ray beam characteristics, such as shape, focus, and spatial distribution is a challenge with current technology. This is especially a problem for experiments that require precise beam positioning and beam shape monitoring to ensure accuracy and reproducibility. Most in-beam detectors currently used have at most four-pixels, or quadrants, which can be used for positioning, but only provide limited information about in-beam features and structure. To overcome these limitations, Advent Diamond developed the ClearXCam^{TM} video-rate, transparent in-beam imaging monitor, with 2304 pixels.

In this presentation, we will show recent analysis highlighting the performance and capabilities of the ClearXCamTM in-beam detector system. Results from the Brockhouse beamline at Canadian Light Source (CLS) and the 17-BM XFP beamline at NSLS-II demonstrate successful imaging of an x-ray beam in real-time across a wide dynamic range from 10^7 to 10^{15} photons per second.

U-58

Performance of KETEK's Multi-channel Silicon Drift Detector Array 3.0 for Large Area XRF-Applications

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[1]KETEK GmbH, 81737 Munich, Germany

KETEK, the leading manufacturer of Silicon Drift Detectors, presents its new Array 3.0, which supports up to seven VITUS SDD modules and combines very large area, excellent energy resolution and high-count rate capability by a single compact system with no need for additional equipment such as vacuum pumps or chillers.

The Array 3.0 can be freely configured with three to seven vacuum-encapsulated SDD modules, with collimated areas between 7 mm² and 50 mm² each, resulting in a total collimated area of up to 350 mm². The system can be operated at input count rates up to 1 Mcps per channel (7 Mcps in total) at peaking times down to 100 ns. Typical energy resolution of each channel is <127 eV @ 1 µs peaking time for Mn Kα-line.

All SDD modules feature KETEK's high-end charge sensitive amplifier (CSA) as well as KETEK's proprietary CH- or CL-Graphene windows with excellent x-ray transmission from 0.1 keV (CL-type) upwards.

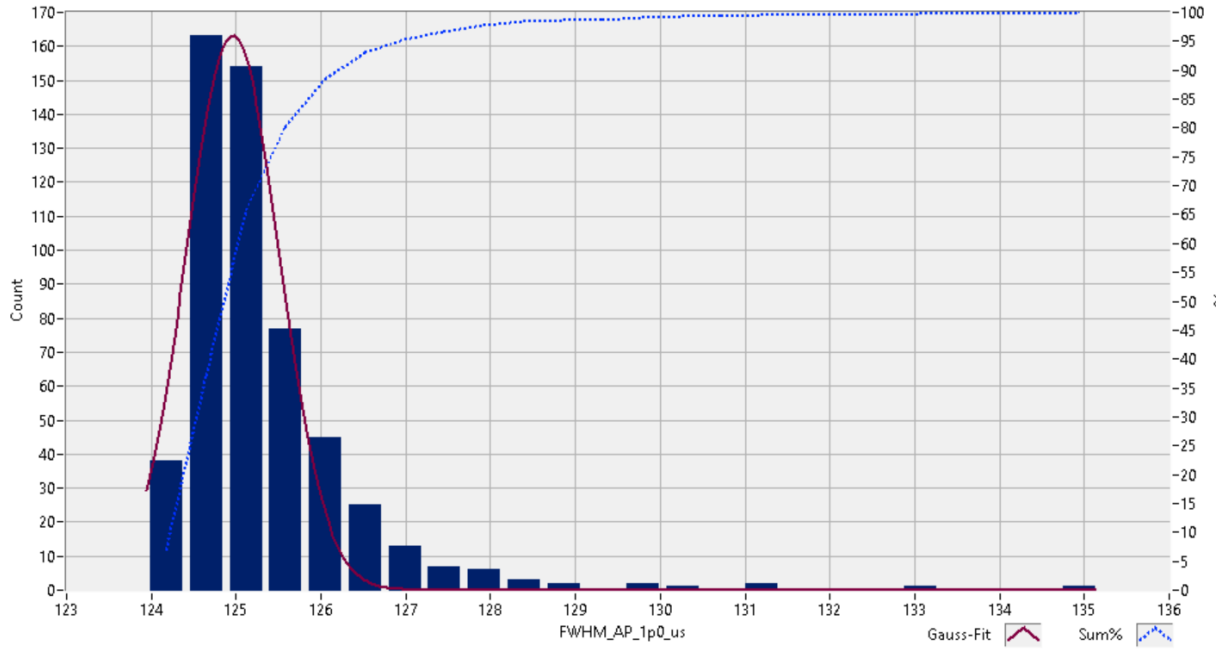
We will further present KETEK's latest electronics generation 3.0, including the brand new DPP3.0 digital pulse processor, offering 25 ns as shortest peaking time for super-high input count rates up to 4 Mcps and throughput at < 60% dead time. On the other side an excellent energy resolution down to 123 eV (Mn Kα-line) can be achieved at 1 µs peaking time.

V000-KGN0-H050-ML6C Messdaten

TDMS-Filter:
Artikelnummer = "V000-KGN0-H050-ML6C"
Messart = "Endtest"
Spezifikation = "passed"

FWHM_AP_1p0_us

Histogramm mit 24 Klassen aus 540 Werten (0 NoValues ignoriert).



Min	Max	Range	Mean	Median	Sigma
123.94	135.17	11.226	125.33	125.05	1.0945

C-45

Lab-ready Instrumentation from Quantum Design

Rick Hapanowicz[1]

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Since 1982, Quantum Designs has been providing lab ready scientific instruments to colleges, universities, and government laboratories around the world. Instruments include the DynaCool Physical Property Measurement System (PPMS), the MPMS3 SQUID Magnetometer and VersaLab Physics Education System. The OptiCool is a large volume, low vibration, low temperature, and high magnetic field cryogen free environment for magneto-optical investigations. The FusionScope is a correlative microscopy system for SEM, AFM and Elemental imaging of materials. These instruments are made in the USA and were designed and developed by Quantum Design's engineering team in San Diego.

The MPMS3 SQUID Magnetometer is Quantum Design's flagship instrument for magnetic measurement; this is the highest sensitivity commercial magnetometer with thousands of installations around the world. The DynaCool and VersaLab systems have over 20 experimental measurement options. These options allow researchers to quickly measure electrical, magnetic, thermal, and optical properties of materials. Many new materials have been analyzed in the PPMS systems including superconductors, quantum magnets, thermo-electrics, magneto-caloric, 2-dimensional materials, and many others.

In addition to Quantum Design’s efforts in building precision scientific instrumentation, we are also active in improving hands-on experimental learning for STEM students. Quantum Design launched The Discovery Lab Initiative which seeks to partner colleges and universities with leading technology companies to develop new curricula emphasizing hands-on experiential learning. By introducing industry-standard research instruments, students are inspired to "take theory into practice," thereby better training themselves to be successful in the next stage of their scientific careers. We created a website, discoveryteachinglabs.com, to promote this collaborative effort between academics and industry.

Quantum Design

MPMS®3

Quantum Design

RECYCLING HELIUM

PPMS®

DYNACOOL

OptiCool®

By Quantum Design

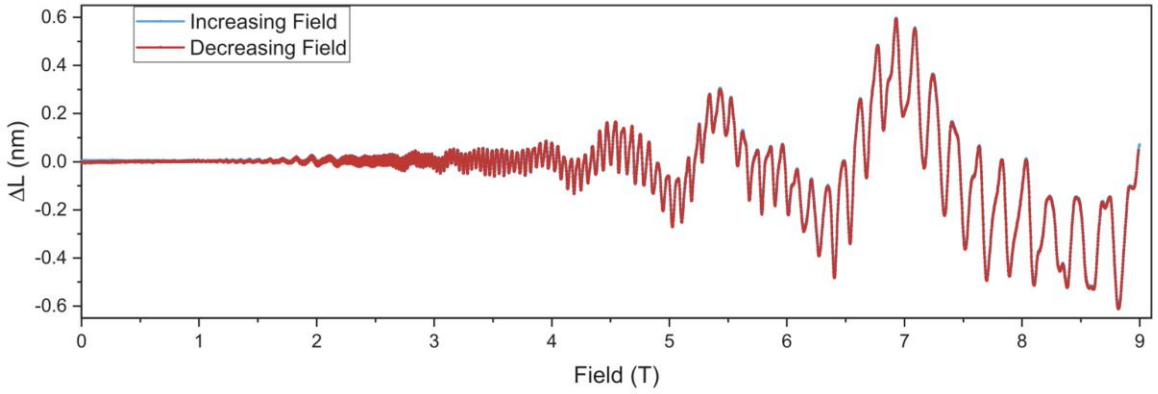
fusion scope

by Quantum Design

DilatoMeter

Aluminum magnetostriction at 2.0 K showing the De Haas-van Alphen Effect

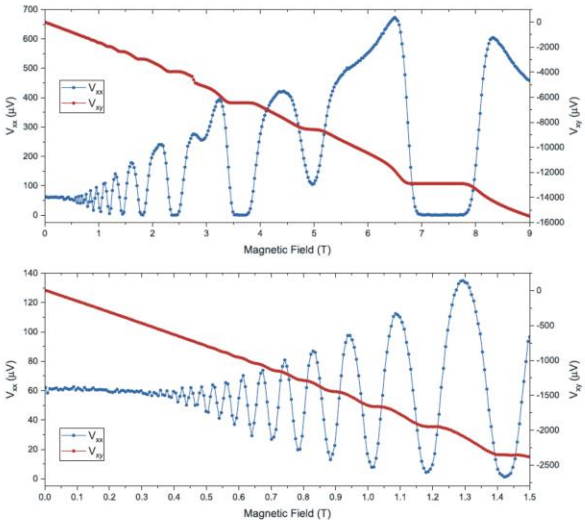
Sample Size:
2mm +/- 0.05
Temperature Range:
1.8 - 400 K
Magnetic Field:
Up to 16T
Noise Level:
70 ppm at 300 K
20 ppm at 2 K



M91 FastHall™ Measurement Controller

Quantum Design

Lake Shore CRYOTRONICS ADVANCING SCIENCE



Sample provided by Dr. M. Pendharkar, Chris Palmstrøm Group, UCSB

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DECTRIS Hybrid Photon Counting High Frame Rate Capabilities

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The latest high frame rate capabilities of DECTRIS's detectors are presented here. This includes the recent complimentary EIGER2 firmware upgrades, enabling features such as 8-Bit Mode readout and Lines-ROI, which pushes the frame rates beyond what was previously possible. In addition, an example of Double Gating Mode in Pump-Probe a diffraction experiment is shown. Finally, we give an outlook on the high frame rate capabilities of our latest developments that will be available soon.

Nuclear Resonant Scattering Studies at APS IXN Group

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Sector 3 at APS/ANL is a high-energy resolution spectroscopy beamline that develops and applies nuclear resonant scattering (NRS) methods to study architype and functional materials. Nuclear resonant inelastic x-ray scattering (NRIXS) method focuses on determining vibrational dynamics and elastic properties of samples, e.g., biomimetic materials like porphyrins as well as proteins and enzymes, materials that are of interest to catalytic cycle, energy storage and energy transmission, minerals and synthetic analogs relevant to Earth and planetary sciences, and samples in current condensed matter physics research. Synchrotron Mossbauer spectroscopies (SMS) in both time and energy domain are also used to reveal information about valence, ligand symmetry, and magnetism.

Sector 30 also offers a NRS program part time. Between the two sectors we have capabilities to measure NRS spectra from five different isotopes (Kr-83, Fe-57, Eu-151, Dy-161, and Sn-119) to cover vast classes of materials relevant to magnetism, superconductivity, piezo and thermoelectricity, high pressure sciences, geosciences, and biological systems.

Efficient high-energy resolution monochromator development is of the utmost importance to these types of spectroscopies. Our research activities also include development of novel instrumentations in new application areas, providing extreme conditions of temperature and pressure, micro-focusing strategies, and launching new applications in microscopy and imaging.

APS-U will deliver various enhancements to NRS programs, including better focused beam size, more flux, and beam quality and stability. A new energy domain Mossbauer microbeam spectrometer is being developed to enhance NRS capabilities at APS-U. We are looking forward to continuing all successful user research while expanding and broadening NRS studies with new users.

X-ray Optics for the Cavity-based X-ray Free-electron Laser Project

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A cavity-based x-ray free-electron laser (CBXFEL) is a possible future direction in the development of high-brilliance x-ray sources. CBXFELs consist of a low-emittance electron source, a magnet system with several undulators and chicanes, and an x-ray cavity. The x-ray cavity stores and circulates x-ray pulses for repeated FEL interactions with electron pulses until the FEL reaches saturation. CBXFEL cavities require low-loss wavefront-preserving components: near-100%-reflectivity x-ray diamond Bragg-reflecting crystals, outcoupling devices such as thin diamond membranes or x-ray gratings, and aberration-free focusing elements.

Here, we report on the design, manufacturing, and characterization of x-ray optical components for a CBXFEL x-ray cavity in a framework of a CBXFEL collaborative R&D project of Argonne National Laboratory, SLAC National Laboratory, and SPring-8. The cavity includes high-reflectivity diamond crystal mirrors with thin drumhead diamond membranes, Beryllium refractive lenses, and channel-cut Si monochromators. Diamond crystals with almost flawless 2×2 mm^2 regions of interest (ROI) were selected and furnished with strain-relief cuts for strain-free mounting. All crystals were characterized and exhibit small Bragg-plane slope errors of $\lesssim$ 0.2 μrad in the ROIs and small wavefront phase errors of $\lesssim \lambda/70$ on a footprint of 100 μm in diameter. In addition, diamond drumhead membranes with a thickness of < 20 μm were successfully manufactured. The Be lenses were characterized by x-ray phase-contrast imaging featuring wavefront errors $\lesssim \lambda/70$ on a footprint of 200 μm in diameter. Furthermore, 2-bounce and 4-bounce channel-cut Si monochromators have been successfully manufactured and tested to meet the desired performance and will be used as x-ray outcoupling monochromators to reduce the spontaneous radiation background and to single out x-rays with a small bandwidth in the peak of the cavity spectral distribution. These optical components are available to be installed in the CBXFEL cavity soon.

We are grateful to Michael Wojcik for support at the Advanced Photon Source 1-BM beamline and Elina Kasman for polishing the Si channel-cut monochromators. Sergey Antipov is acknowledged for machining the diamond drumheads. Ryan Lindberg and Yuanshen Li at ANL, and Gabriel Marcus and Zhirong Huang at SLAC, are acknowledged for helpful discussions. Work at ANL is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract No. DE-AC02-06CH11357.

X-ray Diagnostics for the Cavity-based X-ray Free-electron Laser Project

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A cavity-based x-ray free-electron laser (CBXFEL) is a possible future direction in the development of high-brilliance x-ray sources. CBXFELs are built upon repetitive FEL interactions between electron pulses in an undulator and circulating x-ray pulses in an x-ray cavity, consisting of near-100%-reflectivity x-ray Bragg-reflecting diamond crystal mirrors and aberration-free focusing elements. All cavity optical components have to be aligned with a precision ensuring that the to-be-amplified x-ray beam returning into the undulator (after traveling tens to hundreds of meters in the cavity and being refocused to a spot of a few tens of microns) meets a fresh electron bunch of a similar size and a few tens of microns in length. Here, we report on the design, manufacturing, and evaluation of x-ray diagnostics components for the precise CBXFEL x-ray cavity alignment and characterization performed in a framework of a CBXFEL R&D collaborative project of Argonne National Laboratory, SLAC National Laboratory, and SPring-8. The CBXFEL x-ray diagnostics include x-ray beam position and profile monitors (XBPMs), x-ray beam intensity monitors (XBIMs), and a meV-resolution x-ray spectrograph. Four types of XBPMs are used to monitor the beam position and profile along the entire cavity: coarse-alignment photo-luminescent YAG XBPMs, fine-alignment photo-luminescent minimally-invasive diamond XBPM, x-ray microscopes to monitor x-ray beams on the diamond crystal mirrors, and fast electronic quadrant diamond XBPM. To measure x-ray photon intensity in individual x-ray pulses, both ns-resolution Si-based XBIMs extra-cavity (behind diamond mirrors), and intra-cavity ns-resolution minimally invasive diamond XBIM are used. All the diagnostics have been tested and calibrated with pulsed x-rays at Advanced Photon Source (APS) beamlines 1-BM, 4-ID, and 7-ID, as well as at the LCLS. These diagnostics components will be installed inside or outside the FEL cavity to ensure the accuracy of the initial beam alignment, final fine beam alignment, and to characterize and optimize FEL performance.

We are grateful to Michael Wojcik, Alan Kastengren, Troy Lutes, Jeff Hoffman, Mark Engbretson, Keshab Kauchha, and Daniel Haskel (ANL) for support with experiments at APS beamlines 1-BM, 4-ID, and 7-ID. Kwang-Je Kim, Marion White, Ryan Lindberg, and Lahsen Assoufid (ANL), Zhirong Huang and Gabriel Marcus (SLAC) are acknowledged for helpful discussions and support. Yuri Shvyd'ko is indebted to Andreas Koch and Wolfgang Freund (European XFEL) for extensive discussions of the x-ray beam imagers, as well to Jen Bohon and John Smedley (LANL) for detailed information on the time-resolved diamond-based x-ray sensors. Work at ANL is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract No. DE-AC02-06CH11357. Work at SLAC is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Contract No. DE-AC02-76SF00515.

SparkPix-RT: A 100 kHz Charge Integrating Pixel Detector with On-chip Compression

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The SparkPix-RT is a novel Application Specific Integrated Circuit (ASIC) being developed jointly between Argonne National Laboratory (ANL) and SLAC National Accelerator Laboratory in collaboration to meet the requirement of developing a continuous real-time (RT) readout system with a high bandwidth detector rate. The SparkPix-RT is an advancement over traditional x-ray imaging ASICs implemented in 130-nm technology as it reduces the data size by performing data compression directly on the detector ASIC. It contains an array of 8 columns where each column comprises 4 clusters each having 12 x 6 pixels. Therefore, the total number of pixels are 48 x 48 with 100-micron pitch. Each cluster implements an ADC which samples the data at 8 MSPS. For every 2 cluster columns, one dual compressor is digitally implemented where data compression algorithms compress the high-bandwidth data which is then sequentially passed to a priority FIFO read scheme. At the end, data is encoded, serialized, and read out with the two differential outputs.

This work is based on work supported by the Accelerator and Detector R&D program in Basic Energy Sciences' Scientific User Facilities Division at the U.S. Department of Energy, under contract DE-AC02-06CH11357 and DE-AC02-76SF00515.

Hybrid Pixel Array Detector for Time-resolved and Imaging Applications with 56,000 fps Sustainable Frame Rate

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It has been more than ten years since HPAD (Hybrid Pixel Array Detectors) had been widely utilized as x-ray diffraction and imaging detectors. Thanks to its single photon counting capability, HPAD shows images without background noise and wide dynamic range. Due to limitations of the fabrication process, most HPADs are made with monolithic sensor and tiled readout ICs. In conventional HPAD, there were so-called “inter-chip pixels” on the edges of readout ICs. These inter-chip pixels have 1.5 times or wider width and/or height than non-inter-chip pixels. This means, we are losing position information of a hit of photons on those pixels. We have successfully dealt with this inter-chip pixel problem by use of re-distribution layer on the Silicon sensor. So, in our new detector, non-uniformity in a single sensor module is eliminated.

This new detector is designed based on UFXC32k IC [1] designed by AGH University of Science and Technology and named XSPA-500k [2]. XSPA-500k detector consists of 16 UHXC chips tiled and 1024 x 512 76 um sq. pixels per module. No inter-chip pixels in between ROICs which terribly suffer the image quality.

XSPA-500k is aiming not only for x-ray imaging but also for time-resolved x-ray measurements. Dealing with “inter-chip pixels” is our main feature for imaging, and for time-resolved measurements we understand that frame rate is as important as the size of the pixels and the area of the detector. Thanks to UFXC32k IC’s high count-rate and fast operation capability, combined with our high data throughput backend circuits, XSPA-500k is capable of up to 56,000 fps with full-frame readout and 100,000 fps with 100 lines ROI in the centre of the modules with continuous exposure (zero-deadtime mode operation with 2-bit counter/pixel). If the non-continuous exposure (burst-mode operation [3]) is allowed, it can achieve over 970,000 fps with approximately 2 % duty ratio.

[1] P. Grybos *et al.*, IEEE Trans. Nucl. Sci., vol. **63** (2016), no. 2, pp. 1155-1161.
[2] Y. Nakaye *et al.*, J. Synchrotron Rad. vol. **28** (2021), pp. 439-447.
[3] Q. Zhang *et al.*, J. Synchrotron Rad., vol. **25** (2018), pp. 1408-1416.

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X-ray Calorimetric Multimodal Instrumentation for General Thermodynamics and X-ray Science Applications

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X-ray calorimetric instrumentation combining precision heat capacity and synchrotron x-ray measurements makes a powerful analytical tool for understanding fundamental properties of novel materials. In this work, we discuss our novel spatial design and fabrication of precision calorimetric sensor chips, sensor mounting and packaging, room-temperature precision resistor circuits, cryostat sensor wiring, and precise/delicate placement of small samples under study on to the x-ray transparent membrane platform. Using our calorimetric sensors, we have designed experiments for precision heat capacity measurements of samples below 150 micro-gram in mass. We have characterized the reference and sample cell using relaxation and a.c steady state measurements. The reference and sample side thermometers were balanced during limited differential operation of the cell using SynkTek multichannel lock-in system. The close proximity of the thermometers allows accurate determination of the sample temperature. The measurements utilized six synchronized lock-in amplifiers and three a.c/d.c voltage sources measuring low-noise a.c/d.c currents and voltages of the two balanced on-chip sensitive thermometers and one low-power thin film heater. We report a.c heat-capacity measurements of empty reference cell from room temperature down to 3 K. We have also observed niobium superconducting phase transition using a.c temperature oscillations from the sample cell. We discuss determination of fundamental thermodynamic quantities from the measured low temperature electronic and lattice specific heat of Nb metal. The flexibility of simply placing the small samples without the need of direct electrical contact to the cell allows variety of scientific samples. The versatile x-ray calorimetric instrumentation will enable multimodal measurements of variety of technologically important materials with a range of physical properties including concurrent studies of phase or structural transitions covering a wide temperature range.

This research used resources of the Advanced Photon Source and Center for Nanoscale Materials, U.S. Department of Energy, Office of Science User Facilities operated for the DOE Office of Science by the Argonne National Laboratory under Contract No. DE-AC02-06CH11357. This project supports students from summer GEM fellowship and spring SULI program, 2024. We thank XSD, APS management team for financial support of the students.

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Moving on from VME without Breaking the Bank

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Most APS beamlines are using VME crates as a major part of their control system. These crates mainly provide the following functions:

- Motion control: Mainly OMS-58 and MAXv controllers
- Counters, timers, MCS: Mainly Joerger scalers and SIS-3820 multi-channel scalers
- Analog output: Systran DAC128V and others
- Analog input: Acromag IP-330 and others
- Digital I/O: SBS IP-Unidig and others
- Serial communication: SBS IP-Octal and others

The VME hardware is expensive and becoming obsolete, with replacements for many of the above items no longer available. The VxWorks software is also expensive.

I am leading an effort at Sector 13 to completely eliminate the 7 VME systems during the dark year. Once this is proven at Sector 13, XSD and other CAT beamlines can have confidence in doing this themselves as time and resources permit.

I plan to install the following to replace the VME systems:

- Galil DMC-4183 motor controllers to replace the OMS-58 and MAXv. Many of these will be used with existing Step-Pak or Phytron drivers. Others have on-board microstepper drivers (1.4A or 3.0A max current), or on-board servo drivers. The average cost is 5 times less than the new ACS motion controllers that have been used on new APS beamlines. They have advanced functions such as complex coordinated motion, support for absolute BISS encoders, programmable limit switch behavior, and software control of motor current and microstepping.
- Moxa terminal servers to replace the VME serial communication modules.
- Measurement Computing USB-CTR08 to replace the Joerger and SIS 3820 scalers and multi-channel scaler functions. This has 8 counter channels, 4 programmable frequency generators, and 8 digital I/O. I have written EPICS support for both the scaler record and MCS support like the SIS units. Cost is about $500.
- Measurement Computing USB-3104 to replace the DAC128V for analog output. These have 8 16-bit channels which be operated in voltage output or current output mode, and 8 digital I/O. Cost is about $500.
- Measurement Computing USB-1808X to replace the IP-330. This has 8 18-bit analog inputs, 2 quadrature encoder inputs, 2 programmable frequency generators, and 4 digital I/O. It can stream analog input data at up to 200,000 samples/s. Cost is about $1,000.

The total hardware cost to replace 7 VME systems for 2 FOEs and 5 experimental stations is less than $200K. This includes more than 370 motor channels.

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Advances in *Operando* and *In-situ* High-energy X-ray Scattering for Structural Science

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The Structural Science group at the APS operates a suite of four high-energy x-ray (HEX) beamlines dedicated to *in-situ* and *operando* experiments ranging from synthesis to studies of interaction of atomic order with macroscopic properties in functional materials to *operando* studies of complex real devices. In the last years, the program was highly demanded and contributed to more than 350 publications each year, which reflects the impact of HEX diffraction on fundamental and applied science.

After the upgrade of the APS, beamline 11-ID-D will complement this suite of beamlines, which will enable a combination of total scattering with small angle scattering and focusing in the sub micrometer range. With this, the length-scale gap between the resolution in reciprocal and real space to provide a complete picture of the structure of materials will be closed. Multimodal setups and photon energies between 26 keV and 120 keV with highest flux will enable complex *in-situ* and *operando* diffraction experiments. The priorities will be on the discovery of new materials by automated panoramic syntheses and the mapping of local atomic ordering during operation with high-spatial resolution to understand the role of inhomogeneities and interfaces on macroscopic performance, especially in energy storage and conversion systems.

Upcoming opportunities for research in materials science at the upgraded APS will be discussed.

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High-energy-resolution Inelastic X-ray Scattering Spectrometer at the Advanced Photon Source Beamline 30-ID

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Inelastic x-ray scattering is a powerful and versatile technique for studying lattice dynamics in materials of scientific and technological importance. An overview of the momentum-resolved high-energy-resolution inelastic x-ray spectrometer (HERIX), 1.5 meV energy resolution, at beamline 30-ID of the Advanced Photon Source (APS) will be presented, including the enhancement from the recent APS storage ring upgrade. In addition to the HERIX spectrometer at Sector 30, the beamline offers nuclear resonant scattering (NRS) capabilities. NRS is used to study atomic dynamics and electronic properties of archetype and functional materials. 30-ID NRS instrument focuses on tin-containing samples using isotope Sn-119.

Asymmetric Rowland Circle Geometries for Spherically Bent Crystal Analyzers: (Often) Better, Faster, Cheaper

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Due to their good energy resolution and large collection solid angle, spherically bent crystal analyzers (SBCA) see extensive use in hard x-ray photon-in/photon-out x-ray spectroscopy at synchrotron light sources. This includes especially measurement of high energy resolution fluorescence detection x-ray absorption fine structure (HERFD-XAS) and x-ray Raman scattering (XRS). For both of those methods, it has become common practice to use arrays of five to as many as several dozen SBCA to increase the detection solid angle.

In the recent study by Gironda, et al., (2024) [1], the authors demonstrate that SBCA perform well in asymmetric implementations of the Rowland circle, exhibiting several highly desirable characteristics. First, Johann error can be minimized and sometimes fully eliminated. Second, multiple crystal plane orientations can be used from a single SBCA, greatly increasing the energy range available from a single SBCA while correspondingly decreasing the need for a large number of SBCA exchanges. As a case in point, Zn HERFD-XAFS is performed using the preferred Si (642) reflection accessed asymmetrically from the more readily available Si (211) SBCA. This suggests new approaches to HERFD spectrometers that improve ease of use and decrease cost. Third, XRS is performed using a 0.5-m radius of curvature SBCA in an asymmetric configuration that removes Johann error. These results suggest a greatly simplified paradigm for future XRS spectrometers that can optimize both collection solid angle and compatibility with special sample environments without sacrificing energy resolution.

[1] A.J. Gironda, et al., “Asymmetric Rowland Circle Geometries for Spherically Bent Crystal Analyzers in Laboratory and Synchrotron Applications,” Journal of Analytical Atomic Spectroscopy,” 2024, DOI: 10.1039/D3JA00437F.

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A High-Speed, High-Resolution Transition Edge Sensor Spectrometer for Soft X-ray at the Advanced Photon Source

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This project explores the design and development of a transition edge sensor (TES) spectrometer for soft x-ray measurements in resonant soft x-ray scattering (RSXS) developed in collaboration between Argonne National Laboratory (ANL) and the National Institute of Standards and Technology (NIST). Soft x-ray scattering is a powerful technique for studying the electronic and magnetic properties of materials on a microscopic level. However, the lack of high-performance soft x-ray spectrometers has limited the potential of this technique. TES spectrometers have the potential to overcome these limitations due to their high energy resolution, high efficiency, and broad energy range. This project aims to optimize the design of a TES spectrometer for RSXS measurements and more generally soft x-ray spectroscopy at the Advanced Photon Source (APS) 29-ID, leading to an improved understanding of advanced materials.

We will present a detailed description of the instrument design and implementation. The spectrometer will consist of a large array of approximately 250 high-speed and high-resolution pixels. The pixels will have saturation energies of approximately 2 keV, pulse duration sub ms, and resolution sub eV. The array will be read out using microwave multiplexing chips with MHz bandwidth per channel, enabling efficient data throughput. To facilitate the measurement of samples in situ under ultra-high vacuum conditions at the beamline, the spectrometer will be integrated with an approximately 1 m long snout.

Characterization of prototype pixels will be presented.

This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science user facility, operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DEAC02-06CH11357. Work performed at the Center for Nanoscale Materials, a U.S. Department of Energy Office of Science User Facility, was supported by the U.S. DOE, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

The Hard X-ray *In-Situ* Nanoprobe Beamline at the Advanced Photon Source

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The *In-Situ* Nanoprobe (ISN) beamline at Sector 19-ID is a new feature beamline that is currently under construction as part of the Advanced Photon Source Upgrade (APS-U) in the newly built Long Beamline Building at Argonne National Laboratory [1,2]. The ISN beamline is designed for hierarchical multimodal studies of complex multiscale materials and systems under *in-situ* and *operando* conditions at very high spatial resolution, with close to atomic sensitivity. The ISN enables to tackle pressing research questions, *i.e.*, material stability, degradation processes, correlation of (in)homogeneities and performance, charging and discharging cycles, or defect properties, for applications in photovoltaics, energy storage and batteries, catalysis and microelectronics.

The ISN endstation instrument is placed 218 m from the x-ray source and is designed to deliver hard x-rays in the energy range from 5 – 30 keV via a crystal monochromator enabling access to elemental absorption edges of most elements. Kirkpatrick-Baez (K-B) mirrors are used as nanofocusing optics to focus the hard x-rays into a diffraction-limited spot of 20 nm at 25 keV. Additionally, it allows for a long working distance of 55 mm to study materials under *in-situ* and *operando* conditions. Available sample environments include heating and cooling capabilities, flow of liquids and gases, and applied electric fields.

The major contrast mechanisms are x-ray fluorescence (XRF) to study elemental distribution and trace elements, x-ray beam induced current (XBIC) and x-beam induced voltage (XBIV) to map electronic properties, ptychography for structural imaging with sub-10 nm resolution and auxiliary diffraction capabilities for structural information.

This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science user facility, operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

[1] Maser, J.; Andrade, V. D.; Kearney, S.; Knopp, J.; Lai, B.; Mooney, T.; Preissner, C.; Rebuffi, L.; Reininger, R.; Schmidt, O.; Shi, X.; Roehrig, C.; Shu, D.; Chen, S. Design of the *In-situ* Nanoprobe Beamline at the Advanced Photon Source. In *X-Ray Nanoimaging: Instruments and Methods V*; Lai, B., Somogyi, A., Eds.; SPIE, 2021; Vol. 11839, p 1183905. https://doi.org/10.1117/12.2596609.
[2] Kearney, S. P.; Bean, S.; Maser, J. Analysis of Vibration Isolated Facilities for the *In-situ* Nanoprobe at the Advanced Photon Source. *Synchrotron Radiation News* **2019**, *32* (5), 13–19. https://doi.org/10.1080/08940886.2019.1654827.

Hard X-ray Photoelectron Spectroscopy (HAXPES) in Material Development

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X-ray photoelectron spectroscopy is a powerful method to investigate the chemistry of surfaces and buried interfaces which are critical for batteries, solar cells, etc. As the energies of the excited electrons are not high enough to travel through the material to the detector. Hard x-rays have been increasingly useful in this field [1] due to the higher photon energies and significantly increased information depth. Scienta Omicron's HAXPES Lab uses a monochromatic Ga Ka metal jet source, enabling artefact-free investigations with superior information depth. Combined with a hemispherical electron analyzer with a ±30-degree acceptance angle [2], investigation of buried interfaces, *operando* devices, and real-world samples becomes easily achievable [3].

This presentation will give an overview of HAXPES applications with a focus on buried interfaces in electronic devices. Nanoparticles typically consist of a core surrounded by a protective shell, e.g., passivation layer in quantum dots for optoelectronics and bioimaging. The shell thickness and chemical composition highly influence the material properties. Herein, a series of PTFE nanoparticles with PMMA shell was investigated (nominal shell thicknesses between 4.5 and 35.5 nm). The core material was detected even for particles with greatest shell thickness using Ga Kα HAXPES.

[1] Kalha, J. Phys. Condens. Matter., 33 (2021) 233001.
[2] B. Spencer, Faraday Discuss., 236 (2022), 311.
[3] T. Hashimoto, Vac. Surf. Sci. 64 (2021) 493.

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High-pressure Microfluidic Cells for 3-30 MPa

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Thin membrane-delimited fluid cells are well-known and commercially available for working in vaccum chambers, such as used by electron microscopies, photon beam (e.g., x-rays) analyses or various particle beam spectroscopies. Such fluid cells need to support pressure differences of up to 1 bar, but many times chemical, biochemical, or physical processes happen at pressures of tens to hundreds of bar. Since higher pressures in fluid cells can be reached with thicker and smaller area membranes, this is not desirable, due to increase in beam absorption and signal limitations. Hence, solving a trade-off between membrane thickness and size, on one hand, and supported pressure, on the other hand, needs a careful engineering. We carried out a computer-based optimization of membranes and verified the solution by bulging and bursting experiments on microfabricated SiNx membranes. Finally, fluid cell prototypes were fabricated using 70-nm-thick membranes, engineered to withstand 46 bar (median value), compared to regular (un-engineered) membranes withstanding only 35 bar (median value). The fluid cell prototypes include 8 microchannels for feeding/evacuating the fluids or reactants, two electrodes for electrochemical or conduction measurements in the sample and possible pressure or temperature sensor, customizable for specific experiments. The pressure is supposed to be applied from the exterior through capillary fluid connections.

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Rapid Detection and Location of Rare Events from *In-situ* X-ray Diffraction Data Using Machine Learning

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High-energy x-ray diffraction techniques enable the 3D microstructure and its properties of metallic polycrystalline materials to be mapped non-invasively. These methods, often boosted by external factors like thermo-mechanical forces, facilitate the tracking of changes in material properties over time without damage. Yet, the extensive data volume and the high expenses related to conventional data collection and analysis have been significant hurdles in achieving quick, actionable insights and improving the frequency of these observations. To address these issues, we introduce an innovative, fully automated approach that drastically reduces the time required to identify the beginning of plastic deformation in high-energy x-ray microscopy data, offering processing speeds that are up to 50 times faster. Beyond just identifying rare events, our system also aims to pinpoint the specific grain exhibiting the anomaly. By leveraging simulation outcomes from MIDAS, we expect our method to highlight potential grains for further examination.

MATERIALS SCIENCE

The Role of the Dopant on Electronic Structure of Erbium-doped Oxides for Quantum Memory

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Quantum communication networks rely on qubits, such as photons, for secure long-distance communication. Rare-earth ion (REI) memory systems, particularly those employing erbium (Er^{3+}) in oxide hosts with C-band emission properties, play a pivotal role in synchronizing entanglement for signal amplification within quantum networks [1,2]. Oxides offer advantages such as growth simplicity, compatibility with complementary metal-oxide-semiconductor (CMOS) technology, and long coherence times. However, embedding Er^{3+} ions introduce defects that perturb the host lattice, leading to variations in photoluminescence linewidths and lifetimes in Er-doped oxide films, the causes of which remain unclear [3,4]. In this study, we utilize cutting-edge synchrotron-based x-ray tools from the Advanced Photon Source, including x-ray absorption spectroscopy and diffraction, to investigate the electronic and crystal structures of Er-doped titanium oxides across varying doping levels. This investigation is essential for understanding and controlling the tunability of excited state lifetimes and rare-earth defect linewidths, thereby mitigating decoherence effects in quantum communication systems.

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[1] Zhong, M. et al. Nature, 2015, 517, 177–180.
[2] Kanai S. et al., PNAS, 2022, **119**(15), e2121808119.
[3] Singh, M. K., et al., arXiv, 2022, preprint arXiv:2202.05376.
[4] Phenicie, C.M., et al, NanoLett., 2019, **19**(12): p. 8928-8933.

ESRP-67

ESRP: Proving Einstein's Theory of Relativity through the Michelson-Morley Experiment

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This experiment aims to prove Einstein's Special Theory of Relativity, including its citation of time dilation and length contraction, using the results of the Michelson-Morley experiment as a basis for research. Using the same materials as the Michelson–Morley experiment in the context of these experiments, this team collected data to prove the relationships outlined in the theory to show that when objects approach the speed of light, physical properties of time and space change due to light's constant speed.

The apparatus contained a laser, a half-silvered mirror (beam splitter), two plane mirrors, a slit, and a detector sheet. To begin the experiment, a laser was shot through the half-silvered mirror, splitting it into two separate beams that traveled perpendicularly. Those independent lasers reflected off the plane mirrors and passed back through the half-silvered mirror, reflecting onto the same path. That beam was then shone through a slit and onto a detector sheet, where multiple diffraction patterns presented themselves.

Analysis of the diffraction patterns will allow the determination of whether a present aether affects the speed of light. If a statistically significant difference is noticed between the speed of light and the recorded speed of light relative to the experiment, the presence of an affecting luminiferous aether is proven. This difference can be seen by comparing the placement of the diffraction patterns of the split beam. Identical patterns would suggest a constant speed of light while differing patterns would argue otherwise.

In previous years, this team performed spectroscopy, which provided information about crystalline structures. Through staying within the realm of optics, the study of light's nature is highlighted through this procedure. This experiment attempts to prove the existence of a medium that was theorized to impact the wavelength of light, and thus, its speed.

Through such relationships, the Michelson–Morley experiment establishes a baseline on the view of modern physics as a whole from the validity of Einstein's Theory of Relativity to past theories such as time dilation, the Doppler Effect, and the Photoelectric Effect. Modern-day appliances such as solar panels, home-cooling systems, etc., are derived from the cascading effects the Michelson–Morley experiment brought on.

ESRP-78

ESRP: Using a Wadsley-Roth Phase to Compare Lithium and Sodium Ion Batteries

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Battery performance depends not only on the type of chemicals used for the anode and cathodes, but also on how the chemicals are prepared and the structure of the compound. The Wadsley-Roth phase is a way of arranging the structure of the anode to increase fast charging capabilities and to maintain capacity charging after many cycles. Furthermore, this crystalline structure allows for easier and greater transposition of electrons between the anode and cathode due to the large size of the holes. We have seen in studies a PNb_9O_{25} Wadsley-Roth anode that maintains 90% capacity after 1100 cycles. Here we tried to dope molybdenum and titanium into the Wadsley-Roth phase $V_{1.3}O_{0.7}O_5$ to improve the capacity of the battery.

To test the efficacy of both lithium ion and sodium ion batteries with the Wadsley-Roth phase, we first prepared a sample of the two materials, $V_{1.3}W_{.07}O_5$ and $Mo_{1.5}$ $W_{1.5}Nb_{14}O_{44}$. For the $V_{1.3}W_{.07}O_5$ compound, we combined samples of V_2O_5, V_2O_3, MoO_3, TiO_2, and VO_3. For the $Mo_{1.5}$ $W_{1.5}Nb_{14}O_{44}$ sample, we combined samples of Nb_2O_5, MO_3, and WO_3. We ball milled each sample using a 4:1 ratio for one hour and prepared 0.5-gram batches for pellets. Finally, the powder was pressed into a pellet and heated at 700 degrees C for 24 hours. Next, we used a slurry from each completed pellet to create and assemble a battery. After placing all the materials into the anode and cathode faces to create one solid battery, the cell was then charged, discharged, and recharged multiple times.

Using x-ray diffraction (XRD), we found that the $V_{1.3}W_{.07}O_5$ compound with doping did improve the capacity of the battery. Further characterization is needed, such as scanning electron microscopy and elemental analysis. However, the XRD did provide insight into the phase in which we were interested. The XRD showed the $Mo_{1.5}$ $W_{1.5}Nb_{14}O_{44}$ was not successful, showing other compounds were created.

We also analyzed $V_{1.3}W_{.07}O_5$ batteries' initial capacity and ability to be brought back to original capacity after recharging. This analysis revealed that although both batteries charged, the lithium-ion battery had a much greater capacity and ability to be brought back to original capacity.

A-68

Material Flow and Consolidation Behavior of Metal Cutting Chips during Friction Surfacing

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Friction surfacing is a solid-state deposition technology that is traditionally studied and applied as a coating method but shows promising results as an additive manufacturing process for repairs/remanufacturing and to add features onto large parts. A rotating consumable rod is pressed against a substrate under an applied axial load. The frictional heat generated at the consumable rod tip plastically deforms the rod. The rod is subsequently traversed over the substrate with an axial load that deposits a uniform layer bonded to the substrate. The consumable rod is a conventional form of feedstock for the friction surfacing process, but a loose form of feedstock can be potentially incorporated to produce dense deposits. In the previous work by the authors, machining chips of stainless steel 316L were successfully deposited using friction surfacing by pressing into hollow stainless steel 304L rods [1]. This is the first work to apply the friction surfacing process for recycling metal cutting chips. Fully consolidated deposits were produced with good bonding to the substrate. Post-process, a dense plastically deformed layer of machining chips was observed at the bottom of the consumable friction surfacing rod. However, how the metal cutting chips flow, consolidate, and deform in the friction surfacing process is not fully understood. In the friction surfacing literature, a basic understanding of the material flow dynamics is illustrated by post-processing friction surfacing deposits and rods [2,3,4]. However, the literature lacks some fundamental knowledge about the morphology of the shear zone during deposition that potentially dictates material flow and hot working conditions imposed onto the deformed material layer. These works also do not explain how loose feedstock, like metal cutting chips, consolidate and affect the material flow dynamics of the process.

The objective of this work is to analyze the material flow, consolidation behavior of machining chips, and morphology of the shear zone during the friction surfacing process. Metal cutting chips of mild steel 1045 were pressed in stainless steel 304L rods and the rods were then used as consumables in the friction surfacing process. Fully consolidated layers of mild steel chips and stainless-steel rod were observed with chips material in the middle and bottom of the deposit and rod material around and above it. With increasing the hole diameter, more chips material was found in the deposit vs rod material only on the top layer. Chip-filled rods were investigated post processes where three separate layers of chips were identified based on the compaction level of chips. The cross sections explain the in-process compression and consolidation of chips in the friction surfacing. To analyze the morphology of the shear zone and how chips consolidate in the process, experiments are proposed to observe the process *in situ* with high-energy x-rays. Through *in-situ* observations, the team seeks to understand what the shape of the shear zone is during the deposition, and how and where surface oxides or impurities on chips disturb and redistribute in the friction surfacing process.

[1] A. Deshpande, H. Agiwal, C. Baumann, S. Krall, F. Bleicher, and F. E. Pfefferkorn, “Recycling metal cutting chips into a consolidated deposition with friction surfacing,” *Manufacturing Letters*, vol. 35, pp. 743–749, Aug. 2023, doi: 10.1016/j.mfglet.2023.08.093.
[2] K. Fukakusa, “On the characteristics of the rotational contact plane - a fundamental study of friction surfacing,” *Welding International*, vol. 10, no. 7, pp. 524–529, Jan. 1996, doi: 10.1080/09507119609549043.
[3] H. Rafi, G. Phanikumar, and P. Kalvala, “Material Flow Visualization during Friction Surfacing,” *Metallurgical and Materials Transactions A-physical Metallurgy and Materials Science - METALL MATER TRANS A*, vol. 42, pp. 937–939, Apr. 2011, doi: 10.1007/s11661-011-0614-2.
[4] G. M. Bedford, V. I. Vitanov, and I. I. Voutchkov, “On the thermo-mechanical events during friction surfacing of high speed steels,” *Surface and Coatings Technology*, vol. 141, no. 1, pp. 34–39, Jun. 2001, doi: 10.1016/S0257-8972(01)01129-X.

Enabling Data-centric AI for Synchrotron Datasets Using FAIRification and Knowledge Graphs

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Enabling data-centric AI on synchrotron datasets can unlock an advanced level of analysis and interpretation of its complex experimental data that was previously impossible. In order to achieve this, traditional synchrotron datasets must first undergo modifications in order to adhere to FAIR (Findable, Accessible, Interoperable, Reusable) data standards. Our newly-developed FAIRification framework FAIRifies the synchrotron dataset by generating an ontology from its variables and populating the ontology with data stored in the synchrotron datasets. This ontology is used as a precursor for knowledge graphs that are stored in our Janusgraph database. The resulting knowledge graph allows us to execute complex graph computations on the dataset, perform ML techniques like link prediction and graph learning, and even accelerate data-driven discoveries.

A-70

A Machine Learning Approach to Streamlining Rietveld Refinement

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The neutron and x-ray diffraction of powder samples create a pattern that is characterized by reflections described by Bragg's Law. Rietveld refinement is a technique used in the characterization of crystalline materials. Current software utilized for Rietveld refinements requires expert knowledge as well as several iterations of analysis to obtain a convergent result. Through the use of adaptive neural networks that are specifically tailored to the experimental setup and materials present, the number of iterations to reach adequate convergence is reduced. Reducing the number of iterations improves the ease of use and efficiency of Rietveld refinement softwares. These experiment-specific neural networks are trained utilizing simulated diffractograms using the diffraction simulation functionality present in GSAS II. With these simulated diffractograms, NNs are trained to predict the phase fractions, the lattice parameters, and the phases present. Using this approach, we have predicted phase fractions on average 0.39% from the true value and a maximum deviation of 4.45%. We have predicted lattice parameters with an average error of 0.46% and a maximum error of 4.06%.

Elucidation of Relaxation Dynamics Beyond Equilibrium Using AI-informed X-ray Photon Correlation Spectroscopy

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Understanding and interpreting dynamics of functional materials *in situ* is a grand challenge in physics and materials science due to the difficulty of experimentally probing materials at varied length and time scales. X-ray photon correlation spectroscopy (XPCS) is uniquely well-suited for characterizing materials dynamics over wide-ranging time scales, however spatial and temporal heterogeneity in material behavior can make interpretation of experimental XPCS data difficult. Here, we show how incorporating unsupervised machine learning (ML) with XPCS enables automated extraction and classification of dynamic trends and recurring patterns in large experimental datasets. Visualizing how ML-learned dynamic behaviors evolve through time allows for direct correlation between macroscopic material properties and microscopic dynamics without requiring any background knowledge of the dataset. Finally, we demonstrate the application of our method on a complex experimental system, a shear thickening colloid, and show how ML-informed data analysis has uncovered a physical model describing complex experimental dynamics. This general workflow can be applied for any time-resolved imaging or scattering experiment, providing a new tool for automated experimentation at the APS-U.

X-ray Scattering Techniques to Probe the Structure and Dynamics of Soft Matter

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Soft materials play a critical role in the development of a wide range of emerging technology including energy storage devices, photochromic displays, advanced coatings, and novel robotic sensors. By engineering their rheological and electrical properties, we can achieve desired macroscopic performances in response to applied stimuli. Through a combination of nanoscale synthesis, rheo-electrical measurements, and small-angle scattering, the Richards Lab investigates and manipulates the structure and dynamics of complex soft matter building blocks. Our lab frequently utilizes facilities at various beamlines at Argonne National Laboratory to provide structural and dynamic insight of our systems. This insight enables us to further develop the constitutive relationships necessary to transform our economy toward sustainability and circularity. Additionally, the enhanced capabilities resulting from the upgrade to the Advanced Photon Source will allow us to quantify transient and out-of-equilibrium dynamics that were not previously accessible.

Deep Learning-based Semantic Segmentation on 2D X-ray HEDM/Powder Image Data

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Rapid scanning of materials is essential for high-throughput beamlines at the APS. Techniques such as powder diffraction and high-energy diffraction microscopy require different material and experiment conditions, and developing an automated decision-making workflow is immensely beneficial for beamline setups. The use of advanced detectors combined with improved x-ray beam properties at the APS post-Upgrade is projected to significantly increase data generation, challenging traditional processing methods with the need for automatic and real-time analysis. To overcome this, we introduce a deep learning-based semantic segmentation strategy specifically designed for HEDM/powder diffraction data classification. Utilizing the Segment Anything Model (SAM) as a foundation model for the segmentation task, we refined its performance through model fine-tuning with our simulation data. This enhancement allows for precise HEDM/powder data classification and its ratio assessment, ensuring efficient real-time data processing and analysis.

A-74

Double Gels Based on Interpenetrating Colloidal Particle Networks

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Gels composed of multicomponent building blocks offer promising opportunities for the development of novel soft materials with unique and useful structures. While interpenetrating polymer networks have been extensively studied and applied in the creation of these gels, equivalent strategies utilizing colloidal particles have received limited scientific and technological attention. This study presents a novel class of thermo-responsive apolar double gels from interpenetrating networks of attractive colloidal silica and lipid particles. These double gels are easily assembled and suitable for the fabrication of 3D-printed edible soft constructs. Emphasis is focused on the rheological properties and structure emerging on the dilute regime. Rheological investigations demonstrate that double gels exhibit greater stiffness and resilience to yielding compared to their single lipid gel counterparts. The scaling behavior of the oscillatory linear shear moduli and the critical strain for yielding with volume fraction remain comparable between single and double gels. Creep yielding in double gels exhibits two exponential decay regimes, suggesting the presence of thicker gel strands undergoing flow. Visualization and quantification of the quiescent microstructure confirms the existence of such denser aggregates devoid of larger clusters due to steric hindrance of interpenetrating networks in double gels. This is in stark contrast to lipid single gels where aggregates grow unrestrictedly into larger clusters [1]. Our study constitutes the first demonstration on the assembly of apolar double gel networks as a promising avenue for the design of novel soft materials and foods with tailored structure and mechanics.

[1] B.A. Macias-Rodriguez, R. Gouzy, C. Coulais, and K. P. Velikov. Soft Matter. doi.org/10.1039/D3SM01582C.

PtychoSAXS: Combined X-ray Ptychography and SAXS Imaging for Nanostructure Characterization

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Multi-length scale structure characterization is required for hierarchical samples ranging from energy storage and catalysis to biological and bio-inspired systems because morphology as well as local arrangements of nanostructures affect physical properties of materials. X-ray scattering techniques, such as SAXS, have provided powerful tools to study internal structures, but the obtained information is typically the ensemble average over the sample area covered by the x-ray beam, which would limit a full understanding of local diverse arrangements of nanostructures. The unique capability of x-ray ptychography to probe both surface and internal structures of samples at a high-spatial resolution makes it attractive to combine with SAXS which shares a similar measurement geometry. With this combination of PtychoSAXS, ptychography can provide morphology at nanoscale resolution across micron-to-mm size sample, while SAXS provides reciprocal-space information revealing nanostructure size, shape, and orientation in a length scale smaller than hundreds of nanometers and even down to sub-nanometer. A revolutionary increase in brightness and coherent flux promised by the coming upgrade of the Advanced Photon Source (APS) at Argonne National Laboratory will greatly contribute to improved imaging capabilities via ptychography which requires high coherent flux and SAXS which benefits from high probe intensity. This work presents how by utilizing our proposed combined approach, we can correlate the information collected by the two techniques from a single sample environment ideally with a single detector while minimizing radiation dose. In addition, we will highlight the experimental and computational challenges that arise with the PtychoSAXS technique.

This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science user facility and is based on work supported by Laboratory Directed Research and Development (LDRD) funding from Argonne National Laboratory, provided by the Director, Office of Science, of the U.S. DOE under Contract No. DE-AC02-06CH11357.

Two-dimensional Diffractogram Analysis: Kinematic-diffraction Simulator for Neural Network Training Data Generation

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To fully exploit the information contained in two-dimensional (2D) diffractograms for materials research, we can utilize the tremendous powers of neural networks and deep learning. Since these diffractograms are essentially images, neural networks can – in theory – predict material properties from these "images." A challenge of this approach is to generate suitable training data. If one has to manually refine a huge set of diffractograms to train a neural network, it defeats the purpose. This can be accomplished by generating training data with the help of a "kinematic-diffraction simulator." This simulator is implemented in the Wolfram language within a high-performance computing environment. The KDS applies the theory of diffraction physics to generate *Fraunhofer* diffractograms. It iterates over a user-provided number of grains and adds the Bragg's reflection from every grain until the iterations are complete. It then adds up all the reflections from individual grains to generate a complete diffraction pattern of the system. These simulated diffractograms can then be used to train a neural network and analyze "real" diffractograms.

X-ray Scattering Characterization of Catalyst Inks and Electrodes for the PEFC MEA Integration Study

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The ink composition of electrochemical energy applications, such as solvent ratio, ink processing method and time, ionomer chemistry, and carbon morphology/porosity can impact the final electrode microstructure and performance. Catalyst layers of a polymer electrolyte fuel cell (PEFC) are typically prepared by first dispersing a catalyst powder, comprised of platinum or platinum alloy nanoparticles supported on carbon blacks, with ionomer in solvents, followed by extensive mixing using a variety of methods. This presentation will describe the results of *in-situ/ex-situ* x-ray scattering studies of the evolution of the cathode catalyst layer to determine the effect of ink processing variables and ink composition on the ink properties, especially in the carbon agglomerate break-up. The results integrate ionomers and catalyst inks in various compositions to improve membrane-electrode assembly (MEA) performance and durability.

A-80

Leveraging Deep Neural Networks for Artefact Detection in High-energy Scattering Computed Tomography (XSCT) Diffraction Patterns from APS Beamline Data

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High-energy x-ray scattering tomography is an emerging field of study, increasingly utilized in the spatiotemporal reconstruction and characterization of reactive material systems and bone structures. Despite its promise, heightened sensitivity for phase detection can lead to high-intensity artifacts, or "spots," in XRD patterns. These impurity detections can pose significant challenges in accurate tomographic reconstruction and Rietveld refinement, especially in heterogeneous samples. This study explores the use of convolutional neural networks (CNNs) in classifying XRD patterns according to the presence (or absence) of these “spots.” Beamline data on electrode pencil beam samples were retrieved from the APS and sampled for the training and evaluation of binary classification models. Supervised learning is being performed from annotated XRD patterns, and the outputs will be used to determine subsequent steps in a larger beamline data analysis pipeline.

Multimodal X-ray Characterizations of Structure and Optical Properties in CdTe Thin Films

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Multimodal x-ray-based characterizations provide a thorough investigation of functional materials and relate the functional performance with composition, structural and optical properties in semiconducting materials. In this work, we use a CdTe thin film sample as a testbed for showcasing the benefit of simultaneous microscopic assessment using x-ray excited optical luminescence (XEOL) and x-ray Laue diffraction (XRD) at the nanoscale for the first time in photovoltaic materials research. The nanoprobe x-ray measurements show that the spectrally resolved XEOL measurements reveal the presence of microscopic heterogeneity of optical bandgap in CdTe films. When correlating with 2D Laue reconstructions, we find that the XEOL intensity varies slightly based on grain orientation, with significant intensity variation across general grain boundaries. The Laue measurement was also utilized to track grain boundary misorientation types, including twin boundaries—coincident site lattice (CSL) $\Sigma = 3$—which specifically show a negligible impact on XEOL spectra as opposed to other non-twin boundary sites. Deviatoric strain measurements from the Laue measurements were also compared against the corresponding XEOL spectra, showing little to no direct correlation between estimated strains and optical band gap throughout the sample. Overall, these results showcase the efficacy of correlative multimodal crystal structure measurements using XRD and optical property measurements using XEOL as a promising tool for highlighting potential pathways for improving future semiconductor devices.

ESRP-75

ESRP: Comparative Study of Lithium-Ion Batteries with Organic-Based Carbon Anode

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Many batteries today use graphite-based anodes which are not reusable [1]. In an attempt to further develop solutions to these problems, our group decided to design and test batteries with organic material from activated coconut shells as a carbon source in our anode. Seventy percent of pre-activated carbon from the coconut shells was mixed with 10% NDPH (binding agent), 20% Carbon Black (conductive additive), dissolved with NMP (solvent), and mixed on a vortex for 24 hours to produce a homogeneous slurry. This slurry was then spread using the doctor blade method onto a copper film, dried in an oven, and punched out to form the electrodes. The coin cell was assembled with a lithium-ion cathode in an argon glovebox. The battery's structure, using x-ray diffraction, and performance, using charge/discharge cycling, were compared to those of a non-organic one. The x-ray diffraction revealed the coconut shell anode to be amorphous and the graphite to be crystalline. The specific capacity for both anodes was low but their rate goes back to the original rate after cycling. The specific capacity for the pre-activated carbon was higher than the specific capacity for the graphite anode. This work provides a glimpse into moving towards more eco-environmental functioning batteries.

[1] Natarajan, S., & Aravindan, V. (2020). An Urgent Call to Spent LIB Recycling: Whys and Wherefores for Graphite Recovery. *Advanced Energy Materials*, *10*(37), 2002238. https://doi.org/10.1002/aenm.202002238.

Identifying the Precursors of Ductile Failure via Void Nucleation and Coalescence

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Classical solid mechanics models have limited precision for predicting ductile failure because they are informed by continuum-scale failure metrics. Ductile failure in metals occurs via the nucleation, growth, and coalescence of voids within microstructures that exhibit anisotropy. This work employs multimodal characterization techniques (conducted at the Advanced Photon Source, beamline 1-ID, under DOE Contract DE-AC02-06CH11357) to determine the evolution of grain-scale failure metrics and ductile failure mechanisms in Al2219. *In-situ* near-field and far-field high-energy x-ray diffraction microscopy were employed to track the micromechanical state of the grain structure. Simultaneously, micro-computed tomography was employed to track the state of secondary particles and voids. These datasets were consolidated to investigate the evolution of stress created by microstructural anisotropy. In particular, sites of particle cracking and sites of void nucleation, growth, and coalescence were examined based on the micromechanical evolution of neighboring grains. Sandia National Laboratories are managed and operated by NTESS under DOE NNSA contract DE-NA0003525.

Surface X-ray Studies of Rare Earth Group Ion Transport Pathway on Functionalized Monolayer MoS_2

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Rare earth elements (REs) have been identified as critical elements with short-term supply risks. They are essential in modern technologies and devices such as permanent magnets, optical fiber, and medical imaging agents [1,2]. REs exhibit very similar chemical properties but varied electronic properties, and their application requires high purity of every single element. Therefore, achieving effective and efficient separation among REs from one another has been the challenge since their discovery and this task is especially critical now to enable recycling to secure the REs supply. Currently, the solvent extraction used in the industrial productions induces negative impact on the environment due to drawback of energy and chemical intensiveness [3-5].

Based on the two important properties of REs, ionic radius (decreasing with atomic number) and Lewis acidity (increasing with atomic number) [2], we proposed to create a two-dimensional (2D) solid ionic channels with stacked 2D materials (such as MoS2) that are able to modulate the dehydration, transport, and hydration of REs. To achieve selective transport of REs by rational design, a better understanding of the binding and conduction of REs ions through the 2D channels is required. Hence, we established an understanding of binding thermodynamic on the single monolayer MoS2 with functionalize surface as a model system to determine how lanthanide RE ions contact and interact with pristine and surface functionalized monolayer 2D materials. We combined surface x-ray diffraction (crystal truncation rod) and gracing incidence x-ray absorption spectra to provide a precise local coordination configuration. This allows us to create an accurate molecular-level structural model for the electronic structure computation and modeling, which would be better to reconstruct the ion transport pathway and realize the selectivity control among REs. The outcomes will have immediate impact to enabling new energy efficient separation methods, especially transport-based separation technologies (membrane separation), to be applied to REs extraction, separation, and recycling.

[1] Kostelnik, T. I. & Orvig, C. Radioactive Main Group and Rare Earth Metals for Imaging and Therapy. Chem. Rev. 119, 902 956 (2019).
[2] Cheisson, T. & Schelter, E. J. Rare earth elements: Mendeleev’s bane, modern marvels. Science 363,489 493 (2019).
[3] Liao, C., Wu, S., Cheng, F., Wang, S., Liu, Y., Zhang, B. & Yan, C. Clean separation technologies of rare earth resources in China. Journal of Rare Earths 31, 331–336 (2013).
[4] Wang, L., Huang, X., Yu, Y., Zhao, L., Wang, C., Feng, Z., Cui, D. & Long, Z. Towards cleaner production of rare earth elements from bastnaesite in China. Journal of Cleaner Production 165, 231–242 (2017).
[5] Choi, H., Harvey, D., Ding, Y. & Wang, N.-H. L. Constant-pattern design method for the separation of ternary mixtures of rare earth elements using ligand-assisted displacement chromatography. Journal of Chromatography A 1580, 49–62 (2018).

ESRP-85

ESRP: Analysis of V-Doped Wadsley-Roth Phase $Nb_4W_7O_{31}$ as a Battery Anode

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An increasing global population drives rising energy demand, leading to the use of unsustainable energy solutions and consequent environmental damage. Batteries, by storing chemical potential energy, reduce energy waste and ensure efficient energy utilization, thus mitigating the harmful effects of energy generation. Research concerning novel battery anodes is vital in documenting and progressing these solutions for further exploration. Specifically, the Wadsley-Roth phase's crystallographic shear structure has been explored in the literature as a possible structure for battery anodes, used to create effective, sustainable batteries. Our group previously explored the $(W_{0.2}V_{0.8})_3O_7$ Wadsley-Roth phase as a potential battery anode. We demonstrated that this material, when tested as a battery anode with both lithium and sodium ions, maintains a stable structure during charging and discharging through extended x-ray absorption fine structure (EXAFS) analysis. Additionally, we observed an excellent charge-discharge efficiency during battery cycling testing. This study expands upon previous Wadsley-Roth phase analyses by testing the merits of the crystallographic shear phase $Nb_4W_7O_{31}$ as a battery anode, both independent of doping and when doped with vanadium. After creating the identified phases through ball-milling and sintering, we tested whether the proper phase was created by use of x-ray diffraction (XRD). We then analyzed the performance of the two proposed materials through battery cycling analysis, measuring the performance of the battery through its specific capacity over 350 charge-discharge cycles. We found that the $Nb_4W_7O_{31}$ Wadsley-Roth phase doped with vanadium recorded improved performance as a battery anode when compared to its vanadium-free counterpart, evidenced by an increase in the specific charge capacity across all cycles. Given this, in combination with our previous study, we find promising evidence for the efficacy of Wadsley-Roth phase battery anodes.

C-47

Degradable Luminescent Polymers with High-efficiency Thermally Activated Delayed Fluorescence

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Organic light-emitting diodes (OLEDs) have emerged as the most prevalent display technology. Polymer-based emitters have garnered increasing attention owing to their advantages in processibility and mechanical properties. However, the disposal of light-emitting polymers from displays poses a significant threat to the environment, necessitating the incorporation of degradable properties in these materials. In this study, we developed a novel type of degradable thermally activated delayed fluorescence (TADF) polymers that exhibit high OLED performance and can be degraded under specific conditions. We present a general concept for the development of degradable TADF polymers by incorporating degradable dynamic bonds into the polymer backbone. We designed and synthesized a degradable TADF polymer with degradable bonds and TADF units in the polymer main chain that exhibited high external quantum efficiencies (EQE) for both host-free system and host-guest system, which also demonstrated the degradability under certain conditions. Overall, our study opens up new possibilities for the development of environmentally friendly OLED technologies, thereby minimizing their environmental impact [1].

This work is founded by Laboratory Directed Research and Development (LDRD) program. We appreciate the help from Center of the Nanoscale Materials, Argonne National Laboratory.

[1] W. Liu, Y. Wu, A. Vriza, C. Zhang, H. Jung, et al. Depolymerizable and recyclable luminescent polymers, **2024**, accepted by *Nat. Sustain.*

C-48

Light-controlled Spin Coupling in Doped Hybrid Perovskites

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It is a fundamental challenge to modulate a pair of entangled particles such as the spin-entangled electron and hole in a light-induced exciton. Herein, we show a novel approach to manipulate the exciton lifetime achieved in a Nd^{2+} doped perovskite film through a Kondo-like exciton-spin interaction. This system is an optic mimic to the classic Kondo effect found in metallic system where a localized magnetic impurity dopant is spin-coupled with an overwhelming number of free electrons in the host metal. In stark contrast, our system is composed of magnetic impurity doped in a semiconductor perovskite material where dopants can outnumber the light-induced excitons. As such, the spin-entangled electron and hole have high probability to respectively couple with the opposite localized impurity spins in their proximity as evidenced by the notably prolonged carrier lifetimes. More interestingly, an external magnetic field nullifies the exchange interaction between the exciton and the localized impurity spin because of the vanishing opposite local spins needed for coupling the electron and the hole in an exciton, respectively. The interaction between photoelectrons and the empty $6s^05d^0$ orbital of Nd^{2+} is also evidenced by scanning tunneling microscopy. We are also able to regulate the coupling strength between exciton and magnetic spins by tuning the amount ratio of Nd(II) to incident photons.

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C-49

Autonomous Discovery of Plastic Recycling

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The mounting crisis of plastic waste, coupled with our dependence on fossil-derived materials for commercial polyolefin plastics, jeopardizes our environment. In response, we aim to develop Circular Polyolefin-like Plastics (CPLPs) — materials that match the qualities of current polyolefins but with inherent chemical recyclability.

Our multidisciplinary team is positioned to rapidly innovate in creating sustainable polymers, ones that are recyclable and biodegradable, and yet match or exceed the performance of prevalent commodity polymers. Valuable input from our industrial advisory board ensures our research aligns with real-world commercial needs. By integrating high-throughput testing, autonomous labs, computational modeling, and AI/ML techniques, we're poised to craft a purpose-driven approach for crafting recyclable plastics from waste-derived components. Our focus will remain on scalable and economical processes, emphasizing sustainable feedstocks and avoiding the use of rare catalysts.

A-84

A Comprehensive Framework for X-ray Diffraction Analysis: Deep Learning Insights and Approaches

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Our research designs a comprehensive deep-learning framework deployed on a high-performance computing platform for the efficient and precise analysis of x-ray diffraction (XRD) data, which is important for unraveling material microstructure. The approach includes data preparation, modeling, hyper-parameter tuning, synthetic data generation, and uncertainty quantification. Specifically, we utilize convolutional neural networks (CNN) to analyze video sequences of 2D XRD patterns for Ti–6Al–4V alloy during heat-treatment. The fine-tuning models can determine the β-phase volume fraction in superior performance, highlighting the efficacy of our CNN design in capturing intricate material information. Moreover, our analysis delves into the correlation between 2D XRD data and the properties of CNN. Our investigation reveals that even when trained exclusively on pure α- and β-phase 2D XRD patterns of Ti–6Al–4V, CNN models still can accurately detect 2D XRD patterns with intermediate β-phase fractions. This finding showcases the potential of our approach to mitigate the need for extensive training datasets, thereby addressing resource constraints in real-world experiments.

A-86

Enhancing Low-contrast High-energy Tomography with Machine Learning

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Synchrotron x-ray tomography enables the examination of the internal structure of materials at submicron spatial resolution and subsecond temporal resolution. Unavoidable experimental constraints can impose dose and time limits on the measurements, introducing noise and artifacts in the reconstructed images. Therefore, recovering a high-quality image from noisy indirect measurements is an important problem with many applications. Deep learning, specifically convolutional neural networks (CNNs), has emerged as a powerful tool to remove noise from reconstructed images. However, their training requires collecting paired noisy and high-quality images, which is a major obstacle for most applications. Recently, methods for CNN-based denoising have been proposed that require no separate training data beyond the already available noisy reconstructions. Among these, the Noise2Inverse method is specifically designed for tomography and related inverse problems. The Noise2Inverse method creates multiple statistically independent reconstructions used to pair the data and was shown to theoretically obtain the clean reconstruction. We apply the Noise2Inverse method to high-energy (XX keV) tomography collected at the Advanced Photon Source 1-ID at Argonne National Laboratory. Tomographic data acquisition is performed while a sample is rotated on a rotation axis. Typically, samples are rotated 180°, a half rotation, with the number of projections specified by a fixed offset. We extend the Noise2Inverse method to samples rotated 360°, a full rotation. Furthermore, we compare the denoised samples reconstructed with the filtered back-projection algorithm provided by the TomoPy software package with the grid reconstruction algorithm provided by the MIDAS software. Overall, our method significantly reduces the noise in the reconstructed volume as well as enhancing the features of the sample.

NANOSCIENCE & NANOTECHNOLOGY

C-50

A Systematic Approach to Develop High-performance Colloidal Quantum Dot Infrared Photodetectors

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Colloidal quantum dots (CQDs) are a desirable platform for the development of next-generation infrared (IR) detectors thanks to their scalable synthesis, tunable optoelectronic properties, CMOS compatibility, and monolithic integration. However, CQD-based IR detectors typically have lower quantum efficiencies than epitaxial semiconductors and still require cryogenic cooling to achieve background-limited infrared photodetection. Developing CQD-based IR detectors that achieve state-of-the-art performance could bridge the gap between low-cost and high operating temperature detectors for IR sensing, especially for MWIR capabilities. Such a technology could significantly enable the advancement of compact, lightweight, and low-cost infrared systems for higher volume applications such as unmanned drone surveillance, driver-assisted vehicle navigation in low-visibility environments, and mountable visual systems for advanced situational awareness.

A systematic approach to materials development and detector design that relates material synthesis to detector optoelectronic properties will accelerate the development of CQD-based IR detector technologies. Such a system has not been explicitly established for CQD materials and their IR detectors. In this poster, a process using a combination of empirical and numerical processes will be described to guide and accelerate the development of CQD-based IR detectors. HgTe CQDs, one of the more mature IR CQD materials, was studied as a model system to provide useful feedback for establishing design rules and relationships between synthesis, material properties, and detector performance. Improvements to the performance of HgTe CQD photodetectors as an outcome of this study are demonstrated.

ESRP-52

ESRP: Carbon Capture Effects of Boron-coating on Platinum Single Crystal

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In the face of the global climate crisis, the need for advanced materials and scalable solutions of carbon capturing is more urgent than ever. It's essential to explore the fundamental physical and chemical mechanisms of such materials to realize the next-generation high-performance carbon-capturing systems. Here, we explore functionalized low-dimensional material synthesized on a platinum single crystal for CO_2 capturing and reduction. The project aims to understand how coating a platinum crystal surface with an atomically thin layer of boron affects the capture of CO_2. Utilizing a low-temperature scanning tunneling microscope (LT-STM) to characterize surfaces at the atomic resolution. The performance of CO_2 capturing/reduction of the nanomaterial is evaluated by temperature-programmed desorption (TPD) measurements. The result provides additional insights into a carbon capturing and reduction mechanism.

C-51

Progress in Fabrication of Free-standing Gold Mask for Low-energy Phase-based X-ray Microscopy

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We are developing a laboratory-based intensity-modulation phase-based x-ray microscope, which will provide nanometric information of biological cells through advanced scattering and ptychography imaging modes. The key optical element of the system is the mask with an array of void slit apertures to modulate x-ray beam intensity.

We previously reported the development of a freestanding grating fabrication process based on gold electroplating into a silicon mold coated with various thin films to form a voltage barrier, plating base, and sacrificial layer, followed by mold removal to obtain the gold membrane with void slit apertures. The first mask prototype - a 10-μm-thick gold membrane with an array of micrometer-wide and 400-micrometer-long void slit apertures with 7.5-μm periodicity has been fabricated and an aperture-driven spatial resolution has been demonstrated [1]. This result enables the development of a multiresolution microscope where multiscale samples can be explored on different length scales by adjusting only the mask aperture size, without other modifications.

Here, we present the progress towards obtaining a gold membrane with arrays of sub-micrometer-wide void apertures with various periodicity in one frame. Having a mask with various periods in a system allows the changing imaging conditions in real time when scanning the sample. This improvement will expand the microscope capabilities to provide a range of co-registered, multi-modal images with sensitivity to nanometer features.

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[1] O.V. Makarova, R. Divan, N. Moldovan, D.A Czaplewski, M. Esposito, M. Endrizzi, C-M Tang, J.D. Ferrara and A. Olivo, Nanotechnology 2023, 34**,** 045301.

OTHER

Online Analysis Workflows for X-ray Experiments Using Bluesky and Sirepo

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The design, execution, and analysis of light source experiments requires the use of sophisticated simulation, controls, and data management tools. Existing workflows require significant specialization to accommodate specific beamline operations and data pre-processing steps necessary for more intensive analysis. Recent efforts to address these needs at the National Synchrotron Light Source II (NSLS-II) have resulted in the creation of the Bluesky data collection framework, an open-source library for coordinating experimental control and data collection [1]. A framework has been developed to integrate Bluesky with the Sirepo online application gateway [2,3], to provide user interfaces and more transparent workflow analysis for online data processing and metrics. This interface has been prototyped and tested at the coherent hard x-ray (CHX) and coherent soft x-ray (CSX) beamlines at NSLS-II. Our application leverages Bluesky in combination with a flexible run engine to execute user configurable Python-based analyses with customizable queueing and resource management. We discuss initial demonstrations to support x-ray photon correlation spectroscopy experiments and future efforts to expand the platform's features.

This material is based upon work supported by the U.S. Department of Energy, Office of Science, Office of Advanced Scientific Computing Research under Award Number DE-SC00215553.

[1] D. Allan et al. "Bluesky's Ahead: A Multi-Facility Collaboration for an a la Carte Software Project for Data Acquisition and Management". In: Synchrotron Radiation News 32.3 (May 2019), pp. 19–22.
[2] M. S. Rakitin et al. "Sirepo: an open-source cloud-based software interface for X-ray source and optics simulations". In: Journal of Synchrotron Radiation 25 (2018), p. 1177.
[3] M. S. Rakitin et al. "Introduction of the Sirepo-Bluesky interface and its application to the optimization problems". In: Proc. SPIE. Vol. 11493. Aug. 2020.

The Universal Proposal System (UPS): A Common Platform for the Management of Scientific Proposals

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The Universal Proposal System (UPS) is a configurable shared platform that supports the submission, management, review, and allocation of user scientific proposals. The UPS has been collaboratively developed by three US Department of Energy (DOE) light source user facility stakeholders:

- Advanced Photon Source (APS) at Argonne National Laboratory
- Linac Coherent Light Source (LCLS) at SLAC National Accelerator Laboratory
- National Synchrotron Light Source II (NSLS-II) at Brookhaven National Laboratory

National laboratories are multipurpose research institutions, independently operated for the DOE. Each laboratory is home to scientific user facilities, offering scientists (users) from around the world access to a diverse array of research instruments. To request access to these resources, users submit proposals that undergo a peer-reviewed application process.

This talk will provide an introduction to UPS, describe current structure and functionality, and offer a glimpse into future plans.

This work used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science user facility, operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

ESRP-39

ESRP: Exploring the Evolution of Nuclear Spin-orbit Splittings in Exotic Nuclei

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Recent works exploring the evolution of the 1*p* single-particle energies in neutron-rich (exotic) nuclei around neutron number $N = 20$ [1] reveal a smooth decrease in the energy separation of the $1p_{3/2}$- $1p_{1/2}$ spin-orbit splittings as they become more weakly bound. Stable nuclei are well-bound, and exotic (short-lived, radioactive) nuclei are more weakly bound. The evolution of single-particle energies is a key focus of contemporary nuclear physics research at facilities such as the Argonne Tandem Linac Accelerator System and the new Facility for Rare Isotope Beams at Michigan State University. A realistic, finite mean-field nuclear potential, such as a Woods-Saxon potential, well describes the behavior of spin-orbit splittings described above. Still, it is at odds with the well-accepted scaling of measured spin-orbit splittings across the chart of nuclides for well-bound states [2]. We present experimental data for stable and exotic nuclei and compare them to a mean-field description and the well-established trends for well-bound systems. We reveal a somewhat universal, predictable behavior, where the splitting between spin-orbit partners decreases as nuclei become more weakly bound, particularly for spin-orbit partners with low angular momentum (*p* and *d* orbitals). This observation coincides with the 75th anniversary of Argonne's Maria Goeppert Mayer's seminal 1949 work, which first proposed spin-orbit coupling that led to a robust description of the level ordering in nuclei.

This material is based upon work supported by the U.S. Department of Energy, Office of Science, Office of Nuclear Physics, under Contract Number DE-AC02-06CH11357.

[1] B. P. Kay, et al., Phys. Rev. Lett. 119, 182502 (2017).
[2] G. Mairle, Phys. Lett. B 304, 39 (1993).

APS Additive Manufacturing Facility

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Introduced in 2009, additive manufacturing at the APS has expanded from the original Objet 30 to include PolyJet, FDM, SLA, and Metal BDM Machines. What began as a form, fit, and function endeavor with a modest investment has matured into a full production environment averaging 2,800 parts per year. This includes parts for onsite experimenters, mail-in experiments, and various devices to enable the science at the APS. The novel method of printing components has yielded many benefits including cost savings compared to traditional manufacturing, to a reduction in environmental impact caused by disposal of metal components. This has enabled creative solutions to difficult manufacturing problems via the additive benefits.

Our present collection of Additive Manufacturing machines includes a Desktop Metal Studio System, a Stratasys Objet350 Connex 3, a Stratasys J55, a Stratasys F770, two Stratasys F370's, a FormLabs Form 3, a FormLabs Form 3L, a Markforged Mark Two, and a Wazer Waterjet. Each machine has its niche application allowing us to select the correct equipment to ensure we produce parts cost effectively for the user community. This diverse level of equipment allows us to produce precision parts on PolyJet machines down to the 15-micron layer heights and robust support components on the FDM based machines.

The latest addition of the Desktop Metal Studio System in 2023 has brought a new arena to the facility. This system has opened the community to Bound Deposition Manufacturing of metal components with materials including Copper, 316L, 17-4ph, IN625, and Ti64. Development and testing of design parameters for this system are ongoing.

Automating X-ray Fluorescence Mapping with Differentiable Modeling

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X-ray fluorescence (XRF) has been widely utilized to analyze chemical composition and elemental distribution within samples. The process of fitting raw XRF spectra and obtaining quantified insights (i.e., mapping) is at the core of XRF analysis [1]. At the Advanced Photon Source (APS), there are large amount of diverse XRF datasets from different samples, instruments, and experimental conditions. Extensive parameter tuning is needed to obtain best results for each specific dataset. Typically, the fitting parameters such as energy calibration coefficients or gaussian parameters are tuned by beamline scientists based on their past experiences. This reduces overall throughput and potentially introduces subjective biases. We anticipate parameter tuning will become a major bottleneck especially after the APS-Upgrade, when the data collection rate will be ~100 times higher [2]. Therefore, it is important to develop automated XRF parameter tuning workflows.

The MAPS algorithm [3] and the XRF-Maps software [4] are currently utilized at the APS to analyze XRF data, requiring users to specify element types and fitting parameters for each experiment. To automatically search for optimal parameters, we present a physics-based and data-efficient differentiable modeling (DM) approach by leveraging automatic differentiation (AD), a well-known technique in the field of machine learning [5] and has been applied to computational imaging techniques such as ptychography [6]. When solving optimization problems with complex physical models, AD can produce accurate numerical gradients for all inputs efficiently in a programmatic fashion, eliminating the need for deriving closed forms gradients or approximating finite differences. We implemented a differentiable version of the robust physics-based MAPS algorithm using PyTorch [7]. In the forward pass, each potential element's contribution is calculated and summed to the model spectrum. Then elastic, Compton and escape amplitudes are calculated to produce the model spectrum. The resulting model spectrum is then compared to the experimental spectrum to calculate a loss, which can be backpropagated via AD. Since the amplitudes and all fitting parameters are all free parameters in the DM approach, we can optimize them simultaneously during the fitting process, thus eliminating the need of manual tuning. Besides, the framework can automatically determine which elements present in the sample (i.e., amplitudes are non-zero) from a set of potential candidates. Moreover, with the flexibility of the DM framework, it is possible to design advanced optimization routine to overcome various issues encountered in the current fitting routine and provide better fit to the experimental data. Overall, combining the solid physics foundation of the MAPS algorithm and powerful AD, the DM framework represents a promising route to lift the burden of parameter tuning and can be extended to other spectrum fitting problems such as energy-dispersive x-ray spectroscopy and electron energy loss spectroscopy.

[1] T Nietzold et al., *J. Vis. Exp.* **132** (2018), p. e56042. https://doi.org/10.3791/56042.
[2] The APS Upgrade: Building a Brighter Future, https://www.aps.anl.gov/APS-Upgrade (Feb 13, 2024).

[3] S Vogt. et al., *J. Phys. IV France*, **104** (2023), p. 617-622, https://doi.org/10.1051/jp4:20030156.
[4] A Glowacki, *XRF-Maps*, https://doi.org/10.11578/dc.20210824.5.
[5] A Baydin et al., *J. Mach. Learn. Res.,* **18** (2018), p. 1-43, http://jmlr.org/papers/v18/17-468.html.
[6] M Du et al., *Optics Express* (2021), p. 10000-100035. https://doi.org/10.1364/OE.418296.
[7] A Paszke et al., *NeurIPS* (2019), p. 8024–8035. https://doi.org/10.5555/3454287.3455008.

POLYMERS

C-53

Nanoscale Porosity on Polymeric Sintered Electrospun Capsules for Long-term Controlled Release

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Drug delivery vehicles made through extrusion or film formation are frequently used to control drug release rates. While effectiveness can be limited based on the drug size, incorporation of porogens can improve release rates. Implementing porogens to achieve nanoscale pores can be challenging, as traditional techniques often result in pores larger than the nanoscale, leading to biphasic drug delivery. Incorporating salts into polymer solutions used for electrospinning offers an alternative approach to generate nanoscale porosity onto sintered electrospun capsules for controlled drug delivery. Electrospun polycaprolactone (PCL):polyethylene terephthalate (PET):HEPES salt (70:10:20, 80:10:10, 85:10:5 and 88:10:2) fibers were generated using a Fluidnatek LE-100 unit. Following our previous work, samples were electrospun onto 3 mm rods and sintered at 100°C for 3 h under vacuum to form dense specimens. After being removed from the rod, salts were leached out in water, samples were then sealed with a model drug (rose bengal, RB) and oil carriers (hydrophobic and hydrophilic) to form closed capsules. Microstructure, water uptake and RB release were studied.

Nanoscale pores were observed on electrospun and sintered capsules post-water treatment. Pore size and interconnectivity varied depending on HEPES content with more interconnectivity at higher initial salt loading. Water uptake over 49 days showed no water uptake when a hydrophobic oil was used. Meanwhile, depending on the type of hydrophilic oil used, water uptake increased either 15% or 30% of the initial capsule weight. RB release from capsules following salt removal and using a hydrophilic oil as the carrier showed zero-order *in vitro* delivery over 10 days. Obtained release rates were 6.87 ± 0.23, 10.56 ± 0.09, 33.81 ± 0.56 and 60.73 ± 1.34 $\mu g\ day^{-1}$ for 88:10:2, 85:10:5, 80:10:10 and 70:10:20 PCL:PET:HEPES, respectively. The inclusion of soluble salts in polymeric solutions is a promising approach for creating nanoscale porosity in electrospun fibers. Following densification and post-salt leaching, we can enhance the porosity and interconnectivity of the resulting capsules. These sintered vehicles have the potential to improve drug loading, as they can be tailored to different sizes and can use various carriers to enhance water uptake and drug delivery.

A-18

Kinetic Viscoelasticity during Early Polymer-polymer Spinodal Dewetting

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The dewetting kinetics of a supported polymer bilayer were measured *in situ* using coherent grazing incidence x-ray scattering. X-ray photon correlation spectroscopy (XPCS) provides both the two-time correlation functions and the cross-correlation function which measures the average spatial shift of the speckles produced by the coherent x-rays. The stress in the ultra-thin dewetting top film can be directly observed due to exquisite sensitivity to sample curvature changes provided by the x-ray speckle correlation functions. The hole opening events in the film are found to be associated with significant changes to the stress. These results are interpreted through an analogy between viscoelastic spinodal dewetting and early-stage bulk viscoelastic phase separation. The frequency of hole initiation events during dewetting decreases with time as a power law, the power-law exponent can be linked to non-linear viscoelastic effects, showing similarity in their stress relief dynamics to aftershock decays.

We will also present data on dewetting velocity from holes and late stage coarsening as well.

[1] J. Lal, L. B. Lurio , D. Liang, S. Narayanan, S. B. Darling and M. Sutton, “Kinetic viscoelasticity during early polymer-polymer spinodal dewetting” *Phys. Rev. Research*, **3,** 043162 (2021).
[2] J. Lal, L. B. Lurio, D. Liang, S. Narayanan, S. B. Darling, and M. Sutton, Universal dynamics of coarsening during polymer-polymer thin-film spinodal dewetting kinetics, Phys. Rev. E 102, 032802 (2020).
[3] J. Lal et al. Dewetting in immiscible polymer bilayer films. Phys. Rev. Materials **1**, 015601, DOI: 10.1103/Phys. Rev. Materials.1.015601 (2017).
[4] Hu, X. et al. Resonantly enhanced off-specular x-ray scattering from polymer/polymer interfaces. Eur. Phys. J. E 17, 353-359, DOI: 10.1140/epje/i2004-10147-4 (2005).
[5] Hu, X. et al. Observation of a low-viscosity interface between immiscible polymer layers. Phys. Rev. E 74, 010602, DOI: 10.1103/PhysRevE.74.010602 (2006).

TECHNIQUE

Transport Coefficient Approach for Characterizing Non-equilibrium Dynamics in Soft Matter

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After the APS-Upgrade, with substantially increased beam coherence, x-ray photon correlation spectroscopy (XPCS) possesses the capability to explore structural dynamics previously inaccessible, such as their cooperativity and heterogeneity under external stimuli. However, the established theoretical foundation of analysis lags the temporal and spatial resolution that the upgrade can deliver. Such gap hinders scientists from fully understanding the subtle dynamical changes over time, thereby limiting the scientific productivity of the beamline. We have developed an innovative method to integrate the collective influence of internal and external forces acting on the system within the framework of Markov chain and introduce a universal parameter, transport coefficient, to characterize dynamics over time. This method is verified using molecular dynamics (MD) simulated colloidal system subjected to temperature changes and a complex fluid under experimental conditions known for their complex non-equilibrium characteristics. The results reveal detailed dynamical information in non-equilibrium states and align with previous observation while providing enhanced vision of the dynamical processes. This approach represents an advancement in XPCS analysis, addressing the growing demand to extract intricate non-equilibrium dynamics. Further, the methods presented are agnostic to the nature of the material system and can be potentially expanded to other condensed matter systems.

Opportunities at the Frontiers of Resonant Inelastic X-ray Scattering Science

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The upgrade of the Advanced Photon Source (APS-U) heralds a transformative era, introducing enhancements in brilliance and reduced divergence, imperative for cutting-edge research with resonant inelastic x-ray scattering (RIXS). RIXS has produced essential information across all fundamental degree of freedom in materials, including lattice, charge, spin, and orbital dynamics and provided valuable insights into fundamentally and technologically important materials' electronic and dynamic properties at the atomic and molecular levels, spanning a broad spectrum of scientific disciplines from fundamental physics and materials science to biophysics and geophysics. While numerous intriguing scientific challenges will continue to be addressed effectively with well-established spectroscopic methodologies, the APS-U era represents an opportunity to explore new possibilities in RIXS techniques to align with cutting-edge research. This presentation highlights the latest technical advancements in the RIXS beamline at sector 27-ID-B, showcasing the introduction of a high-resolution monochromator, nano-scale x-ray focusing capabilities, and a high-resolution analyzer. These advancements elevate the RIXS beamline at APS-U to the leading edge of RIXS research, facilitating the exploration of quantum materials phenomena with unprecedented resolution in the meV range, high-pressure RIXS studies, wavefront manipulation RIXS techniques, and time-resolved RIXS investigations.

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A-62

An Innovative Integration of High-resolution Synchrotron X-ray Laminography Fluorescence Imaging

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Computed laminography (CL) is developed and executed to achieve high spatial resolution in three-dimensional imaging of flat, laterally extended objects. This study explores the synergy of synchrotron laminography, x-ray fluorescence (XRF), and efficient data analysis software, aiming to enhance x-ray imaging capabilities at beamline 2-BM and 2-ID-E of the Advanced Photon Source (APS) at Argonne National Laboratory. Unlike the conventional computed tomography (CT) setup, our study leverages the pivotal innovation of laminography to streamline large sample scans by minimizing cutting procedures in this study. The investigation encompasses diverse subjects, including mammalian neural tissues and bones, plant tissues, and integrated circuits, investigated by synchrotron CL and synchrotron XRF techniques with a tilted and rotary stage along the beam direction. After data acquisition, the TomocuPy software was employed for laminography reconstruction. Subsequently, the Dragonfly ORS software was used to produce high-quality interactive volume and multichannel visualizations, facilitating the visualization and analysis of the 3D reconstructed data. The integration of these techniques, facilitated by the advanced synchrotron source, holds the potential for substantial advancements in imaging technology.

Enhancing Depth Resolution in Multi-slice Ptychography Using Advanced Reconstruction Techniques

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In recent years, x-ray ptychography has emerged as a crucial imaging technique to unveil the intricate structures of materials offering unprecedented resolution. With the Advanced Photon Source Upgrade, feature beamlines such as the PtychoProbe will greatly benefit from the increased brightness and be capable of imaging large specimens at sub-10 nm resolution. The reconstruction of such datasets requires an approach that takes wavefield propagation effects into account. Multi-slice ptychography stands out as a significant advancement as it overcomes the inherent limitations of the conventional single-slice model by accommodating the complex scattering effects within thick objects. However, experimental data often lacks sufficient information that guarantees an exact recovery of the 3D structure, resulting in artifacts such as feature blurring, crosstalk, or even random noise. In this study, we delve into several novel approaches to improve the depth resolution of multi-slice ptychography. Reconstruction methodologies such as virtual depth sectioning and dynamic reconstruction strategies on the reconstruction quality are assessed. Moreover, we examine deep learning techniques that attempt to mitigate crosstalk artifacts between the reconstructed slices. The systematic evaluation of such novel reconstruction strategies on the simulated and experimental datasets will be discussed in detail.

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Deep Learning of Structural Morphology Imaged by Scanning X-ray Diffraction Microscopy

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Scanning x-ray nanodiffraction microscopy is a powerful technique for spatially resolving nanoscale structural morphologies by diffraction contrast. One of the critical challenges in experimental nanodiffraction data analysis is in disentangling the crystalline lattice information from the effects of the zone plate optics. The convergence angle of nanoscale focusing optics creates simultaneous dependency of the far-field scattering data on three independent components of the local strain tensor – corresponding to dilation and two rigid body rotations of the unit cell. All three components are in principle resolvable through a spatially mapped sample tilt series; however, traditional data analysis is computationally expensive and prone to artifacts. In this study, we implement NanobeamNN, a convolutional neural network that learns lattice strain and rotation angles from simulated diffraction of a focused x-ray nanobeam by an epitaxial thin film. NanobeamNN predicts these parameters from experimental data without the need for additional fine-tuning. We demonstrate that this approach represents a significant advancement in computational speed over conventional methods, as well as a potential improvement in accuracy over the current standard.

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