NSLS-II & CFN USERS' MEETING

2016 NSLS-II & CFN Users' Meeting Poster Session

Abstracts

XFM: A Versatile and Multi-modal X-ray Fluorescence Microprobe

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XFM is unique as a spectroscopy beamline because it spans the tender (2-5 keV) and hard (5-23 keV) energy range (P to Tc K-edges), and also offers a polychromatic "pink" mode ideal for rapid 2D and 3D fluorescence imaging and tomography. It employs compound focusing to achieve a user-tunable spot size from 1 to 10 µm while maintaining a long working distance for physically large samples and custom in situ environmental cells. If desired, a pre-focused bulk (1×1mm) beam is available at the sample position for medium energy powder XRD and bulk EXA FS measurements.

Elucidating the Mechanism of Nanoparticle Uptake by Higher Plants

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Nanoparticulate cerium oxide (CeO_2) is new diesel fuel additive used in Europe to increase fuel efficiency and decrease air pollution. CeO_2 has been deposited onto soils through fuel emissions and biosolids application; however, little is known about its impact on environment or potential for trophic transfer in food web. In particular, the role of surface chemistry has not been investigated. The objective of this study was to understand the role of polymer coating charge on the uptake, translocation and subcellular distribution of CeO_2 in the tomato (Solanum lycopersicum cv Micro-Tom). Dextran (DEX; neutral), diethylaminoethyl dextran (DEAE; positive) and carboxymethyl dextran (CM; negative)-coated CeO_2NP (3-5 nm primary particle size) were synthesized and characterized for primary particle size (TEM), hydrodynamic diameter (DLS) and zeta potential (PALS) in exposure media. Tomato seedlings were grown in 10% Hoagland solution are exposed to DEAE, DEX and CM CeO_2NPs and control media for 2 weeks. The bulk tissue concentrations of Ce were determined in shoots and roots by inductively coupled plasma mass spectrometry (ICP-MS). Tissues were also collected from the root tip region and prepared by thin sectioning for elemental mapping using synchrotron based X-ray fluorescence (XRF) at the Hard X-ray Nanoprobe beamline (HXN) at the National Synchrotron Light Source-II (Upton, NY, USA). The preliminary results indicate that CeO_2NP with positively charged coatings have the greatest impact on seedling growth. There is a trend of decreasing values of bioconcentration factor for all three CeO_2NP with increasing exposure concentration. Ce was concentrated mostly in the root tissue although there is significant translocation of Ce from the roots to shoots. The distribution of Ce in the root tip cells (down to 10-15 nm resolution) vary with the charge states of the coatings. NPs with neutral coatings were transported between cell wall and plasma membrane, suggesting an apopolastic transport mechanism to endodermis. NPs with negative coatings have a nearly uniform distribution in the root cortex. NPs with positive coatings accumulate preferentially at rhizoplane.

Argon Blocks the Way

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CO at elevated pressures penetrates readily through two-dimensional zeolite model systems consisting of a nanoporous crystalline ultra-thin (0.5 nm) aluminosilicate films to adsorb on a Ru(0001) surface. In this work we carry out infrared reflection absorption spectroscopy (IRRAS) experiments to show that, for the case of an all-Si 2D-zeolite model, trapping Argon atoms within the nanopores blocks the passage of CO molecules. This then prevents the adsorption of CO on the Ru(0001) surface. The trapping of Ar atoms on the 2D-zeolite model was also demonstrated by ambient pressure XPS at the CSX-2 beamline of NSLS-II.

Chain Conformation near the Substrate Interface in Nanoparticle Stabilized Polymer Thin Films

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Addition of a diverse array of inorganic nanoparticles to ultrathin polymer films is vital to achieve the necessary attributes for the next-generation polymer thin film technology including sensors, energy conversion devices, and coatings. At the same time, nanoparticles have been used to stabilize ultrathin polymer films against dewetting: they often migrate to the film-substrate interface and form a "diffused immobile interfacial layer", which serves to screen the polymer-substrate interaction, imparts pining to the contact line, and suppress dewetting. An unsolved question is how the conformations of the polymer chains in the immobile layer are affected by the nanoparticles and the relationship with the improvement of film stability. To address the question, dodecane thiol-functionalized gold nanoparticles (2.8 nm in diameter) and polystyrene (PS, Mw = 30 kDa and 50 kDa) were used as a rational model. We found that the Au nano-particles induce complete dewetting suppression of 20 nm-thick PS/Au thin films on cleaned Si substrates at 0.25% (volume of the particle/volume of the polymer). To investigate the interfacial structures at the polymer-solid interface, we rinsed the annealed PS/Au thin films with toluene and characterized the residual interfacial layers by using a suite of surface sensitive experimental techniques including X-ray reflectivity, grazing incidence small angle X-ray scattering, X-ray photon correlation spectroscopy (at NSLS/NSLS-II), atomic force microscopy, X-ray photoelec-tron spectroscopy, and scanning electron microscopy (at CFN). The results indicate that when nanoparticles are added, the conformation of the polymer chains becomes elongated and the ad-sorbed chains can act as "connector molecules" to stabilize the thin film.

High Data-Rate Image Processing for Macromolecular Crystallography

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The very high data rates and data volumes now being seen with the new generation of pixel-array x-ray detectors at synchrotron beam-lines tax our computing resources severely. This will require tuning and reorganization of computing infrastructure, and well-established processing pipelines, to make much more aggressive use of parallelism and to avoid unnecessary motion of data. Standard cluster environments, such as SGE, need to be re-tuned to work on much shorter time scales than usual. Standard spot-finding approaches need to be revised to make more use of "regions of interest", binning, and image summing when appropriate.

Multiple conversions of images need to be avoided and compressions need to be very carefully chosen to balance the need for compact data streams against competing demands for CPU access. Data, that once could have resided on single ordinary rotating disks, now need to be in memory as much as possible, on SSDs, or when necessary, on heavily striped RAIDS of rotating disks. Networks need to be several times faster than in the past. Each of the major processing flows, such as in DIALS and XDS, need to be re-tuned to allow as much parallel processing of images as possible.

We appreciate the cooperation shown by Dectris, the DIALS group, especially Nicholas K. Sauter, Graeme Winter, Gwyndaf Evans, and James Parkhurst, and by the XDS project, especially Kay Diederichs, as well as by collaborators at many other facilities, and the assistance and support of Robert M. Sweet and Sean McSweeney of BNL, and Kaden Badalian of Binghamton University.

Financial support for this work comes principally from the US Department of Energy Offices of Biological and Environmental Research and of Basic Energy Sciences grants DE-AC02-98CH10886 and E-SC0012704 and from NIH grants P41RR012408, P41GM103473, and P41GM111244. H. J. Bernstein also has support from Dectris Ltd.

Two Highly Integrated Experimental tations for FMX/AMX Beamlines at NSLS-II

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We present the updated design and fabrication status of two highly integrated experimental end stations for the micro-focusing(FMX) and the highly automated (AMX) MX beamlines at the National Synchrotron Light Source II. These experimental end stations are designed in-house to meet the challenging requirements from the small beam size of 1µm and the extremely short working distance of only 190mm from the beam exit window to the FMX focal spot. We designed these experimental stations with high modularity for future upgrades and extensions. Central components of the in-house-developed experimental stations [3] are a horizontal axis goniometer with a 100 nm sphere of confusion, piezo-slits for dynamic beam size changes during diffraction experiments, a dedicated secondary goniometer for data collection from crystallization plates, and a next generation pixel array detector.

FMX and AMX will support a broad range of biomedical structure determination methods from serial crystallography on micron-sized crystals, to structure determination of complexes in large unit cells, to rapid sample screening and room temperature data collection of crystals in trays.

This work is supported by the US National Institutes of Health and the US Department of Energy.

Balancing Intracellular Metal Homeostasis with Compartmentalization

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Trace nutrients such as Zn, Cu, Fe and Mn present a unique challenge to life. On the one hand, these metal ions are indispensable cofactors for a large number of proteins that catalyze essential chemistry within the cell. On the other hand, overabundance or improper handing of these ions can result in toxicity such as protein inactivation and the generation of reactive oxygen species. As a result, organisms have evolved to fine-tune morphological, physiological, and molecular responses to meet their catalytic demand for metals while avoiding toxicity. An under-appreciated component of metal homeostasis is subcellular compartmentalization. An organism can not always control the flux of nutrients in its environment, but it can prepare for times of insufficiency by stockpiling essential nutrients during times of plenty. At the Submicron Resolution X-ray Spectroscopy Beamline (SRX) at NSLS-II, we are taking a glimpse into the otherwise invisible world of subcellular metal compartmentalization. Combined with transcriptomics, genetics and molecular biology we are building a systems-level view of trace nutrient homeostasis.

Fabrication of High Efficiency Focusing Optics for Hard X-ray Microscopy

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Multilayer Laue lenses (MLLs) optics offer great promise for focusing hard x-rays to nanometer level. Fabrication of MLLs rely on accurate depth-graded multilayer film deposition of thousands of layers over tens of microns as well as high quality sectioning of the film to the appropriate thickness for operation. Here we report on our developments which led to the fabrication of MLL optics for the HXN beamline[1] at NSLS-II, delivering a 15 x 15nm[2] focus at 12keV, and the fabrication of the largest MLL, 102 microns, reported to date[2]. To reach higher spatial resolution and efficiency, wedged MLLs optics are needed[3]. Wedged MLLs are based on the challenging deposition of profiled and depth-graded layers to form the multilayer. Our recent advances on wedged MLLs fabrication will also be presented.

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Work carried out at National Synchrotron Light Source II and the Center for Functional Nanomaterials at Brookhaven supported by the Department of Energy, Office of Basic Energy Sciences under contract DE-SC00112704.

Development of V_2O_5 Nanostructure for Aqueous Energy Storage Applications via Scalable Exfoliation of Bulk Material

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Aqueous electrochemical energy storage (EES) devices are a promising technology for a wide range of applications due to their low cost and superior safety compared to Li-ion batteries. However, their low energy density has inhibited implementation. Materials with layered structures possess favorable characteristics for improving the energy density of aqueous EES devices, in particular aqueous EES using cations with larger radii (e.g. Na+ and K+) which are more abundant and cheaper than Li+ but also have smaller radii of solvation in aqueous solutions. In this study the exfoliation of bulk vanadium pentoxide was achieved via a wet chemical method using a conductive polymer. The resulting exfoliated V_2O_5 material was characterized through various techniques and was shown to possess a layered structure with large interlayer spacing. Electrochemical investigations were performed utilizing a neutral, aqueous, 1M KCl electrolyte in a three electrode half-cell. Cyclic voltammetry measurements of the exfoliated V_2O_5 material exhibited a significant increase in capacitance as compared to the bulk V_2O_5 material at both fast and slow sweep rates. This work demonstrates top down approach for the development of high capacity electrode materials for aqueous EES devices.

Energy Transfer from Isolated Perovskite Nanoparticles to Single-layer Graphene

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Metal halide perovskites have attracted enormous attention recently due to their advanced applications in photovoltaic, light-emitting and optoelectronic devices. Hence, we have investigated the interaction between cesium lead halide, $CsPbI_3$, perovskite nanoparticles (PNPs) and single-layer graphene (SLG). Nonradiative energy transfer from isolated PNPs to SLG has been verified by photoluminescence (PL) blinking dynamics coupled with PL quenching of intensity and lifetime. Our results indicate that graphene can be utilized as a better acceptor material than TiO_2 for perovskite-NPs based optoelectronics. The role of energy transfer instead of charge transfer is truly important in the PNPs/SLG system. As a result, the combination of PNPs nanomaterials with two-dimensional (2D) graphene substrates establishes a new possibility in hybrid 0D-2D systems.

In-situ X-ray Characterization of Multi-alkali Antimonide Photocathode-Grown by Tripe Evaporation and Sputtering Methods

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Multi-alkali antimonide K2CsSb is an excellent photocathode material for electron sources in the next generation light source, such as LCLS, ERL and FEL. It has outstanding performance in the aspects of quantum efficiency, emittance, life time and response time. Traditionally-grown K2CsSb generally has a rough surface (up to 25 mm rms roughness), which is not favorable in the future electron gun. Therefore it is necessary to explore novel growth methods. This study includes X-ray characterization (XRD, XRR, XRF) on K2CsSb grown by triple evaporation method and sputtering. Results show that to achieve a photocathode with high QE and low roughness by these two methods is possible.

X-ray Beam Stabilization System Utilizing Diamond Beam Position Monitors

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The mechanical, optical, electronic and thermal properties of diamond make it an ideal material to address the x-ray beam monitoring needs of modern synchrotrons. Diamond Beam Position Monitors (DBPMs) have demonstrated to yield position resolutions of 25 nm for stable beams and a have shown linear flux responses of at least 11 orders of magnitude [1]. Readout electronics tailored to suit the performance and integration needs of DBPMs are needed to fully harness the potential of the technology. Sydor Instruments LLC in collaboration with Brookhaven National Laboratory (BNL) has advanced novel readout electronics package based on BPM readout systems developed for the National Synchrotron Light Source II (NSLS-II).

The SIEPA3P advanced electrometer is a fully integrated DBPM x-ray beam readout and stabilization system. The system features digital controls for DBPM operation, configurable internal feedback routines, digital to analog outputs for device control and a fast 10kHz readout for x-ray beam diagnostics. The system controls are Ethernet based and compatible with the Experimental Physics and Industrial Control System (EPICS), utilizing a custom built Control System Studio (CSS) user interface. The system has been deployed at the NSLSII CHX beamline for x-ray beam diagnostics and stabilization. The SIEPA3P utilizes a Sydor Instruments' DBPM for beam characterization in conjunction with a horizontal mirror and a Double Crystal Monochromator for x-ray beam stabilization. A general overview of the system and its beam stabilization performance will be presented.

Acknowledgements

This material is based upon work supported by the U.S. Department of Energy Office of Science, Office of Basic Energy Sciences, Small Business Innovation Research (SBIR) and Small Business Technology Transfer (STTR) award Number DE-SC0007482. Use of the National Synchrotron Light Source II, Brookhaven National Laboratory, was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-SC0012704.

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Characterization of Ba-rich, Contaminant Laden Nano-colloids and Nano-aggregates

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We performed x-ray nano-spectroscopy imaging at NSLS2's HXN beamline to investigate arsenate and arsenite uptake during barium sulfate precipitation at high salinities typical of wastewater produced as part of shale gas operations. High-resolution fluorescence maps of the relative abundance of arsenate and arsenite species within individual barite particles provide novel insights into the time evolution of arsenic incorporation into barite colloids. Images of individual barite platelets reveal low arsenic within 1-2 micron diameter cores, indicating relatively low arsenic incorporation rates during barite nucleation and early platelet growth. Although both nanoscale oxidation state mapping of individual particles and x-ray absorption fine structure (XAFS) spectra averaged over ensembles of particles indicate that pentavalent arsenate is preferentially incorporated into barite, trivalent arsenite can be taken up and preserved within barite. Complementary light scattering and electron microscopy results show how increasing salinity inhibits the onset of barite precipitation and promotes the formation of sub-micron diameter colloids with relatively slow settling times that could defeat sedimentation approaches to wastewater treatment. In addition to rethinking treatment strategies for hyper-saline wastewater, this work explores how the latest advances in x-ray spectroscopy imaging, which have enabled the mapping of element speciation within an individual particle, can be used to study interfacial phenomena at colloid surfaces.

On-axis Microscopy

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A series of versatile on-axis X-ray microscopes with large working distance, high resolution, large magnification, for in-situ sample alignment and X-ray beam visualization have been developed for beamlines at NSLS-II. The microscopes use reflective optics, which minimizes dispersion, and allows imaging from Ultraviolet (UV) to Infrared (IR) with specifically chosen objective components (coatings, etc.).

Influence of Cadmium Overpressure on the the Crystalline Quality of Cadmium Zinc Telluride Single Crystals

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Cadmium Zinc Telluride (CZT) single crystals are the material of choice for the room temperature detection of γ-rays and hard x-rays in security, medical and space applications. Obtaining high quality single crystal is a challenge in the growth of CZT crystals, and the high density of structural imperfections limits the detection power of CZT-based detectors and their energy resolution. Further a critical requirement for CdZnTe detector applications is high electrical resistivity to reduce the bulk leakage current. By growing CdZnTe under controlled Cd over-pressures, high resistivity has been reproducibly obtained. However, it is also necessary to obtain a thorough understanding of this growth modification on the structural quality of the crystals grown. In this study, the influence of Cadmium (Cd) overpressure on the growth of CZT single crystals by vertical directional solidification (Bridgman technique) method is evaluated. To that end, boules with different Cd over-pressures were grown. Axial and transverse slices cut from the boules and polished were investigated using a combination of synchrotron white beam x-ray topography (SWBXT), Infrared spectroscopy and double axis x-ray diffraction. Preliminary SWBXT results reveals the presence of twining and a uniform distribution of a network of subgrain boundaries and dislocations. Infrared mapping revealed a high density of Te inclusions, which decorated twin boundaries and aligned along subgrain boundaries and dislocations. The Te inclusions have circular and triangular shapes, and their size is between 10 to 60µm. These results will shed light on the influence of Cd overpressure on the structural perfection of CZT crystals.

Characterization of Heterogeneity in Nuclear Waste Glass

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Cleanup of legacy tank waste at the Hanford Site is one of America's most pressing environmental challenges. Efforts are underway to increase the loading of radioactive liquid wastes in glass; that is, increasing the specific concentration of a particular constituent of interest within the glass form while maintaining adequate processability, regulatory compliance and product quality. The rate of glass production during vitrification greatly impacts the cost and schedule of nuclear waste treatment and immobilization. A fundamental tenet underlying the current research efforts is an attempt to be less conservative than previous work that is currently implemented in vitrification facility operations. This requires that all of the characteristics related to long-term stability, such as the solubility of fission products in glass, state of the glass matrix under waste loading, the structure and composition of each separated phase, and the interaction of glass with separated phases, be fully understood. The research outlined here is motivated by the potential for substantial economic benefits (e.g., significant increases in waste throughput and reductions in glass volumes) that can be realized with advancements in glass formulation. Information obtained via synchrotron studies will enable a better understanding of how variations in composition and processing contribute to the formation of heterogeneities (crystals and/or separated phases). Ultimately, processes to control the nature of heterogeneities and their impact on the physical and chemical properties of glasses (chemical durability, thermal and electrical conductivities, rheological and mechanical properties) can be developed. Redox ratios of a few key elements in simplified and surrogate waste glasses were obtained using XAFS. XRF mapping was performed to investigate the spatial distribution of elements and, in particular, detect regions of non-homogeneity.

Study of Stress Relaxation during CVD Homoepitaxial Growth of 4H-SiC for Power Electronics Application

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Interfacial dislocations (IDs) and half-loop arrays (HLAs) present in the epilayers of 4H-SiC crystal are known to have a deleterious effect on device performance. Synchrotron X-ray Topography studies carried out on n-type 4H-SiC offcut wafers before and after epitaxy growth show that in many cases BPD segments in the substrate are responsible for creating IDs and HLAs during CVD growth. This paper reviews the behaviors of BPDs in the substrate during the epitaxy growth in different cases:(1) screw-oriented BPD segments intersecting the surface replicate directly through the interface during the epitaxy growth and take part in stress relaxation process by creating IDs and HLAs(Matthews-Blakeslee model [1]); (2) non-screw oriented BPD half loop intersecting the surface glides towards and replicates through the interface, while the intersection points convert to threading edge dislocations (TEDs)and pin the half loop, leaving straight screw segments in the epilayer and then create IDs and HLAs; (3) edge oriented short BPD segments well below the surface get dragged towards the interface during epitaxy growth, leaving two long screw segments in their wake, some of which replicate through the interface and create IDs and HLAs. The driving force for the BPDs to glide toward the interface is thermal stress and drive force for the relaxation process to occur is the lattice parameter difference at growth temperature which results from the doping concentration difference between the substrate and epilayer.

High-pressure PDF Experiments Using High-energy X-ray Microbeam

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We have developed a micro-focusing optics for high-energy x-rays by combining a sagittally bent Laue crystal monochromator with Kirkpatrick-Baez (K–B) X-ray focusing mirrors. The optical system is able to provide a clean, high-flux X-ray beam suitable for pair distribution function (PDF) measurements at high pressure using a diamond anvil cell (DAC). A focused beam of moderate size (10-15 µm) has been achieved at energies of 66 and 81 keV. PDF data for nanocrystalline platinum (n-Pt) were collected at 12.5 GPa with a single 5 s X-ray exposure, showing that the in-situ compression, decompression, and relaxation behavior of samples in the DAC can be investigated with this technique. PDFs of n-Pt and nano Au (n-Au) under quasi-hydrostatic loading to as high as 71 GPa indicate the existence of substantial reduction of grain or domain size for Pt and Au nanoparticles at pressures below 10 GPa. The coupling of sagittally bent Laue crystals with K–B mirrors provides a useful means to focus high-energy synchrotron X-rays from a bending magnet or wiggler source.

First Measurement of Lubberts Effect in CsI(Tl) Scintillators at NSLS-II

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Background: The spatial resolution of x-ray intensifying screens (e.g. Gd_2O_2S:Tb, CsI:Tl) varies as a function of x-ray interaction depth z within the screen, due to differences in the lateral diffusion of light. This variation is describes the Lubberts effect, which is a fundamental source of noise in scintillating x-ray detectors. The Lubberts effect has been estimated in various scintillators through indirect means, but has never been measured directly. Understanding this effect is crucial to the optimization of scintillating detectors used in medical radiography, which are challenged to maximize image quality while minimizing patient dose.

Methods: Samples of columnar CsI:Tl scintillators with thickness 200-1000 µm were evaluated. The point spread function (PSF) of individual x-ray interactions was measured as a function of z using a high-sensitivity II-EMCCD camera and a 32 keV scanned slit beam (30 µm thickness). The ensemble of PSFs collected at each z were averaged and used to compute the modulation transfer function (MTF). Resulting estimates of MTF(z,f) were then used to determine the Lubberts function L(f) of each scintillator under 32 keV irradiation.

Results: Variation in MTF(z,f) increased with scintillator thickness. At 5 cycles/mm, MTF(z,f) varied by 12.8%, 30.7%, and 112% over the depth extrema of the 200, 600 and 1000 µm samples, respectively. The average MTF at 5 cycles/mm was 0.124, 0.072 and 0.049 in the 200, 600, and 1000 µm samples, respectively. Under 32 keV excitation, L(f) was calculated to be 0.993, 0.988 and 0.947 in the 200, 600 and 1000 µm samples.

Conclusions: This work directly measures the Lubberts effect for the first time, and provides preliminary estimates of its extent in columnar CsI:Tl scintillators. Future work will compare MTF(z,f) and L(f) of powder phosphors such as Gd_2O_2S:Tb with CsI:Tl, and will guide optimization of scintillator design for medical imaging applications.

Environmental Transmission Electron Microscopy Study of Complex Transition Metal Oxide Battery Electrodes

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Layered transition metal oxides are highly desirable battery electrodes. And yet, these materials suffer from thermal runaway caused by deleterious oxygen loss and surface phase transitions at highly overcharged and overheated conditions, prompting serious safety concerns. Using in situ environmental transmission electron microscopy techniques, we demonstrate surface oxygen loss and structural changes in the highly overcharged particles are suppressed by exposing them to an oxygen-rich environment. The onset temperature for the loss of oxygen from the electrode particle is delayed to 350 °C at oxygen gas overpressure of 400 mTorr. Similar heating of the particles in a reducing hydrogen gas demonstrated a quick onset of oxygen loss at 150 °C and rapid surface degradation of the particles. The results reported here illustrate the fundamental materials science governing the failure processes of electrode particles and highlight possible strategies to circumvent such issues.

NSLS-II Biomedical Beamlines forMicro-crystallography, FMX, and for Highly Automated Crystallography, AMX: New Opportunities for Advanced Data Collections

Edwin Lazo, Jean Jakoncic, Martin R. Fuchs , Dieter K. Schneider, Wuxian Shi, Babak Andi, Alexei Soares, Vivian Stojanoff, Dileep K. Bhogadi, Herbert Bernstein, Stuart Myers, Bruno Seiva Martins, John Skinner, Robert M. Sweet, Lonny E. Berman and Sean McSweeney

The two new beamlines at the National Synchrotron Light Source-II, for microfocus (FMX) and highly automated (AMX) crystallography, have seen first light on March 8, 2016, and will begin scientific commissioning in summer 2016. The exceptionally low emittance of the NSLS-II storage ring is the basis for providing previously unattainable beam parameters [1,2] to address current and new challenges in crystallography. With a flux of 0.6 – 0.8×1013 ph/s at 1 Å, and beam sizes from 1 – 50 µm (FMX) and 4 – 100 µm (AMX), the new beamlines' dose rates are up to two orders of magnitude higher than those of the current brightest MX beamlines. They will cover a wavelength range from 0.4 Å (FMX) and 0.7 Å (AMX) to 2.5 Å.

A focus in designing the beamlines lay on supporting a broad range of structure determination methods. The highly flexible design of the experimental stations and of the beam control will support serial crystallography on micron- and sub-micron sized crystals, structure determination of complexes in large unit cells, rapid sample screening and room temperature data collection. The associated wide variety of samples includes frozen crystals in standard loops and meshes, single crystals or up to 20 acoustically deposited crystals, crystals in SBS trays, specialized sample holders such as silicon nitride membranes, micro-fluidic- or LCP-plates & jets.

Probing Charge Transfer in a Novel Class of Luminescent Perovskite-Based Heterostructures Composed of Quantum Dots Bound to RE-Activated CaTiO3 Phosphors

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Here we highlight the synthesis and structural characterization of novel semiconducting heterostructures composed of cadmium selenide (CdSe) quantum dots (QDs) attached onto the surfaces of novel high-surface area, porous rare-earth-ion doped alkaline earth titanate micron-scale spherical motifs, i.e. both Eu-doped and Pr-doped CaTiO3, composed of constituent, component nanoparticles. These unique metal oxide perovskite building blocks were created by a multi-pronged synthetic strategy involving molten salt and hydrothermal protocols. Subsequently, optical characterization of these heterostructures indicated a clear behavioral dependence of charge transfer in these systems upon a number of parameters such as the nature of the dopant, the reaction temperature, and particle size. Specifically, 2.7 nm diameter ligand-functionalized CdSe QDs were anchored onto sub-micron sized CaTiO3-based spherical assemblies, prepared by molten salt protocols. We found that both the Pr- and Eu-doped CaTiO3 displayed pronounced PL emissions, with maximum intensities observed using optimized lanthanide concentrations of 0.2 mol% and 6 mol%, respectively. Moreover, the ligand-capped CdSe QD-doped CaTiO3heterostructures exhibited effective charge transfer between the two individual constituent nanoscale components, an assertion corroborated by the corresponding quenching of their measured PL signals.

Reaction Kinetics of Spinel Cobalt Oxide for Lithium Ion Battery

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Among of the available kinds of batteries, lithium-ion batteries have attracted extensive attention because of their high energy density. Spinel metal oxide have been widely studied as anode materials due to their high theoretical capacity and good capacity retention, such as Co3O4. However, the reaction mechanisms of those materials that occur during lithiation still remain unclear.

We combined galvanostatic intermittent titration techniques (GITT), cycled coin cell as well as in-situ transmission electron microscopy (TEM) to get a better understanding of lithiation kinetics of Co3O4. Therefore, we confirmed that the over all reaction is Co3O4+ Li+ -> Li2O +Co and after first charge back to 3V, it generate CoO rather than pristine Co3O4. Moreover, because of irreversible incorporation of Li ions pushed the Co2+ ions that were present in the 8a site to neighboring 16c sites, a intermediate phase (LixCo3O4) occurred during initial lithiation process. Above all, understanding the reaction mechanisms of electrode materials provides needed insight into the different performances observed in different material.

In situ Probing of the Active Site Geometry of Ultrathin Nanowires for the Oxygen Reduction Reaction

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To create truly effective electrocatalysts for the cathodic reaction governing proton exchange membrane fuel cells (PEMFC), namely the oxygen reduction reaction (ORR), necessitates an accurate and detailed structural understanding of these electrocatalysts, especially at the nanoscale, and to precisely correlate that structure with demonstrable performance enhancement. To address this key issue, we have combined and interwoven theoretical calculations with experimental, spectroscopic observations in order to acquire useful structural insights into the active site geometry with implications for designing optimized nanoscale electrocatalysts with rationally predicted properties. Specifically, we have probed ultrathin (~2 nm) core−shell Pt~Pd9Au nanowires, which have been previously shown to be excellent candidates for ORR in terms of both activity and long-term stability, from the complementary perspectives of both DFT calculations and X-ray absorption spectroscopy (XAS). The combination and correlation of data from both experimental and theoretical studies has revealed for the first time that the catalytically active structure of our ternary nanowires can actually be ascribed to a PtAu~Pd configuration, comprising a PtAu binary shell and a pure inner Pd core. Moreover, we have plausibly attributed the resulting structure to a specific synthesis step, namely the Cu underpotential deposition (UPD) followed by galvanic replacement with Pt. Hence, the fundamental insights gained into the performance of our ultrathin nanowires from our demonstrated approach will likely guide future directed efforts aimed at broadly improving upon the durability and stability of nanoscale electrocatalysts in general.

Multilayered Block Copolymer Architectures Probed by GISAXS

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The formation of periodic structures via diblock block copolymer (BCP) thin film self-assembly is an elegant realization of the "bottom-up" fabrication concept and a facile approach to surface nano-patterning. Unfortunately, the principles of self-assembly of BCP, e.g., a demand for minimization of interfacial area between the two blocks, are limiting the number of available morphologies to a handful (i.e., lamellae, hexagonally-packed cylinders, cubically-packed spheres). To expand the library of structural motifs realized by BCP self-assembly, we are investigating the formation of multilayered structures composed of BCPs with various morphologies and domain orientation. The multilayered structures are realized by a step-by-step approach; the first BCP layer is annealed and crosslinked or converted to an inorganic replica before the next layer is coated processed. Grazing incidence small angle X-ray scattering (GISAXS), scanning electron microscopy (SEM) and atomic force microscopy (AFM) are used to characterize the morphology of the obtained layered nanostructures and to elucidate the epitaxial mechanism underlying the registry of the periodic lattices between the layers.

Identifying Sulfur-microphase in Block Copolymer Membranes Using Tender Resonant X-ray Scattering (TReXS)

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The last few years have seen a new twist in fabrication of next generation materials. Thus, developing a tool to probe the complexity of such materials is needed. Tender Resonant X-ray Scattering (TReXS) is a nano-probe technique for soft and hard matter, utilizing the strong resonant edges primarily at few keV energy range to elucidate the chemical structure of polymers, biological membranes and energy storage materials. Recently, a block copolymer (BCP) electrolyte membrane has been used as a drug capture [1]. A series of triblock copolymer membranes have been fabricated and thoroughly characterized using TReXS and scanning transmission electron microscopy (STEM), in order to understand the relationship between membrane morphology and doxorubicin capture. TReXS revealed the morphological underpinnings of porous and nonporous polystyrenesulfonate-block-polyethylene-block-polystyrenesulfonate membranes, as the hydrophilic polystyrene sulfonate microphase was found to be the main responsible for doxorubicin capture. This study demonstrates that TReXS is not only a powerful tool to directly identify the microstructure of sulfur-phase, as it can also be used to probe and elucidate the chemical structure of the species in block copolymer membranes. The gap between data collection and real time data treatment will be addressed in the present poster.

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Images in Chemistry in the Natural World

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An X-ray fluorescence microprobe can image patterns of chemistry in heterogeneous materials. Presented here are examples of natural materials imaged in the "tender" energy range (1-5 keV), which permits measurement of light elements (Mg through Ca) as well as heavier elements using their L and M fluorescence emission.

Simulating Beamlines and User Experiments with "Synchrotron Radiation Workshop"

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The “Synchrotron Radiation Workshop” (SRW) physical optics computer code has been very successfully used for design and commissioning of X-ray optical beamlines at NSLS-II and other Light Source facilities. Among new functionalities recently implemented in the code one can mention physical optics based “propagators” for grazing-incidence focusing mirrors that are extensively used in hard and soft X-ray beamlines, for perfect crystals used in hard X-ray monochromators, and for variable-line-spacing gratings that are the key elements of monochromators for soft X-rays. Detailed partially-coherent emission and wavefront propagation simulations are now routinely performed using the SRW code for the hard and soft X-ray beamlines of NSLS-II at BNL (e.g. HXN, CHX, IXS, SRX, FMX, SMI, ESM, SIX), allowing for better optimization and matching of insertion devices and X-ray optics to scientific goals of these beamlines, and the most efficient use of the high brightness and coherence of the NSLS-II radiation beams. A complement open-source cloud-based computing platform named “Sirepo” is being developed in collaboration with RadiaSoft LLC., allowing scientists to execute SRW code from a web-browser using a convenient user-friendly web-interface. This development will significantly improve user experience and usability of SRW code at NSLS-II and other facilities.

The present work was supported by US DOE contract No. DE-AC02-98CH10886, US DOE grant No. DE-SC0011237 and SBIR grant No. DE-SC0011237.

0D-2D Semiconducting Hybrids with Enhanced Absorption and Charge Mobility

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Emerging 2-D semiconductors, especially layered metal dichalcogenides (LMDs) such as MoS2, WS2 and WSe2 have shown great potential applications in photocatalysis, photon detectors and energy harvesting. These materials show band structure dependence on the number of layers and band-gap inversion at monolayer with intense visible photoluminescence, making them interesting candidates for photosensing applications. However, their low dimensionality makes photon absorption poor. In this study we present a straightforward method to increase photon absorption in such LMDs. We show that doping of single and few-layer of tin disulphide (SnS2), with colloidal CdSe/ZnS quantum dots (QDs) results in 0D-2D hybrids with large photon absorption cross-section and high charge mobility. Using time resolved single nanocrystals photoluminescence microscopy and monitoring the blinking dynamics of QDs alone and part of the LMD, we were able to unveil the interaction between the two nanoscale components as non-radiative energy transfer and to find that the rate of this process increases with the increase in the number of SnS2 layers [1]. We also found that such 0D-2D hybrids, when incorporated into field effect transistor devices, outperformed the LMD only devices by 500% in the photocurrent response and displayed substantially increased spectral responsivity [2].

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Improving Charge Transport in Solid State Avalanche Amorphous Selenium Detector

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A flat panel imager with avalanche gain is being developed to achieve quantum noise limited x-ray imaging at low doses. To overcome the electronic noise of the thin film transistor (TFT) readout, the signal must be amplified prior to storage in the pixel's capacitor. This can be accomplished using a thin layer of avalanche amorphous selenium (a-Se) referred to as High Gain Avalanche Rushing Photoconductor (HARP). In this work we successfully deposited this HARP structure onto a 24 x 30 cm2 TFT array with a pixel pitch of 85 um. An electric field (ESe) up to 105 V/um was applied across the a-Se layer without breakdown. In indirect mode with a 150 um thick structured CsI scintillator, an optical gain of 76 ± 5 was measured at ESe = 105 V/um. Image quality at low dose increases with the avalanche gain until the electronic noise is overcome at a constant exposure level of 0.76 mR. We demonstrate the success of the first solid-state HARP X-ray imager as well as the largest active area HARP sensor to date. Our future work in conjunction with the CFN is to improve charge transport within the HARP structure to eliminate charge build-up and ghosting.

Self-assembly of Block Copolymers at the Polymer-solid Interface

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Block copolymer thin films offer a simple and effective route to fabricate highly ordered periodic microdomain structures. The fundamental, yet unsolved question is whether these highly oriented microdomain structures persist even near an impenetrable solid wall. We here report adsorbed structures of polystyrene-block- poly (4-vinylpyridine) (PS-block-P4VP, Mw =41,000,weight fraction of PS=0.81) formed on planar silicon substrates. Perpendicularly aligned cylindrical microdomains were created by a solvent vapor annealing process as a model and the strongly bound polymer layer onto the substrate surface was derived by solvent leaching with chloroform, a good solvent for the polymers, and thereafter characterized by using atomic force microscopy, scanning electron microscopy (at CFN), grazing incidence small angle X-ray scattering, and x-ray reflectivity at NSLS. The results showed that both PS and P4VP chains lie flat on the substrate, forming a heterogeneous two-dimensional structure without long-range order. Moreover, a spin-coated PS-block-P4VP thin film annealed under vacuum at 190 °C (T>T g of both blocks, but below the order-disorder temperature) showed a similar microscopic heterogeneous structure on the substrate, indicating the generality of the interfacial self-assembling block copolymer structures.

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Sodium-manganese Oxide Electrode Materials for Aqueous Electrochemical Energy Storage

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Sodium-ion aqueous electrochemical energy storage devices have been widely studied recently due to their low-cost and environmental-friendliness compared with commonly used non-aqueous Li-ion batteries. Here, sodium-manganese oxide materials were synthesized by thermal treatment of the mixture of Mn3O4 and NaOH solids. Energy-dispersive x-ray spectroscopy (EDS) tests confirmed atomic ratio of sodium to manganese varied from 0.02 to 0.29 by increasing the NaOH amount during the synthesis. X-ray diffraction (XRD) analysis showed the structural evolution from Mn5O8 to MnO2 with the increased sodium concentrations. Cyclic Voltammetry (CV) tests showed that the specific capacitance of sodium-manganese oxide was 211 F g-1 at a scan rate of 5 mV s-1 in a 0.1 M Na2SO4 electrolyte, 60% higher than that of pure manganese oxide (132 F g-1) via three-electrode half-cell measurements. Electro-kinetics analysis showed that more capacitive contribution was observed in sodium-manganese oxide compared with that of pure manganese oxide. In-situ XRD measurements showed crystalline structure change and the d-spacing of (001) diffraction peak of sodium-manganese oxide varied 4% during charge and discharge processes. Our synthesis methods as well as structure and electrochemical studies of sodium-manganese oxide materials provided an insight into the development of innovative electrode materials for aqueous electrochemical energy storage.

Integrated Synchrotron Biophysics at Case Center for Synchrotron Biosciences

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The Case Center for Synchrotron Biosciences is participating in the development of world-class synchrotron resources at the NSLS-II in three areas: X-ray footprinting, X-ray absorption spectroscopy and macromolecular crystallography. Our overall vision of a Biology Village at NSLS-II includes an Integrated Synchrotron Biophysics approach where users are introduced to new technologies to help them find the best way to solve their overall scientific problem, as opposed to simply providing access to beamlines. We are constructing a new beamline, XFP, to conduct X-ray footprinting for in vitro and in vivo structural studies of biological macromolecules. Now we are expanding the capability of the XFP beamline to also support X-ray absorption spectroscopy. We are partner users at the two advanced crystallography beamlines, FMX and AMX, which are starting scientific commissioning with users in the summer of 2016. In addition, to maintain our user base and pilot the integrated analysis approach we have developed a virtual synchrotron resource model where we support synchrotron users at remote sites. We have successfully established outposts at ALS, where we support footprinting users, and at SSRL, where we support x-ray absorption spectroscopy users, while NSLS-II facilities are being brought on line.

Optimizing Cryogenic Sample Environments for X-Ray Spectroscopic Microscopy of Hydrated Specimens

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Understanding and managing synchrotron beam damage to hydrated specimens can be a complex problem because the system of interest is often being

changed by the measurement itself. Understanding or extrapolating to the initial state can be difficult with the many factors involved like flux density, temperature, hydration level, concentration, and the nature of the specific element/complex being studied. It's becoming more and more important to understand and manage beam damage as new X-ray sources come online with higher X-ray beam brilliance. Lowering the sample temperature to cryogenic levels is thought to help mitigate physical beam damage [1,2], creating a need for cryo sample environments in routine measurements.

However, it is not known if sample cooling can mitigate chemical changes caused by beam damage.

We present recent developments of a cryo sample environment for X-ray fluorescence micro/nanoprobe applications that is being used to study low temperature effects of beam-induced changes to metal oxidation states and speciation of frozen, hydrated specimens. The criteria we used for the device development were: (1) relatively easy to use for routine measurements, (2) large range of low temperatures (down to -150 C or lower), and (3) low X-ray fluorescence background signal suitable for trace element analysis. To meet these criteria, we modified a commercial cryostage with X-ray transparent windows and we managed interfering background fluorescence with shielding of the cooling surface that maintains the necessary cryogenic temperatures. Lastly, we developed a mechanism for quickly changing samples and fiducializing them with light microscopy prior to X-ray data collection.

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Far-infrared Studies of Electromagnons at U4-IR NSLS (MET NSLS-II)

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Electromagnons are electric dipole-active magnetic excitations which attracted considerable attention during the last decade due to non-trivial optical properties such as directional dichroism as well as possibility to control magnetic oscillations with electric field. We utilized two spectroscopic techniques, namely far-IR magneto-spectroscopy and mueller matrix ellipsometry (MME), to study electromagnons in DyFeO3 and TbMnO3 multiferroics, correspondingly. Two electromagnons were revealed at ~20 and 55 cm-1 in the transmission spectra of DyFeO3 below the temperature of antiferromagnetic ordering of Dy moments TN2=4.2K. We showed that the electromagnons give a significant contribution to the changes in static permittivity upon cycling of the magnetic field H. Earlier optical transmission and reflectivity studies of TbMnO3 revealed electromagnons at 19 and 62 cm-1 below Tc = 27K. On example of electromagnon positioned at 62cm-1 we demonstrate the capabilities of MME in determining parameters of magnetoelectric excitations. Also we present design and possibilities of future optical setup for generation of vortex beams with non-zero orbital angular momentum as well as toroidal moments for electric and magnetic fields utilizing far-IR synchrotron radiation of NSLS-II.

Selenium Multi-Well Detectors: Fabrication and Characterization

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Direct conversion Active Matrix Flat Panel Imagers (AMFPIs) with a-Se thickness of 200 to 1000 μm have been a great success for mammography and radiography. The nominal electric field across a-Se is E_Se = 10 V/μm, with electron (e-) and hole (h+) blocking layers at the electrode interface to minimize dark current due to charge carrier injection. High-definition High-gain Avalanche Rushing Photodetector (HARP) video cameras with a-Se up to 35 μm thickness operated in avalanche mode (i.e., E_Se > 80 V/μm, gain from 20 to 2000) have been used in broadcast for over two decades. [Ref. 1]

We propose direct-conversion amorphous selenium (a-Se) radiation detector with separate absorption and avalanche regions, for both energy integration and photon counting. We recently demonstrated the first two-dimensional (2D) multi-well solid-state detector (MWSD), using micro-strip grid electrodes over insulating pillars, for substantially improving the time response of a-Se detectors. [Ref. 2] This design, however, is not optimized for avalanche operation because the highest electric field in the well develops at the interface between the semiconductor and the pixel electrode which results in high dark current due to large charge injection and potentially irreversible damage to the detector.

We propose a novel direct conversion a-Se detector structure which combines the large-area direct conversion layer with “low E_Se” and the avalanche gain property of a thin a-Se layer with “high E_Se”. The a-Se layer is divided into two separate regions: the bulk for x-ray interaction (up to 1 mm thick for x-ray absorption), and the well for avalanche multiplication. While the electric field E_Se in the bulk is ~ 10V/μm (identical to that in existing direct conversion AMFPI detectors), the E_Se in the center of the wells are > 80 V/μm to enable avalanche multiplication. During exposures, the x-rays absorbed in the bulk will generate electron-hole pairs (ehp), with the faster moving holes driven towards the readout structure. The grids embedded in the walls of each well will bend the electric field lines for all the holes to enter the wells, where they would undergo avalanche multiplication due to the strong E_Se. The thickness of the avalanche region (~15 μm) is at least one order-of-magnitude thinner than that of the bulk, ensuring uniform avalanche gain for x-ray interactions occurring at different depths of the bulk. This design is inspired by F. Sauli's gas electron multiplier (GEM). [Ref. 3]

We have successfully fabricated the first practical multi-well structure optimized for a direct conversion avalanche a-Se detector. The embedded grids in the well walls which create the field shaping will allow for tune-able gain within the wells and unipolar time differential sensing, overcoming a-Se’s inherent limitations of low carrier mobility and low charge conversion gain. By overcoming these inherent limitations, our structure permits a-Se based solid state detectors to be a potential low cost option for various medical imaging applications which require high spatial and temporal resolution, such as photon counting and time of flight imaging.

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Chemistry and Formation of Chlorine Derived MAPbI3 from in-situ X-Ray Measurements

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Methylammonium Lead Iodide ($CH_3NH_3PbI_3$) has very quickly become a promising low cost alternative photovoltaic absorber material with current record power conversion efficiencies exceeding 20%. At room temperature this structure adopts a tetragonal structure; however, at modest temperatures (~60°C) the structure transforms into a cubic structure. However, the tetragonal-to-cubic transition temperature varies within the literature from 37°C, reported from PL, to ~55 °C reported from XRD and DSC. Therefore, it is important to not only understand the structural behavior at this transition and its impact on device performance but also if this transition occurs in an actual operational device. Here we report structural characterization of an operational $CH_3NH_3PbI_3$ photovoltaic device.

XRF and XANES Studies on Individual Aerosol Particles

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Atmospheric aerosols are important constituents of our atmosphere that have an uncertain effect on climate and a deleterious effect on human health. In this study, Sub-micron Resolution X-ray Spectroscopy (SRX) at the National Synchrotron Light Source-II (NSLSII) was used to probe well-characterized aerosol sample sets. The first ambient set of samples, taken on the Island of Okinawa, was analyzed to gain additional information regarding the mineralogy of iron. It is hypothesized that the iron in these samples is characteristic of aerosols that increase bioavailable iron in the world's oceans. The source of these Fe-rich particles is thought to be industrial rather than from the large asian deserts. The aerosol samples from the asian outflow are compared to standard asian dusts. The second set of samples was taken from an industrial region of Mexico City where high Pb concentrations were observed. The SRX beamline is being used to probe the bonding environment of Pb in these samples to better characterize potential effects the particles have on human health.

Symbiotic Changes in Nutrient Distribution in the Poplar Rhizosphere Observed with FTIR-Imaging

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Plant based biomass is an efficient renewable energy in the United States, but its availability is not large. It has been shown that the addition of microbes to the plant system can help increase biomass production. The symbiotic relationship between a plant, its root hairs and microbes is crucial to understanding the transport of nutrients through changing environments. In this study, seedlings of the Quaking Aspen tree (Populus Tremuloides) are grown in a thin layer of Woody Plant Medium, which is a gel-like matrix controlling nutrient transport in the rhizosphere of the plant roots. This project aims to image the nutrient distribution in the rhizosphere both in the presence and absence of bacteria (Pseudomonas Fluorescens) in order to assess how different combinations (nutrient-enhanced vs. nutrient-starved) affect the production of biomass.

Using FTIR microspectroscopy for imaging of the root hairs and surrounding few hundred microns of the rhizosphere, the movement of nutrients in the plant root hairs can be monitored through the use of their unique IR signatures. Specifically, changes in sugars, phosphates and nitrates are being investigated. The ultimate goal of this study is to understand and optimize this process to increase bioenergy production from plants grown on marginal lands.

Probing the Dependence of Electron Transfer on Size and Coverage in Carbon Nanotube–Quantum Dot Heterostructures

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As a model system for understanding charge transfer in novel architectural designs for solar cells, double-walled carbon nanotube (DWNT)−CdSe quantum dot (QD) (QDs with average diameters of 2.3, 3.0, and 4.1 nm) heterostructures have been fabricated. The individual nanoscale building blocks were successfully attached and combined using a hole-trapping thiol linker molecule, i.e., 4-mercaptophenol (MTH), through a facile, noncovalent π−π stacking attachment strategy. Transmission electron microscopy confirmed the attachment of QDs onto the external surfaces of the DWNTs. We herein demonstrate a meaningful and unique combination of near-edge X-ray absorption fine structure (NEXAFS) and Raman spectroscopies bolstered by complementary electrical transport measurements in order to elucidate the synergistic interactions between CdSe QDs and DWNTs, which are facilitated by the bridging MTH molecules that can scavenge photoinduced holes and potentially mediate electron redistribution between the conduction bands in CdSe QDs and the C 2p-derived states of the DWNTs. Specifically, we correlated evidence of charge transfer as manifested by (i) changes in the NEXAFS intensities of π*resonance in the CK-edge and CdM3-edge spectra, (ii) a perceptible outer tube G-band downshift in frequency in Raman spectra, as well as (iii) alterations in the threshold characteristics present in transport data as a function of CdSe QD deposition onto the DWNT surface. In particular, the separate effects of (i) varying QD sizes and (ii) QD coverage densities on the electron transfer were independently studied.

First Principles Study of Core Level Binding Energy Shift and Noble Gas Adsorption in Two-dimensional Zeolite

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Due to its extraordinary properties of radiation hardness, thermal conductivity and large bandgap, diamond is an ideal material for X-ray detectors. Traditional diamond X-ray detectors are fabricated with metal contacts, such as platinum, and provide absolute flux calibration and position resolution better than 50nm. However, for many energy scanning applications the absorption lines from the metallic contacts effect the downstream experiment. Therefore, we have incorporated a conducting ultra-thin nano-crystalline diamond (UNCD) as the contact material, which shares all the benefits of diamond without the absorption lines associated with metal contacts. We will present results from several detectors fabricated with UNCD contacts where the contacts have been patterned with reactive ion etching into quadrant or single channel devices. The UNCD is grown at the Center for Nanoscale Materials at Argonne National Laboratory. The design, preparation and patterning is done at the Center for Functional Nanomaterials. The detectors were tested at BNL and CHESS and they display uniform response and full collection.

Coherent Hard X-ray (CHX) Scattering Beamline

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The Coherent Hard X-ray Scattering (CHX) beamline [1], currently undergoing commissioning at Brookhaven National Laboratories National Synchrotron Light Source II, is dedicated to experiments such as X-ray Photon Correlation Spectroscopy (XPCS) that exploit the extraordinary source brightness. The beamline's optical scheme [2] relies exclusively on refractive optics to tailor the beam sizes to match the experimental requirements. The beamline optics and their optimization for both high stability and wavefront preservation will be presented. Physical optics simulations of key performance parameters such as coherent flux and beam size will be compared against early commissioning results.

The beamline endstation instrumentation is designed to collect as much of the available scattering information as possible, allowing for covering simultaneously a Q-range corresponding to atomic sizes to hundreds of nanometers. The integrated endstation approach combines an 18 axes diffractometer, enabling horizontal and vertical scattering geometries, with a small angle scattering instrument with up to 16m sample-to-detector distance and in-vacuum detectors. The endstation instrumentation and possible scattering geometries will be described along with the available sample environments, featuring an in-vacuum optical on-axis microscope.

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Engineering and Assembly of Protein Complexes

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Proteins are the basic functional units in cells and carry out a wide variety of functions, from catalyzing metabolic reactions, synthesizing DNA and RNA, to responding to and transducing signals, and transporting molecules. Protein engineering, though a young discipline, provides a vital tool for advancing the understanding of protein stability, folding, and interactions, as well as solving problems in industrial and pharmaceutical biotechnology. By integrating protein engineering with approaches from biology, chemistry, biophysics, and statistics, our interest is to engineer and assemble protein complexes and proteins of related functions in order to understand the fundamental questions on the underlying mechanisms and to develop new protein systems for solving problems in human health, sustainable energy, and environment. This poster shows a few projects based on two protein assembly/engineering platforms we have been developing as well as their potential applications in drug delivery, biosynthesis, and bioremediation.

Enhanced Electro-Kinetics of CO_2 Generation for Ethanol Oxidation using Catalysts with a Partially Oxidized Pt and Rh Core and a SnO_2 Shell

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Direct ethanol fuel cell (DEFC) is a promising technology for generating electricity via the electro-oxidation of liquid ethanol. Its implementation requires the development of anode catalysts capable of producing CO2 and yielding 12-electron transfer through breaking C-C bond of ethanol. Here we presented comprehensive studies of electro-kinetics of the CO2 generation on Pt/Rh/Sn ternary catalysts. Our studies showed that the tri–phase PtRhOx-SnO2 catalysts with a partially oxidized Pt and Rh core and a SnO2 shell, validated by X-ray absorption analyses, coincided with a 2.5-fold increase in the CO2 generation rate towards ethanol oxidation reaction, compared with the bi-phase PtRh-SnO2 catalysts with a metallic PtRh alloy core and commercial Pt. These in situ studies provided insight on the design of a new genre of electro-catalysts with a partially oxidized noble metal.

Assessment of Factors Controlling the X-ray Penetration Depth in Studies of 4H-SiC using Monochromatic Synchrotron X-ray Topography in Grazing Incidence Geometry

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Synchrotron X-ray Topography has been shown to be a vital tool for the nondestructive characterization of defects in 4H-SiC crystals [1-2]. Techniques utilizing reflection geometry such as the grazing incidence geometry are particularly useful for discerning defects at different depths below the crystal surface particularly for off-cut 4H-SiC wafers. For example, in studying defects in SiC PiN diode structures, which typically comprise a buffer layer homoepitaxially grown on a substrate, with the drift layer grown on top of the buffer layer, it is important to be able to discriminate the depth at which particular defect configurations reside. This is particularly important for the characterization of defects resulting from relaxation processes such as interfacial dislocations and half loop arrays [3]. The operating penetration depth for grazing incidence X-ray topography is generally a function of the level of perfection in the crystal. In perfect regions it is determined by dynamical theory through the extinction length which predicts particularly shallow penetration depths for these geometries. In deformed regions (such as around dislocation cores) the penetration depth is determined by photoelectric absorption. However, the penetration depths calculated theoretically by just considering a dislocation as simple line defect are much smaller than those measured experimentally. In our study we have developed and compared two models involving different diffracting volumes to account for this discrepancy. In one model, the diffracting volume for kinematical diffraction is defined by the surface over which the effective misorientation associated with the dislocation is equal to the rocking curve width [4] as demonstrated in Fig. 1 for basal plane (BPDs) and deflected threading (TDs) dislocations . In the second model, the image contrast is formed by simulating the density map of the diffracted beam according to the local lattice plane tilt assuming the dislocation image formation is dominated by orientation contrast. This is the same as the ray tracing simulation that is used to predict image contrast of a dislocation of a particular Burgers vector [5]. Fig 2, Fig 3 and Fig 4 compare the recorded and simulated images from BPDs and TDs using this assumption. We will present a comparison between measured penetration depths in grazing incidence geometry with these two approaches and develop an optimized model to explain our observations.

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Effects of Thermal Annealing on Structural and Optical Properties of ZnO-Polymer Hybrid Thin Film Generated by Infiltration Synthesis

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Infiltration synthesis is a process derived from atomic layer deposition, where the sequential infiltration of vapor-phase material precursors into polymer matrix enables the generation of organic-inorganic hybrids with enhanced properties, such as mechanical strength, etch resistance, triboelectric performance, and photoluminescence. Most of the demonstrated hybrids are however based on the infiltration of aluminum oxide that has relatively limited intrinsic functionalities compared with other electro-active counterparts, such as zinc oxide (ZnO), a wide band semiconductor having favorable features including photo-catalytic activity, visible optical luminescence, and piezoelectricity. Infiltration synthesis of ZnO in polymers therefore has a potential for creating a new type of organic-inorganic hybrid with useful multi-functional properties. In this work, we generated hybrid thin films consisting of infiltration-synthesized ZnO in SU-8, a epoxy-based negative-tone polymer photoresist, and investigated the influence of precursor infiltration and post-synthesis thermal annealing conditions on the structural and optical properties of the hybrid thin films. Specifically, the sequential vapor infiltration of diethylzinc (DEZ) and water was applied into SU-8 thin films at 85 oC, with varying precursor infiltration protocol and the number of synthesis cycles. Cross-sectional scanning electron microscopy confirmed the infiltration of ZnO in SU-8 matrix. After annealing the hybrid film at 250 oC, we observed the highest photoluminescence (PL) intensity with its peak centered around 520 nm. Further increasing the annealing temperature up to 400 oC, however, decreased the PL intensity, suggesting the presence of interaction between the infiltrated-ZnO molecules and the polymer matrix. Fourier-transform infrared spectroscopy (FTIR) and ultraviolet-visible spectroscopy (UV-VIS) showed that the infiltrated ZnO mainly reacted with epoxy groups in SU-8 at the higher temperature, subsequently resulting in a red shift in the optical absorption spectrum of the hybrid film. The current study not only demonstrates the formation of visible-luminescent ZnO-infiltrated hybrid thin film but also improves the understanding of the interaction between organometallic precursor and polymer and its correlation with the infiltration synthesis protocol and thermal annealing conditions, providing the information critical for generating multi-functional inorganic-organic hybrid materials by infiltration synthesis process.

Detecting the Future: Diamond X-ray Detector

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Diamond is the ultimate material for x-ray detecting due to its large bandgap (low thermal noise, Schottky barrier), low absorption (transmission mode), radiation hardness (long lifetime) ,high carrier mobility (quick signal response) and the highest thermal conductivity (low heat build-up and expansion). Unlike traditional radiation detectors, diamond is not suffering from electronics saturation and heat dispersion problems as in silicon diodes, and it is not environmental sensitive as in ion chambers and does not require delicate electronics. We have built various transmission mode diamond detectors for beam flux, beam position and real-time imaging in NSLS, NSLS II, APS, CHESS, LANL and etc. Furthermore, thin diamond membrane (less than 10um) detector fabricated by deep RIE could be used for soft x-ray detection. Ultra-nanocrystalline diamond (UNCD) contacts are under developing for even less absorption. In this poster, a detailed study of diamond detector performance is included; in addition, the preliminary results of diamond response under Compton region (over 30keV) and diamond membrane fabrication are discussed.

Diamond X-ray Detector with Ultrananocrystalline Diamond Contacts

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Due to its extraordinary properties of radiation hardness, thermal conductivity and large bandgap, diamond is an ideal material for X-ray detectors. Traditional diamond X-ray detectors are fabricated with metal contacts, such as platinum, and provide absolute flux calibration and position resolution better than 50nm. However, for many energy scanning applications the absorption lines from the metallic contacts effect the downstream experiment. Therefore, we have incorporated a conducting ultra-thin nano-crystalline diamond (UNCD) as the contact material, which shares all the benefits of diamond without the absorption lines associated with metal contacts. We will present results from several detectors fabricated with UNCD contacts where the contacts have been patterned with reactive ion etching into quadrant or single channel devices. The UNCD is grown at the Center for Nanoscale Materials at Argonne National Laboratory. The design, preparation and patterning is done at the Center for Functional Nanomaterials. The detectors were tested at BNL and CHESS and they display uniform response and full collection.

Point-of-Care (POC) Micro Biochip for Cancer Diagnostics

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Cancer remains the second most common cause of death in the US, accounting for nearly 1 of every 4 deaths. The early detection of cancers can enhance preventive measures, increase curability of the disease, reduces health care costs and finally improves the quality of life for patients. To achieve the early detection the specific cancer type, the Point-of-care (POC) biochip is coated with a highly sensitive and specific set of antibodies and incorporated with an innovative sensing technology. To increase the sensing capability, the biochip is fabricated with microchannels. The microchannel in the biochip is designed to control the flow mechanism and amplify the capillary effect of the blood flow. The flow is self-driven using the natural phenomenon called surface tension of the blood flow. Cancer antibodies with enhanced specificity and affinity are specially developed and ligated in the microchannel. When the blood sample flows in the microchannel over the cancer antibody, the cancer antigen from the blood forms antigen-antibody complex. This antigen and antibody interaction is captured using state of the art sensing technology which is developed using highly sensitive nano-circuit design in the biochip. Also, the cancer severity is sensed depending on the intensity of the signal output. Therefore, the cancer and its severity can be diagnosed with the POC biochip, using both qualitative and quantitative results of the sensing technology.

New N-G/ZIF Catalyst Synthesized by High Energy Wet Ball Milling for Electrochemical Systems

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The nitrogen-doped graphene (N-G) catalyst, which is one of the promising non-platinum group metal catalysts, is investigated to replace platinum group metal catalysts in electrochemical systems or industries. This work is the second stage of a research targeted on investigating new N-G catalyst with high electrochemical performance. N-G catalysts, which were synthesized by high energy wet ball milling in the first stage, are modified by a metal organic framework material named ZIF-8 in this work. The final compound, N-G/ZIF, is expected to have advantages of both N-G and ZIF-8 such as high catalytic activity, high specific surface area, high porosity, and high thermal and chemical stability. The modification effects of ZIF-8 on the properties of N-G catalysts and the reaction mechanism are studied through the characterization analyses included XPS, BET, SEM, TEM, and Zetasizer.