Novel Trends in Synchrotron and FEL-Based Analysis Focus Topic

Room Hall B - Session SA-ThP

Novel Trends in Synchrotron and FEL-Based Analysis Focus Topic Poster Session

SA-ThP1 Relative Sensitivity Factors in Hard X-ray Photoelectron Spectroscopy up to 10 keV for Quantitative Analysis, ***Satoshi Yasuno***, Japan Synchrotron Radiation Research Institute, Japan; *N. Ikeno,* Aichi Synchrotron Radiation Center, Japan; *H. Oji,* Nagoya University Synchrotron Radiation Research Center, Japan

Hard X-ray photoelectron spectroscopy (HAXPES) has been attracting considerable attention since it can probe the chemical and electronic states of the bulk and buried interface lying at depths of several tens of nm due to its large probing depth.[1] In the last decade, HAXPES have been applied to various research fields, such as electronic devices, organic materials, and rechargeable batteries. However, the quantitative analysis of the HAXPES measurement related to the relative sensitivity factors(RSFs), standard materials and theoretical calculations in hard X-ray region, have not previously been reported in detail. Therefore, in this study, we investigated the procedures of quantitative analysis for HAXPES and development of the RSFs data base in the hard X-ray region. Here, we focused the database of the RSFs of compounds provided by Wagner.[2] According to Wagner's RSFs principle, the absolute value of the sensitivity factor will vary with the matrix because of the variability of the mean free path λ, by contrast the relative sensitivity factor will hardly vary because the ratio λ_1/λ_2 for element 1 and 2, is only slightly with matrix dependence. Therefore, with the RSFs of compounds, the corrections related to the mean free path which largely influenced the number of photoelectron (signal intensity) are not needed for the quantitative analysis. For HAXPES, the core level peaks can be measured in a wide kinetic energy. Thus, it is suggested that the RSFs obtained by the compound is suitable for HAXPES measurement. In this study, the empirical sensitivity factors for the 1s, 2s, $2p_{3/2}$, $3d_{5/2}$ and 4f levels relative to O 1s were derived from HAXPES measurements with the photon energy of 6, 8, 10 keV. Comparing with the theoretical RSFs calculated from Hartee-Slater cross sections reported by Scofield with combined energy dependence on the spectrometer function and the inelastic mean free path, the good agreement between the empirical and the theoretical RSF values were observed with several notable exceptions, while the discrepancies were observed in several energy regions.

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References

[1] K. Kobayashi, Nucl. Instrum. Methods Phys. Res. A601, 32-47 (2009).

[2] C.D.Wagner et at., Surf. Interface Anal. 3, 211 (1981)

SA-ThP2 In Situ Characterization of Freeze-Cast Metal Nanowire Aerogels, ***Tyler Fears***, *J.A. Hammons, F. Qian, T. Braun, A.L. Troksa, M.H. Nielsen, J.B. Forien, T.F. Baumann, T.Y. Han, S.O. Kucheyev, M. Bagge-Hansen,* Lawrence Livermore National Laboratory

Metal nanowire aerogels are a new class of nanoporous materials desirable for a number of applications in energy storage, generation, and utilization. These materials are made by freezing suspensions of high-aspect-ratio (a ≈ 1000) metal nanowires and gently removing the solidified matrix, e.g., via freeze-drying, to prevent collapse of the porous nanowire network. As such, the porosity in the final aerogel is intrinsically linked to solvent phase separation and crystallization during freezing which is highly sensitive to the conditions under which it takes place, e.g., temperature, solvent composition, and sample geometry.

Herein will be discussed recent developments at Lawrence Livermore National Laboratory to produce high-quality ultra-low-density (1-30 mg/cm^3) metal aerogels via a facile freeze-casting approach. Due to the hierarchical structure of the aerogels (1-100 µm micropores in a nanoporous matrix of 3-30 nm diameter nanowires) it was necessary to use a wide variety of complementary in situ/ex situ analysis techniques to ascertain the structure and origin of these hierarchical features. This presentation will discuss the unique properties of these aerogels and the advanced analysis techniques used in their characterization, e.g., USAXS/SAXS/WAXS, X-ray tomography, XPS, optical microscopy, and electron microscopy. This work was performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344.

SA-ThP3 In situ Probing of the Potential Distribution in a Thin Film All-solid-state Li-ion Battery, ***Evgheni Strelcov***, National Institute of Standards and Technology (NIST)/University of Maryland; *E.J. Fuller,* Sandia National Laboratories; *W. McGehee, N.B. Zhitenev, J. McClelland,* National Institute of Standards and Technology (NIST); *A. Talin,* Sandia National Laboratories

The next generation of portable electronic devices, electric vehicles, power grids, and robots require safer, smaller, lighter, cheaper, and more stable batteries. Of special importance are all-solid-state power sources that do not use conventional, flammable electrolytes and are intrinsically safer. Rational design of such batteries is challenging without in-depth understanding of the chemical and physical processes in electrochemical cells at the microscopic, nanoscopic, and eventually, atomic levels. Particularly important structural elements of solid-state Li-ion batteries (SSLIBs) that control the overall device performance are the interfaces that form between the electrodes and the cathode/anode materials and solid electrolyte. Despite decades of studies with classical electrochemical techniques, spectroscopic and microscopic tools, the interfacial characteristics of batteries, including the origins of high impedance often observed at solid state interfaces, are still poorly understood. Here, we employ in situ Kelvin Probe Force Microscopy (KPFM) to probe the potential distribution in a SSLIB as a function of its charge state. The battery was fabricated by sequentially depositing thin layers of Pt (110-130 nm), $LiCoO_2$ (280-420 nm), LIPON (1100-1200 nm), Si (50-240 nm) Cu or Pt (150-200 nm) onto a Si/SiO_2 wafer (oxide thickness 100 nm). The fabricated battery was cleaved in an Ar atmosphere to expose the stacked layers, mounted on a holder, wired, and safely transferred without exposing to air into a dual-beam instrument that combines a scanning electron microscope (SEM), a Ga-ion focused ion beam (FIB) and an atomic force microscope (AFM) in one vacuum chamber (residual pressure of 10^{-4} Pa). The stacked battery was milled to expose a cross-section of the layers, and imaged using SEM and KPFM, while cycling the battery. The acquired potential maps reveal a highly non-uniform interelectrode potential distribution, with most of the potential drop occurring at the electrolyte-Si anode interface in the pristine battery. During the first charge, the potential distribution gradually changes, revealing complex polarization within the LIPON layer due to Li-ion redistribution. The acquired data shed light onto the interfacial Li-ion transport in SSLIBs and its reversibility.

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SA-ThP4 A New Route for the Determination of Protein Structure in Physiological Environment through Coherent Diffraction Imaging., ***Danny Fainozzi,*** university of Trieste / Elettra Synchrotron, Italy

Revealing the structure of complex biological macromolecules, such as proteins, is an essential step for understanding the chemical mechanisms that determine the diversity of their functions. Synchrotron based x-ray crystallography and cryo-electron microscopy have made major contributions in determining thousands of protein structures even from micro-sized crystals. They suffer from some limitations that have not been overcome, such as radiation damage, the natural inability to crystallize of a number of proteins and experimental conditions for structure determination that are incompatible with the physiological environment. Today the ultrashort and ultra-bright pulses of X-ray free-electron lasers (XFELs) have made attainable the dream to determine protein structure before radiation damage starts to destroy the samples. However, the signal-to-noise ratio remains a great challenge to obtain usable diffraction patterns from a single protein molecule. We describe here a new methodology that should overcome the signal and protein crystallization limits. Using a multidisciplinary approach, we propose to create a two dimensional protein array with defined orientation attached on a self-assembled-monolayer . We develop a literature-based, flexible toolbox capable of assembling different proteins on a functionalized surface while keeping them under physiological conditions during the experiment, using a water-confining graphene cover.

SA-ThP5 The League of European Accelerator-Based Photon Sources: New strategic partnerships in Europe and beyond*, **Maya Kiskinova**,* Elettra-Sincrotrone Trieste, Italy

The grand challenges of our century to evolve from extensive wasteful development to sustainable economies is full understanding of the mechanisms which control the behavior of complex natural and man-made systems. This can be attained only through development of an integrated multidisciplinary approach. Many of the breakthroughs in investigations of a broad range of complex functional material systems have been made using the state-of-the art experimental techniques undergoing continuous developments at the synchrotron and free electron laser large scale facilities.

Recognizing the leading roles of these large scale research centers in paving the road to discoveries and further technological advancements, the recently established new research consortium in Europe called "*The League of European Accelerator-Based Photon Sources (LEAPS)"* is aiming at reaching a new level of cooperation, coordination and integration to better cope with cross-cutting scientific and technological challenges for knowledge-based design of advanced materials and better drugs. The poster will present an overview of the LEAPS strategy, goals and expected impacts in science and innovation.

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