Chemical Analysis and Imaging Interfaces Focus Topic
Room Union Station B - Session CA-ThP

Chemical Analysis and Imaging at Interfaces Poster Session

CA-ThP1 Probing Solid-liquid Interfaces with Tender X-rays, *Nicolò Comini, Z. Novotny, B. Tobler,* University of Zuerich, Switzerland*; D. Aegerter, E. Fabbri,* Paul Sherrer Institute, Switzerland*; U. Maier,* Ferrovac GmbH, Switzerland*; L. Artiglia, J. Raabe, T. Huthwelker,* Paul Sherrer Institute, Switzerland*; J. Osterwalder,* University of Zuerich, Switzerland

Many important chemical and biological processes occur at the interface between a solid and a liquid. Despite its importance, it is very difficult to collect meaningful signals from this buried interface. We recently built a new instrument at the Swiss Light Source that combines ambient-pressure X-ray photoelectron spectroscopy with in-situ electrochemistry. With this new setup, we can stabilize a thin liquid film by a dip&pull method [1] and using tender X-rays, we can probe the solid-liquid interface while having potential control over the electrolyte film. We will present results from the first commissioning beamtime and outline the future direction we are going to pursue.

[1] S. Axnanda, E. J. Crumlin et al., Sci. Rep. 5, 09788 (2014).

CA-ThP2 Using AES, EDS, and FIB to Detect, Identify, and Image Buried Metallic Particles, *Ashley Ellsworth, D. Paul, J.G. Newman,* Physical Electronics

Auger Electron Spectroscopy (AES) is a powerful analytical tool that provides quantitative elemental information from surfaces of solid materials. The average depth of analysis for an AES measurement is approximately 5 nm with lateral spatial resolution as small as 8 nm. The information AES provides about surface layers or thin film structures is important for many industrial and research applications where surface or thin film composition plays a critical role in performance including: nanomaterials, photovoltaics, catalysis, corrosion, adhesion, semiconductor devices and packaging, magnetic media, display technology, and thin film coatings used for numerous applications.

In this work, we demonstrate the use of AES in conjunction with a focused ion beam (FIB) to produce site specific imaging of microscale features beneath a sample surface. The combination of the two techniques allows for high spatial resolution analysis of buried particles and defects. This information can be very useful in helping to determine important parameters such as the origin of defects, corrosion mechanisms, coating problems, etc. We will highlight the advantages of FIB milling compared to traditional depth profiling. Energy dispersive x-ray spectroscopy (EDS) is also a powerful complementary technique to Auger analysis as it provides information from much deeper in the sample surface (few μm). With the combination of AES, FIB, and EDS, we show that buried metal particles can be first located and characterized with EDS, followed by the subsequent FIB milling and high spatial resolution Auger spectroscopy and imaging of the particles.

CA-ThP3 Secondary Ion Mass Spectrometry Designed for Ultra-sensitive Molecular Analysis of Solids and Liquids, *Stanislav Verkhoturov, D.S. Verkhoturov, E.A. Schweikert,* Texas A&M University

SIMS is a method of choice for elemental and molecular surface thin layer analysis. We present here significant enhancements in SIMS capability with regards to a) detection sensitivity, and b) applicability to liquid samples.

Detection of atto to zeptomole amounts of analyte is demonstrated with deposits of fractional monolayers of organics on double layer graphene. Three innovations make ultrasensitive analysis possible. The graphene support minimizes interference between analyte and substrate signals. Secondly, the analysis is run in a unique experimental setup. The sample on graphene is bombarded with 50 keV C_{60}^+ in transmission mode, i.e. the ejecta are mostly in the forward direction where they are collected in a time-of-flight mass spectrometer, ToF MS. Thirdly, the bombardment is reduced to a series of single C_{60} impacts, each coupled with ToF measurement. This event-by-event bombardment-detection mode allows the selection of specific impacts on analyte at the exclusion of signals from supporting materials. In this approach, unusually high ionization of analyte molecules (e.g. ~10%) was observed. A distinct ejection-ionization mechanism operates here. The molecules "trampoline" off the graphene following a C_{60} impact. The mass spectra contain abundant peaks of molecular ions. The proposed mechanism of ionization involves tunneling of electrons from the vibrationally exited area around the hole to the molecules⁻. Another proposed mechanism is a direct proton transfer exchange.

Liquid samples are inherently incompatible with SIMS, which operates under vacuum. We demonstrate the feasibility of storing liquids in carbon nanotube, CNT, sponges. We found that within a practical experimental time of 30 min, liquids with vapor pressure of < 1 torr can be analyzed. The CNT sponge (97% porosity) was made from multiwall carbon nanotubes (CNT cross-section ~6 layers). Hydrophilic and hydrophobic sponges were examined. For the hydrophobic case, we examined the lubricant fluids (vapor pressure ~100 millitorr). The mass spectra of pure fluids were used as a blank for comparison with mass spectra of wear tracks on metals. For the hydrophilic case, pure glycerol and the glycerol-water mixture have been examined. Glycerol evaporates slowly from the sponge. The method allows to investigate the organic molecules dissolved in glycerol and glycerol water mixture. Thus, the event-by-event technique applied on CNT sponges is a promising method for analysis of liquid and soft materials by Cluster SIMS.

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