

Thursday Afternoon, September 22, 2022

Beyond SIMS

Room Great Lakes C - Session BS+SS-ThA2

Polymers & Multi-Technique

Moderators: Andrew Giordani, Procter & Gamble Company, Michaeleen Pacholski, Dow Chemical Company

2:00pm **BS+SS-ThA2-1 Multidimensional Chemical Imaging of Polymeric Materials Using TOF-SIMS with GCIB Sputtering**, Paul Vlasak, M. Clark, R. Drumright, J. Harris, M. Pacholski, H. Ying, Dow **INVITED**

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) aided by gas cluster ion beam (GCIB) sputtering has become an important tool for studying morphology of multiphase systems. While electron microscopy (EM) remains a workhorse approach, SIMS allows specific detection of low concentration components such as additives, catalysts, colorants, or crosslinkers that are not easily detected by other methods, and 3D SIMS imaging can reveal how these trace components are distributed relative to the overall morphology of multiphase systems. While SIMS cannot yet match the ultimate spatial resolution of EM methods, these results have been critical to orient the electron micrographs by identifying features directly based on mass spectrometry that would otherwise be differentiated only by single channel contrast mechanisms, often relying on heavy metal staining strategies. Because the 3D SIMS depth profiles can be acquired reasonably quickly over relatively large areas, SIMS can identify features existing on larger length scales and verify uniformity in a single analysis whereas similar information obtained by EM would require preparing and imaging many cross-sections.

This presentation will highlight an industrially important multiphase polymer system. In this example, complex coating phase morphology existing on the single micron scale shifts dramatically with changes in proportions of the raw materials or with the addition of various compatibilizers, sometimes at low concentration. These coatings derived from polyolefin dispersions (POD's) have been developed as an attractive alternative to coatings from bisphenol A based epoxies for next generation aluminum beverage can interior linings. Various compatibilizers and dispersants with polar functionalities are compounded with the nonfunctional polyolefin resins to achieve stable waterborne emulsion formulations. Key performance requirements demonstrated by POD derived coatings include superior adhesion to the aluminum surface, effective barrier properties, and preservation of the canned products' flavor. These properties, including flavor scalping, or the absorption of key flavorants from the beverage into the coating, are influenced by coating morphology.

2:40pm **BS+SS-ThA2-5 Mixed Actinide Glasses as Working Reference Materials for Spatial Analyses**, David Willingham, J. Matzel, P. Weber, Lawrence Livermore National Laboratory; E. Groopman, National Institute for Science and Technology (NIST); D. Weisz, J. Wimpenny, J. Caseres, K. Knight, Lawrence Livermore National Laboratory

Secondary ion mass spectrometry (SIMS) has long been applied to the analysis of isotopic heterogeneities in nuclear materials. Few other methodologies challenge the ability of SIMS to measure the isotopic composition of nuclear materials with high accuracy and precision with micrometer/nanometer spatial resolution. While a number of certified/standard reference materials exist for bulk actinide concentration and isotopic analytical techniques, there are few, if any, working reference materials available for spatially resolved analyses, such as SIMS. These working reference materials must be well-characterized for actinide concentration and isotopic composition, homogeneous at the lateral resolution appropriate for the application, and representative of the real-world elemental concentrations and isotopic compositions of the materials of interest.

For this study, two working reference materials were developed in a glassy matrix containing both uranium and plutonium. The first, UPI, was composed of 496 ppm of uranium with a ^{235}U enrichment of 92.3% and 50 ppm of plutonium with a $^{240}\text{Pu}/^{239}\text{Pu}$ ratio of 0.0054 ± 0.00001 . The second, UPO, was about 8x less concentrated than UPI and was composed of 60 ppm of uranium with a ^{235}U enrichment of 79.6% and 8 ppm of plutonium with a $^{240}\text{Pu}/^{239}\text{Pu}$ ratio of 0.05541 ± 0.00001 . In addition to SIMS analyses, these glasses were analyzed by traditional bulk methods to determine their elemental concentrations and isotopic compositions. These methods include chemical dissolution of the bulk glasses following the principles of Isotope Dilution Mass Spectrometry (IDMS) and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).

In addition to traditional SIMS, these mixed actinide glasses were analyzed by the Naval Ultra-Trace Isotope Laboratory's Universal Spectrometer (NAUTILUS) developed at the U.S. Naval Research Laboratory, which combined the best attributes of SIMS and Single-Stage Accelerator Mass Spectrometry (SSAMS). The NAUTILUS is comprised of a SIMS instrument that provides micrometer resolution ion imaging and high precision isotope ratio measurements couple to a SSAMS that enables the dissociation of molecular isobaric interferences common to mass spectrometry.

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 and was supported by the LLNL-LDRD Program under Project No. 20-SI-006.

3:00pm **BS+SS-ThA2-7 An Overview of Automotive Coatings and the Analytical Tools that Drive Innovation**, Sabrina Peczonczyk, N. Hosking, C. Peters, T. Misovski, C. Seubert, M. Nichols, Ford Motor Company

The development and implementation of high-quality, robust automotive coatings requires a critically fundamental understanding of coating properties, process conditions, and durability in automotive environments. To achieve this Ford Motor Company employs a suite of surface analytical techniques and expertise. This talk will focus on the use of Auger electron spectroscopy (AES), time-of-flight secondary ion mass spectrometry (ToF-SIMS), and x-ray photoelectron spectroscopy (XPS) for the evaluation of automotive coatings. Case studies highlighting applications in Research and Development and Corporate Support will be discussed.

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