

## SIMS Solutions in Materials and Life Sciences Room Great Lakes B - Session SS-MoM2

### Industrial Applications I

**Moderators:** Derk Rading, IONTOF GmbH, Alan Spool, Western Digital Corporation

#### 10:00am SS-MoM2-10 The Characteristics of Multi-material Depth Profiles with Low-Energy Atomic and Diatomic Ion Beams and Cluster Ion Beams of Ar and O<sub>2</sub>, *Albert Fahey, M. Zhang*, Corning Inc. **INVITED**

The newest IONTOF instruments feature several sputter sources that can be used for depth profiling. One of the sources, an O<sub>2</sub>-Gas Cluster source, is not commonly used. However, we have found it invaluable when sputtering into insulating materials, like glass, where it is important to preserve the fidelity of the profiles of alkali and other mobile species. Data will be shown illustrating the value of different ion beams for measurements of a variety of species, even at trace levels.

Depth profiles of sputtered coatings are performed commonly, but can be problematic for a variety of reasons, not only due to sputtering artefacts. Use of low energy ion beams and cluster ion beams allows improved resolution and sensitivity. In addition, ToF-SIMS depth profiles can show previously unnoticed contaminants both in the bulk of each layer as well as between layers. Contaminants can be significant if they affect interlayer adhesion and other properties. Some examples will be shown and discussed.

#### 10:40am SS-MoM2-14 Analysis of Alkali and Trace Species in Silicate Glasses, *Timothy Dimond, A. Fahey, C. Mahoney, C. Cushman*, Corning Inc.

Data artifacts associated with insulating materials can present significant challenges for analysis by SIMS. In particular, alkali species can be elusive especially when the use of some common techniques, like O<sub>2</sub><sup>+</sup> sputtering, for promoting ionization have been employed. A societal move towards an interactive glass display world requires chemical alteration by surface treatments and coatings on commonly insulating materials and the need to be able to characterize those surfaces. Several commercially available tools for mitigating charging issues such as the use of an electron gun, gas flooding, and some novel sputtering beams conclude to some best practices for producing viable data when working with insulating materials, in particular silicate glasses and oxide thin films. Some practical explanations of these tools being used to generate HR imaging, quantitative depth profiles, and other practical data will be discussed to promote their efficacy in a growing interactive glass display industry. Our results show the use of oxygen Gas Cluster Ion Beam (GCIB) and Cs sputtering to enable ideal conditions for most positive mode analyses in silicates and other oxides.

#### 11:00am SS-MoM2-16 TOF-SIMS Surface Hydroxyl Measurements on Multicomponent Glasses, *Cody Cushman, N. Smith, J. Banerjee, C. Mahoney, A. Fahey, T. Dimond*, Corning Incorporated; *M. Linford*, Brigham Young University

Surface hydroxyls (primarily Si-OH) are thought to govern surface mediated properties and processes on multicomponent glass surfaces including particulate adhesion, surface contamination, and water adsorption. While ToF-SIMS protocols for measuring surface hydroxyls have been previously reported, they have seldom been applied to multicomponent glass surfaces. In this presentation, we will discuss ToF-SIMS surface hydroxyl measurements as applied to calcium aluminosilicate glass, including measurement reproducibility, fundamental measurement limitations, and the influence of hydrocarbon surface contamination on these measurements. Further development of ToF-SIMS surface hydroxyl measurements will ultimately provide a powerful tool for understanding the fundamental surface science of glasses and oxides.

#### 11:20am SS-MoM2-18 Dynamic SIMS Imaging of Impurities in Cold Spray Copper Coating, *Jonas Hedberg*, Surface Science Western, Western University, London, Ontario, Canada; *F. Filice, X. Li*, Department of Chemistry, Western University, London, Ontario, Canada; *S. Ramamurthy*, Surface Science Western, Western University, London, Ontario, Canada; *J. Noël*, Department of Chemistry, Western University, London, Ontario, Canada; *M. Behazin, P. Keech*, Nuclear Waste Management Organization, Toronto, Ontario, Canada

The Nuclear Waste Management Organization (NWMO) in Canada is developing and implementing a strategy for safe disposal of used nuclear fuel. The proposed multi-barrier system includes metallic used fuel

containers surrounded by highly compacted bentonite buffer boxes. These containers will be emplaced in a deep geologic repository located at a depth of about 500 m in a suitable host rock. The current NWMO UFC design specifies a 3 mm copper (Cu) corrosion barrier, applied by electrodeposition and cold spray (CS) technologies onto a low alloy steel inner vessel. The cold spray coating will be applied on-site after the used fuel bundles are placed inside the container and the lid is welded shut.

Cold spray coating is produced by impingement of Cu power at a high velocity on the container surface. Adhesion is through the plastic deformation of Cu particles. The Cu powders used for CS coating can contain both metallic (Fe, Bi, Pb, Sn, Zn, and Ag) and non-metallic (O, S, C, N, and P) impurities. Even when present in trace quantities, some of the impurities may precipitate within the grain boundaries and affect the corrosion behavior of Cu coating. Hence, a major objective of this study is to determine the corrosion behavior of CS Cu coating containing known amounts of various impurities and determine acceptable tolerances for the CS Cu coatings.

Cold spray Cu coatings containing known amounts of O, S, and Fe, were applied on steel plates. Dynamic secondary ion mass spectrometry (DSIMS) was used to analyze the coated samples because the impurities were present at very low levels (~100 to 700 ppm). The DSIMS results showed that all CS Cu samples exhibited oxygen enrichment at the Cu particle boundaries, regardless of oxygen content of the Cu power used for CS Cu coating. The DSIMS images were complemented with accelerated corrosion measurements, which indicated that increased oxygen content in Cu increased the tendency for corrosion under aggressive conditions.

DSIMS images for sulfur were mainly focused on the results from 34S due to the possible overlaps with molecular ions of oxygen (16O<sub>2</sub>) for the 32S mass. Sulfur images showed stronger signals from the samples with added sulfur as well as the standard CS Cu without any impurities. However, further work is needed to determine the distribution of S within CS Cu coatings and its effect on Cu corrosion behavior.

In summary, DSIMS imaging is a valuable tool in assessing the presence and the distribution of trace amounts of impurities in CS Cu coating. SIMS images were also useful in understanding the observed corrosion behavior of CS Cu.

#### 11:40am SS-MoM2-20 Surface Characterization of High Entropy Alloys with Sea Water and Sulfuric Acid Corrosion Test Using Hard X-Ray Photoelectron Spectroscopy and Time-of-Flight Secondary Ion Mass Spectroscopy, *Hsun-Yun Chang*, ULVAC-PHI, Inc., Taiwan; *W. Lin*, Department of Photonics, National Sun Yat-sen University, Taiwan; *G. Fisher*, Physical Electronics; *S. Iida*, ULVAC-PHI, Inc., Japan

High entropy alloys (HEAs) are known to be composed of five or more principle elements with concentration commonly in equal or near-equal atomic percent. The application of HEAs has substantially gained attention in recent years due to greater fracture resistance, tensile strength and corrosion resistance than conventional alloys. Researchers have developed HEAs with various components in order to strengthen the desirable mechanical or other properties for industrial applications, such as nuclear and aerospace fields. Corrosion resistance is one of important properties to design novel HEA materials, because the cost of corrosion is known to decrease the gross domestic product (GDP) and has no good to economic benefit of industries. With the entropy increase of a larger number of elements in the mix, HEAs shows a stable solid solution phase with no intermetallic phases. The random arrangement of multiple elements results in a particular locally-disordered chemical environment, which leads to unique corrosion-resistance properties. To investigate the corrosion behavior of designed HEA materials, surface characterization on the corrosion area of HEAs is necessary. In this work, hard X-ray photoelectron spectroscopy (HAXPES) and time-of-flight secondary ion mass spectroscopy (ToF-SIMS) are utilized to examine the corrosion behaviors of a commercial AlFeCoCrNi (AFCCN) HEA under sea water or sulfuric acid treatment. Using HAXPES analysis, the greater energy range (Cr K $\alpha$  5414.8 eV) allows us to study the chemical state of alloys without Auger peaks interference in conventional XPS. Also its deeper detection depth (~30 nm) enables us to examine the thicker surface oxidized/passivated layer of corrosion area without the concern of sputter damage. Using ToF-SIMS analysis, the chemical imaging and the surface morphology of HEAs corroded area can

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be observed. With the information of chemical state quantification and chemical imaging, the combination of HAXPES and ToF-SIMS analyses facilitates better understanding on the variation of HEAs surface before and after corrosion.

12:00pm **SS-MoM2-22 Understanding the Retention and Distribution of Anti-Microbial Compounds on Solid Surfaces**, *Michael Clark, Jr.*, Dow, Core R&D Analytical Science; *D. Miller, A. Jayaraman*, Dow, Core R&D Formulation, Automation & Material Science; *A. Karikari, C. Schultz*, Dow Home and Personal Care; *B. Cressman*, Dow, Core R&D Analytical Science  
Disinfection of surfaces has been of great interest over the past two years due to the onset of the coronavirus disease (COVID-19) pandemic. Most commercial surface disinfectant products contain a cationic surfactant as their active ingredient along with other formulation components. This presentation will focus on the application of X-ray photoelectron spectroscopy and secondary ion mass spectrometry technologies to understand how the application method and formulation modifications influence the retention and distribution of the active ingredients across glass surfaces. Such information will aid in the design of commercial disinfectants.

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