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Expanded Materials Sample Platforms for Advanced Surface Analysis of Energy Materials

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As analysis techniques mature, with wider acceptance of the relevance that the information gleaned from their results provides, and broadened use across a wider swath of disciplines and applications, this drives the development of more specialized sample handling and enhanced analysis techniques to address specific applications needs. With new materials systems being combined for a multitude of applications, the recognition of the importance that surface chemical analysis of the materials and their interfaces provides to the understanding of the function and long-term performance of the materials systems has greatly expanded across the research community over the last decade. This has driven focused development of both technique capabilities and special sample handling for several active research emphases.

The increased demand for energy storage systems to support a number of industries (EV, solar, electronics and even petrochemical) generates active interest in methods to characterize the materials. Recent advances in the ability to investigate the interfaces of layered of materials through improvements in analytical capabilities has engendered the ability to expand sputter etching for soft and sensitive materials not previously possible, utilizing clusters of inert gas atoms. These new sources have proven to be particularly advantageous for the analysis of materials with mobile light alkali metals, such as lithium-based battery materials and technological glasses, and results reveal that traditional monoatomic etching alters these systems and introduces artifacts in the data [1] (Figure 1). When coupled with improvements in the ability to use higher energy x-ray sources and the greatly enriched automation capabilities and specialized sample handling of modern instrumentation, these advances allow the investigation of interface development during accelerated testing.

Specialized electrochemical sample platens have been developed to allow samples to be mounted and transferred via an inert environment and loaded into the instrument load lock and evacuated for measurement without ever exposing to atmosphere, which is critical in many materials systems, and particularly when investigating lithium battery materials. Coupled with the inert loading is the ability to make electrical connection to the sample for bias and/or current application, to simulate operational environments for live or accelerated testing. The same inert, multi-contact, electro-chemical sample platens also have heating capability to provide additional accelerated testing capabilities. Utilizing these mounting capabilities, test specimens can be analyzed during operation so that elemental migration and chemical changes can be observed [2]. Specimen heating capability further allows accelerated aging effects on the elemental distributions and chemistry of the interface to be studied, which will be discussed.

In addition to the improvements in information quality that the cluster ion sources add to the energy materials fields, these same sources now allow depth profiling of soft materials, such as polymers, adhesives, and biological systems that were not previously possible, now allowing mixed organic and inorganic materials systems to be depth profiled. Unlike the need to heat for accelerated aging in the

energy materials, biological materials often need cooling capability to minimize evaporation and sublimation of the sample materials of interest. This capability has developed such that once samples are pre-cooled in an inert environment and then evacuated, these can be transferred for analysis using the same sample automation capabilities, improving the ease of performing these types of analyses. Now buried interfaces within soft materials can be chemically investigated while still preserving the structure of the materials as they are profiled, and examples of this expanding applications environment will be given.

Finally, specialized sample preparation and reaction capabilities have been developed to address the ever-growing application space for heterogeneous catalysis, critical to energy and materials industries across the spectrum. Modern materials and technological developments now allow broader ranges for testing reactions of surfaces with specialized catalysts reaction cells. These allow the heating of specimens while exposed to reaction gases under pressure, up to 30 bar for the Fisher-Tropsch process, with the ability to directly transfer to the analysis chamber after the exposure for XPS and other analyses, providing insight into improvements in catalytic efficiency for improved design, which will also be presented.

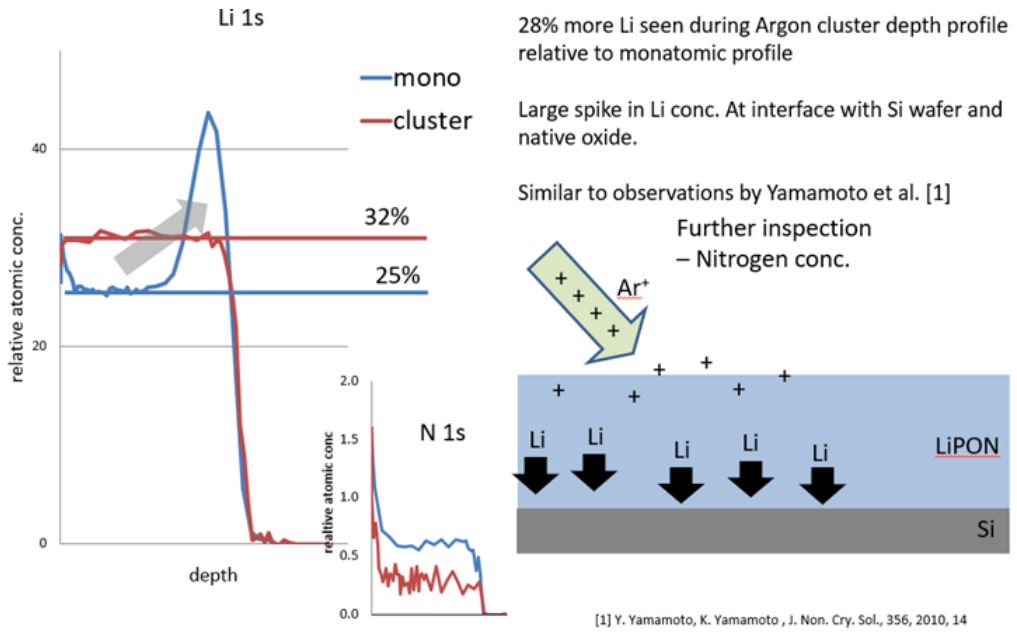

Figure 1. Comparison of elemental lithium concentration as function of depth in a Li_xPO_yNz film when profiled using traditional monoatomic argon ion sputtering versus when the film is profiled utilizing an argon gas cluster ion source.

References:

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[2] C-M Wang et al, J. Adv. Surf. Anal. **24** (2017), p. 141.

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