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Advances in surface chemical analysis of thin film solid-state battery materials and development of operando measurement capability

Chris Moffitt[1], Jonathan Counsell[2] and Simon Hutton[2]

[1]Kratos Analytical, Corpus Christi, Texas, United States, [2]Kratos Analytical, United States

Demand for energy storage over a range of applications and operating environments is driving broad research for improvements in materials and efficient design in battery technologies. As with so many physical systems, the interfaces between materials have large bearing on the operation, performance, and lifetime of the individual battery.

Surface chemical analysis via electron spectroscopy allow the functionality of the exposed surface to be investigated. Combining X-ray photoelectron spectroscopy (XPS) with inert ion sputtering has traditionally been used to study material composition and chemistry into the bulk of the materials. Ions of light elements such as Li, Na and K have been observed to easily migrate in various solid systems, which is one of the reasons why Li has great utility as a charge carrier in solid state batteries. These same ions are observed to migrate under the influence of the surface charge buildup during conventional inert gas ion sputtering, skewing the concentration in the bulk and causing a buildup of Li at the underlying interface. The development of inert gas cluster ion sources, which allows profiling of organic materials not previously possible, has had profound impact on the quality of information obtained from profiling of LiPON thin film battery structures1. We will show that not only does the cluster source relieve the issue of the light ion migration, it also reveals that a chemical state previously observed in traditional mono-atomic depth profiling is actually an artifact of the process and is not observed when using an optimized cluster ion source for the profiling.

To utilize these improvements in chemical characterization over a wider range of conditions, the mounting of samples such that bias voltages can be applied while heating the materials at controlled temperatures during analysis becomes essential for understanding surface and interface changes that take place during device operation. Many of these battery materials systems encountered are very sensitive to oxygen exposure, so operando sample holders that can be mounted in a glove box environment have been developed to provide surface analysis of these solid battery systems while applying potential and heating, in replication of more actual application environments. In addition, an enclosure system which allows for loading in a glove box physically remote from the instrument, and transport to it for analysis, was developed for these bias-capable, temperature-controlled sample holders. Once in the vacuum environment of the load lock, the cover can be removed, and the unexposed sample surface can be automatically loaded and investigated in the Axis Supra+, utilizing all the benefits of the ArN+ gas cluster ion source for cleaning and sputter-profiling the materials.

References

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