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Three-Dimensional Imaging and Interface Analysis of Battery Materials Via Plasma FIB-SEM

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Abstract

In the past decades, the rapid growth of electric vehicles and consumer electronics market leads battery technology to attract significant attention. In order to further advance battery technology, in-depth understanding on the electrode and interface structure of battery materials are essential. Focused ion beam – scanning electron microscope is an analytical method combines ion beam for materials processing and electron column for imaging. It enables both 2D interface analysis and 3D imaging capability as well as multimodal information collection, which is an effective analytical approach for battery structure analysis.

To date, conventional Ga⁺ FIB-SEM has been widely employed for battery materials development including interface/interphase characterization, 3D quantification and simulation analysis. However, Ga⁺ FIB materials removal efficiency limits its capability to access representative area and volume in some battery materials systems, e.g. electrode with tens of micron particle size. In addition, the reactive property of Ga⁺ ion with alkali metals, e.g. Li metal, results in the complex experimental procedure to limit the Ga⁺ contamination during FIB-SEM characterization.

Recently, the emerging Plasma FIB-SEM (PFIB-SEM) technology has been developed with different ion source and high removal efficiency. It promises great potential for battery materials characterization, due to accessing representative 2D area and 3D volume via 40 times faster (than Ga⁺ system) milling rate as well as enabling Ga⁺ free sample preparation on advanced battery system with alkali metal electrode through non-reactive ion source (Xe⁺ and Ar⁺ ion). In this presentation, PFIB-SEM has been used to perform 2D interfacial structure analysis and 3D imaging on different battery systems. First, to evaluate the performance of plasma ions processing with Ga⁺ reactive

battery system, lithium metal was selected for the study. Furthermore, the interfacial structure between Li-metal and sulfide based solid electrolyte in all solid-state battery system was investigated. In order to demonstrate the capability of accessing large representative 3D volume through PFIB-SEM, the microstructure of thick NMC 811 cathode ($>100\text{ }\mu\text{m}$ field of view with $>70\text{ }\mu\text{m}$ thickness) was selected for 3D reconstruction and quantification. The microstructural characteristics extracted from representative 3D volume helps the understanding of correlation between electrode microstructural evolution and performance deterioration.

The success demonstration of PFIB-SEM opens up exciting opportunities for exploring both current and future generation advanced battery technologies, which is expected to become a critical analytical approach for battery development.