Reliable Microscopy and Microanalysis Strategies for Real-World Batteries

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Rechargeable batteries, including Li-ion batteries (LIBs) and beyond-Li batteries, are essential energy storage devices to electrify the modern society, improving the utilization of renewable energy sources and achieving the carbon neutralization mission. Transmission electron microscopy (TEM) is an indispensable analytical methodology to characterize materials structure and composition at atomic resolution, which is particularly powerful for battery research to investigate the crystal lattices, microstructures, local defects, and chemical compositions of materials used in battery electrodes, electrolytes, and other components [1]. Recent advancement of in situ TEM has enabled the real-time observation of various dynamical phenomena and chemical processes during battery cycling and the associated phase transformations, manifesting itself a rapidly growing area in this field [2]. However, there are several pressing challenges that need to be addressed in order to obtain accurate and reliable results. Two of the most significant issues are the radiation damage by the high-energy electron beam and the side reactions between lithium (or other alkali metals) compounds and moisture and oxygen in the air. In addition, the capability to perform operando tests under the same working conditions is vital to the new battery design and development. Therefore, it is desired and urgently needed to develop reliable electron microscopy and microanalysis strategies for characterization, analysis, and diagnosis of real-world battery materials.

We have developed and demonstrated effective methodology and instrumentation to tackle with the scientific questions in realistic batteries. Figure 1 shows a schematic illustration summarizing the critical issues and the relevant strategies to overcome or mitigate those problems. Specifically, to minimize the impact of electron beam radiation, TEM techniques with low-kV and low-dose settings are typically useful to maintain the original material structures from knock-on damage, while cryo-TEM to freeze the Li-containing sample at low temperatures can effectively preserve the intrinsic structures to allow for atomic-scale imaging of the sensitive materials and interfaces such as Li metal and solid-electrolyte interphase (SEI) [3]. From another aspect, Li and alkali metals and their compounds inside batteries are also extremely sensitive to ambient environments, easily causing unwanted by-products through side reactions with oxygen and moisture during sample preparation and transfer. In this regard, a necessary need of air-free transfer system is required for an ideal workflow of battery characterization and diagnosis. A special air-free TEM holder has been demonstrated its effectiveness in assuring excellent protection during FIB preparation and the subsequent transfer between microscope vacuum chambers and inert gas-filled glovebox [4]. Furthermore, MEMS-based platform that enables in situ TEM with operando electrochemical testing functions can provide direct relationship linking the battery structures and compositions with the functionality and performance [5]. Overall, rationally choosing appropriate experimental setups and workflows will promise effective and precise electron microscopy and microanalysis for sensitive materials relevant to real-world applications [6].

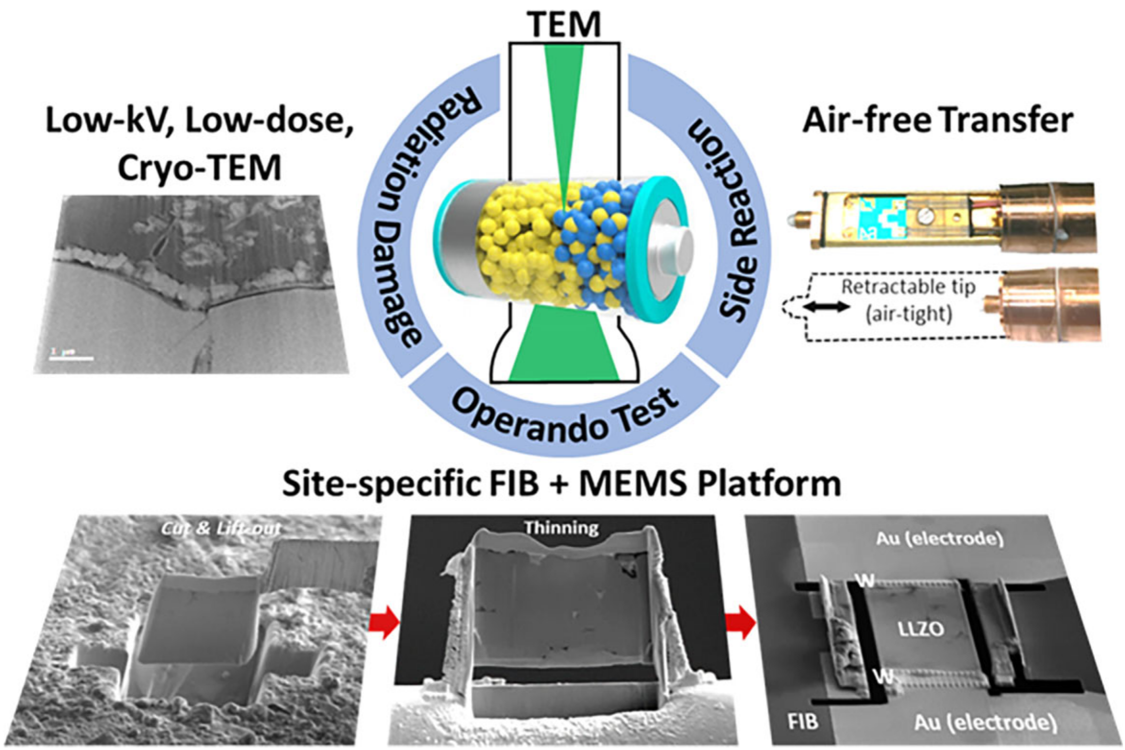

Fig. 1. Schematic illustration showing critical issues for TEM characterization of battery materials and relevant approaches to mitigate these fundamental challenges.

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