doi:10.1017/S1431927620014117
Microsc. Microanal. 26 (Suppl 2), 2020

SEM and Raman In-situ Stages Applied on Air Sensitive Lithium Ion Battery Materials Inspection

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Lithium ion battery (LIB) with excellent properties such as high power density as well as gravimetric capacity among other types of battery [1] plays as a crucial energy converting carrier between renewable energy and electric grid. Hence, investigating and dealing with the safety and long life time topics of LIB are of great importance for building up a safe and energy efficiency electric grid application. Due to lithium is a highly active and flammable element and can react with the water and oxygen in atmosphere. Therefore, using the sample carrier with the air isolation function during the sample transferring has become an essential procedure while investigating air sensitive lithium-based battery materials. Due to the isolation from the water and oxygen in atmosphere, the possibility of active lithium reacting with water and oxygen is suppressed which ensure the pristine information, for instance, composition, bonding state, morphology could be obtained from the sample during microscopy related analysis procedures. In some scenario of finding the reasons caused lithium ion battery failed, however, it often need acquiring the complementary information from specific sample position instead of from entire specimen, and therefore the sample carrier should also have the isolation function after the sample was loaded out from the instrument vacuum chamber so that ensure the same position on sample could be inspected under pristine condition via various analytic instrument. To overcome this bottleneck, we develop the common in-situ stage for compatible with both SEM and Raman spectrometer which provide more abundant information such as morphology, element distribution, and bonding type at same sample area comparing with single environmental protected stage in SEM [2] and Raman [3] spectrometer respectively.

The configuration of in-situ SEM/Raman stage in fig. 1(a) shows the relative positions between SEM objective polepiece, EDS detector, and in-situ stage. The sample was transferred and fix on the in-situ stage in glove box (oxygen≤ 10 ppm and water≤ 5 ppm) then rotated the sealing window to the sample position which would protect the sample under Ar atmosphere free from oxidation before the stage was load into SEM chamber. Till the SEM chamber vacuum at 10-4 mbar order, we used the remote electrical control to open the sealing window so that the electron beam can scan the sample surface through the observation window. Fig. 1(b) shows the EDS result of pure lithium metal test samples with and without stage protection. It clear shows that the stage could prevent the oxygen react with lithium so that the oxygen signal is suppressed down to negligible level, the inset also shows that the original lithium metal surface still preserves the slightly scratched morphology, however, if without stage protection, it shows particle-like morphology.

Fig. 2 demonstrates the integrated SEM & Raman analysis result of same sample area for graphite anode via in-situ stage. As shown in fig. 2(a), the original graphite anode shows clearly carbon D band and G band signals before carrying out charge/discharge cycling test at Raman shift of 1311.7 and 1579.3 cm-1 respectively. Increasing the power density during cycling or cycling times, the D and G band peaks width gradually increased and peaks height reduced which indicated that the graphite crystal structure gradually decomposed due to the continuing lithium ion insertion/desertion of graphite crystal [3]. As indicated in fig. 2(b), the D and G band peaks already disappeared at 15 charge/discharge cycles. After the Raman experiment, we compared the optical microscope image and SEM image to proceed the EDS analysis and morphology photography as the same location as acquiring Raman spectrum as shown in fig. 2(c~f). The SEM images show the coarse dendrite for lower power density and thinner dendrite for higher power density. The EDS mapping result also shows more amount of fluorine signal at higher power density charge/discharge cycle than at that lower level.

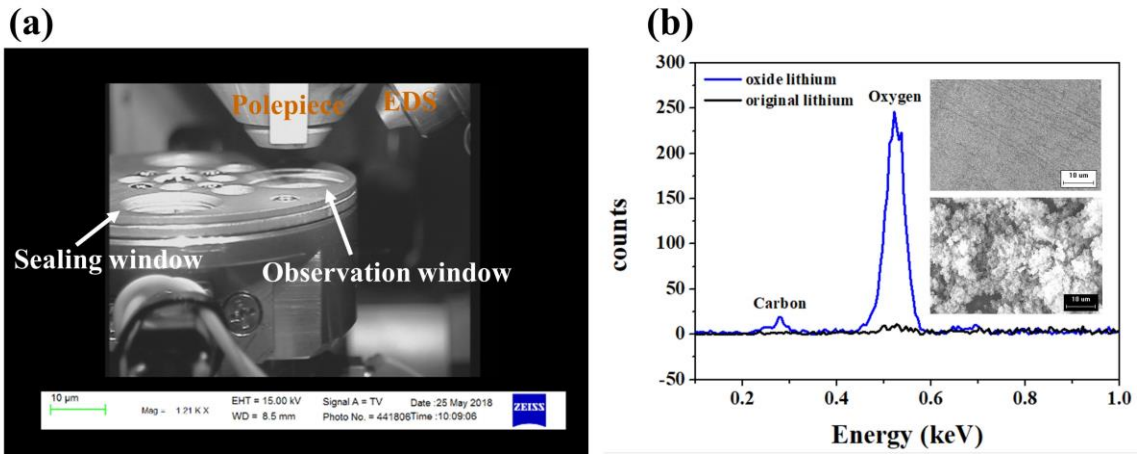

Figure 1. (a) The relative positions of stage, EDS detector, SEM polepiece (b) Oxygen contaminating examination of original lithium (environment-protected), and oxide lithium (air-exposed). The corresponding inset of SEM images for both original/oxide lithium.

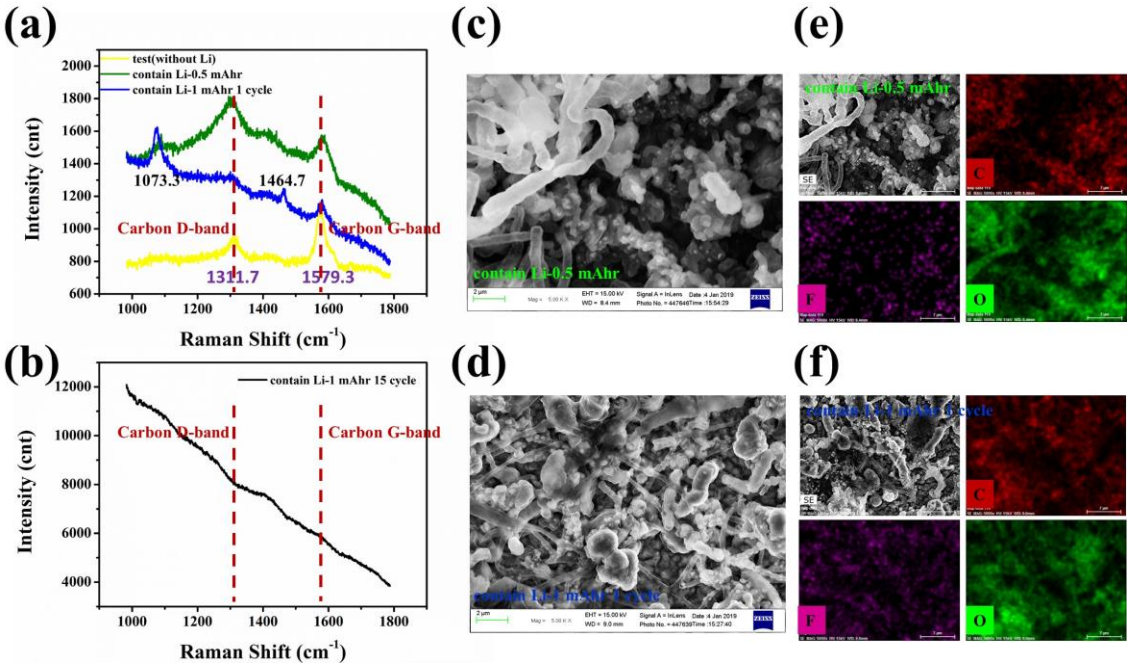

Figure 2. In-situ common stage for Raman/SEM inspection (a,b) The Raman spectrum of graphite anode at different charge/discharge cycle condition (b~f) SEM result of the same location in Raman inspection

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https://doi.org/10.1017/S1431927620014117 Published online by Cambridge University Press