doi:10.1017/S1431927622003737
Microsc. Microanal. 28 (Suppl 1), 2022

Battery in situ Electrical Testing in FIB-SEM

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We present a method and equipment for battery preparation in FIB-SEM and consequent electrical testing of the battery inside FIB-SEM chamber. Electrochemical cells were prepared from lithium metal and lithium titanate (LTO) using Xe PFIB (Helios 5 Hydra) and placed on the electrical testing holder [1] mounted on SEM stage. Ionic liquid EMIM BF_4 was selected as the high vacuum compatible electrolyte [2] for in situ battery testing. After the electrodes had been prepared on the electrical testing holder, the ionic liquid was moved between them using FIB (Figure 1). Capacity measurement as well as charging and discharging of the micron sized battery was possible in FIB-SEM [3].

Two different transfer modules were designed for airless transfer of the electrical testing holder from glove box to SEM and back. First module, which is shown in Figure 1, is based on the Clean Connect device [4]. The modified shuttle contains the electrical testing holder and a stub for bulk material, from which the battery can be created by FIB. The shuttle is transferred under argon overpressure in the Clean Connect capsule. When connected to SEM chamber and pumped to high vacuum, the shuttle is loaded into a SEM stage adapter, which provides electrical connection between the battery testing holder and a connector on the air side of a SEM chamber.

Second module is a standalone transfer box shown in Figure 2. The box is sealed by the overpressure of surrounding air after it is closed and pumped either in glove box or in FIB-SEM. The operation inside the microscope is assured just by the SEM stage. The box gets opened during pumping of the SEM chamber and its bottom part with the electrical testing holder can be moved under the electron or ion beam. After battery preparation, imaging or in situ electrical testing in FIB-SEM, the transfer box is closed in high vacuum before chamber venting. Air sensitive samples inside the box are then transferred from FIB-SEM under vacuum.

The MEMS holder [1], which was so far described as the electrical testing holder, also provides possibility for sample heating and enables in situ SEM imaging of battery materials transformation at high temperatures (40°C to 1200°C). The heating option can further be combined with delivery of reactive gases into the sample area [5]. This approach allows for in situ SEM imaging of battery materials synthesis controlled by the sample temperature and by the composition of injected gas.

Each of the transfer modules can protect air sensitive (e.g. lithium) samples for several tens of minutes during the transfer among glove box, FIB-SEM and other tools. The presented transfer modules can accommodate future customized testing holders serving for in situ testing of different types of batteries in SEM.

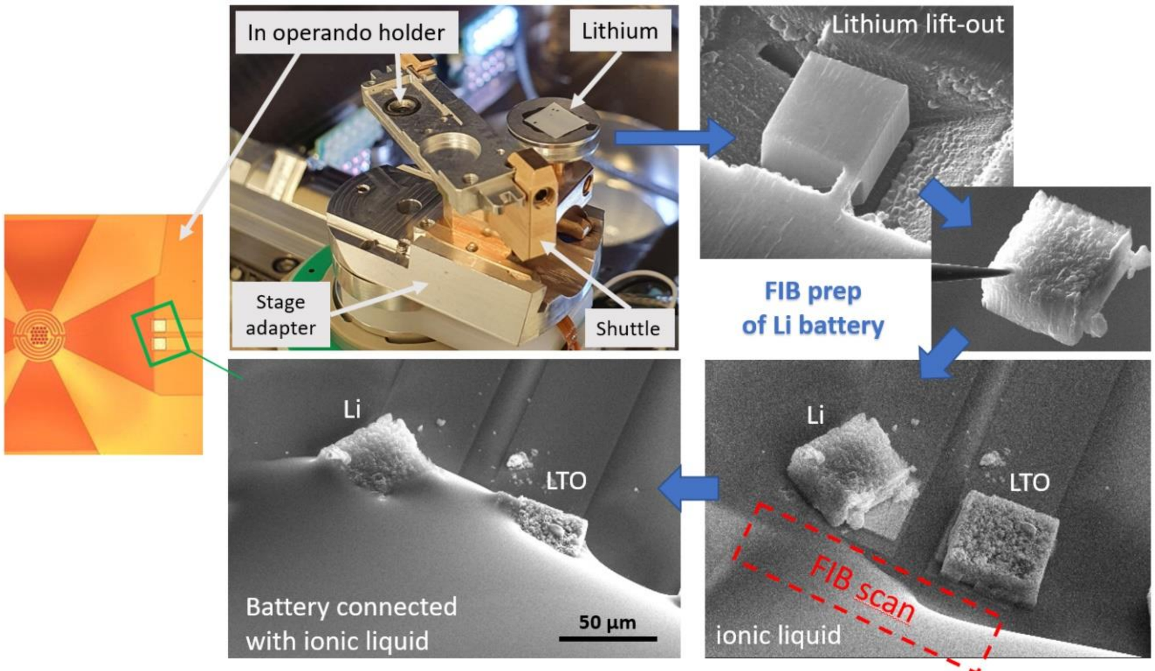

Figure 1. FIB preparation of LTO battery on electrical testing holder. Ionic liquid (EMIM BF_4) was used as the electrolyte in high vacuum. The battery testing holder was electrically connected with a SEM feedthrough via a SEM stage adapter. A shuttle containing the testing holder and a stub for bulk battery materials was transferred to SEM under argon using Clean Connect transfer device [4].

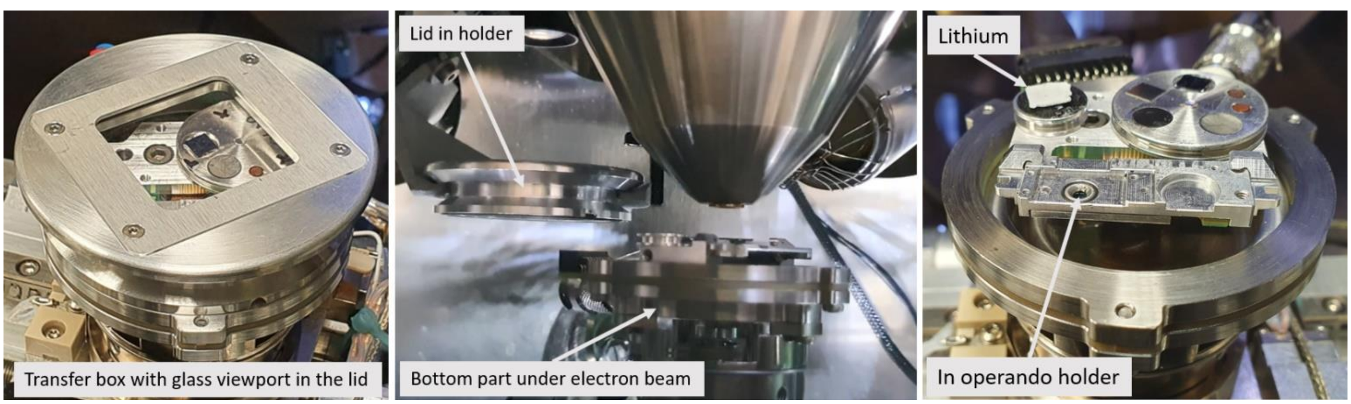

Figure 2. Left: transfer box closed and pumped during transfer from glove box to SEM. Middle: box opened in high vacuum in SEM, sample moved under electron beam. Right: the box contains electrical testing holder and stubs for bulk samples. FIB-SEM battery preparation and in situ testing is possible in the transfer box.

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https://doi.org/10.1017/S1431927622003737 Published online by Cambridge University Press