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IN-SITU SEM ANALYSIS OF LITHIUM METAL POLYMER BATTERY

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Lithium metal polymer battery (LMP) is an “all solid” energy storage technology that uses only very light components. This leads to a very high energy/weight ratio making it one of the best candidates for use in both electric and hybrid cars. The main physical characterisation tool used is the SEM because the total thickness of a unit cell is less than 100µm. SEM characterisation has played a key role in the discovery of the causes for certain types of failure modes (ex.: dendritic growth on the anode, dissolution of cathode materials in the electrolyte, etc.). However, for “dynamic failure modes”, like power fades and area specific impedance (ASI) increases, in-situ characterization (i.e. characterization of a cell during cycling) must be preferred. This work will present in-situ SEM analysis techniques applied to LMP cells. Attention will be given mainly to the lithium plating and unplating processes, to the salt gradient concentration during cycling and to the evolution of morphology during lithium plating and unplating.

An LMP cell typical microstructure is shown in Figure 1. The total thickness of a single fully charged cell (V=3.5V) is around 100µm. The Figure shows a cross section of an electrochemical cell (EC) developed for use in electric vehicles (150Wh/Kg per EC). The major advantage of the LMP cells over Li-ion cells in term of SEM characterization is that all components (anode, cathode and electrolyte) are solid. In LMP, the anode is metallic lithium and the cathode is a mixture of electrochemical active oxide, polymer and lithium salt. The same polymer and salt are also used for the solid polymer electrolyte (SPE). Because of the great diversity of materials, a special preparation technique has been developed to efficiently prepare cells for SEM examination. Specimen preparation is done in a dry room (relative humidity below 50ppm). Samples can be transferred from the glove box to the SEM using a specially designed controlled atmosphere holder. This keeps the metallic lithium from being attacked by water from the ambient air.

In order to increase electrochemical activity, LMP must be cycled at around 80°C. Either a heating film or a Pelletier device is used to heat the cell in the SEM. A holder that applies pressure while heating has also been designed. In-situ SEM examination can either be made on a plane view (looking at the back of the lithium film) or in a cross section. In cross section, tungsten carbide (WC) probes with a tip diameter under 1µm are used to probe the desired cell assembly (see Figure 2). The probes allow us to obtain the cell voltage, measure the ASI and cycle the cell. During cycling, the cell voltage and performance are recorded using a standard cell cycling apparatus. The cycling protocol can be ajusted to the phenomena being studied. SEM images are recorded during cycling using a time laps video recorder. The configuration of the probes relative to the x-ray detector allows for lithium salt concentration measurements during cycling.

In conclusion, in-situ SEM cycling of an LMP cell improves the use of SEM characterization and allows for cell component examination under “working” conditions. This new technique has been shown to be essential for failure mode characterization. It is also a powerful tool for investigating lithium plating mechanisms. A video of a cycling cell will be presented.

Références :
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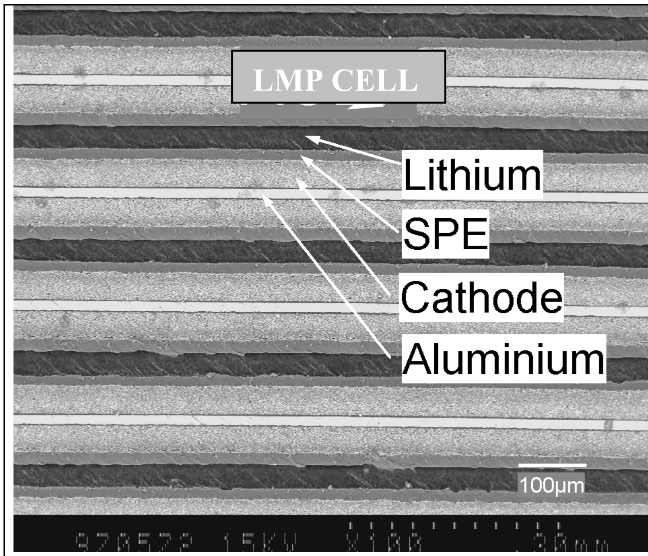

Fig. 1

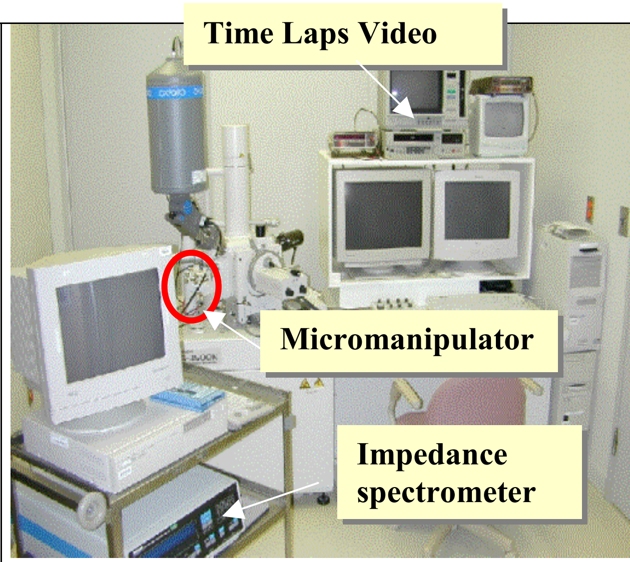

Fig. 2

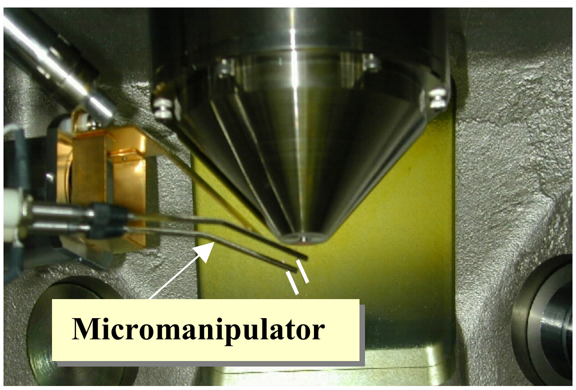

Fig. 3

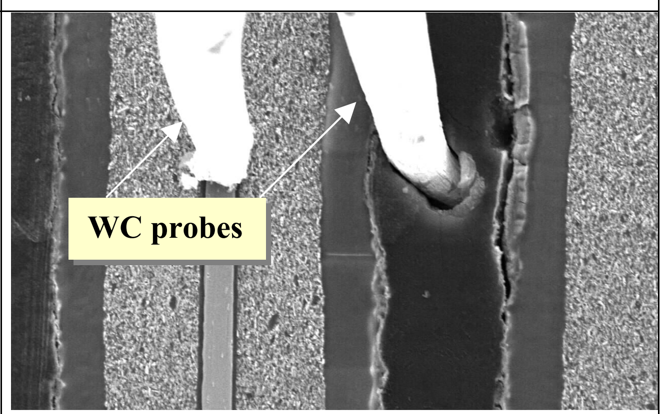

Fig. 4

Figure 1: Cross-section view of a LMP EC showing the stacking of individual battery
Figure 2: Photos showing the SEM and the different apparatus used for IN SITU experiment
Figure 3: Photos showing the SEM pole piece and the micromanipulator
Figure 4: Micrograph showing both the WC probes and the cell under IN SITU cycling

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