Development of Operando SEM technique for fabrication of lithium ion battery

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Lithium-ion battery is one of the most important energy storage devices. To improve the performance, silicon (Si) is expected as good anode material because of very high theoretical specific capacity (4200 mAh/g). However, when a large amount of lithium ions (Li^+) are inserted into the Si by charging, the volume changes by more than 300% and damage the electrode. Therefore, much effort has been devoted to avoid such mechanical damage to realize more stable cycles of charge and discharge without reducing the capacity.

In the previous ex-situ scanning electron microscopy (SEM) study, it was found that the silicon particles broke into several fragments, and solid electrolyte interface (SEI) layer was sequentially formed around these fragments. However, the process of expansion and contraction of the silicon particles and the process of the SEI formation have not been observed microscopically. Such microscopic in-situ observation has been required to understand and improve the battery material. Transmission electron microscope is a powerful tool to obtain local structure at high spatial resolution, but it has the disadvantage that it is necessary to prepare an observation sample that is thin enough to allow electron beams to pass through.

In this study, we have designed a special cell for in-situ SEM observation of lithium ion battery during charging and discharging processes. By in-situ SEM observation, we can dynamically observe the expansion and contraction process and degradation process of silicon particles. Comparing the different processes under different components utilization can help us to have a better understanding on the growth process of silicon particles.

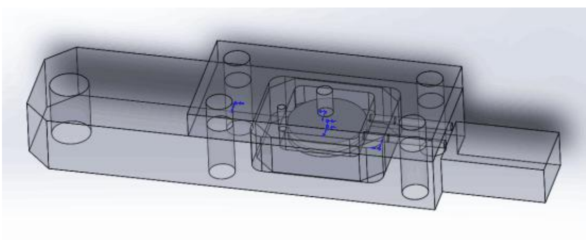

Figure 1 A design of sample stage of our developed SEM holder

Figure 1 shows the basic structure of the sample stage of our developed SEM holder. It consists of three parts (body, inner stage and cover). Both body and cover are made of stainless steel 304 and the inner stage is made of insulating material. The cover has the observation holes and electrolyte injection holes. The body has a hole for fixing with the SEM holder. The cell maintains good air tightness as a whole. The cathode and anode materials can be connected with electrical wires, respectively. It means that we can obtain SEM images simultaneously with measuring cyclic voltammetry. The assembled battery cell was confirmed to have low internal resistance.