Air-Isolated Sampling of Solid-State Battery for TEM

Solid-state Battery

A solid-state battery is made of cathode, anode and electrolyte (Fig. 1). This type of battery doesn't use liquid state electrolyte, so it tends to avoid the issues associated with leakage of electrolyte and ignition/explosion. Recently, silicon has been used as an anode material to improve the battery charge capacity (can store ten times more charge as compared to graphite anodes), but some challenges remain in terms of volume expansion during cycling, low electrical conductivity, and instability of the SEI (solid electrolyte interphase) layer caused by repeated volume changes of the Si material.

For the purpose of observing structure and shape of anode silicon grains (charged 90 %), a TEM sample was prepared under air isolated condition.

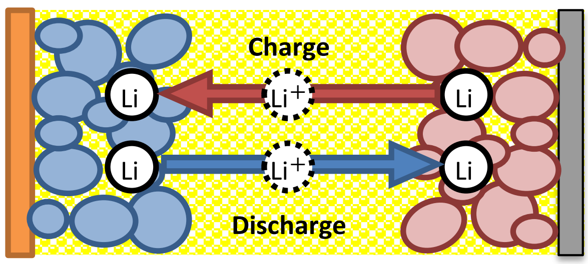

Fig. 1. The schematic of a structure of solid-state battery and charge/discharge

TEM Sampling Process under Air-Isolated Condition

A lithium battery sample was made under air-isolated conditions from start of preparation to observation to avoid reaction with air and potential oxidation (Fig. 2). For the sample transfer between instruments, a transfer vessel and slide cover holder were used. For the TEM sample preparation by FIB, an in-chamber manipulator (OmniProbe350, Oxford Instruments) was used. A cross section was first prepared by Ar-ion beam Cross-section Polisher (CP) to expose a large area showing the inner grain part of the battery, and subsequently decide the FIB processing area. JEOL air-isolated preparation methodology allows movement between all steps - CP process, SEM observation/analysis and TEM sampling - by using just one sample holder.

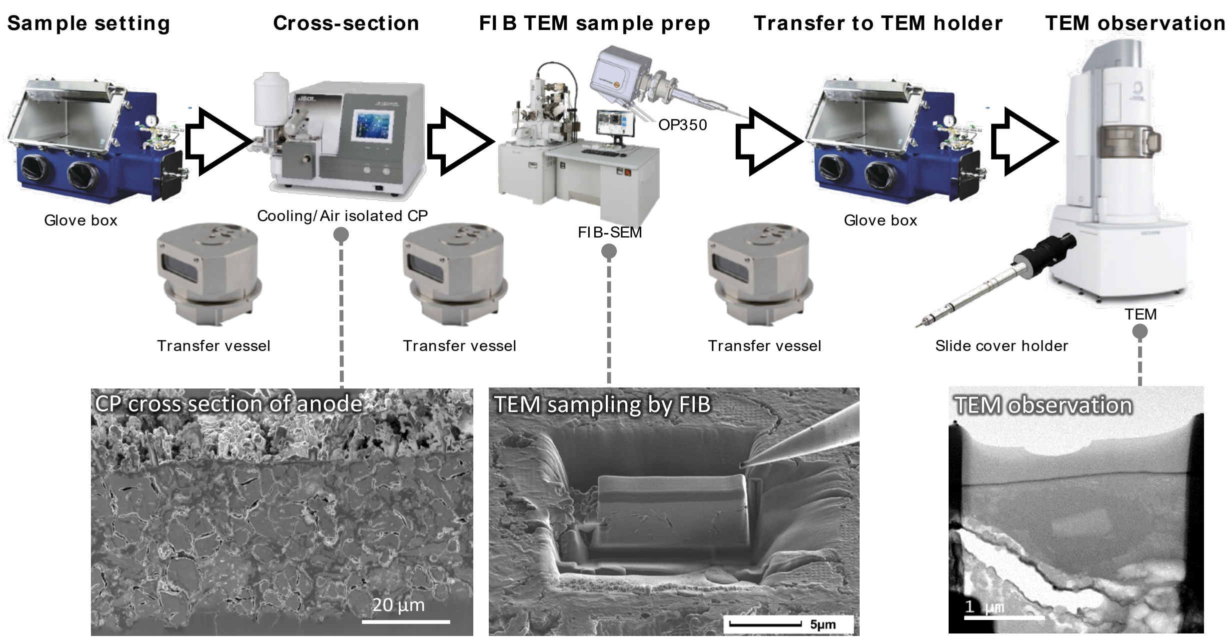

Fig. 2. Process of FIB TEM sample preparation under air-isolated condition

TEM sample preparation from CP-processed surface

A TEM sample of silicon anode material of charged all solid-state battery was made under air-isolated process which is shown in Fig. 2. Fig. 3 shows backscatter electron compositional (BEC) image and EDS map. This surface was prepared by CP. FIB process used this CP surface for choosing the correct position on the anode grain to subsequently prepare a thin TEM lamella (Fig. 4).

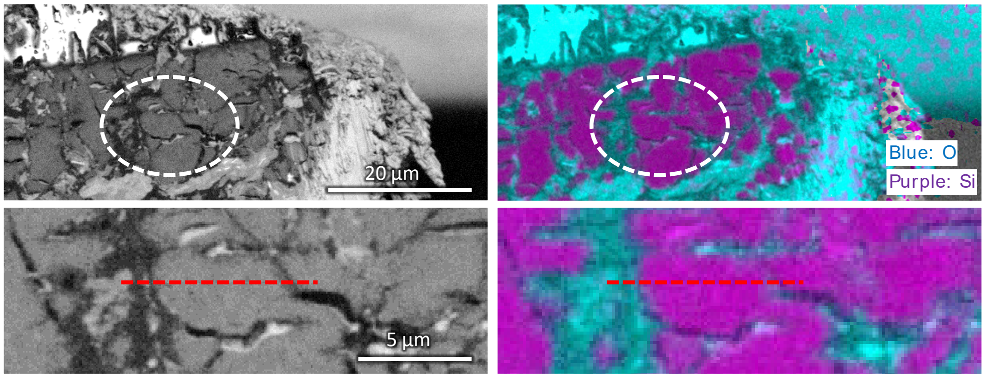

Fig. 3 SEM BEC image (left) and EDS map (right) of CP processed surface.

The silicon grain position was found from SEM BEC image and EDS map. TEM lamella position is identified by the red line.

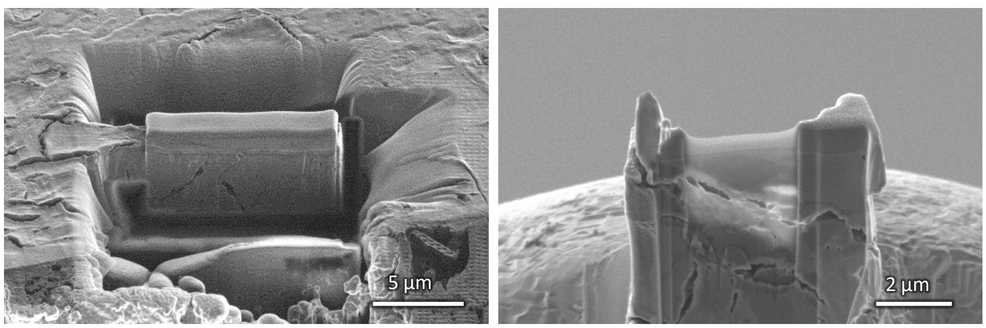

Fig. 4 Images during FIB process of making TEM sample. Sample block (left) and TEM lamella (right)
The sample block was made at the silicon grain position (left), and it was fixed to the FIB grid by using OmniProbe350, Oxford Instruments. After that, it was made into a lamella sample for TEM observation (right).

TEM observation and confirmation of air-isolated condition

Fig. 5 shows BF-STEM image of TEM sample. The red line encompasses the targeted silicon grain. After TEM observation, the sample was exposed to air, causing a change due to oxidation (Fig. 6). This result indicates that the entire sampling processes was indeed performed under air isolated condition.

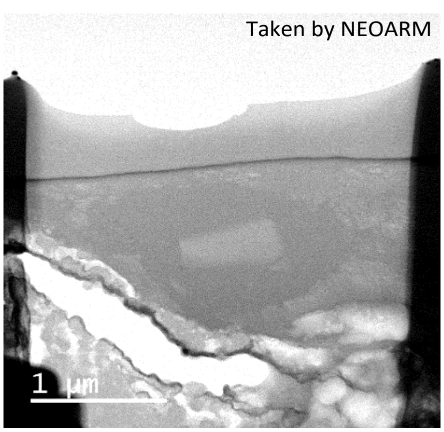

Fig. 5 BF-STEM image
There were three layers in silicon grain inner part from the center to outside. It is speculated that the center part was a silicon single crystal, and the lithium density increases from the center to the outside.

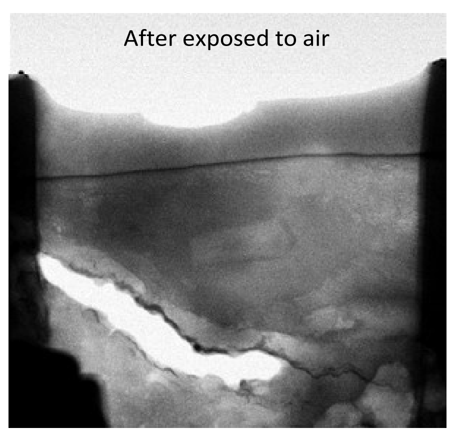

Fig. 6 BE-STEM image after exposed to air
After observation, the sample was exposed to air. Then retaken TEM image shows that it has reacted with air.

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