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3D Structural Analysis of Lithium Ion Secondary Battery Material

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The interior of the LIB consists of cathode, anode, and separator, and when these are impregnated with electrolyte, network of ionic and electronic conduction is connected to allow electrochemical reactions to proceed and the battery to operate. The cathode, anode, and separator have complex 3D structure consisting of multiple components. The 3D-SEM method using an focused ion beam and a scanning electron microscope (FIB-SEM) has been applied, and case studies of the cathode have been reported. On the other hand, there are limited reports on the anode and separator. This is due to the fact that the constituent elements of the anode and separator are similar to those of the resin used for embedding, making it difficult to distinguish between them by contrast in the SEM images. In this study, we developed and applied proprietary pretreatment methods of 1) anode: high-contrast resin embedding and 2) separator: simultaneous multi-layer staining, in order to visualize and quantify the 3D structure of the anode and separator, which had been difficult in the past. Two commercial consumer LIBs (high-capacity type and high-power type) were dismantled, and the anode was extracted, washed with diethyl carbonate, and vacuum-dried. After staining the binder, the obtained specimens were encapsulated in the proprietary high-contrast resin, transported to the FIB-SEM system, and continuous slice images were obtained using SEM. Using the obtained serial slice images, we constructed a trivalued 3D model consisting of active material, binder, and pore, and analyzed the 3D structure. A comparison of SEM images of graphite anodes of high-capacity LIBs obtained by normal and high-contrast resin embedding is shown in Fig. 1. In both images, the white areas show the distribution of the stained binder. In the conventional resin-embedding method, it is impossible to distinguish the resin part (pore) from the active material, but in the high-contrast resin-embedding method, it is possible to clearly distinguish the gray part as the resin and the black part as the active material. The results of 3D analysis of the high-capacity and high-power graphite anode are shown in Fig. 2. Compared to the high-capacity type, the high power type anode tends to have a larger pore size. Quantitative correlation with battery characteristics will also be investigated. The presentation will also show the results of the separator analysis

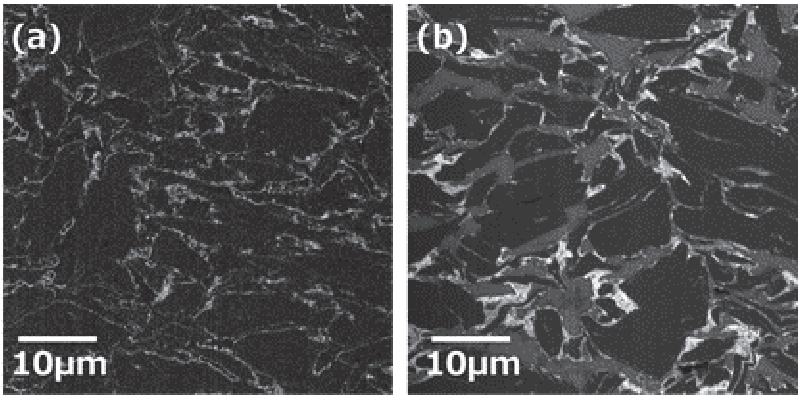

Fig.1 SEM image of high-capacity anode (a) conventional resin-embedded (b) high-contrast resin-embedded

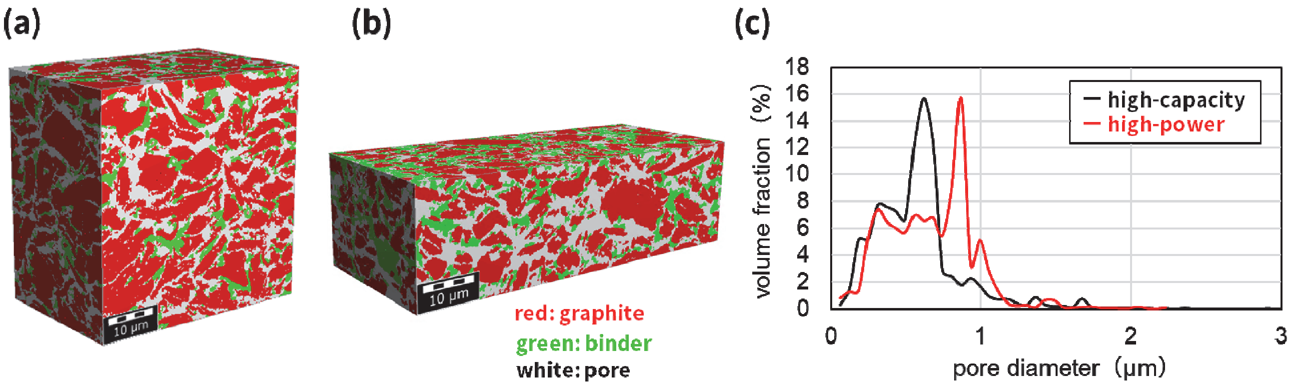

Fig.2 3D model (a) high-capacity anode (b) high-power anode and (c) pore diameter distribution