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Electron Channeling Contrast Imaging (ECCI) of Ion Battery Cathode Materials

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The microstructural characterization of ion battery cathode materials is often studied by transmission electron microscope (TEM) due to its high spatial and angular resolution which enables precise analysis of the battery materials. However, TEM studies present several challenges that might hinder their suitability for numerous potential applications. The conventional processes of preparing thin foils for TEM analysis, such as twin jet electropolishing, are time consuming and expensive. Moreover, when high-energy electron beams interact with the specimen in TEM, the studied sample may experience damage through various mechanisms, including knock-on displacement, radiolysis, and heating, leading to changes in the microstructure and properties of materials, particularly those sensitive to beams [1,2]. Here, another interesting approach for bulk specimens is presented that offers both a high spatial resolution and a large field of view at relatively low accelerating voltages: the use of ECCI in a field emission scanning electron microscope (FESEM). ECCI is an imaging method that relies on the change of backscattered electron (BSE) intensity caused by differences in the angle between the incoming electron beam and the crystallographic orientation of the lattice planes in crystalline samples. When the incoming electrons are parallel (or very close to parallel) to the lattice planes, low BSE intensity and hence a darker area can be anticipated, while with the increase in the angle, higher intensity and a brighter area are expected. ECCI technique allows us to figure out how the specimen's crystallographic orientation changes, along with identifying features like grain boundaries and cracks, as well as individual lattice defects such as dislocations [3,4].

In this study, the microstructural evolution of two different layered Li-ion based (composed of secondary particles) and Na-ion based (composed of primary particles) cathode materials in their pristine state was investigated. High resolution secondary electron (SE) and ECC images were acquired with the use of Hitachi SU8000 dedicated FESEM at a relatively low accelerating voltage of 4 kV. For this purpose, cross-sectional samples were prepared using a Hitachi IM4000 ion milling machine.

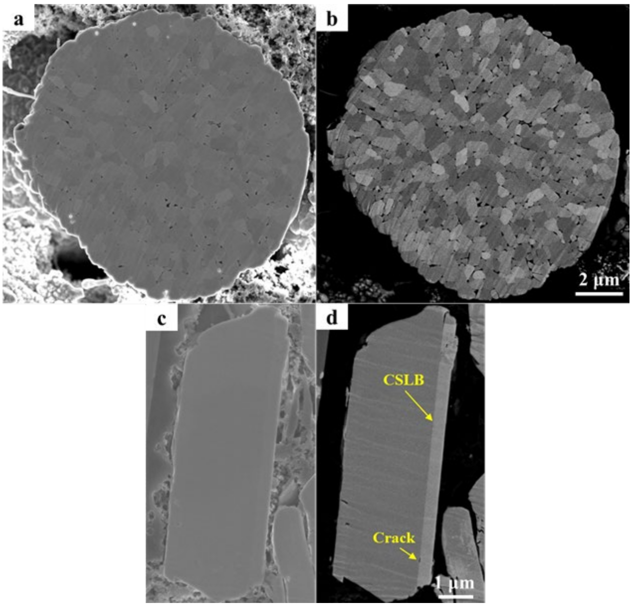
Figures 1a and b show the SE and ECC cross-sectional images of a Li-ion based cathode polycrystalline secondary particle. The secondary particle comprises many single crystalline primary particles. Primary particles are simply like the 3-dimensional grains found in conventional polycrystalline metallic materials with different crystal orientations. As can be seen in Figures 1a and b, the primary particles cannot be well recognized in the SE image (Figure 1a), while in the ECC image they are completely clear with very well-defined grain boundaries thanks to the ECCI technique (Figure 1b). Furthermore, the imperfections between the primary particles such as voids and pores are more evident with sharp edges in the ECC image compared to the SE image.

The SE and ECC cross-sectional images of a Na-ion based cathode single crystalline primary particle are represented in Figures 1c and d. The SE image displays the primary particle with an irregular shape without any distinguished lattice features (Figure 1c). However, the ECC image depicts the same particle with two clearly visible features shown by yellow arrows: I) coincidence site lattice boundary (CSLB) and II) intragranular crack along the CSLB as a preferential site for crack initiation (Figure 1d). In an earlier study by the authors [5], the

presence of CSLBs in the same specimen has been shown with the aid of Hitachi SU9000 scanning transmission electron microscope (STEM).
Conclusively, this work unraveled the unique role of ECCI technique as a perfect microanalysis tool for reasonably priced and rapid investigation of the cathode battery materials.

Figure 1. a) SE and b) ECC cross-sectional images of a Li-ion based cathode polycrystalline secondary particle. c) SE and d) ECC cross-sectional images of a Na-ion based cathode single crystalline primary particle.

Graphic:



Keywords:

ECCI, Battery, Cathode, Grain Boundary

Reference:

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