Lithium analysis of electrode materials of Li-ion batteries using analytical electron microscope

Noboru Taguchi[a]

[a] *Research Institute of Electrochemical Energy, Department of Energy and Environment, National Institute of Advanced Industrial Science and Technology (AIST), Ikeda, Osaka 563-8577, Japan*

E-mail: n-taguchi@aist.go.jp

Electrodes of lithium-ion batteries are composite devices made of various materials. Analysis methods that employ X-rays, such as X-ray photoelectron spectroscopy (XPS) and X-ray absorption fine structure (XAFS), which have high energy resolution, have generally been used for evaluating the composition and chemical state of materials.

Electron microscopy is known to be an effective technique to examine elemental distribution at the nanometer level. Analysis techniques with high spatial resolution is also becoming important. In particular, in scanning transmission electron microscopy (STEM), the analysis of chemical state by mapping, using a combination of analytical techniques such as electron energy loss spectroscopy (EELS) and energy dispersive X-ray spectrometry (EDS), with probes of nanometer size are powerful analysis techniques. We focus attention on the distribution of lithium elements in electrode materials, and we perform measurements by electron microscope using EELS. The STEM-EELS has the advantage of being able to evaluate very low-energy–loss region including Li K-edge with high spatial resolution. Combining the Li distribution with information on other elements and knowledge of the structure makes it possible to utilize it for analysis such as reaction distribution of battery materials [1, 2]. Such a STEM-EELS approach is becoming a well-known tool in the field of battery research.

Meanwhile, the electrode of an actual battery has a thickness on the order of tens of microns, and further analysis is desired with a wider view such as an attempt to further increase the thickness of the electrode. However, TEM(STEM) observation has some limitations especially for sample size. Therefore, Li evaluation method based on scanning electron microscopy (SEM) was examined. We have found that Li in compounds can be easily detected by reflection EELS (REELS) measurement using backscattered electron. REELS is easier to use for Li measurement in compounds compared with scanning auger electron spectroscopy (AES), which is an SEM-based analytical method. Currently, we are developing a measurement method for Li analysis using REELS. By using the Li K-edge signal, the chemical state of Li can be evaluated, and the Li distribution can be visualized [3, 4].

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References:

[1] J. Kikkawa, T. Akita, M. Tabuchi, M. Shikano, K. Tatsumi, M. Kohyama, Electrochem. Solid-State Lett. 11 (2008) A183.
[3] N. Taguchi, T. Akita, H. Sakaebe, K. Tatsumi, Z. Ogumi, J. Electrochem. Soc. 160 (2013) A2293.
[3] N. Taguchi, M. Kitta, H. Sakaebe, T. Akita, J. Electron Spectrosc. Relat. Phenom. 203 (2015) 40.
[4] N. Taguchi, M. Kitta, H. Sakaebe, T. Akita, Surf. Interface Anal. 48 (2016) 1144.