

A novel 2-dimensional combinative analytical method (IR imaging-Raman mapping-SEM) for the characterization of solid electrolyte interphases (SEIs)

Woo Sung Jeon,* Changhoon Jung, Eui-seong Moon,
Sunjung Byun and Hyouksoo Han
Samsung Electronics (SAIT), Yongin, Republic of Korea

The solid electrolyte interphase (SEI) is a protecting layer formed on the electrodes of Li-ion batteries as a result of electrolyte decomposition. As well-known, battery performance, such as irreversible charge “loss”, rate capability, cyclability, exfoliation of graphite and safety are highly dependent on the quality of the SEI. Therefore, understanding the actual nature and composition of SEI is of prime interest. However, the composition and the nature of SEI are still the subject of much controversy.¹⁻²

From an analytical point of view, the averaged composition or the components of the SEIs acquired from the conventional FT-IR or XPS technique might have not described well the whole picture of the SEIs, because the SEIs are chemically and morphologically inhomogeneous, namely a mixture of organic-inorganic composites.

Thus, a 2-dimensional (2D) mapping or imaging analysis would be needed for distinguishing between components and their distribution.² Electron Probe X-ray Microanalysis (EPMA) or scanning electron microscopy (SEM) equipped with Energy Dispersive Spectrometer (EDS) might be widely used for 2D-elemental mapping analysis.

FT-IR imaging spectroscopy, combining the spatial resolution of microscopy and the chemical specificity of conventional infrared spectroscopy, has already been known as a powerful tool for analyzing organic mixtures, such as blended polymers, human and animal tissues.³ However, FT-IR imaging spectroscopy has never been reported yet for analyzing the SEIs, probably because the SEIs are highly sensitive to contamination, air, and humidity.

In this presentation, we propose FT-IR imaging spectroscopy as a novel analytical technique for investigating the spatial distribution of the various organic lithium carbonates in the SEIs. In order to overcome the problems in analyzing SEIs, several instrumental modifications were devised. Firstly, the whole system of FT-IR imaging microscope is installed in a glove box (Ar purged) with a sample transfer chamber which allows the sample to be transported from the atmosphere to the analysis chamber of FT-IR imaging spectroscopy without exposure to air and humidity. As shown in Figure 1, a visible optical image, a secondary electron image (SEI) from SEM, and an FT-IR image of Li dendrites are successfully acquired at the same position of the same sample. Although the quality of these images differs because of the inherently different spatial resolutions of the images (e.g., ~1 μm for the optical microscope, ~100 nm for the SEM and 3.1 μm for the FT-IR image), the detailed FT-IR images clearly revealed that, among many plausible organic carbonates, the major component of the SEI on Li dendrites is ROLi in this experimental condition.

Further evolution of a combinative analytical method for the analysis of SEI using this unprecedented analytical tool, IR imaging, will be presented at the meeting along with other 2D-mapping techniques, such as micro Raman spectroscopy and SEM. The combined use of micro FTIR imaging spectroscopy, micro Raman spectroscopy and scanning electron microscopy (SEM) techniques for the

characterization of SEIs provides complementary information on the physicochemical characteristics of the interface between the electrode and electrolyte. Our approach for developing a new analytical methodology, 2D-imaging analysis, may provide new opportunities for the study of the SEI.

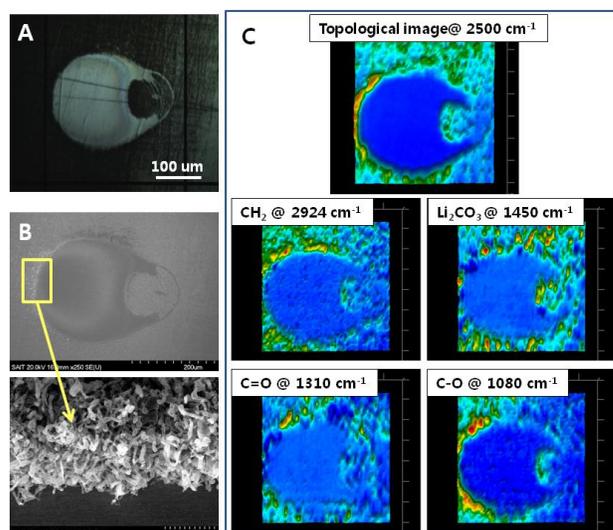


Figure 1. Visible optical image (A), secondary electron image from SEM (B), FT-IR image (C) of the same Li-dendrites on Ni foil. The chemical distribution of organic carbonates can be seen by color difference (Red: the higher peak intensity at the denoted wavenumber, Blue: almost no signal intensity at the same wavenumber.)

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