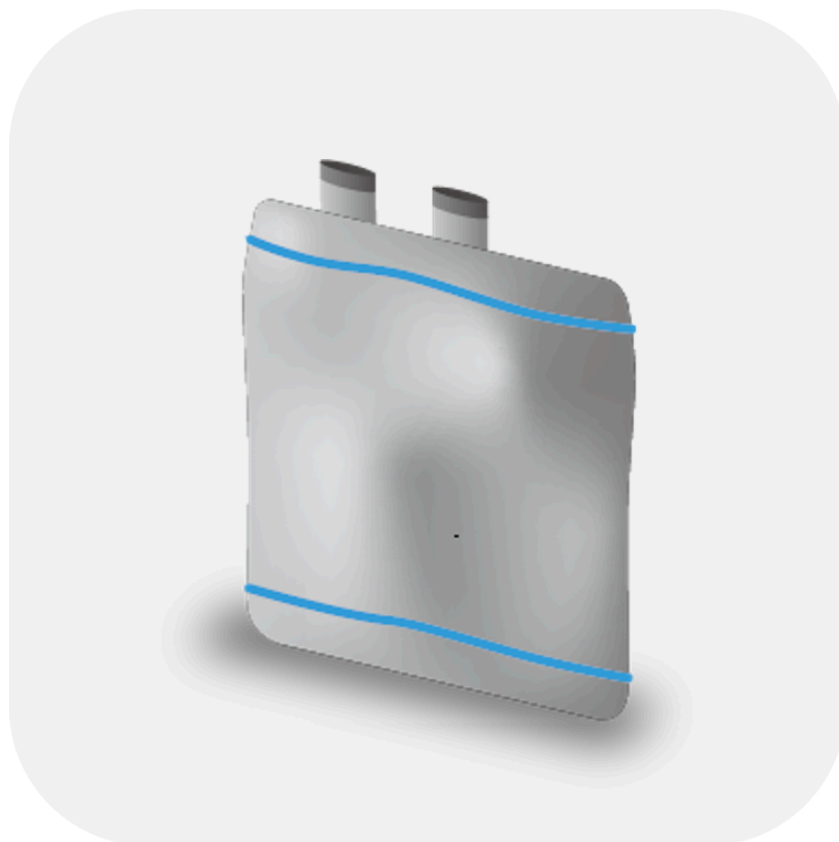


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Battery Performance



With the synthesis of each new battery material, the change in crystalline phase and deterioration behavior during repeated charge/discharge cycles are investigated to determine the effectiveness of the new material. Utilizing X-ray diffraction (XRD) or X-ray microscopy (XRM) measurements, materials characteristics and behavior can be observed non-destructively during these charge/discharge cycles.

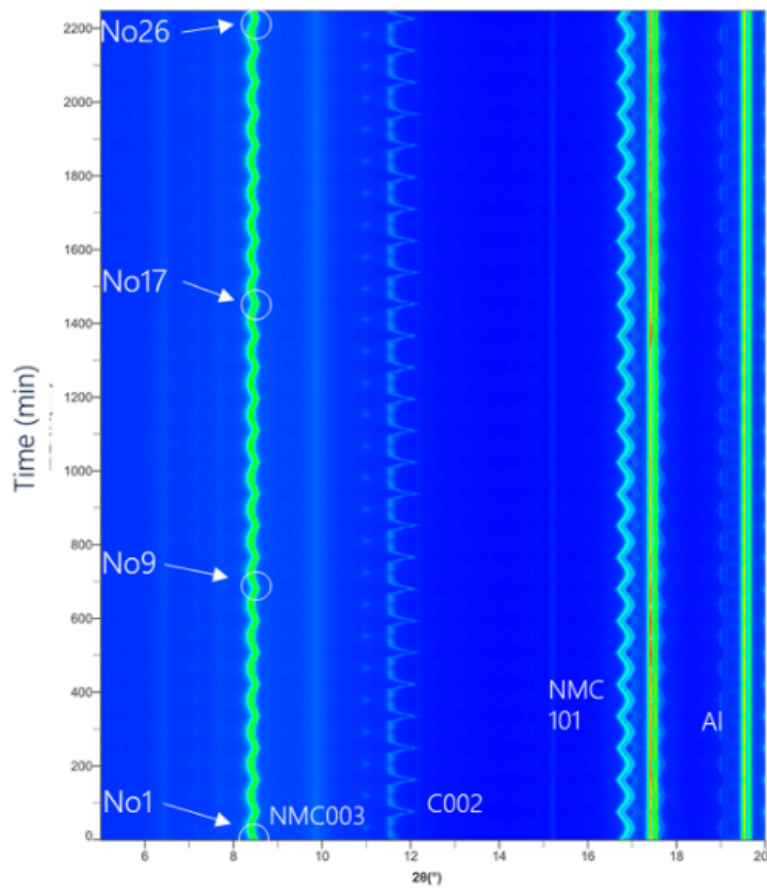
IN-OPERANDO MEASUREMENT OF POUCH CELL AND IN-SITU OBSERVATION OF BATTERY INTERIOR

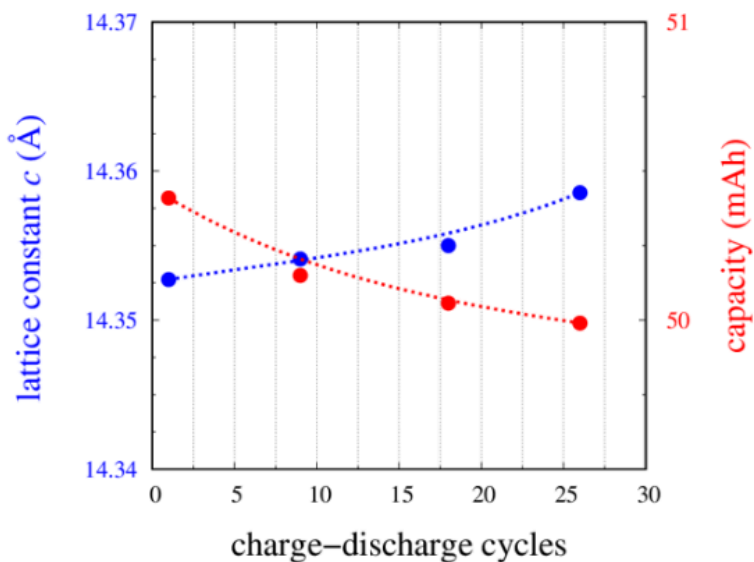
1. In-operando measurement of pouch cell with XRD

The reaction behavior of cathode and anode materials can be analyzed by in-operando X-ray diffraction (XRD) measurements during the discharge and charge process. In addition, the material's degradation behavior can be directly observed during the repeated cycling. As an example, in-operando XRD measurements were performed on a lithium metal

oxide battery focusing on the behavior of the cathode material (NMC). The study was performed through repeated charging and discharging cycles in order to investigate the deterioration of NMC. As the number of discharge and charge cycles were increased, it was found that the lattice constant expanded while the battery storage capacity decreased. The lattice expansion is due to Li-ions from the cathode being captured by carbon in the anode. The captured Li-ions are removed from the charging and discharging process, effectively reducing battery storage capacity. As the amount of captured Li-ion increases, metal dendrites will eventually be generated, causing the battery to short-circuit.

Waterfall plot of cathode material (NMC) with charge-discharge cycles



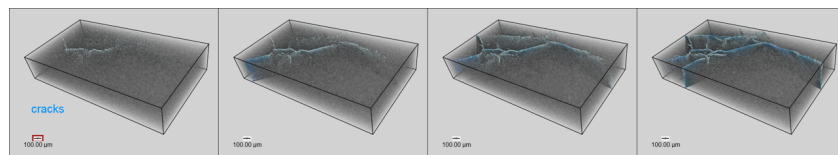


Lattice constant 'c' vs capacity with charge-discharge cycles

Samples provided by Toray research center, Inc.

2. Fine in-situ observation of battery interior with XRM

The degradation of a battery is caused by a variety of factors. When considered macroscopically rather than microscopically, there are problems such as vacancies, cracks, particle decay, and interfacial peeling. X-ray computed tomography (CT), an internal observation method, can be used for in-situ observation of the inside of a battery without disassembling it. Rigaku's nano3DX system enables high-contrast and higher-resolution measurement than X-ray micro CTs and can capture and visualize these phenomena.



3D rendered X-ray CT images of solid electrolyte. 2.5 micron/voxel

With X-ray tomography, it is possible to non-destructively visualize crack growth within a solid electrolyte during battery operation - providing key insights into potential degradation mechanisms.

More information available in: **Visualization and Control of Chemically Induced Crack Formation in All-Solid-State Lithium-Metal Batteries with Sulfide Electrolyte**

Misae Otoyama, Motoshi Suyama, Chie Hotehama, Hiroe Kowada, Yoshihiro Takeda, Koichiro Ito, Atsushi Sakuda, Masahiro Tatsumisago, and Akitoshi Hayashi

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<https://doi.org/10.1021/acsami.0c18314>

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