HORIBA

- O/N/H Analyzer EMGA Series –
- C/S analyzer EMIA Series –

Measurement of Oxygen Deficiency in Lithium Manganate

Background / Challenges - Surface contamination leads to the formation of an oxide film.

Background

Spinel-type lithium manganate ($LiMn_2O_4-\sigma$) is said to change its discharge characteristics due to slight differences in composition caused by oxygen deficiency. The differences in the discharge characteristics of lithium manganate batteries are also observed in the particle size and crystal structure, but attention is also paid to the difference in the oxygen bonding ratio, as samples with less oxygen bonding have poorer discharge characteristics. A customer asked us if we could confirm the slight difference in oxygen valence.

Challenges

The material is in powder form, and there are few instruments capable of accurate quantitative analysis of oxygen. In addition, oxygen analysis of powder samples with a large surface area requires a method to reduce the influence of oxygen present in the atmosphere, which requires "rapid measurement," "encapsulation to reduce contact with the atmosphere," and "encapsulation that can be performed quickly and accurately.

Solution from HORIBA

A need from a wafer manufacturer

"We do not want to require special pretreatment or advanced analytical skills. We also need to measure a large number of samples, so we want to analyze them easily and accurately…"
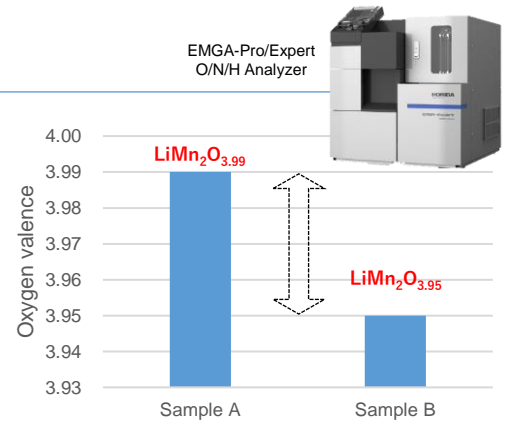
By sealing the sample in a Ni capsule using a special jig and a transfer vessel to reduce the atmospheric effects, a very high quantitative analysis was possible, and the difference in valence could be confirmed from the analysis results.


Capsule press jig

Transfer vessel

EMGA-Pro/Expert O/N/H Analyzer

$LiMn_2O_{3.99}$

$LiMn_2O_{3.95}$

	Oxygen(%)	Oxygen Valence
Sample A	35.29	3.99
Sample B	35.94	3.95
Theoretical value	35.39	4.00

Result of Oxygen Analysis

Related Application: the carbon concentration in the cathode materials

The EMIA-Expert can help control the carbon concentration in the cathode materials at ppm level. It can also accurately measure sulfur concentration, which affects battery lifetime, capacity and degradation. Analysis time is as short as approximately 1 minute, enabling highly accurate quantitative analysis with a small sample volume.

EMIA-Pro/Expert C/S Analyzer
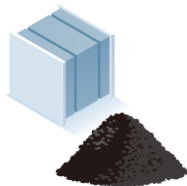
Sample weight	Carbon(mass %)
0.3059	0.0200
0.3061	0.0201
0.3055	0.0199
0.3057	0.0198
0.3059	0.0197
Average	0.0199
Standard deviation	0.0002
Coefficient of variation(%)	0.7900

Result of carbon content in $LiCoO_2$

Product Website

Inquiry Form

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