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Application News

SALD™ -2300 Laser Diffraction Particle Size Analyzer
iSpect™ DIA-10 Dynamic Particle Image Analysis System

Evaluation of Particle Properties of Lithium-Ion Battery Anode Materials —Particle Size Distributions and Shapes—

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User Benefits

- Particle properties (size distributions and shapes) can be easily evaluated to achieve battery performance targets or improve the quality of battery materials.
- The SALD-2300 can quickly measure particle size distributions.
- The iSpect DIA-10 can not only measure particle size distributions but also shape of particles, and you can make use of images of particles.

■ Introduction

Lithium-ion batteries (LiB) are rechargeable batteries that are charged or discharged by transferring lithium ions between cathodes and anodes. Because they are now widely used in everything from smartphones to automobiles, they are the subject of active research aimed at improving their capacity, service life, cost, and safety. The main components used in these batteries are cathodes, anodes, separators, and electrolyte solutions. Some battery materials can affect battery performance due to their particle properties (such as size distribution, shapes, density, specific surface area, and pore distribution), so their respective property values need to be optimized.

This article describes an example of evaluating anode materials using a laser diffraction particle size analyzer and a dynamic particle image analysis system.

■ Samples

Five types of natural spherical graphite powder that are used as an anode material were evaluated. The samples were provided by Dainen Material Co., Ltd. Detailed information about the samples used for this article is indicated in Table 1. Note that A-2 and B-2 are carbon-coated samples of A-1 and B-1 respectively.

Table 1 Sample Information

Sample	Product	Coating	Characteristics	Application Example	Particle Diameter
A-1	DG10-095	-	Output prioritized	HV	10 to 11 µm
A-2	DG-B10M1	✓	-	-	
B-1	DG15-097	-	Balance prioritized	EV	15 to 16 µm
B-2	DG-B15M1	✓	-	-	
C-1	DG22-098	-	Efficiency (life) prioritized	Gaming devices	22 to 23 µm

■ Measurement Instruments and Conditions

The SALD-2300 laser diffraction particle size analyzer (Fig. 1) was used to measure the distribution of particle size, and the iSpect DIA-10 dynamic particle image analysis system (Fig. 2) was used to evaluate particle shapes. The measurement parameters are listed in Tables 2 and 3.

Fig. 1 SALD™-2300 Laser Diffraction Particle Size Analyzer

Table 2 Particle Size Distribution Measurement Conditions

Instrument:	SALD-2300 Laser Diffraction Particle Size Analyzer
Unit:	Sampler
Dispersion Medium:	Purified water
Dispersant:	Surfactant (Tween 20)
Dispersion Method:	Circulation only
Refractive Index:	2.00 - 0.05i

Fig. 2 iSpect DIA™-10 Dynamic Particle Image Analysis System

Table 3 Measurement Conditions for Particle Shape Evaluation

Instrument:	iSpect DIA-10 Dynamic Image Analysis System
Dispersion Medium:	Purified water
Dispersant:	Surfactant (Tween 20)
Dispersion Method:	Stirring only
Frame Rate:	8 fps
Image Acquisition Efficiency:	97 %
Binarization Value:	110
Flowrate:	0.1 mL/min
Liquid Volume Measured.	50 µL
Concentrations Measured:	0.05 wt% (C-1) and 0.01 wt% (A-1, A-2, B-1, and B-2)

■ Measurement Results of Uncoated Natural Spherical Graphite

Fig. 3 shows the paticle size distribution of A-1, B-1, and C-1, which are without carbon coating, measured by SALD-2300. All three Samples were mono-dispersions with different particle distributions, and the larger the particles were, the wider the particle diameter range was.

Fig. 4 shows both the scatter plots of area based diameter and circularity, and the particle images, all of which were obtained from the iSpect DIA-10. These results are summarized in Table 4.

Fig. 4 shows a similar particle size difference as observed with the SALD-2300. The standard deviation values in Table 4, along with the scatter plots and particle images in Fig. 4 demonstrate that samples A-1, B-1, and C-1 have increasingly wider circularity ranges, with particles of larger diameters exhibiting greater shape variability.

Table 4 Particle Size Distribution and Particle Shape Measurement Results for Samples without Carbon Coating

		A-1	B-1	C-1
Median Diameter (μm)		10.174	16.035	22.187
Circularity	Mean	0.972	0.952	0.927
	Std. Dev.	0.037	0.044	0.051

Median diameter results were measured with the SALD-2300 and the circularity with the iSpect DIA-10.

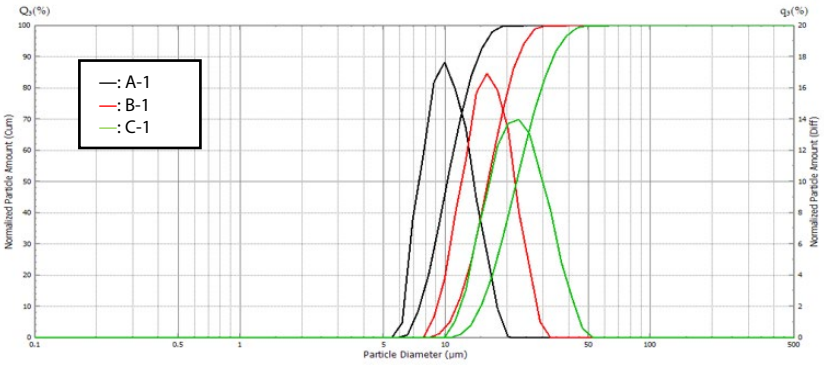

Fig. 3 SALD-2300 Particle Size Distribution Measurement Results for Samples without Carbon Coating

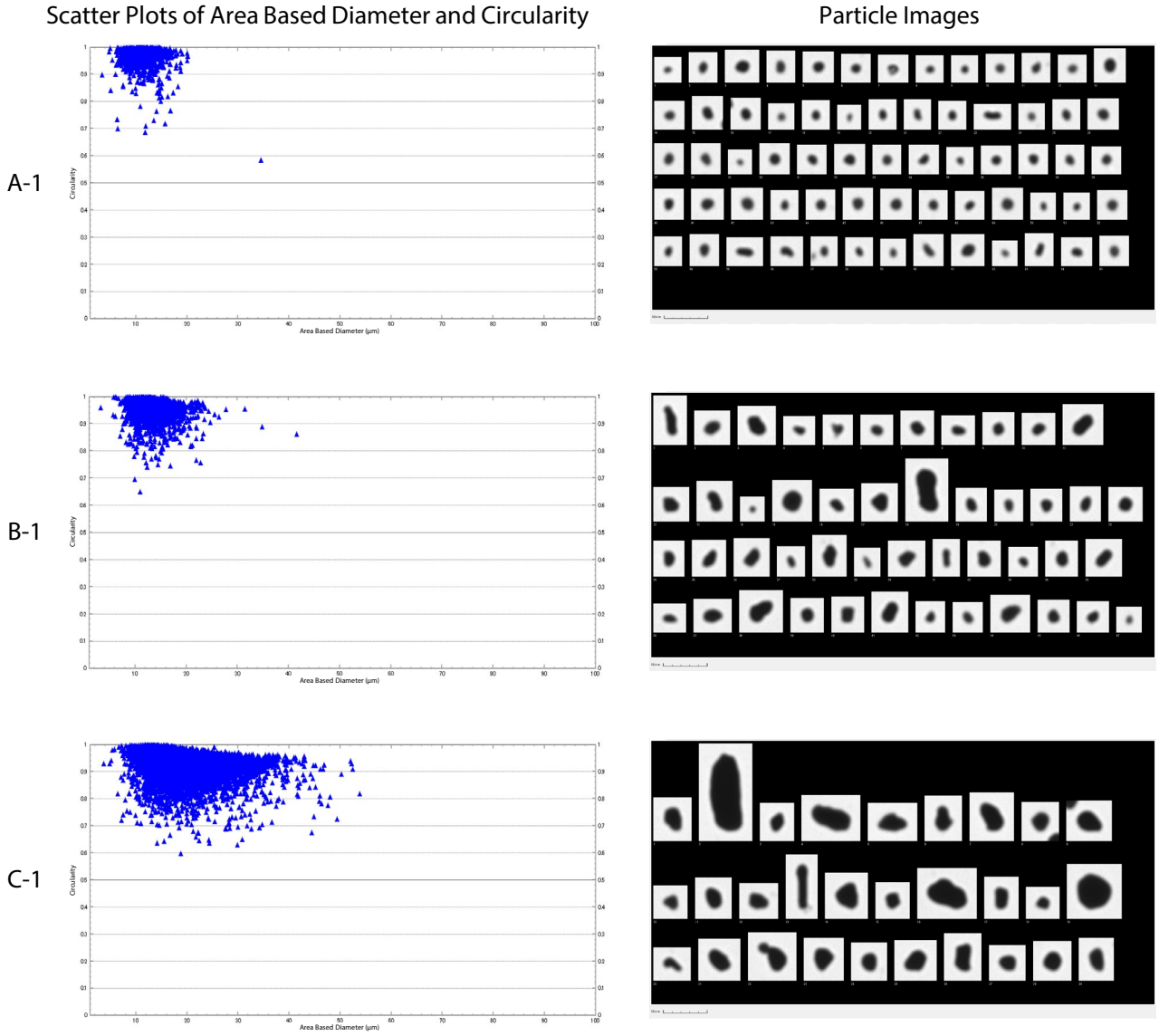

Fig. 4 iSpect DIA-10 Particle Shape Evaluation of Samples without Carbon Coating
Left: Scatter Plots of Area Based Diameter and Circularity; Right: Particle Image Examples (In Order of Acquisition)

■ Measurement Results of Coated Natural Spherical Graphite

The particle size distributions and particle shapes of samples with carbon coatings were compared with samples without coatings.

Fig. 5 shows the particle size measurement results acquired from the SALD-2300 analyzer. There were minimal differences between B-1 and B-2 particle size distributions, whereas the A-2 distribution was skewed towards larger particles. The median diameter values (Table 5) also show that A-2 particle diameters were larger.

Figs. 6 and 7 present the particle shape measurements for samples A-1, A-2, B-1, and B-2, which were conducted using the iSpect DIA-10. From the scatter plot, it can be confirmed that the number of coarse particles over 20 µm in A-2 has increased compared to A-1. In particular, it can be seen that the amount of particles with low circularity has increased.

In contrast, such differences were not as clearly apparent between B-1 and B-2 as between A-1 and A-2. Figs. 6 and 7 also show some of the particle images in descending order of area based diameter. The images of both A-2 and B-2 show particles that are apparently clusters of multiple smaller particles. This suggests agglomeration occurred during the coating process.

The above findings from the comparisons of A-1 and A-2, as well as B-1 and B-2, measured by the iSpect DIA-10 were also reflected in the results from the SALD-2300. It is thought that the particles larger than 20 µm observed in the particle size distribution of A-2, as measured by the SALD-2300, correspond to those observed in the scatter plots from the iSpect DIA-10.

Table 5 Particle Size Distribution and Shape Measurement Results Measured before/after Carbon Coating

		A-1	A-2	B-1	B-2
Median Diameter (µm)		10.174	12.307	16.035	16.122
Circularity	Mean	0.972	0.952	0.952	0.944
	Std. Dev.	0.037	0.054	0.044	0.050

The median diameter results were measured with the SALD-2300 and the circularity with the iSpect DIA-10.

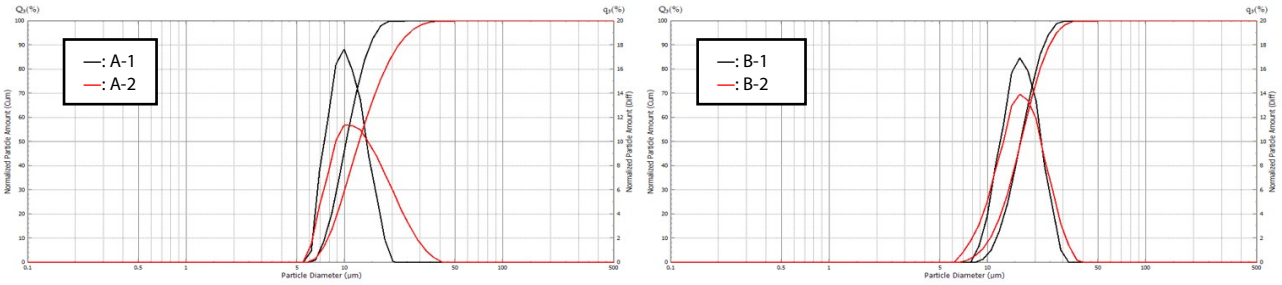

Fig. 5 SALD-2300 Particle Size Distribution Results Measured before/after Carbon Coating

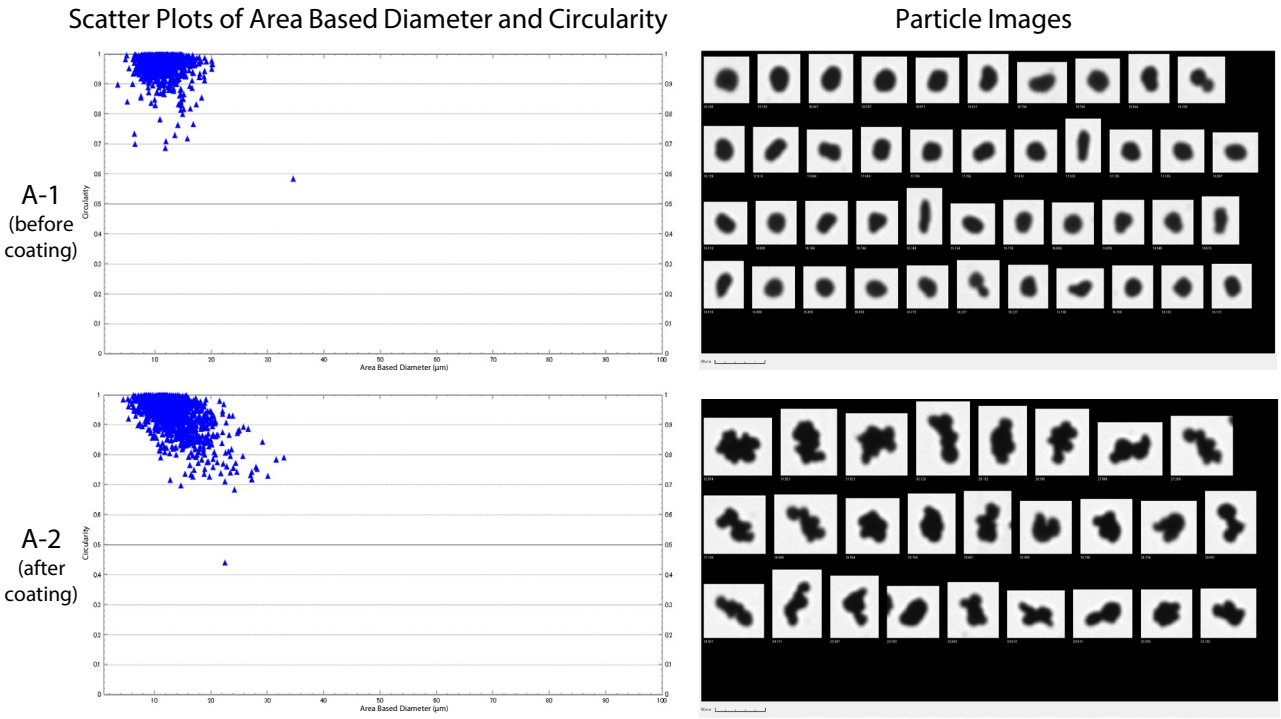

Fig. 6 iSpect DIA-10 Particle Shape Evaluation of Samples A-1 and A-2
Left: Scatter Plots of Area Based Diameter and Circularity; Right: Particle Image Examples (In Descending Order of Area Based Diameter)

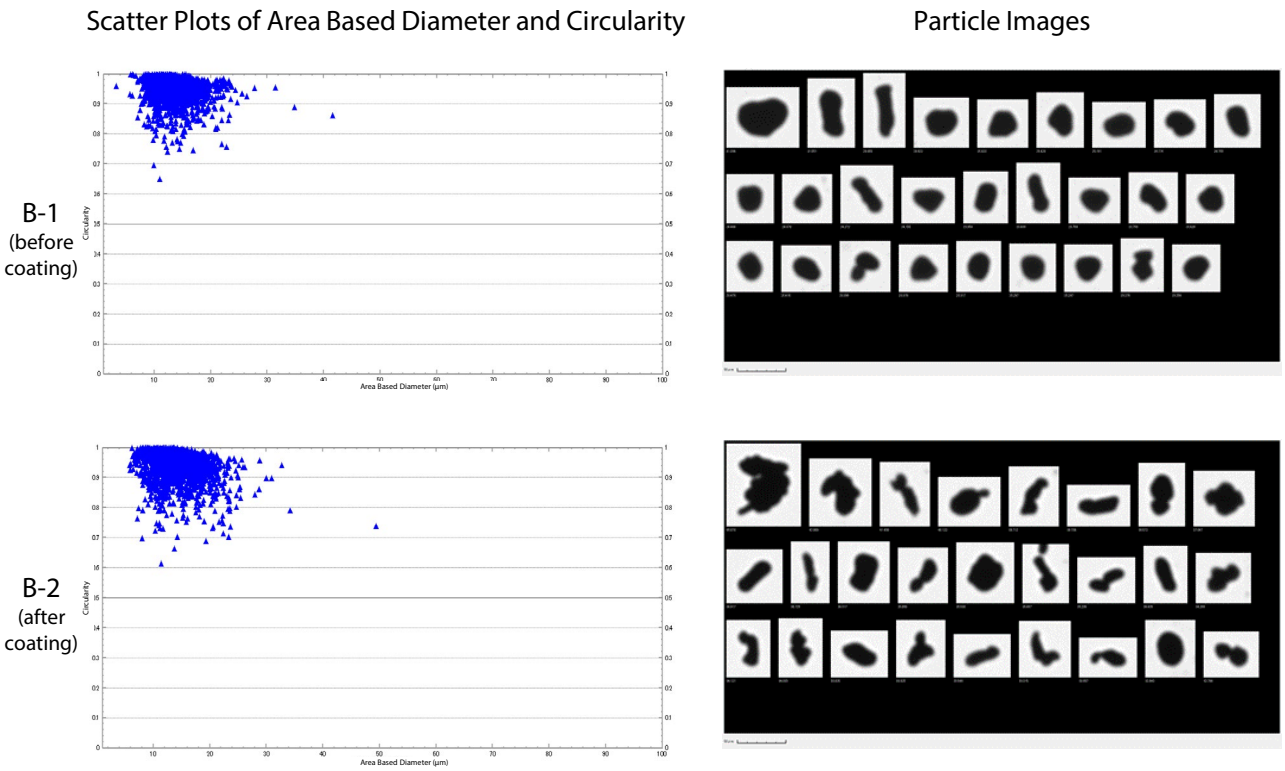

Fig. 7 iSpect DIA-10 Particle Shape Evaluation of Samples B-1 and B-2
Left: Scatter Plots of Area Based Diameter and Circularity; Right: Particle Image Examples (In Descending Order of Area Based Diameter)

■ Conclusion

The particle diameter and circularity results in this article are summarized in Table 6. In this report, we were able to measure the particle size distribution and the particle shape properties of the negative electrode material using the SALD-2300 and the iSpect DIA-10, respectively. In addition, we were able to confirm changes in the particle size distribution and particle shapes due to the coating process.

In the iSpect DIA-10 measurement, we were able to capture shape information that couldn't be understood with the SALD-2300. On the other hand, in the SALD-2300 measurement, we benefited from a wide range of particle size measurements that wasn't possible with the iSpect DIA-10. By conducting these two measurements, we were able to complement the information that couldn't be understood with one method alone, making a comprehensive evaluation possible. In the anode and cathode material, the particle size and shape significantly contribute to the performance of a battery, such as output and lifespan. By conducting a comprehensive evaluation of powder properties using methods such as SALD and DIA, which we introduced in this report, it can be helpful to the further maintenance and improvement of battery material quality.

Acknowledgments
We are deeply grateful to Dainen Material Co., Ltd. for providing samples.

Table 6 Anode Material Measurement Results

Sample	A-1 (DG10-095)	A-2 (DG-B10M1)	B-1 (DG15-097)	B-2 (DG-B15M1)	C-1 (DG22-098)
Median Diameter (µm)	10.174	12.307	16.035	16.122	22.187
Circularity (Mean Value)	0.972	0.952	0.952	0.944	0.927

The median diameter results were measured with the SALD-2300 and the circularity with the iSpect DIA-10.

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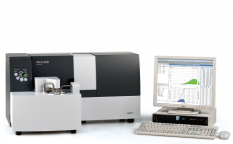

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