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MCT™-510 Micro Compression Testing Machine

Application News

Compression Tests for Anode Material for Lithium-Ion Batteries

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User Benefits

- The deformation strength can be calculated by performing compression tests on anode material one particle at a time.
- This provides a useful method for correctly analyzing the physical properties of particulate material, without being affected by the experience of the analyst.
- This provides an index for evaluation of strength, for development of new anode materials.

■ Introduction

Lithium-ion batteries are a type of rechargeable battery that is charged or discharged by desorption or insertion of lithium ions (Li^+) from or into the active material structure. In recent years, the uses of lithium-ion batteries have expanded dramatically, and research to increase their capacity, extend their life, reduce their cost, and increase their safety is being actively undertaken. The main materials of lithium-ion batteries are the cathode, anode, separator, and electrolytic solution, with the active material, the main material from which the electrodes are composed, being the most important element for improving performance.

In recent years the use of silicon and metal oxides as anode materials has been investigated. Silicon anodes have a higher theoretical capacity than conventional carbon anode materials, so they are expected to contribute to higher capacity lithium-ion batteries. On the other hand, charging and discharging can easily be accompanied by volumetric expansion and contraction, so there is the issue that aging of the battery itself is faster compared with carbon anode materials.

Hence evaluation of the properties of new materials is required in order to improve the performance of anode materials. In this article a case of measurement of deformation strength[*1] in compression tests is introduced, as an example of quantitative evaluation of physical properties. The MCT-510 micro compression testing machine is used for compression tests on minute samples and can be applied to flexible anode materials. The deformation strength is quantitatively evaluated by high-accuracy measurement of both displacement and force.

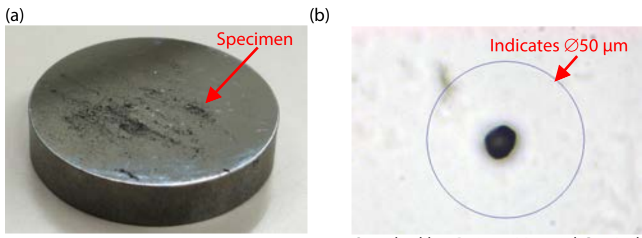
*1 Deformation strength
The strength as defined by JIS Z 8844 (Test method of fracture and deformation strength of a fine particle), and is obtained by calculation from Equation (1).

■ Test Specimens

Table 1 shows information regarding the test specimens used in this test, and Fig. 1 shows an image of a test specimen. When anode material is dispersed on the test specimen stand with a spoon, each particle can be observed using the instrument's microscope, as shown in Fig. 1(b).

Table 1 Test Specimen Information

Specimen	:	Three types of anode material (anode material a, anode material b, anode material c)
Material	:	Carbon (graphite)
Particle diameter	:	10-15 µm



Supplied by: Dainen Material Co., Ltd.

Fig. 1 Test Specimen

(a) On the lower compression plate
(b) Particle observed with a 400 fold microscope

■ Test Conditions

Fig. 2 shows a view of the instrument and a schematic diagram of a test, and Table 2 shows the test conditions. With the MCT-510 it is possible to perform a compression test on single particles, and use the Side Observation Kit to view the compression on video.

The anode material used in this case was graphite, so it was soft, and with this sample it was difficult to correctly determine the start of the compression test when the particle was contacted. To resolve this problem, the soft material measurement mode[*2] was used. The following equation shows the calculation of deformation strength.

$$\sigma_{10\%} = \frac{F_{10\%}}{A} \quad \cdots (1)$$

$$A = \frac{\pi d^2}{4}$$

$\sigma_{10\%}$: Deformation strength for a 10 % compression displacement of particle size [Pa]
$F_{10\%}$: Force for a 10 % compression displacement of particle size [N]
A : Representative area [m^2]
(Area equivalent to a circle, found from the particle size measured before compression)
d : Particle size [m]

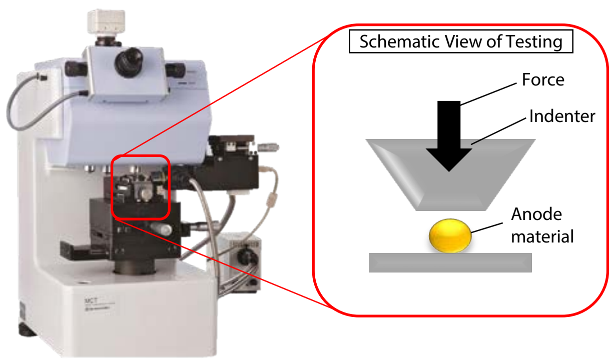


Fig. 2 Test Apparatus and Schematic View of a Test

Table 2 Test Conditions

Testing machine	MCT-510 Micro Compression Testing Machine, Side Observation Kit, and Length Measurement Kit
Flat indenter (μm)	∅50
Test mode	Soft material measurement mode
Test force (mN)	10
Loading speed (mN/sec)	0.2231
Number of measurements (times)	10

*2 Soft material measurement mode
In the standard measurement mode, the point at which the indenter contacts the sample is automatically recognized, so the compression test is started and measurements are taken. However with soft samples it is difficult to correctly detect the point at which the sample is contacted. In the soft material measurement mode, all the displacement - force data is collected from before contact with the test sample until a set force is reached. Therefore even when the point at which the compression has started is unclear, the displacement - force data during the test can be properly acquired.

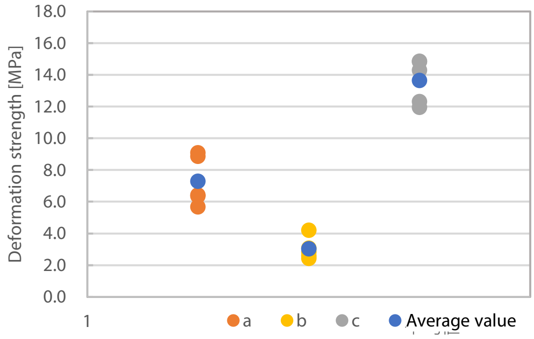


Fig. 3 Test Results (Deformation Strength at Each of the 5 Points)

■ Results

Table 3 shows the test results (average values), Fig. 3 shows the test results (deformation strength at 5 points), and Fig. 4 shows the force - displacement graphs. In these tests more spherical particles were selected, and after taking measurements on each 10 particles for each material, the 5 points nearest to the central value were selected. No clear failure point could be detected from Fig. 4, so the deformation strength σ10% was calculated from the force when the deformation was 10 % of the particle diameter, and used as a reference for comparison. The order of the value of deformation strength of each material was as follows, and clear differences were found between each test sample.

Anode material c > Anode material a > Anode material b

The surface condition of each test sample was different; anode material c was a carbon coating type, and anode material b was uncovered, so differences were observed in the deformation strengths, and each test sample had its own characteristics.

Fig. 5 shows anode material a during the test, and the corresponding force - displacement graph. When the Side Observation Kit is used it is possible to relate the movement at each point shown on the force - displacement graph to video taken during the compression test. Each photo is an image extracted from the video taken using the Side Observation Kit, and the red points on the force - displacement graphs indicate the force and displacement on each photo. The photos from (1) to (4) show the compression of the particle in time series sequence, from which it can be seen that at about 0.84 to 3.63 N the particle deformed significantly.

Table 3 Test Results (Average Values)

Sample	Anode material a	Anode material b	Anode material c
Average diameter [μm]	13.32	12.54	13.11
Deformation strength σ10% [MPa]	7.29	3.04	13.66

■ Conclusion

This article introduced an example of quantitative evaluation of anode material using deformation strength, which is one of the mechanical evaluations. There is a method of performing compression tests on powder particles in which a certain quantity of powder is placed in a container and overall compression is applied. However in this method compression is applied to each individual particle, so the differences in strength between particles can be clearly seen. In this case compression tests were conducted using the MCT-510, so differences in strength due to the anode material could be obtained in units of one particle, so it is expected that in the future this will be applied for improving the quality of anode materials. The MCT-510 micro compression testing machine is useful for evaluation of not only minute particles of anode material, but also of cathode material, etc.

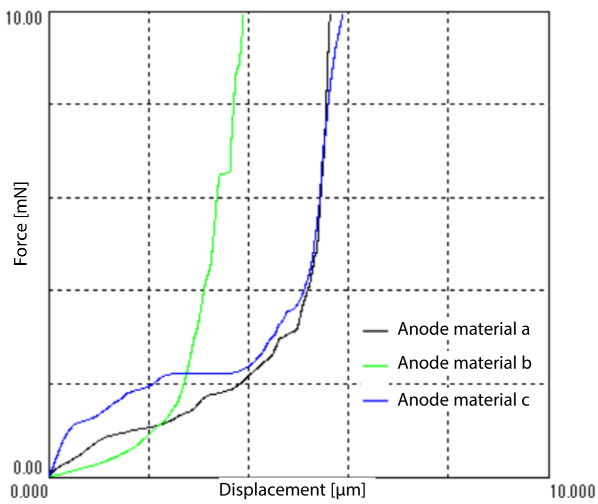


Fig. 4 Force - Displacement Graph

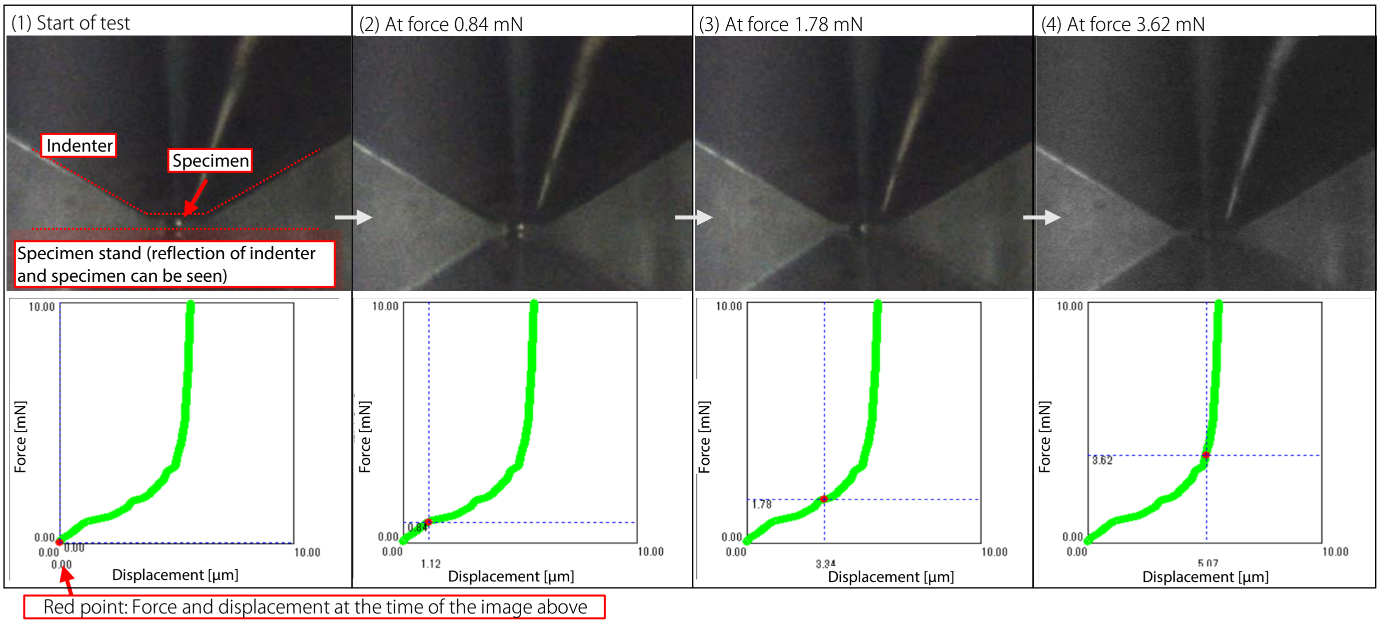


Fig. 5 Anode Material a during Test, and Corresponding Force - Displacement Graph

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