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Electrochemical Performance Test of Lithium Nickel Manganese Oxide - Test Method for the Initial Discharge Specific Capacity and Initial Efficiency

镍锰酸锂电化学性能测试

首次放电比容量及首次充放电效率测试方法

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Electrochemical Performance Test of Lithium Nickel Manganese Oxide - Test Method for the Initial Discharge Specific Capacity and Initial Efficiency

1 Scope

This document describes the test method for the initial discharge specific capacity and initial charge and discharge efficiency of lithium nickel manganese oxide - lithium-ion battery positive electrode material.

This document is applicable to the test of the initial discharge specific capacity and initial charge and discharge efficiency of lithium nickel manganese oxide - lithium-ion battery positive electrode material. The test method includes button-type half-cell method and button-type full-cell method.

2 Normative References

This document does not have normative references.

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Reagents and Materials

4.1 Lithium nickel manganese oxide: spinel type.

4.2 Artificial graphite negative electrode material.

4.3 Lithium-ion battery electrolyte: high-voltage resistant type, with a moisture content not greater than 0.002%, free acid (HF) not greater than 0.005%, and electrical conductivity (25 °C) not less than 7.0 mS/cm.

4.4 Ethanol: industrial grade.

4.5 Binder: polyvinylidene fluoride (PVDF), battery grade, with a weight-average molecular weight not less than 5×10^5 and moisture content not greater than 0.10%; sodium carboxymethylcellulose (CMC), battery grade, with a viscosity of 500 mPa • s ~ 1,200 mPa • s; styrene-butadiene rubber (SBR) emulsion.

4.6 *N*-methylpyrrolidone (NMP): battery grade, with a purity not less than 99.9% and a moisture

5.11 Inert atmosphere (argon) glove box: with a moisture content and oxygen content not greater than 0.0001%.

5.12 Button-type battery packaging machine.

5.13 Lithium-ion battery electrochemical performance tester: with a current and voltage full-scale accuracy of 0.1%.

5.14 Thermostat: with a temperature of 25 °C ± 1 °C and a relative humidity less than 40%.

5.15 Pre-treatment drying room: with a temperature of 25 °C ± 2 °C and a relative humidity less than 5%.

5.16 Desktop digital display thickness gauge: with a resolution of 1 μm.

5.17 Myriameter.

5.18 Tweezers.

5.19 Insulated tweezers.

6 Test Procedures

6.1 Button-type Half-cell Method

6.1.1 Pre-treatment

6.1.1.1 Lithium nickel manganese oxide (4.1), polyvinylidene fluoride (PVDF) (4.5), conductive agent (4.7): put in the oven (5.1), at 85 °C ~ 120 °C, bake for 4 h ~ 20 h. After cooling to room temperature, place it in the desiccator (5.3).

6.1.1.2 Lithium-ion battery separator (4.12): place it in the oven (5.1), at 50 °C ~ 70 °C, bake for 4 h. Take it out and transfer to the inert atmosphere (argon) glove box (5.11).

6.1.1.3 Standard battery structural parts (4.13): use ethanol (4.4) to ultrasonically clean the standard battery structural parts for 1 ~ 3 times, 30 min each time. After the operation is completed, take out the standard battery structural parts and place them in the oven (5.1); at 85 °C, bake for above 12 h, then, transfer to the inert atmosphere (argon) glove box (5.11).

6.1.2 Preparation of positive electrode sheet

6.1.2.1 Test conditions

The operation shall be carried out in the pre-treatment drying room (5.15), and the instruments and equipment used shall be clean.

6.1.2.2 Weighing

Weigh-take the pre-treated lithium nickel manganese oxide, polyvinylidene fluoride (PVDF) and conductive agent in 6.1.1.1, with a total amount of 4.0 g ~ 25.0 g. The mass fractions of the three are respectively: (90% ~ 96%), (2% ~ 5%) and (2% ~ 5%); use the electronic balance (5.4) for weighing; the amount of *N*-methylpyrrolidone (NMP) (4.6) is 30% ~ 75% of solid content; use the electronic balance (5.4) for weighing.

NOTE: the solid content is the ratio of the mass of the positive electrode active material lithium nickel manganese oxide, polyvinylidene fluoride (PVDF) and conductive agent to the mass of positive electrode slurry.

6.1.2.3 Slurrying

Add the weighed *N*-methylpyrrolidone (NMP) and polyvinylidene fluoride (PVDF) into the beaker, place the beaker under the dispersing mixer (5.6), disperse and stir, until the polyvinylidene fluoride (PVDF) is completed dissolved. Thus, prepare it into a colorless and transparent glue solution. Add the weighed conductive agent to the above-mentioned colorless and transparent glue solution, mix and stir it. Slowly add the weighed lithium nickel manganese oxide, evenly disperse and stir it, so that the various materials are evenly mixed.

6.1.2.4 Coating

Use the flat coating machine (5.7) to evenly coat the mixed positive electrode slurry on one side of the aluminum foil (4.9). Put the coated positive electrode sheet into the oven (5.1), at 110 °C ± 5 °C, bake it for 2 h ~ 3 h.

6.1.2.5 Production of positive electrode sheet

Take the positive electrode sheet that has been dried and reached the requirements for processability in 6.1.2.4, use the sheet-punching machine (5.9) to punch out the positive electrode sheet with a diameter (D) of 12.0 mm ~ 15.0 mm. Use the electronic balance (5.5) to weight the mass m_c of the positive electrode sheet; use the desktop digital display thickness gauge (5.16) or the myriameter (5.17) to measure the thickness d_c of the positive electrode sheet. Use the sheet-punching machine (5.9) to punch out the aluminum foil substrate with the same diameter as the positive electrode sheet. Use the electronic balance (5.5) to weigh the mass m_{Al} of the aluminum foil substrate; use the desktop digital display thickness gauge (5.16) or the myriameter (5.17) to measure the thickness d_{Al} of the aluminum foil substrate.

The compacted density of the positive electrode sheet shall be calculated in accordance with Formula (1):

$$\rho_c = \frac{(m_c - m_{Al}) \times 10^4}{\pi \left(\frac{D}{2}\right)^2 \times (d_c - d_{Al})} \quad \cdots\cdots (1)$$

Where,

ρ_c---the compacted density of the positive electrode sheet, expressed in (g/cm^3);

gasket) and place it in the center of the negative electrode case;

c) Use the tweezers (5.18) to pick up the metal lithium sheet (4.11) and place it on the foam nickel sheet (or spring support sheet and gasket); maintain the metal lithium sheet (4.11) center-aligned with the foam nickel sheet (or spring support sheet and gasket);

d) Use the pipette (5.10) to dropwise add 20 μL ~ 200 μL of lithium-ion battery electrolyte (4.3) onto the surface of the metal lithium sheet (4.11);

e) Use the tweezers (5.18) to place a piece of lithium-ion battery separator (4.12) in the center of the negative electrode case, so that it completely covers the metal lithium sheet (4.11);

f) Use the pipette (5.10) to dropwise add 20 μL ~ 200 μL of lithium-ion battery electrolyte (4.3) onto the surface of the separator;

g) Use the tweezers (5.18) to place the positive electrode sheet (see 6.1.2.5) in the center of the lithium-ion battery separator (4.12), with the aluminum foil side facing up;

h) Use the insulated tweezers (5.19) to pick up the positive electrode case over the negative electrode case, and manually press and fasten it;

i) Place the negative electrode case facing up, transfer it onto the button-type battery packing machine (5.12), and crimp it for packaging. The packaging pressure is 35 kg/cm^2 ~ 45 kg/cm^2;

j) Use the dust-free paper (4.14) to wipe the electrolyte leaking outside the button-type cell case;

k) Number the assembled cells one by one and keep records.

6.1.4 Battery test

Put the prepared test battery into the thermostat (5.14), let it stand for 2 h ~ 12 h, then, adopt the lithium-ion battery electrochemical performance tester (5.13) to conduct the test. The recommended charge and discharge system is as follows:

a) Charge cut-off voltage: constant-current charge to 4.95 V, and the constant-voltage charging cut-off current is 0.05 C;

b) End of discharge voltage: constant-current discharge to 3.0 V;

c) Constant-current charging and discharging current: 0.1 C.

NOTE 1: when adopting other charge and discharge systems, it shall be determined by the demand-side and the supply-side through negotiation.

NOTE 2: the current t C represents the magnification number of completion of charging or discharging $\frac{1}{t}$ h.

6.2 Button-type Full-cell Method

6.2.1 Pre-treatment

6.2.1.1 Lithium nickel manganese oxide (4.1), artificial graphite negative electrode material (4.2), conductive agent (4.7): put in the oven (5.1), at 85 °C ~ 120 °C, bake for 4 h ~ 20 h. After cooling to room temperature, place it in the desiccator (5.3).

6.2.1.2 Polyvinylidene fluoride (PVDF) (4.5), sodium carboxymethylcellulose (CMC) (4.5): place it in the oven (5.1), at 70 °C ~ 90 °C, bake for 4 h ~ 6 h. After cooling to room temperature, place it in the desiccator (5.3).

6.2.1.3 Lithium-ion battery separator (4.12): place it in the oven (5.1), at 50 °C ~ 70 °C, bake for 4 h. Take it out, then, transfer to the inert atmosphere (argon) glove box (5.11).

6.2.1.4 Standard battery structural parts (4.13): use ethanol (4.4) to ultrasonically clean the standard battery structural parts for 1 ~ 3 times, 30 min each time. After the operation is completed, take out the standard battery structural parts and place them in the oven (5.1); at 85 °C, bake for above 12 h, then, transfer to the inert atmosphere (argon) glove box (5.11).

6.2.2 Preparation of positive electrode sheet

The preparation of the positive electrode sheet shall be carried out in accordance with the stipulations of 6.1.2. The surface density of the positive electrode sheet shall be calculated in accordance with Formula (3):

$$\sigma_c = \frac{m_c - m_{Al}}{\pi\left(\frac{D}{2}\right)^2} \qquad \cdots\cdots (3)$$

Where,

σ_c---the surface density of the positive electrode sheet, expressed in (g/cm^2);

m_c---the mass of the positive electrode sheet, expressed in (g);

m_{Al}---the mass of the aluminum foil substrate, expressed in (g);

D---the diameter of the positive electrode sheet, expressed in (cm).

6.2.3 Preparation of negative electrode sheet

6.2.3.1 Weighing

The ratio (ε) of positive and negative electrode capacities per unit area (hereinafter referred to and graphite negative electrode material, then, add the above-mentioned colorless and transparent glue solution. In accordance with the designed solid content, add deionized water, disperse and stir for 1.5 h ~ 2.0 h. Finally, dropwise add the weighed styrene-butadiene rubber (SBR) emulsion, and stir it at a low speed for 20 min ~ 30 min, so as to evenly mix the various materials.

6.2.3.3 Coating

Use the flat coating machine (5.7) to evenly coat the mixed negative electrode slurry on one side of the copper foil (4.10). Put the coated negative electrode sheet into the oven (5.1), at 70 °C ~ 90 °C, bake it for 2 h ~ 4 h.

6.2.3.4 Production of negative electrode sheet

Take the negative electrode sheet that has been dried and reached the requirements for processability in 6.2.3.3, use the sheet-punching machine (5.9) to punch out the negative electrode sheet with a diameter larger than that of the positive electrode sheet. Use the electronic balance (5.5) to weight the mass m_a of the negative electrode sheet; use the desktop digital display thickness gauge (5.16) or the myriameter (5.17) to measure the thickness d_a of the negative electrode sheet. Use the sheet-punching machine (5.9) to punch out the copper foil substrate with the same diameter as the negative electrode sheet. Use the electronic balance (5.5) to weigh the mass m_{Cu} of the copper foil substrate; use the desktop digital display thickness gauge (5.16) or the myriameter (5.17) to measure the thickness d_{Cu} of the copper foil substrate.

The compacted density of the negative electrode sheet shall be calculated in accordance with Formula (6):

$$\rho_a = \frac{(m_a - m_{Cu}) \times 10^4}{\pi \left(\frac{D_0}{2}\right)^2 \times (d_a - d_{Cu})} \qquad \cdots\cdots (6)$$

Where,

ρ_a---the compacted density of the negative electrode sheet, expressed in (g/cm^3);

m_a---the mass of the negative electrode sheet, expressed in (g);

m_{Cu}---the mass of the copper foil substrate, expressed in (g);

D_0---the diameter of the negative electrode sheet, expressed in (cm);

d_a---the thickness of the negative electrode sheet, expressed in (μm);

d_{Cu}---the thickness of the copper foil substrate, expressed in (μm).

Design in accordance with the compacted density of the negative electrode sheet 1.4 g/cm^3 ~ 1.7 g/cm^3, and calculate the theoretical thickness of each negative electrode sheet. Use the roller machine (5.8) to roll the dried negative electrode sheet (see 6.2.3.3) to the target thickness. Use the sheet-punching machine (5.9) to punch out a sufficient number of negative electrode sheets with a diameter larger than that of the positive electrode sheet. Place the negative electrode sheet into the vacuum oven (5.2), at 105 °C ± 5 °C, bake it for above 4 h.

6.2.4 Cell assembly

Cell assembly shall be performed in the inert atmosphere (argon) glove box (5.11). The stacking sequence of assembled button-type full-cell is from bottom to top, successively: negative electrode case, spring support sheet, gasket, negative electrode sheet, separator, positive electrode sheet and positive electrode case. The cell assembly may take the following steps as a reference:

a) Take the standard battery structural parts (4.13), with the negative electrode case opening facing up, and flatly place it on the horizontal table;

b) Use the tweezers (5.18) to pick up the spring support sheet and place it on the negative electrode case. The large opening of the spring support sheet faces upward and is flatly placed in the center of the negative electrode case;

c) Use the tweezers (5.18) to pic up the gasket and place it on the spring support sheet, with the burr side facing down;

d) Use the tweezers (5.18) to pick up the negative electrode sheet (see 6.2.3.4) and place it in the center of the gasket, with the copper foil side facing down; maintain the negative electrode sheet, the gasket and the spring support sheet center-aligned;

e) Use the pipette (5.10) to dropwise add 50 μL ~ 200 μL of lithium-ion battery electrolyte (4.3) onto the surface of the negative electrode sheet;

f) Use the tweezers (5.18) to place a piece of lithium-ion battery separator (4.12) in the center of the negative electrode case, so that it completely covers the negative electrode sheet and is centered;

g) Use the pipette (5.10) to dropwise add 50 μL ~ 200 μL of lithium-ion battery electrolyte (4.3) onto the surface of the separator;

h) Use the tweezers (5.18) to place the positive electrode sheet in the center of the separator, with the aluminum foil side facing up;

i) Use the insulated tweezers (5.19) to pick up the positive electrode case over the negative electrode case, and manually press and fasten it;

j) Place the negative electrode case facing up, transfer it onto the button-type battery packing machine (5.12), and crimp it for packaging. The packaging pressure is 35 kg/cm^2 ~ 45 kg/cm^2;

k) Use the dust-free paper (4.14) to wipe the electrolyte leaking outside the button-type cell case;

l) Number the assembled cells one by one and keep records.

6.2.5 Battery formation and test

The test process of the test battery shall be carried out in the thermostat (5.14). Adopt the lithium-ion battery electrochemical performance tester (5.13) to carry out the test. The recommended battery formation and capacity setting procedures are as follows:

a) Let it stand for not less than 3 h;

b) Constant-current charge: charge to 3.4 V with a current of 0.02 C;

c) Constant-current charge: charge to 3.75 V with a current of 0.1 C;

d) Constant-current and constant-voltage charge: charge to 4.8 V with a current of 0.333 C, then, switch to constant-voltage charging, with a cut-off current of 0.05 C;

e) Let it stand for not less than 10 min;

f) Constant-current discharge: discharge to 3.0 V with a current of 0.333 C;

g) Let it stand for not less than 10 min;

h) Constant-current discharge: discharge to 3.0 V with a current of 0.05 C.

NOTE: when adopting other charge and discharge systems, it shall be determined by the demand-side and the supply-side through negotiation.

7 Test Data Processing

7.1 Initial Discharge Specific Capacity

The initial discharge specific capacity of lithium nickel manganese oxide shall be calculated in accordance with Formula (7):

$$C_{\mathrm{m}} = \frac{Q_{\mathrm{ID}}}{m} \quad \cdots\cdots (7)$$

Where,

C_{m}---the initial discharge specific capacity, expressed in (mA • h/g);

Q_{ID}---the initial discharge capacity, expressed in (mA • h);

m---the mass of the active material lithium nickel manganese oxide in the positive electrode formula, expressed in (g).

The calculation results shall retain one decimal place.

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