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

钴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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Foreword

This document was drafted in accordance with the rules of GB/T 1.1-2020 *Directives for Standardization - Part 1: Rules for the Structure and Drafting of Standardizing Documents*.

This document serves as a replacement of GB/T 23365-2009 *Electrochemical Performance Test of Lithium Cobalt Oxide - Test Method for Specific Capacity and Charge-discharge Efficiency of the First Cycle*. In comparison with GB/T 23365-2009, apart from structural adjustments and editorial modifications, the main technical changes are as follows:

a) The scope of application is modified (see Chapter 1; Chapter 1 of Version 2009);

b) The requirements for “binder”, “lithium ion battery separator”, “aluminum foil”, “metal lithium sheet” and “electrolyte for lithium ion battery” are modified (see 5.3, 5.4, 5.7, 5.9 and 5.10; 2.5, 2.9, 2.7, 2.8 and 2.1 of Version 2009);

c) The requirements for “vacuum oven”, “dryer”, “oven”, “electronic balance”, “thickness gauge”, “roller press”, “insulated tweezers”, “pipette”, “button cell packaging machine” and “thermostatic box” are added (see 6.1, 6.2, 6.7, 6.9 ~ 6.15); the requirements for “inert atmosphere (argon) glove box” are modified (see 6.3; 3.4 of Version 2009);

d) The requirements for “reagent or material pre-treatment” and “data recording” are added (see 7.1 and 7.5);

e) The requirements for “cathode sheet preparation”, “battery assembly” and “battery testing” are modified (see 7.2 ~ 7.4; 4.1 ~ 4.3 of Version 2009);

f) The requirement for the precision of calculation results is added “the calculation results shall retain one decimal place” (see 8.1 and 8.2);

g) “allowable difference” is added (see Chapter 9).

Please be noted that certain content of this document may involve patents. The institution issuing this document does not undertake the responsibility of identifying these patents.

This document was proposed by China Non-ferrous Metals Industry Association.

This document shall be under the jurisdiction of National Technical Committee 243 on Nonferrous Metals of Standardization Administration of China (SAC/TC 243)

The drafting organizations of this document: Tianjin Guoan MGL New Materials Co., Ltd.; Beijing Easpring Material Technology Co., Ltd.; Chengdu B&M Science and Technology Co., Ltd.; Hunan Changyuan LICO Co., Ltd.; Guangdong BRUNP Recycling Technology Co., Ltd.; XTC New Energy Materials (Xiamen) Co., Ltd.; Jiangmen Keheng Industry Co., Ltd.; BASF Shanshan Battery Materials Co., Ltd.; Amperex Technology Limited; Tianjin Lishen Battery Joint-Stock Co., Ltd.; Hunan Zhongwei New Energy Technology Co., Ltd.; Gem (Wuxi)

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

1 Scope

This document describes the test method for the initial discharge specific capacity and initial charge and discharge efficiency of lithium cobalt oxide, which is a lithium ion battery cathode material.

This document is applicable to the test of the initial discharge specific capacity and initial charge and discharge efficiency of lithium cobalt oxide, which is a lithium ion battery cathode material. The test of the initial discharge specific capacity and initial charge and discharge efficiency of other lithium ion battery cathode materials may take this document as a reference.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in this text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

3 Terms and Definitions

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

4 Test Conditions

The test procedures specified in this document, unless otherwise specified, should be carried out in a drying room (the ambient dew point temperature is not greater than –20 °C); for the circumstance where there are no conditions for such drying room, the various test procedures shall be carried out under environmental conditions with a relative humidity not greater than 40% and a temperature of 20 °C ~ 30 °C.

5 Reagents or Materials

5.1 Cathode active material: lithium cobalt oxide.

5.2 Conductive agent: acetylene black or conductive carbon black.

5.3 Binder: polyvinylidene fluoride (PVDF), battery grade, with a relative molecular mass not less than 5×10^5, and a moisture content (mass fraction) not greater than 0.10%.

5.4 Lithium ion battery separator: polyolefin porous membrane, with a porosity of 35.0% ~ 60.0%, an average pore diameter not greater than 1.0 μm, a diameter of 16 mm ~ 22 mm, and a thickness of 9.0 μm ~ 25.0 μm.

5.5 Standard structural members of button cells: commonly used models CR2016, CR2025, CR2032 or CR2430, including cathode case, anode case, gasket, spring support sheet or foam nickel sheet.

5.6 Absolute ethanol: industrial grade.

5.7 Aluminum foil: battery grade, with a thickness of 10 μm ~ 20 μm.

5.8 N-methylpyrrolidone (NMP): battery grade, with a purity not less than 99.9% and a moisture content (mass fraction) not greater than 0.02%.

5.9 Metal lithium sheet: with a diameter of 14 mm ~ 18 mm and a thickness of 0.40 mm ~ 0.80 mm.

5.10 Electrolyte for lithium ion battery: made from battery-grade lithium hexafluorophosphate ($LiPF_6$) dissolved in an organic solvent, with a moisture content (mass fraction) not greater than 0.002%, and free acid (HF) content (mass fraction) not greater than 0.005%.

6 Instruments and Equipment

6.1 Vacuum oven: with a temperature control accuracy of ± 1.0 °C.

6.2 Desiccator: contain appropriate desiccant (such as: color-changing silica gel and phosphorus pentoxide, etc.).

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

6.4 Electronic balance: the division value of display is 0.0001 g.

6.5 Dispersing mixer.

6.6 Small coating machine or film applicator.

6.7 Oven: with a temperature control accuracy of ± 1.0 °C.

6.8 Sheet-punching machine.

6.9 Electronic balance: the division value of display is 0.00001 g.

6.10 Thickness gauge: with a resolution of 1 μm.

6.11 Roller press: for button lithium cells only.

6.12 Insulated tweezers.

6.13 Pipette: 1 mL.

6.14 Button cell packaging machine.

6.15 Thermostatic box: with a temperature control accuracy of ± 1.0 °C.

6.16 Lithium ion battery electrochemical performance tester: the voltage and current accuracy is not less than 0.1% of the measuring range.

7 Test Procedures

7.1 Reagent or Material Pre-treatment

7.1.1 Cathode active material (5.1), conductive agent (5.2) and binder (5.3): place in a vacuum oven (6.1), at 85 °C ~ 120 °C, bake it for 4 h ~ 20 h, then, place in a desiccator (6.2) to cool to room temperature.

7.1.2 Place the lithium ion battery separator (5.4) into a vacuum oven (6.1), at 60 °C ~ 90 °C, bake it for 4 h ~ 8 h. After taking it out, transfer it to an inert atmosphere (argon) glove box (6.3) for storage.

7.1.3 Use absolute ethanol (5.6) to ultrasonically clean the standard structural members of button cells (5.5) for 3 times, 30 min each time. After the operation is completed, take out the standard structural members of button cells (5.5) and place them in a vacuum oven (6.1) to bake for 15 h at 90 °C. Then, transfer them to an inert atmosphere (argon) glove box (6.3) for storage.

7.1.4 Use absolute ethanol (5.6) to wipe and clean the aluminum foil (5.7).

7.2 Cathode Sheet Preparation

7.2.1 Weighing

Weigh-take the total amount of cathode active material, conductive agent and binder pretreated in 7.1.1 as 5.0 g ~ 30.0 g. The mass fraction ratio of the three is 90% ~ 98% : 1% ~ 5% : 1% ~ 5%; use an electronic balance (6.4) to weigh it. The mass of NMP (5.8) is 50% ~ 80% of the solid content; use an electronic balance (6.4) to weigh it.

Take the rolled-pressed sheet in 7.2.4, use the sheet-punching machine (6.8) to punch out a cathode sheet with a diameter not greater than the lithium ion battery separator (7.1.2); place it in a vacuum oven (6.1), at 80 °C ~ 120 °C, bake it for 8 h ~ 12 h, and remove traces of residual solvent. After baking is completed, take out the cathode sheet, and use an electronic balance (6.9) to measure the mass of the cathode sheet, and record it as m_c.

The mass of the active material lithium cobalt oxide in the cathode sheet is calculated in accordance with Formula (2):

$$m = (m_c - m_{Al}) \times w \qquad \cdots\cdots (2)$$

Where,

m---the mass of the active material lithium cobalt oxide in the cathode sheet, expressed in (g);

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

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

w---the mass fraction of the active material lithium cobalt oxide in the cathode sheet.

Calculate the mass m of the active material lithium cobalt oxide in the cathode sheet, number and record it. Then, transfer the cathode sheet to an inert atmosphere (argon) glove box (6.3) for storage and save it for later use.

NOTE: the mass fraction of the active material lithium cobalt oxide in the cathode sheet in this document is the ratio of the active material lithium cobalt oxide to the sum of the active material lithium cobalt oxide, conductive agent and binder.

7.3 Assembly of Test Battery

The assembly of test battery shall be carried out in an inert atmosphere (argon) glove box (6.3). The assembly process may take the following steps as a reference:

a) The opening of the anode case faces upward and is flatly placed on the horizontal platform;

b) Use insulated tweezers (6.12) to clamp the metal lithium sheet (5.9) and place it in the anode case. It is in plane contact with the anode case and lies flat in the center of the anode case;

c) Use insulated tweezers (6.12) to clamp the lithium ion battery separator (see 7.1.2), so that it completely covers the metal lithium sheet and is centered;

d) Use a pipette (6.13) to take 50 μL ~ 200 μL of the electrolyte for lithium ion battery (5.10) and inject it into the anode case;

e) Use insulated tweezers (6.12) to clamp the cathode sheet prepared in 7.2.5 and place

it in the middle of the lithium ion battery separator (see 7.1.2);

f) Use a pipette (6.13) to take 50 μL ~ 200 μL of the electrolyte for lithium ion battery (5.10) and inject it into the anode case containing gasket, spring support sheet or foam nickel sheet, cathode sheet, lithium ion battery separator and metal lithium sheet;

g) Use insulated tweezers (6.12) to successively pick up the gasket, spring support sheet or foam nickel sheet, and place them on the cathode sheet; make sure that the gasket, spring support sheet and cathode sheet are centered and aligned;

h) Use insulated tweezers (6.12) to pick up the cathode case and place it on the anode case;

i) Carry out a translational movement to the button cell packaging machine (6.14), press and seal it;

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

k) Number the assembled test batteries one by one and make records.

The above-mentioned steps a) ~ h) can also be reversed. Place the cathode case first, then, respectively place the cathode sheet, lithium ion battery separator, metal lithium sheet, gasket and anode case.

7.4 Test of Test Battery

Put the prepared test battery into the thermostatic box (6.15) and control the temperature at 25 °C ± 1 °C. After leaving it for 4 h ~ 12 h, adopt the lithium ion battery electrochemical performance tester (6.16) to perform the test. The following process may be taken as a reference for the charge and discharge test:

a) Perform constant-current charge, until reaching the limited charge voltage (see Table 1 for battery test charging and discharging parameters);

b) Perform constant-voltage charge, until reaching the cut-off current of 0.01 C ~ 0.05 C;

NOTE: C is the 1 h discharge rate rated capacity of the battery, 0.01 C is the current value corresponding to the 100 h discharge rate, and 0.05 C is the current value corresponding to the 20 h discharge rate.

c) Leave it aside for 5 min ~ 10 min;

d) Perform constant-current discharge, until reaching the discharge cut-off voltage (see Table 1 for battery test charging and discharging parameters).

The constant-current charging and discharging current is 0.1 C (0.1 C is the current value corresponding to the 10 h discharge rate), and its value can be calculated with reference to

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