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**Electrochemical performance test of lithium iron phosphate
- Test method for the initial discharge specific capacity and
the initial efficiency**

磷酸铁锂电化学性能测试 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 iron phosphate

- 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 the initial charge-discharge efficiency of lithium iron phosphate, i.e., the cathode material for lithium-ion batteries.

This document applies to the test for the initial discharge specific capacity and the initial charge-discharge efficiency of lithium iron phosphate, i.e., the cathode material for lithium-ion batteries.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 20522-2014 Lithium cobalt oxide

3 Terms and definitions

For the purpose of this document, the terms and definitions defined in GB/T 20522-2014 apply.

4 Test conditions

Unless otherwise specified, each test step should be carried out at a relative humidity not greater than 40.0 % and an ambient temperature of 20 °C ~ 30 °C. The rolling process should be carried out at a relative humidity not greater than 30.0 % and an ambient temperature not greater than 30 °C.

5 Reagents and materials

5.1 Lithium iron phosphate: the particle size characteristic value D_{50} is 0.5 μm ~ 8.0 μm , and the specific surface area is 6 m^2/g ~ 30 m^2/g .

5.2 Conductive agent: conductive carbon material.

5.3 Polyvinylidene fluoride (PVDF): battery grade, the weight average molecular weight is not less than 5×10^5 , and the moisture (mass fraction) is not greater than 0.10 %.

5.4 N-methylpyrrolidone (NMP): battery grade, the purity is not less than 99.9 %, and the moisture (mass fraction) is not greater than 0.02 %.

5.5 Aluminum foil: the thickness is $8 \mu\text{m} \sim 20 \mu\text{m}$.

5.6 Ethanol: analytical reagent.

5.7 Lithium-ion battery separator: polyolefin porous membrane, the porosity is 35.0 % $\sim$ 60.0 %, the air permeability is 100 s/100 mL $\sim$ 500 s/100 mL, the average pore size is not greater than $1.0 \mu\text{m}$, the diameter is 16.0 mm $\sim$ 18.0 mm, and the thickness is $9.0 \mu\text{m} \sim 32.0 \mu\text{m}$.

5.8 Metal lithium sheet: the diameter is 12.0 mm $\sim$ 16.0 mm, and the thickness is 0.40 mm $\sim$ 0.80 mm.

5.9 Battery standard structural parts: model CR2016, CR2025 or CR2032, including positive electrode case, negative electrode case, gasket, and spring support piece (or nickel foam sheet).

5.10 Lithium-ion battery electrolyte: lithium-ion battery electrolyte composed of lithium hexafluorophosphate (LiPF_6) and mixed carbonate-based organic solvents [ethylene carbonate (EC), dimethyl carbonate (DMC), ethyl methyl carbonate (EMC), etc.], the moisture is not greater than 0.002 %, the free acid (HF) is not greater than 0.005 %, and the conductivity (25 °C) is not less than 7.0 mS/cm.

5.11 Dust-free paper.

5.12 Nitrogen (or argon): the purity (volume fraction) is not less than 99.99 %.

6 Instruments and equipment

6.1 Vacuum oven: 0 °C $\sim$ 200 °C, and the temperature deviation is ± 2 °C.

6.2 Oven.

6.3 Electronic balance: the accuracy is 0.0001 g.

6.4 Electronic balance: the accuracy is 0.00001 g.

6.5 Dispersing mixer or pulp mixer.

6.6 Small coating machine for lithium-ion battery electrode sheets.

7.2.1 Weighing

Calculate the lithium iron phosphate, conductive agent, and PVDF pretreated in 7.1 according to the mass fractions of 80 % ~ 97 %, 1 % ~ 10 %, and 2 % ~ 10 % respectively, and weigh them with an electronic balance (6.3). Calculate the amount of NMP (5.4) according to the design requirements of solid content (mass fraction) of 25 % ~ 65 %, and weigh with an electronic balance (6.3).

7.2.2 Pulping

The pulping process is as follows:

- a) Add the weighed NMP into the mixing tank of a dispersing mixer or pulp mixer (6.5); gradually add the weighed PVDF; disperse and stir until completely dissolved; to prepare a transparent glue, of which the mass fraction of PVDF is 2 % ~ 10 %;
- b) add the weighed conductive agent to the above transparent glue; vacuum, disperse and stir evenly;
- c) gradually add the weighed lithium iron phosphate in portions; vacuum, disperse and stir evenly;
- d) add another NMP according to the designed solid content, to control the slurry viscosity at 3000 mPa · s ~ 20000 mPa · s; vacuum, disperse and stir evenly; complete the pulping process.

NOTE: The solid content in this document is the ratio of the mass of the cathode active material lithium iron phosphate, conductive agent and PVDF to the mass of the cathode slurry.

7.2.3 Coating

Use a small coating machine for lithium-ion battery electrode sheets (6.6) to evenly coat the cathode slurry mixed in 7.2.2 on one side (matte side) of the aluminum foil (5.5) or directly on the carbon-coated aluminum foil, and the thickness of wet slurry coating is 100 μm ~ 300 μm . After the coating is completed, transfer the positive electrode sheet to a vacuum oven (6.1) for drying. During drying, vacuum or circulate in a nitrogen (or argon) (5.12) atmosphere, the baking temperature is controlled at 90 °C ~ 150 °C, and the baking time is 0.5 h ~ 18 h.

7.2.4 Preparation of positive electrode sheets

Take the electrode sheets that have been dried in 7.2.3 and meet the processability requirements, use a punching machine (6.7) to punch out positive electrode sheets of appropriate size; use an electronic balance (6.4) and a desktop digital thickness gauge (6.8) to measure the mass m_c and thickness d_c of the positive electrode sheets, respectively.

vacuum or circulate in a nitrogen (or argon) (5.12) atmosphere. The baking temperature is controlled at 90 °C ~ 120 °C, and the baking time is 12 h ~ 18 h. Cool down and then transfer them to an inert atmosphere (or argon) glove box (6.10) for storage.

7.3 Battery assembly

Battery assembly shall be performed in an inert atmosphere (or argon) glove box (6.10). To assemble a button battery, please refer to the following steps:

- a) Place a negative electrode case flatly on a horizontal table with the opening upward;
- b) use an insulated tweezers (6.11) to pick up a metal lithium sheet (5.8) and place it into the negative electrode case, so that it is in plane contact with the negative electrode case and lies flat in the center of the negative electrode case;
- c) use an insulated tweezers (6.11) to pick up a lithium-ion battery separator (5.7) and make it completely covers the metal lithium sheet and lies in the center;
- d) use an injector (6.12) to inject 50 μL ~ 200 μL of lithium-ion battery electrolyte (5.10) into the negative electrode case;
- e) use an insulated tweezers (6.11) to pick up a positive electrode sheet prepared in 7.2.4 and place it in the middle of the lithium-ion battery separator (5.7);
- f) use an insulated tweezers (6.11) to pick up a gasket and a spring support piece in sequence and place them on the positive electrode sheet, ensuring that the gasket, spring support piece, and positive electrode sheet are aligned and centered;
- g) use an injector (6.12) to take 200 μL of lithium-ion battery electrolyte (5.10) and inject it into the negative electrode case containing the gasket, spring support piece, positive electrode sheet, lithium-ion battery separator, and metal lithium sheet;
- h) use an insulated tweezers (6.11) to pick up a positive electrode case and place it on the negative electrode case;
- i) move to the button battery packaging machine (6.13), press and seal;
- j) wipe the electrolyte leaked outside the button battery case with dust-free paper (5.11);
- k) number the assembled test batteries one by one and make records.

7.4 Battery test

Place the prepared test battery into a thermostat (6.14), control the temperature at 23 °C $\pm$ 2 °C, let stand for 2 h ~ 12 h, and use a lithium-ion battery electrochemical

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